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A NEW PROCEDURE FOR THE
IDENTIFICATION OF HYDROCARBONS

by

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CHAPTER I

INTRODUCTORY DISCUSSION

INTRODUCTORY DISCUSSION

The original object of this research was to develop a procedure for the systematic identification of hydrocarbons which might replace the present "Genus IX" of Volume I of the "Identification of Pure Organic Compounds" written by Professor S. P. Mulliken. It was our hope that a procedure might be developed which would divide the hydrocarbons rather sharply into natural families and that general procedures for the specific characterization of individual species of such families might then be made to supplement the general sectional tests.

The system at present adopted in Vol. I of Mulliken's works for the qualitative analysis of hydrocarbons depends upon the general rule that the aromatic and condensed ring hydrocarbons have a specific gravity greater than 0.85 at 20°-4°, and that of the other hydrocarbons, in general the paraffins are not readily attacked by bromine or by fuming sulphuric or nitric acids. The difficulties which present themselves with respect to this fundamental method of division necessitate the inclusion of some hydrocarbons, whose gravities are on the border line, in two groups, as for instance, the terpenes and para cymene. It

also requires the inclusion of α - and β -terpene tetrahydrides, cycloheptadiene, naphthalene octahydride, anthracene perhydride, and other exceptionally heavy non-aromatic hydrocarbons with the aromatics. Again, some paraffin hydrocarbons with branch chains, like diisooamyl, brominate hot and react with fuming nitric and sulphuric acids. Moreover, in applying the nitration hot the presence of dissolved NO_2 in paraffins may disguise two layers. Cycloparaffins with side chains react with fuming acids in the cold. This requires the inclusion of such compounds as naphthalene decahydride and the hexahydroxylenes with the unsaturates. Finally, the presence of ethers interspersed among the hydrocarbons is undesirable. These difficulties and illustrations present themselves with the Genus as at present constituted. With enlargement of the Genus to include the more important of the compounds prepared since the first appearance in print of this volume, the number of objections is increased, and the number of misfits is necessarily considerably multiplied. The work reviewed in this thesis has led to a procedure which divides the hydrocarbons according to the following scheme;

DIVISION A -- Solid Hydrocarbons

Section 1 -- Non-aromatics

Section 2 -- Chiefly aromatics

DIVISION B -- Liquid Hydrocarbons

Section 1 -- Aromatics, fulvenes,
quino-methides

Section 2 -- Acyclic ethers

Section 3 -- Polyolefines, acetylenes,
cyclic olefines, terpenes

Section 4 -- Olefines

Section 5 -- Naphthenes

Section 6 -- Acyclic paraffins

DIVISION C -- Gaseous Hydrocarbons

Details of the procedure, together with tables of the hydrocarbons known up to certain specified dates, are given at the back of this thesis. It should be clearly understood that the writer by no means guarantees this procedure for all the compounds listed in it. Some two hundred hydrocarbons have been handled in the course of this work. Not all of the tests necessary for the placing of a compound in a given section have been applied to all of these hydrocarbons, but what appear to be the important, or in any particular case doubtful tests have been applied. The body of the

thesis gives exactly the tests applied. The compounds which were located in their proper sections by actual experiment are starred in the tables. Something over two thousand hydrocarbon species are tabulated. The compounds obtained in this work include representatives of most of the classes known. The compounds prepared have been synthesized from two points of view -- the desire to obtain certain series, that trends might be noticed in them, and the desire to obtain "borderline" cases of doubtful sectional location, in order that exact sectional limits might be placed. The foregoing statements amount to saying that work has been done with ten per cent of the known hydrocarbons, and the procedure herein given has been found to apply satisfactorily to this ten per cent.

No attempt will be made in this work to give a complete literature review. A few references to the literature are scattered through the description of experimental work. It need only be said here that several hundred references dealing with the properties and reactions of hydrocarbons have been collected, weeded out by use of the abstracts, a number of original articles consulted, and a few thoroughly digested. The first appendix contains references principally to these last articles. Over seventy properties or reactions which might be applicable to this work have

these last articles. Over seventy properties or reactions which might be applicable to this work have been considered, and of these, all but those mentioned in the course of this work have been discarded.

A tempting procedure for both sectional division and specific identification is the use of three physical constants, density, refractive index, and boiling point. A division on the basis of refractive index, or of density and refractive index combined, leads to almost the same results as the present division in Mulliken's works, based on density, alone. The following table shows this. Only a few of the possible examples are given.

Hydrocarbon	n_D^{20}	D (20°-4°)	B.P.
2,4-Dihydro-toluene	1.468	0.827	106
Myrcene	1.470	0.802	167
trans-Decahydro-naphthalene	1.470	0.872	185
n-Amyl-benzene	1.473	0.860	202
Dipentene	1.473	0.865 (18°)	176
1,3-Cyclo-hexadiene	1.474	0.842	80.5
1,3-Dihydro-toluene	1.476	0.835	110
β -Phellandrene	1.479	0.852	171
p-Cymene	1.488 (24.5°)	0.857	176

If 1.473 be taken as the dividing line for the refractive index, those compounds of lower index include the paraffins, naphthenes, olefines, and light acetylenes, together with some terpenes. Those of higher index include aromatics, a few unsaturated cyclics, and some terpenes. Because of the number of border-line compounds which would have to be included in two groups, illustrated by this table, it is impossible to divide the hydrocarbons into any very distinct families on the basis of physical constants only. There are many compounds which would be distinctly misplaced; thus, the terpenes, sesquiterpenes, cyclohexyl acetylenes, and other types.

That these compounds can be specifically identified on the basis of physical constants, only, is also entirely impracticable. While constants are helpful as confirmatory evidence, it must be considered first of all that, although identification work should always be done with pure compounds, the degree of purity ordinarily met is rarely sufficient to warrant reliance on the boiling point to $\pm 2^\circ$ and on the density and refractive index to ± 2 in the third decimal. Furthermore, the methods ordinarily used for the determination of these constants do not permit of reliance upon the boiling point to ± 1 or 2° and on the

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density and refractive index to ± 1 in the third decimal. The refractive index can, of course, be determined to a high degree of accuracy if the refractometer is accurate, but this is not always the case, as the following results show.

Benzyl Acetate

I - 1.5053

II - 1.4965

Hydrocarbon Samples

	<u>B.P.</u>	<u>n_D</u>	<u>D (20-4)</u>
I	134. - 4.5° (Unc.)	1.4971 (25°)	0.887 (20-4)
II	137.5° (Corr.)	1.4960 (20.5°)	0.8997
Ethyl- benzene	136.5° (777 mm.)	1.4959 (20°)	0.868
m-Xylene	139.0°	1.4973 (20°)	0.865

Of the values above given for the refractive index of benzyl acetate, I and II were determined on the same sample by two different men in the M.I.T. Research Laboratory. Each used a different instrument, one being a rather ancient Abbe refractometer, the other a new one.

Of the hydrocarbon samples, values listed as I and II were determined by different men on different samples of the same compound. One guessed

ethyl benzene, the other meta-xylene. Both had ethyl benzene.

Also, as the table on page 16 shows, the constants reported in the literature are frequently not reliable beyond these limits for the same reasons as given above. These are by no means the worst examples that could be found, the constants shown are the best values reported for these compounds.

See Paragraph 13

Olefine	B.P.	D	$\frac{n}{D}$	Method of Preparation
Hexene (2)	68.0-8.2°	0.6863 (15/4)	1.3981 (15°)	Hexanol (2) + p-toluene Sulphonic acid (1)
	677-68°	0.66888 (15.5/4)	1.38319 (15.5°)	Hexanol (1) + Ni + H ₂ (220°) (2)
2,3-Dimethyl-butene (2)	72 -3° (767mm)	0.712 (20/4)	1.41285 (20°)	Bromide + KOAc (3)
	70 -1° (757mm)	0.6984 (20/0)	1.4055 (20°)	Dimethyl isopropyl Carbinol and crystalline oxalic acid (4) (7)
		0.7007 (20/0)	1.4080 (20°)	
Octene (1)	121-2°	0.716 (19/4)	1.4085 (19)	Allyl bromide + n-C ₅ H ₁₁ MgBr (5) Octene (1) carbonic acid (1) + heat (6)
		0.7207 (18/4)	1.4133 (18)	
Hexene (1)	63.35± 0.05°	0.67875 (15/4))	n-C ₃ H ₇ MgBr + allyl bromide (3)
	62°	0.684 (18/4))	
	60.5-1.5° (756mm.)	0.6830 (20/20)	1.3821 (20°))
	64°	0.6734 (20/0)	1.3870 (20°)	Allyl acetone hydrazone + KOH + Pt catalyst (8)
62 -3°	0.6686 (20/20)			Distillation of trimethyl n-hexyl ammonium hydroxide (9)
3-Methyl-pentene (2)	(I 65.1-5.7°	0.7220 (15/4)	1.3997 (15)	Diethyl methyl carbinol + p-toluene sulphonic acid (10)
	(II 69.9-70.2°	0.7022 (15/4)	1.4072 (15)	
	67 -8° (771mm)	0.6934 (20/20)		3 Brom 3 methyl pentene + alcoholic KOH (11)

Added to these facts it must be remembered that not all of these constants have been determined even approximately on many reported compounds, and that in many cases several compounds have about the same constants, as shown by the following table.

<u>Hydrocarbon</u>	<u>B.P.</u>	<u>D</u>	<u>$\frac{n}{D}$</u>
2-Methyl-pentene-(2)	66.5-7.5	0.691(15-4)	1.403(15)
2-Ethyl-butene-(1)	67 -8	0.694(20.5)	1.404(20.5)
3-Methyl-pentene-(2)	69 -70	0.698(19)	1.407(15)
2,3-Dimethyl-butene-(2)	71 -2	0.698(20-0)	1.406(20)
2,3,6-Trimethyl- heptene-(5) or -(6)	159.5-61	0.758(19)	1.432(19)
2,6-Dimethyl-octene-(6)	162-3	0.749(20-4)	1.427(20)
2,6-Dimethyl-octene-(X)	164-5	0.752(20-0)	1.432(20)
2,6-Dimethyl-nonene-(4) or -(5)	165-9	0.753(25-4)	1.428(25)

The olefines of these two groups (par. 14) are selected from the tables. One could not possibly be distinguished from the others by the use of physical constants only. The summation of these considerations immediately destroys any hope of specific identification by means of these constants only.

The writer feels that this brief introduction is sufficient to point out a few of the things to be guarded against in considering the identification of hydrocarbons. The following chapter deals at once with experimental work.

CHAPTER II

EXPERIMENTAL WORK

A

Introductory

EXPERIMENTAL WORK

Introductory

The experimental work involved in this thesis may be divided into three portions. 17

1. - That dealing with methods of dividing the hydrocarbons into groups.

2. - That dealing with methods of specific characterization of individual hydrocarbons.

3. - That dealing with the preparation and purification of hydrocarbons and their intermediates.

The work in the first two sections cannot be clearly divided. Methods of specific identification may often prove useful for group distinction. Nevertheless the work will be here described under three separate headings. 18

In the first two sections all reactions and tests applied, whether positive or negative, will receive attention. The beginning of each section will deal with methods later abandoned. The end of each section will consider methods adopted. 19

The third section will describe only successful preparative work, and only work relevant to the final form of the thesis. 20

A considerable amount of work which later proved 21

to have no relation to the results of this research has not been described herein, and throughout the writer has tried to be clear but concise. Important details are given. Wordy discussions have been omitted as far as possible.

B

Methods of Dividing the Hydrocarbons

into Groups

Methods of Dividing the Hydrocarbons
into Groups

A tempting procedure for sectional division, as well as for specific identification, is the use of physical constants. The practicability of this has already been discussed. (Paragraph 6.)

The use of physical constants being excluded, search was made for reactions which might distinguish open chains from rings, or which might indicate the presence of unsaturation. According to the literature, aromatic hydrocarbons and other unsaturated ring hydrocarbons give a precipitate when they are treated, cold, with concentrated sulfuric acid and formaldehyde (40% aqueous^(1,2)). The so-called 'Formolite reaction' has been considerably experimented with as a method of analyzing petroleum fractions for ring unsaturates, the weight of precipitate formed under specified conditions being taken as a measure of the hydrocarbons present.⁽³⁾ Working with chemical individuals, Nastjukoff obtained formolite precipitates as follows:

Hexane	-	Capylene	-
Ethylene	-	Diallyl	-
Amylene	-	Hexamethylene	-
Trimethylethylene	-	Methylhexamethylene	-
Hexylene	-	'Dicyclohexylnaphene'	-

'Dimethyldicyclohexylnapthene'	-
Cyclohexane	+
Methylcyclohexene	+
Menthene	+
Limonene	+
Camphene	+
Phellandrene	+
Benzene	+
Toluene	+

The results in this table appear to make an unusually sharp division. However, work reported by other investigators is not entirely corroborative of these results. Thus, decahydronapthalene is said to give a formolite precipitate. In addition to this, it is extremely difficult to get consistent results when formolite numbers of a given hydrocarbon mixture are determined, so that the procedure has been abandoned by many laboratories. Little is known concerning the structure of the compounds, or mixtures formed. They appear to vary greatly in composition. Nevertheless, it was thought best to test this procedure.

About 5ccs. of cold formalin were slowly added to a cold mixture of about one-half cc. of hydrocarbon and 5ccs. of sulfuric acid, with cooling and shaking. The reaction mixture was diluted with about 50ccs. of water

and then poured into an excess of ammonium hydroxide and filtered. ⁽³⁾ Kerosene and petroleum ether gave no precipitates. Amylene gave a light yellow precipitate. Benzene, toluene, and xylene gave gummy precipitates which hardened on standing. The precipitation in the case of amylene, the reported precipitation with decalin, the possible polymerization of formaldehyde to form a precipitate which might be confused with a positive test, and the inability of the formolite precipitates to serve as specific derivatives of the compounds tested appeared to make this reaction of little use, so that work was discontinued on it.

Various inorganic halides give colors with certain hydrocarbons. ^(5,7,8,9) It was thought possible that some compound might be found which would give color with unsaturated hydrocarbons ^{only}. Stannic chloride, two phosphorus halides, and sulfuric acid were tried. The results follow. The compounds used were slightly impure, they corresponded in most cases to what might be met ordinarily in identification practice.

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Action with stannic chloride:

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Petroleum ether	-	soluble,	colorless
Hexahydrobenzene	-	"	"
Benzene	-	"	"
Xylene	-	"	"
- - - - -	-	-	-

Amylene	-	deep red color, violent reaction
Cetene	--	" " "
p - Cymene	-	" " "
Diphenyl-methane	-	" " "
Tetraphenylethylene -		
(in benzene solution)-		golden yellow color

Action with phosphorus oxychloride:

29

Petroleum ether	-	insoluble, no color
n - Octane	-	" " "
2,2,4-Trimethylpentane	-	" " "
2,7-Dimethyloctane	-	" " "
n-Hexane	-	soluble, " "
Hexene -(1)	-	" " "
Heptene - (1)	-	" " "
Benzene	-	" " "
p - Cymene	-	" " "
Diphenylmethane	-	" " "
Diphenyl ether	-	" " "
Amylene	-	violent reaction, deep red color
Cetene	-	" " " "

Action with phosphorus trichloride:

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Amylene	-	heat evolved, yellow solution
Hexene-(1)	-	colorless solution.

Action with sulfuric acid (d.:1.84)^(4,5)

Amylene - violent reaction (with brown color)

Hexene -(1)- slow, colorless solution, heat evolution

Heptene -(1)- " , " " , " "

Hexene-(1) and heptene-(1) on standing with sulfuric acid again gave rise to two immiscible layers.

These results apparently do not permit of any class division on such lines. Coupled with this is the difficulty of telling whether a color reaction is due to the compound at hand or to a slight amount of impurity in it. Hence the idea of using this type of color reaction was abandoned.

Another reagent frequently used in the rough estimation of unsaturates in petroleum fractions is mercuric acetate.^(5,7,10,11) This reagent has not, to the present, been found satisfactory in distinguishing all types of olefines. Tausz has classified a good many olefines according to the way in which they react with mercuric acetate.⁽¹²⁾ Either they form addition products, they are oxidized, or they fail to react. Several attempts to make mercuric acetate a useful reagent in the characterization of olefines failed. These will be described later. Because of the ill success of these experiments it was thought useless to try to adopt this reagent as a means of group division.

Of various methods of titrating unsaturation

three have been seriously investigated in this work. The solutions used were Hanus, benzoyl hydroperoxide, and bromide-bromate.

While Hanus solution (iodine bromide) was made up and used to titrate a few olefines, its extensive application was not attempted because the rather extensive work already done upon it and reported in the literature is sufficient to indicate that it cannot be used systematically as a quantitative measure of unsaturation and that results are difficult to duplicate unless scrupulous observance of very detailed conditions is insisted upon. ^(13,14,28)

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According to Nametkin and Lydia Brussoff, ⁽¹⁵⁾ unsaturated hydrocarbons may be titrated with benzoyl hydroperoxide by weighing 0.1 - 0.3 gms. of the hydrocarbon into a glass stoppered Erlenmeyer and treating it with twice the theoretical amount of benzoyl hydroperoxide in chloroform solution, containing 0.4 - 0.5% active oxygen. The reaction is slower in ether than in chloroform. 38 hours is the time allowed for the reaction, the temperature being 23°. At the end of the reaction 20 cc. of 10% potassium iodide solution are added and the iodine thus liberated titrated with 1/10 N. thiosulfate. Results differ usually by not more than 0.5 - 1.0%. Meerwein ⁽¹⁶⁾ measured the rates of oxidation by benzoyl hydroperoxide

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at 0° of several phenyl substituted ethylenes and various terpenes. He found that pinene reacted most rapidly of those measured, and that, whereas this reaction was nearly complete in one hour, others were complete only after long standing. Prileschaeff⁽¹⁷⁾, the original worker in this field, found that the peroxide yielded the oxides of the hydrocarbons with which he worked and that these could be hydrated to give the glycols. He does not give his method of hydration. Oxides have been reported hydrated by several hours (six) refluxing with 1 % sulphuric acid.⁽¹⁸⁾

The benzoyl hydroperoxide solution used in this work was prepared according to the method described in Beilstein,⁽¹⁹⁾ by dissolving dibenzoyl peroxide (Eastman) in ether (20 grams per 300 cc. ether) and adding in a large separatory funnel an equimolecular amount of sodium ethylate (2 grams sodium per 20 gms. peroxide, dissolved in the necessary amount of reagent alcohol), then dissolving the precipitated salt in water and after discarding the ether layer washing the water solution at least four times with ether, or until no further odor of ethyl benzoate could be detected. The solution was then acidified with hydrochloric acid, extracted by chloroform, washed, dried with sodium sulfate, and diluted to make the active oxygen content approximately 0.5%.

The titrations^{were} run as follows: The sample was

withdrawn with a one cc. graduated pipette and 0.2 cc. added to an eight ounce glass stoppered, narrow mouth bottle to which had already been added 14 cc. (or the requisite amount) of the hydroperoxide solution. The mixture was shaken occasionally during the period of reaction. In cases of long times of reaction the bottles were covered with collodion. To titrate, 15 cc. of 15% potassium iodide solution and five cc. of concentrated hydrochloric acid (D.1.19) were added and shaken well for thirty seconds, then titrated with thiosulfate, running back with Hanus solution if necessary. The results are calculated as % unsaturated in the compound.

Heptene(1) of specific gravity 0.690 at 24.5° gave the following:

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<u>Temperature of Reaction</u>	<u>Time of Reaction</u>	<u>% Unsaturation</u>
24.5°	30 minutes	36.3 %
35°	30 "	63.2 %
50°	30 "	97.3 %
50°	30 "	73.6 %
50°	30 "	79.2 %
ice chest	16 hours, 35 minutes	100.5 %
ice chest	137 " , 40 "	82.2 %

A titration with Hanus solution gave 86.7 % unsaturation.

A bromide=bromate titration with 0.2 cc. of hydrocarbon gave 97.8 % unsaturation, with 0.5 cc., 94.2%, and with 1 cc.,

92.3 % unsaturation.

Hexene(1), specific gravity 0.682 (approximately) at 25°, gave the following results:

<u>Temperature of Reaction</u>	<u>Time of Reaction</u>	<u>% Unsaturation</u>
50°	30 minutes	96.1 %
50°	30 minutes	101.3 %

Cetene, specific gravity 0.784 (approximately) at 25°, gave the following results:

<u>Temperature of Reaction</u>	<u>Time of Reaction</u>	<u>% Unsaturation</u>
50°	30 minutes	74.2 %
50°	30 minutes	82 %

A later titration of this sample with bromide-bromide solution gave as the average of two determinations using 0.5 cc. of cetene, 102.4 % unsaturation, as the average of two determinations using 1.0 cc., 98.5 % unsaturation.

N-octane gave the following results:

Ccs. of thiosulfate required to titrate liberated iodine:

average of two blanks - 24.61 cc.

average of two determinations - 24.84 cc.

It has been reported that the rate of reaction of hydrogen peroxide with unsaturates is greater in acetic acid than in ether solution, and gives glycols directly. Accordingly a concentrated chloroform solution of benzoyl

hydroperoxide was prepared and diluted with glacial acetic acid and water. With cetene, time of reaction 2 hours, temperature that of the room, an average of two determinations gave 36 % unsaturation.

Therefore, since it was found that decomposition of the benzoyl hydroperoxide solution at 50° reaches 16% in thirty minutes, and since, as shown above, consistent results with this temperature and time of heating cannot be obtained, and since a long time of standing in the cold is not feasible, it seemed best to abandon the hope of using this reagent to titrate unsaturation. 46

Bromide-bromate solution of a standard normality has been used for some years in the titration of unsaturated compounds. ^(20,29) Its advantage lies in the fact that the bromine is liberated only gradually, once the solution is acidified, so that a high concentration of bromine is never present, and consequently substitution does not take place to such a marked degree as in other procedures where the halogen to be added is all present at the beginning of the titration. ⁽²¹⁾ For some reason this reagent does not appear to have been used much until Francis adapted it to the titration of unsaturates in cracked petroleum distillates. ⁽²²⁾ The equation for the reaction is:



Francis' procedure follows:

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A slight excess (preferably not more than one cc. as estimated from a trial titration) of the bromide-bromate solution (14 gms. KBrO_3 , 50 gms. KBr /liter) is measured into a small Erlenmeyer flask, and the sample of oil, 3 to 50 cc., depending upon the unsaturated content, is pipetted in. The solution is quickly acidified with about five ccs. of 10% H_2SO_4 , and the flask is stoppered. It is shaken for one minute as vigorously as may be necessary to keep the color a pale yellow. If the color is dark yellow in spite of violent shaking, too much bromide-bromate has been added, and the analysis should be considered only a trial titration. In any case, in order to complete the liberation of bromine, 15 cc. more of acid are added and the shaking is continued for another minute. If the solution remains completely colorless, a little more bromide-bromate should be added. The final color should be light yellow. Then titration is completed with thiosulphate, with vigorous shaking toward the end. (0.2 N thiosulphate)

Cortese has recently titrated a number of pure hydrocarbons by a modification of this procedure. ⁽²⁹⁾ For this work it was felt necessary to adopt a procedure which should not require more than one titration for a correct result, nor more than 0.5 cc. of compound. After several variations had been tried, the following method of titration was finally adopted.

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The approximate molecular weight of the compound is determined by adding twenty-five degrees to its observed boiling point and then referring to the tables of hydrocarbons. From the tables the olefine of highest molecular weight of the boiling point thus found is taken as the basis of calculation for the amount of bromide-bromate solution to be used. This is done to allow for possible errors in the determination of the boiling point and to make certain that the amount of solution taken is not greater than that required to react with the olefine used. The amount of bromide-bromate solution required to titrate 0.5 cc. of the olefine is given in ccs. from the formula $\frac{1000G}{MN}$, where G is the specific gravity of the hydrocarbon, M its molecular weight, N the normality of the standard solution. This quantity of solution is run from a burette into a four ounce glass stoppered, narrow-mouthed bottle, 0.5 cc. of hydrocarbon is added from a graduated 1 cc. pipette, and 15 ccs. of ten per cent. sulfuric acid are added quickly, the bottle tightly stoppered, and shaken vigorously until when shaking is temporarily stopped bromine color no longer develops at once. If the color has all disappeared more bromide-bromate solution is added, one cc. at a time, and the bottle shaken as before until enough has been added so that a faint yellow color (the faintest detectable) remains after the bottle has been

shaken for two minutes after the final addition. At this point 5 cc. more of 10 % sulfuric acid are added, and the bottle shaken for two minutes longer, or until color disappears. *(if the solution becomes colorless in less than two minutes)* The addition of bromide^{bromate} solution is continued until color remains, as before, after two minutes of shaking, then 5 ccs. of 15% potassium iodide solution are added, and the iodine titrated with 0.2 N. thiosulfate, using starch indicator.

If the substance to be titrated is extremely volatile, it is advised to put ice in the bottle before titration begins. The titration solution must warm up to room temperature before the titration is concluded, however, as the acid liberates bromine very slowly from the cold solution so that a considerable excess of the latter may be added without any color developing until it has warmed up to room temperature. It will perhaps be necessary to run a trial titration to find the approximate amount of solution required if the olefine boils below about 40°.

The titrations here reported were run using this modification, with the exception of Eastman's octene -(2), in which case the second addition of sulfuric acid, and shaking, given here, was omitted, but all of the acid was added at the beginning. It is doubtful that this change made any difference in the values obtained. Diolefines

and acetylenes were titrated as though they were olefines, i. e., the amount of solution added at the beginning of the titration was computed as if the compound were an olefine. This was done because if the compound were an unknown this is the assumption that would have to be made.

The results of these titrations are reported here in terms of actual and theoretical "bromide-bromate numbers". The "bromide-bromate number" is the number of centigrams of bromine added by one gram of hydrocarbon. The results are recorded in this way rather than in terms of the customary "bromine number" that the procedure by which the value was obtained may be clear from the name. If 0.5 cc. of sample be used, the "bromide-bromate number" is given by the formula $\frac{16VN}{G}$, where V is the volume of standard solution consumed, N its normality, and G the specific gravity of the hydrocarbon.

In the following determinations the densities of the compounds titrated were assumed to be equal to the best densities reported in the literature extrapolated to the experimental room temperature (except in the cases of new compounds where the experimentally determined densities were used). This assumption probably involves an error in many cases, but the error is probably always within the limits of accuracy of the procedure. The limit of accuracy in this titration is generally considered to be

2 - 3%. The densities of these hydrocarbons are in the neighborhood of 0.7. An error in the assumption of the density of 14 parts in the third decimal place would still leave the results correct to 2%, or within the experimental error. The values for the densities were taken from the tables in this thesis, 1 part in the third decimal being taken as the approximate dd-dt. The tables follow on the next page.

BROMIDE-BROMATE TITRATIONS

Compound	Volume of Compound	Normality of B.B. Solution	Volume of Solution Consumed	B.B. No. (Found)	B.B. No. (Calc.)
<u>Olefines</u>					
1. Pentene-(2)				227 ^{XX} 227 ^{XX} 227	229 229 229
2. 2-Methyl-butene-(2) <i>(Trimethylethylene)</i>				236 ^{XX} 227 ^{XX}	229 229
3. Hexene-(1)	0.5	0.5152	15.72 ^x	192 ^x	190
4. 4-Methyl-pentene-(1)	0.5	0.5152	14.83 ^x	173 ^x	190
5. Heptene-(1)	0.5	0.5059	13.73	157	163
	1.0	0.5059	26.56	154	163
6. Heptene-(2)	0.15	0.5152	4.79	182	163
7. Heptene-(3)	0.5	0.5152	13.67	162	163
	0.5	0.5152	13.63	161	163
8. 4-Methyl-hexene-(1)	0.5	0.5152	13.68 ^x	159 ^x	163
9. 5-Methyl-hexene-(1)	0.5	0.5152	13.67 ^x	164 ^x	163
10. 4,4-Dimethyl-pentene-(1)	0.5	0.5152	14.84 ^x	177 ^x	163
11. 2,4-Dimethyl-pentene-(2)	0.5	0.5152	18.89	224	163
	0.5	0.5152	18.82	224	163
12. Octene-(2) (Eastman's)	1.0	0.5059	22.97	126	143

Compound	Volume of Compound	Normality of B.B. Solution	Volume of Solution Consumed	B.B. No. (Found)	B.B. No. (Calc.)
13. Nonene-(1)	0.5	0.5152	11.61	126 ^x	127
	0.5	0.5152	11.28	129	127
14. Nonene-(4)	0.5	0.5152	11.15	126	127
	0.5	0.5152	11.06	125	127
15. 4-Methyl-octene-(2)	0.5	0.5152	11.26	128	127
	0.5	0.5152	11.05	125	127
16. 4,6-Dimethyl-heptene-(2)	0.5	0.5152	11.69	133	127
	0.5	0.5152	11.70	133	127
17. Decene-(1)	0.5	0.5152	10.47	116	114
	0.5	0.5152	10.46	116	114
	0.5	0.5152	10.75	120	114
18. Hexadecene-(1)	0.5	0.5152	6.63	69.9	71.4
	0.5	0.5152	6.61	69.9	71.4
19. Cyclohexene	0.5	0.5152	18.79	193	195
	0.5	0.5152	18.72	192	195
20. 1-Methyl-cyclohexene-(1)	0.5	0.5152	15.45	158	167
	0.5	0.5152	15.56	160	167
21. 1-Cyclohexyl-propene-(2)	0.5	0.5152	12.25	125	129
	0.5	0.5152	12.08	124	129
	0.5	0.5152	12.29 ^{xxx}	125 ^{xxx}	129
22. d-Menthene	0.5	0.5152	15.23	159	116

Compound	Volume of Compound	Normality of B.B. Solution	Volume of Solution Consumed	B.B. No. (Found)	B.B. No. (Calc.)
23. 4-Cyclohexyl-pentene-(2)	0.5	0.5152	11.31	112	105
24. 4-Cyclohexyl-heptene-(2)	0.5	0.5152	8.37	82.2	89.9
<u>Diolefines</u>					
25. Pentadiene-(1,3)	0.5	0.5152	38.45	470	471
26. 2-Methyl-butadiene-(1,3)	0.5	0.5152	33.82	415	471
	0.5	0.5152	33.36	410	471
27. Hexadiene-(1,5)				378 ^{XX} 378 ^{XX}	390 390
28. Hexadiene-(2,4)				xx 378 ^{XX} 378	390 390
29. 2-Methyl-pentadiene-(2,4)				370 ^{XX} 370 ^{XX}	390 390
30. 2,3-Dimethyl-butadiene-1,3)	0.5	0.5152	33.60	385	390
31. Heptadiene-(2,4)	0.5	0.5152	28.59	325	333
	0.5	0.5152	28.70	326	333
32. Octadiene-(2,4)	0.5	0.5152	25.27	281	291
	0.5	0.5152	25.01	277	291

Compound	Volume of Compound	Normality of B.B. Solution	Volume of Solution Consumed	B.B. No. (FOUND)	B.B. No. (CALC.)
33. 4-Methyl-heptadiene-(1,5)	0.5	0.5152	21.55	244	291
			21.77	247	291
34. 2,2-Dimethyl-hexadiene-(3,4)	0.15	0.5152	7.92	294	291
			7.65	285	291
35. 4,5-Dimethyl-octadiene-(2,6)	0.5	0.5152	17.81	193	232
36. 4-Propyl-heptadiene-(1,5)	0.5	0.5152	16.16	178	232
			16.74	183	232
37. Dicyclopentadiene	0.5	0.5152	35.58	300	242
38. 4-Propene-(4 ^o)-yl-octene-(2)	0.5	0.5152	15.57	170	210
39. 4,5-Di-n-propyl-octadiene-(2,6)	0.5	0.5152	13.79	146	165
40. 4,5-Di-n-butyl-octadiene-(2,6)	0.5	0.5152	12.08	125	144

Terpenes

41. Dipentene

214^{xx}
218^{xx} 235
235

Compound	Volume of Compound	Normality of B.B. Solution	Volume of Solution Consumed	B.B. No. (Found)	B.B. No. (Calc.)
42. Carvene	0.5	0.5152	24.95	242	235
43. Pinene (Eastman pract.)	0.5	0.5152	26.28	253	118
44. d- α -Pinene (Kahl.)	0.5	0.5152	25.19	243	118
	0.5	0.5152	25.67	248	118
45. d- α -Pinene (Sch.)	0.5	0.5152	26.37	254	118
46. l- α -Pinene (Kahl.)	0.5	0.5152	17.69	171	118
	0.5	0.5152	17.61	170	118
47. l- α -Pinene (Sch.)	0.5	0.5152	24.52	236	118
48. l- β -Pinene	0.5	0.5152	26.81	256	118
49. Phellandrene (B.D.H.)	0.5	0.5152	17.99	173	235
50. d-Phellandrene (Sch.)	0.5	0.5152	20.28	198	235
51. l-Phellandrene (Sch.)	0.5	0.5152	15.88	155	235
52. Sabinene	0.5	0.5152	25.54	250	118
53. Cedrene	0.5	0.5152	13.63	121	?
54. Caryophyllene (B.D.H.)	0.5	0.5152	14.60	134	157
	0.5	0.5152	15.61	143	157
55. Caryophyllene (Sch.)	0.5	0.5152	7.06	64.8	157
	0.5	0.5152	6.92	63.4	157

<u>Compound</u>	<u>Volume of Compound</u>	<u>Normality of B.B. Solution</u>	<u>Volume of Solution Consumed</u>	<u>B.B. No. (Found)</u>	<u>B.B. No. (Calc.)</u>
<u>Acetylenes</u>					
56. Heptine-(2)	0.15	0.5152	3.28	122	333
57. Heptine-(3)	0.5	0.5152	21.59	235	333
	0.5	0.5152	21.17	230	333
58. Octine-(1)	0.5	0.5152	12.98	142	291
59. Octine-(2)	0.5	0.5152	15.40	163	291
	0.5	0.5152	14.76	157	291
60. Nonine-(4)	0.5	0.5152	9.89	106	258
61. Hexadecine-(1)	0.5	0.5152	2.97	30.9	144

x Titrations made by Charles W. Schroeder, M.I.T., Class of '31.

XX Values reported by Cortese, Rec. trav. Chim. ⁴⁸~~36~~, 564 (1929), obtained by a different modification of the titration procedure, and recalculated in these terms.

XXX Determined by Gilbert C. Toone.

The results here reported show that although in many cases, especially in the terpene and acetylene series, this titration gives anything but the theoretical figure, yet the results are fairly reproducible and with the olefines and diolefines they approach the calculated values in most cases. The exceptions to this latter statement appear to be such compounds as possess an active hydrogen due to the presence of branched chains in the vicinity of the double bonds, such as 2,4-dimethyl-pentene-(2), this being the only olefine with an open chain which gave markedly different results from the theoretical. Whether any general rule can be established by which exceptional compounds can be picked out from those which act normally cannot be told from these results.

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No aromatic compounds have been titrated because it is known that certain labile hydrogen in such compounds as mesitylene is quantitatively substituted by bromine with extreme ease.

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The use to which this titration has been put will be apparent in the succeeding pages which describe the procedure for sectional division. It will be referred to again in the discussion of specific characterization.

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A consideration of other methods of dividing the hydrocarbons into groups, suggested by the literature, indicated that the selective solvent action of certain types

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of more or less indifferent solvents might offer some hope.

The use of solubilities, generally, in organic qualitative analysis by both Staudinger (24) and Kamm (25) acted as a guide-post to this idea. Kamm recommends dimethyl sulfate as a means of distinguishing paraffins (they are insoluble in it).(25) Picric acid is mentioned by Rosenthaler as being insoluble in some aliphatic hydrocarbons, but soluble in aromatics (5). Perkin refers to the solubility of phenol in aromatic hydrocarbons and its insolubility in the aliphatics.(26) The similar use of levulinic acid, (27), phenylhydrazine, (27), ethylene-glycol-monoacetate, (27), and furfural (27) is mentioned in various places in the literature. These are all reported to dissolve only aromatics. Liquid sulfur dioxide (30, 31, 32) and ethyl sulfate (33) are reported to dissolve olefins, but not paraffins. Benzyl alcohol has been said to dissolve naphthenes, but not acyclic paraffins, (34, 35), and the critical solution temperatures of a few paraffins have been determined in nitrobenzene (36, 37, 38), and still more in aniline. (39, 40, 41, 42, 43) The use of some of these solvents has been popular in various procedures for estimating the olefine, aromatic, or naphthene content of various petroleum fractions and similar products. (44, 45, 46) Most of these solvents, together with some dozen others were investigated, with the results shown in the table on the following page.

Solubilities of Types of Hydrocarbons in Various Solvents
(see paragraph 59)

<u>Solvent</u>	<u>Petroleum Ether</u>	<u>Hexahydrobenzene</u>	<u>Amylene</u>	<u>Benzene</u>
Nitrobenzene	insoluble	soluble	soluble	soluble
Aniline	insoluble	insoluble	soluble	soluble
Benzyl alcohol	insoluble	soluble		soluble
Phenol	insoluble	soluble		soluble
Picric acid	insoluble	insoluble	partially soluble	soluble
o-Nitrophenol	insoluble	soluble	soluble	soluble
Triphenyl phosphate	insoluble	soluble	soluble	soluble
Trimethyl borate	soluble		soluble	soluble
Ethyl carbonate	soluble		soluble	soluble
Chlorpikrin	soluble			
Formamide, $H(CO)NH_2$	insoluble	insoluble	insoluble	insoluble
Urethane, $H_5C_2O(CO)NH_2$	insoluble		insoluble	soluble
Methyl sulphate	insoluble	insoluble	soluble	soluble
Ethyl sulphate	insoluble	insoluble	soluble	soluble
Ethyl nitrate	insoluble	insoluble	soluble	soluble
Isoamyl nitrite	soluble		soluble	
Isoamyl nitrate	soluble			
<hr/>				
Nitromethane	insoluble	insoluble	insoluble	soluble (15°)

By "soluble" is meant that the two liquids when mixed volume for volume were completely miscible at room temperature, or in the case of the solids, at their melting points. Picric acid was considered "soluble" if it gave a yellow color to the hydrocarbon at room temperature.

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A review of the solubilities above the line shows that certain organic esters of inorganic oxygenated acids have valuable selective solvent properties, that this property becomes most specific with decreasing length of an aliphatic chain, that the nitro group on an aromatic ring imparts valuable selective solvent properties to benzene, and that a saturated oxygen linkage increases solubility, witness the change in solvent properties of formamide caused by the introduction of the ethoxy group in place of hydrogen to give urethane. Of these solvents, ethyl and methyl sulphates and ethyl nitrate are the best to distinguish aromatics, but these also dissolve amylene and certain other low boiling olefines, although the higher ones are insoluble. These principles suggest that nitromethane should have valuable solvent properties - it has but one carbon, it has the nitro group, and it is similar to ethyl nitrate, but lacks a saturated oxygen linkage.

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The results of testing this solvent have proven very satisfactory. The structure of the carbon chain has

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a very marked effect on the solubility of hydrocarbons in this solvent. This influence should be borne in mind in the following discussion. The following table (page 50) shows the solubility of a number of hydrocarbons in this solvent.

In the ~~following~~ solubility determinations on page⁵⁰ 63 the procedure used was to measure from a 1.0 cc. graduated pipette (or from a dropper of capillary tubing, graduated to hold 0.1 cc.) 0.1 cc. of solvent into a glass stoppered tube of 3-4 cm. length and 7 mm. bore. 0.1 cc. of hydrocarbon was then added. If the critical solution temperature was below 30° it was determined by using a water bath, if above, by using a nujol bath. The tube was strapped to the bulb of the thermometer by a rubber band. Agitation was constant, but not hurried. The tube used was readily made in a few minutes by grinding a short piece of stirring rod into a short, closed tube of the right diameter. (*Carborundum for grinding was #600, from the Carborundum Co., Niagara Falls.*)

By "soluble" is meant completely miscible. 64
By "insoluble" is meant that the two liquids are not completely miscible, regardless of their partial miscibility. By the critical solution temperature is meant the temperature

at which the solution first shows turbidity upon slowly cooling to below the temperature at which both of its components are completely miscible.

In this table and in those following the sources of the compounds tested are given. Abbreviations are used as follows:

B.D.H.: British Drug Houses, London

Buc: Dr. H. E. Buc, Standard Oil Development Co., Elizabeth, N. J.

C.: Dr. Ing. H. Cohen, Organische Chemische Laboratorium, Berlin

E.: Eastman Kodak Co., Rochester, N. Y.

K.: Kahlbaum through Akatos, N. Y.

S.: Schimmel and Co., Miltitz, bei Leipzig, through Pres. Louis E. Watermeyer of Fritche Bros., N. Y.

syn.: synthesized in the course of this work

In describing the solubility properties, the following abbreviations have been used.

C.S.T.: Critical Solution Temperature

Insol.: Insoluble

Sol.: Soluble

Their meanings have been earlier described (par. 64).

Tables follow on the next page.

SOLUBILITIES OF HYDROCARBONS IN NITROMETHANE

Liquids

Paraffins:

- | | |
|---|---------------------|
| 1. n-Hexane...(syn.) | C.S.T.:106° |
| 2. n-Heptane...(E.) | Insol. at 23° |
| 3. n-Octane... (syn.) | Insol. at 23° |
| 4. 2,2,4-Trimethyl-pentane...(A.D.Little) | Insol. at 105° |
| 5. n-Nonane...(C) | Sol. at about 94.3° |
| 6. n-Decene...(E) | Insol. at 23° |
| 7. 2,7-Dimethyl octane...(syn.) | Insol. at 23° |
| 8. n-Tetradecane...(E) | Insol. at 23° |

Naphthenes:

- | | |
|--|-----------------------------|
| 9. Hexahydrobenzene...(H.W.Underwood) | Insol. at 20° |
| 10. Decahydronaphthalene...(Newport Chemical Works, Passaic, N.J., through H. W. Underwood, Jr.) | Insol. at 20° |
| 11. Methyl cyclohexane...(E., purified) | Insol. at 23° |
| 12. p-Menthane...(E.) | Insol. at 23° |
| 13. Dimethyl cyclohexane...(C.) | C.S.T.:107°
(on heating) |
| 14. ter.-Amyl-cyclohexane...(Toone) | C.S.T.:72.8° |

Aliphatic Oxygenated Compounds:

- | | |
|-----------------------------------|---------------|
| 15. Methyl n-butyl ether...(E.) | Sol. at -17° |
| 16. Ethyl n-butyl ether...(E.) | Sol. at -15° |
| 17. Ethyl isoamyl ether...(Toone) | Sol. at -15° |
| 18. di-n-Butyl ether...(E.) | Insol. at 20° |

- | | | |
|-----|--------------------------|---------------|
| 19. | di-n-Amyl ether...(E.) | Insol. at 25° |
| 20. | di-iso-Amyl ether...(E.) | Insol. at 20° |

Olefines:

- | | | |
|-----|-------------------------------------|--|
| 21. | Pentene-(1)...(syn.) | Insol. at 40° |
| 22. | Pentene-(2)...(Bus.) | Insol. at 40° |
| 23. | Amylene...(E., tech.) | C.S.T.:18° |
| 24. | Hexene-(1)...(syn.) | Insol. at 70° |
| 25. | 4-Methyl-pentene-(1)...(syn.) | Insol. at 25° |
| 26. | 4-Methyl-pentene-(2)...(syn.) | Insol. at 75° |
| 27. | Heptene-(1)...(syn.) | Insol. at 70° |
| 28. | 4-Methyl-hexene-(1)...(syn.) | Insol. at 25° |
| 29. | Heptene-(2)...(syn.) | C.S.T. at 66.9° |
| 30. | 2,4-Dimethyl-pentene-(2)...(syn.) | C.S.T.:64.7° |
| 31. | Heptene-(3)...(syn.) | C.S.T.:64.7° |
| 32. | Octene-(2)...(E.) | Insol. at 25° |
| 33. | 4-Methyl-heptene-(2)...(syn.) | C.S.T.:80.7° |
| 34. | Diisobutylene...(H.S.Davis) | Insol. at 25° |
| 35. | Nonene-(1)...(syn.) | C.S.T.:83.7° |
| 36. | 4-Methyl-octene-(2)...(syn.) | Difficultly sol.
in boiling CH_3NO_2
at 101° |
| 37. | 4,5-Dimethyl-heptene-(2)...(syn.) | Insol. at 100° |
| 38. | 4,6-Dimethyl-heptene-(2)...(syn.) | C.S.T.:97.9° |
| 39. | 4,5,5-Trimethyl-hexene-(2)...(syn.) | C.S.T.:88.2° |
| 40. | Nonene-(4)...(syn.) | C.S.T.:84° |
| 41. | Decene-(1)...(syn.) | Insol. at 100° |
| 42. | 4-Butyl-octene-(2)...(syn.) | Insol. at 107° |

43.	Triisobutylene...(G.Rigby)	Insol. at 107°
44.	Cetene...(syn.)	Insol. at 120°
45.	Tetraisobutylene...(G.Rigby)	Insol. at 120°
46.	3-Ethyl-eicosene-(2) or (3) together with 3-ethyl-octadecene- (2) or (3)...(W.E.Messer)	Insol. at 125°

Cyclic Olefines:

47.	Cyclohexene...(E.)	C.S.T.:52°
48.	1-Methyl-cyclohexene...(E.)	Insol. at 25°
49.	Allyl-cyclohexane...(syn.)	C.S.T.:74.5°
50.	4-Cyclohexyl-pentene-(2)...(syn.)	Insol. at 106°
51.	4-Cyclohexyl-heptene-(2)...(syn.)	Insol. at 100°

Terpenes:

52.	Pinene...(E., pract.)	Inso. at 65°
53.	1-Pinene...(N.)	C.S.T.:81.9°
54.	d-Pinene...(N.)	C.S.T.:75.8°
55.	β -Pinene...(S.)	C.S.T.:70°
56.	Sabinene...(S)	C.S.T.: abt. 52°
57.	Limonene...(laboratory stock)	Insol. at 20°
58.	Dipentene...(E.)	Insol. at 20°
59.	Phellandrene...(B.D.H.)	C.S.T.:37°
60.	Caryophyllene...(B.D.H.)	C.S.T.:85°
61.	Cedrene...(B.D.H.)	Insol. at 105°

Diolefines:

62.	Pentadiene-(1,3)...(E.)	Sol. at -20°
63.	Hexadiene-(2,4)...(F.Cortese)	S.C.T.: -9°

64.	2-Methyl-pentadiene-(2,4)...(F.Cortese)	C.S.T.: -2°
65.	Diallyl...(F.Cortese)	C.S.T.: +5°
66.	Heptadiene-(2,4)...(syn.)	C.S.T.: 17.2°
67.	Octadiene-(2,4)...(syn.)	C.S.T.: 36.3°
68.	2,2-Dimethyl-hexadiene-(3,4)...(syn.)	C.S.T.: 20.2°
69.	4-Methyl-heptadiene-(1,5)...(syn.)	C.S.T.: 52.9°
70.	4-Propyl-heptadiene-(1,5)...(syn.)	C.S.T.: 76.8°
71.	4,5-Dimethyl-octadiene-(2,6)...(syn.)	C.S.T.: 78.3°
72.	4-Allyl-octene-(2)...(syn.)	C.S.T.: 82.4°
73.	4,5-di-n-Butyl-octadiene-(2,6)...(syn.)	Insol. at 103°

Acetylenes:

74.	Pentine-(1)...(Addison)	Sol. at -17°
75.	Heptine-(3)...(syn.)	C.S.T.: 23.8°
76.	Octine-(1)...(syn.)	Sol. at -17°
77.	Octine-(2)...(syn.)	C.S.T.: -2.5°
78.	Nonine-(4)...(syn.)	C.S.T.: 45.4°
79.	Hexadecine-(1)...(syn.)	C.S.T.: 92°

Aromatics:

80.	Benzene---(laboratory stock)	Sol. at -20°
81.	Toluene...(laboratory stock)	Sol. at -20°
82.	o-Xylene...(E.)	Sol. at -20°
83.	m-Xylene...(E.)	Sol. at -20°
84.	p-Xylene...(E.)	Sol. at -20°
85.	Ethyl-benzene...(student preparation)	Sol. at 20°
86.	n-Propyl-benzene...(E.)	Sol. at -17°

87.	Pseudocumene...(stock room)	Sol. at 20°
88.	Mesitylene...(stock room)	Sol. at 20°
89.	Hydrindene...(K.)	Sol. at -18°
90.	n-Butyl-benzene...(E.)	C.S.T.:+4°
91.	sec.-Butyl-benzene...(E.)	C.S.T.: -1°
92.	ter.-Butyl-benzene...(K.)	Sol. at -17°
93.	p-Cymene...(A.E.Schneider)	C.S.T.: -4°
94.	m-Diethyl-benzene...(E.)	C.S.T.: -3°
95.	p-Diethyl-benzene...(E.)	C.S.T.: -5°
96.	Tetrahydronaphthalene...(Newport Chem. Works, Passaic, N.J., through H. W. Underwood, Jr.)	C.S.T.: -16°
97.	ter.-Amyl-benzene...(E.)	C.S.T.:13.5
98.	Phenyl-cyclohexane...(E.)	C.S.T.:23.5°
99.	Styrene...(E.)	Sol. at -10°
100.	Ph.CH=CH.C ₃ H ₇ (n)...(Prof. J.B.Conant, through E.H.Huntress)	Sol. at -21°
101.	Ph.CH=C(C ₃ H ₇ (n)) ₂ ...(J.B.Conant)	Sol. at -18°
102.	Ph.CH=C(CHMe ₂) ₂ ...(J.B.Conant)	C.S.T.:33.9°
103.	Ph.C(Me):C(CHMe ₂) ₂ ...(J.B.Conant)	C.S.T.:48.9°
104.	Phenyl-acetylene...(E.)	Sol. at -18°
105.	Dihydronaphthalene...(E., pract.)	Sol. at -15°
106.	Dimer of 1-phenyl-butadiene-(1,3)... (M.E.Betts)	C.S.T.:26.5°
107.	Diphenyl-methane...(student preparation)	Sol. at -24°
108.	Ph ₂ C=CH.Me...(W.E.Higbee)	Sol. at -16°
109.	α-Methyl-naphthalene...(E., pract.)	Sol. at -17°
110.	β-Methyl-naphthalene...(E., pract.)	Sol. at -17°

111.	1,6-Dimethyl-naphthalene...(C.)	Sol. at -15°
112.	Anisole...(E.)	Sol. at 20°
113.	Phenetole...(E.)	Sol. at 20°
114.	o-Cresyl methyl ether...(Stock room)	Sol. at -18°
115.	m-Cresyl methyl ether,,,(Stock room)	Sol. at -18°
116.	p-Cresyl methyl ether...(C.)	Sol. at -19°
117.	Benzyl methyl ether...(E.)	Sol. at -17°
118.	Resorcinol dimethyl ether...(K.)	Sol. at -15°
119.	o-Cresyl ethyl ether...(E.)	Sol. at -18°
120.	m-Cresyl ethyl ether...(K.)	Sol. at -19°
121.	n-Butyl phenyl ether...(E.)	Sol. at -17°
122.	Eugenol methyl ether...(E.)	Sol. at -17°
123.	Isoeugenol methyl ether...(E.)	Sol. at -17°
124.	Safrole...(E.)	Sol. at 20°
125.	Isosafrole...(E., pract.)	Sol. at -17°
126.	Phenyl isoamyl ether...(J. Farnum)	C.S.T.:23.7°
127.	Diphenyl ether...(E.)	Sol. at 20°
128.	Dibenzyl ether...(E.)	Sol. at 20°
129.	α -Naphthyl methyl ether...(E.)	Sol. at -17°
130.	β -Naphthyl isoamyl ether...(E.)	C.S.T.: -1.5°

Solids

Non-aromatics:

131.	Laurone... (E.)	C.S.T.:110°
132.	Myristone... (S.P.Mulliken)	Insol. at 130°
133.	Stearone... (S.P.Mulliken)	Insol. at 130°
134.	Cetene... (syn.)	Insol. at 20°
135.	Hexadecine-(1)... (syn.)	C.S.T.:92°
136.	Dicyclopentadiene... (C., purified by G. Thomson).	C.S.T.:60°
137.	Camphene... (B.D.H.)	C.S.T.:76.5°

Aromatics:

138.	Durene... (E.)	Sol. at 100°
139.	Hexamethyl-benzene... (E.)	Sol. at 100°
140.	Hexaethyl-benzene... (E.)	Sol. at 83°
141.	Octahydroanthracene... (C.)	C.S.T.:75.8°
142.	Dibenzyl... (E.)	Sol. at 50°
143.	p,p'-Ditolyl... (L.V.Peakes)	C.S.T.:69°
144.	Ph ₂ C=CHPh... (C.B.Wooster)	C.S.T.:21°
145.	Tetraphenyl-ethylene... (J.M.Farnum)	Sol. at 100°
146.	Diphenyl... (E.)	Sol. at 31°
147.	Naphthalene... (Stock room)	Sol. at 46°
148.	2,6-Dimethyl-naphthalene... (C.)	C.S.T.:61.6°
149.	Anthracene... (Stock room)	Sol. at 100°
150.	9-Isoamyl-anthracene... (H.T.Gerry)	Sol. at 20°
151.	Diphenyl ether... (E.)	Sol. at 20°
152.	Neroline... (K.)	Sol. at 18°

SOLUBILITIES OF HYDROCARBONS IN ANILINE

Paraffins:

1. Petroleum ether	Insol. at 20°
2. n-Hexane...(syn.)	C.S.T.:64.5°
3. n-Octane...(syn.)	C.S.T.:66.5°
4. 2,2,4-Trimethyl-pentane...(A.D.Little)	C.S.T.:77.5°
5. n-Decane...(E.)	Insol. at 35° ^x
6. Diisoamyl...(syn.)	C.S.T.:77.2°
7. n-Tetradecane...(E.)	Insol. at 60°

Naphthenes:

8. Hexahydrobenzene...(H.W.Underwood, Jr.)	Insol. at 20°
9. Hexahydrotoluene...(E.)	Insol. at 20° ^x
10. Decahydronaphthalene...(H.W.Underwood, Jr.)	Insol. at 20°
11. p-Menthane...(E.)	Insol. at 35°

Olefines:

12. Amylene...(E., tech.)	Sol. at 20°
13. Hexene-(1)...(syn.)	Insol. at 20°
14. 4-Methyl-pentene-(2)...(syn.)	C.S.T.:41°
15. Heptene-(1)...(syn.)	Insol. at 20°
16. Heptene-(2)...(syn.)	C.S.T.:28°
17. Heptene-(3)...(syn.)	C.S.T.:39°
18. 4-Methyl-hexene-(1)...(syn.)	C.S.T.:28°
19. 5-Methyl-hexene-(1)...(syn.)	C.S.T.:32°

20.	2,4-Dimethyl-pentene-(2)...(syn.)	Insol. at 20 ^x
21.	Octene-(2)...(syn.)	C.S.T.:32.7°
22.	4-Methyl-heptene-(2)...(syn.)	C.S.T.:42°
23.	Diisobutylene▼▼▼(syn.)	Insol. at 25°
24.	Nonene-(1)...(syn.)	C.S.T.:40°
25.	Nonene-(4)...(syn.)	C.S.T.:45°
26.	4-Methyl-octene-(2)...(syn.)	C.S.T.:50°
27.	4,5-Dimethyl-heptene-(2)...(syn.)	C.S.T.:52°
28.	4,6-Dimethyl-heptene-(2)...(syn.)	C.S.T.:55.4°
29.	4,5,5-Trimethyl-hexene-(2)...(syn.)	C.S.T.:52°
30.	Decene-(1)...(syn.)	C.S.T.:48°
31.	4-Butyl-octene-(2)...(syn.)	C.S.T.:65°
32.	Cetene...(syn.)	Insol. at 20°
33.	Cyclohexene...(E.)	Sol. at 20°
34.	1-Methyl-cyclohexene-(1)...(E.)	Sol. at 20°
35.	Allyl-cyclohexane...(syn.)	C.S.T.:8.0
36.	4-Cyclohexyl-pentene-(2)...(syn.)	C.S.T.:37°
37.	4-Cyclohexyl-heptene-(2)...(syn.)	C.S.T.:44°
38.	Pinene...(E., tech.)	Sol. at 20°

Diolfines and Acetylenes:

39.	Pentadiene-(1,3)...(syn.)	Sol. at -20°
40.	Diallyl...(F.Cortese)	Sol. at -20°
41.	Heptadiene-(2,4)...(syn.)	Sol. at -18°
42.	Heptine-(1)...(syn.)	Sol. at 20° ^x
43.	Octadiene-(2,4)...(syn.)	C.S.T.: -11°

44.	4-Methyl-heptadiene-(1,5)...(syn.)	C.S.T.:12°
45.	2,2-Dimethyl-hexadiene-(3,4)...(syn.)	C.S.T.: -14°
46.	Octine-(1)...(Syn.)	Sol. at -25°
47.	Nonine-(4)...(syn.)	Sol. at 35° ^x
48.	4,5-Dimethyl-octadiene-(2,6)...(syn.)	C.S.T.:31°
49.	4-Propyl-heptadiene-(1,5)...(syn.)	C.S.T.:29.7°
50.	4-Allyl-octene-(2)...(syn.)	C.S.T.:32°
51.	4,5-di-n-Butyl-octadiene-(2,6)...(syn.)	C.S.T.:65°
52.	Limonene...(E.)	Sol. at 35° ^x

Aliphatic ethers:

53.	Ethyl ether...(stock purified)	Sol. at 20°
54.	Methyl butyl ether...(E.)	Sol. at 20°
55.	n-Butyl ether...(E.)	Sol. at 20°
56.	Isoamyl ether...(E.)	Sol. at 20°

Aromatics:

57.	Benzene...(stock)	Sol. at 20°
58.	Toluene...(stock)	Sol. at 20°
59.	Xylene...(stock)	Sol. at 20°
60.	Ethyl benzene...(student preparation)	Sol. at 20°
61.	p-Cymene...(A.E.Schneider)	Sol. at 20°
62.	Tetrahydronaphthalene...(H.W.Underwood, Jr.)	Sol. at 20°
63.	Diphenyl-methane...(Student preparation)	Sol. at 20°
64.	Phenetole...(E.)	Sol. at 20°
65.	Diphenyl-oxide...(E.)	Sol. at 20°

x Results by Gilbert C. Toone in the course of work in his course in Advanced Qualitative Organic Analysis in 1929-30.

SOLUBILITIES OF HYDROCARBONS IN DIMETHYL SULFATE

1. Petroleum ether...(stock)	Insol. at 20°
2. Kerosene...(stock)	Insol. at 20°
3. 2,2,4-Trimethyl-pentane...(A.D.Little)	Insol. at 100°
4. Amylene	Sol. at 20°
5. Decene-(1)	C.S.T.:129°
6. Heptadecene-(8)	Insol. on warming to 100°
7. Benzene	Sol. at 20°
8. Toluene	Sol. at 20°
9. Xylene	Sol. at 20°

SOLUBILITIES OF HYDROCARBONS IN DIETHYL SULFATE

Aromatics:

1. Benzene...(stock)	Sol. at 20°
2. Toluene...(stock)	Sol. at 20°
3. Xylene...(stock)	Sol. at 20°
4. Pseudocumene...(stock)	Sol. at 20° (orange color)
5. Mesitylene... (stock)	Sol. at 20° (purple color)
6. p-Cymene...(stock)	Sol. at 20°
7. Diphenyl-methane...(student preparation)	Sol. at 20°
8. Anthracene...(stock)	Difficultly sol. on warming
9. Triphenyl-methane...(E.)	Sol. at 20° (yellow color)
10. Tetraphenyl ethylene...(J.Farnum)	Difficultly sol. on warming
11. Tetrahydronaphthalene...(H.W.Underwood, Jr.)	Sol. at 20°
12. Diphenyl ether...(E.)	Sol. at 20°

Paraffins:

13. Petroleum ether...(stock)	Insol. at 20°
14. Kerosene...(stock)	Insol. at 20°
15. n-Hexane...(syn.)	C.S.T.:69°
16. n-Octane...(syn.)	C.S.T.:79°
17. 2,2,4-Trimethyl-pentane...(A.D.Little)	C.S.T.:73°

18.	Diisoamyl...(syn.)	C.S.T.:90°
19.	Vaseline...(stock)	Insol. on warming
20.	Paraffin...(stock)	Insol. on warming

Naphthenes:

21.	Hexahydrobenzene...(H.W.Underwood, Jr.)	Insol. at 20°
22.	Decahydronaphthalene...(H.W.Underwood, Jr.)	Insol. on warming

Unsaturation:

23.	Amylene...(stock)	Sol. at 20°
24.	Hexene-(1)...(syn., Tech.)	C.S.T.:10°
25.	4-Methyl-pentene-(2)...(syn.)	C.S.T.:22°
26.	Heptene-(1)...(syn. Tech.)	C.S.T.:8°
27.	Heptene-(2)...(syn.)	C.S.T.:14°
28.	4-Methyl-hexene-(1)...(syn.)	C.S.T.:4-5°
29.	5-Methyl-hexene-(1)...(syn.)	C.S.T.:16°
30.	Octene-(2)...(E.)	C.S.T. 12°
31.	4-Methyl-heptene-(2)...(syn.)	C.S.T.:37°
32.	Diisobutylene...(H.S.Davis)	Insol. at 25°
33.	Nonene-(1)...(syn.)	C.S.T.:38°
34.	4-Methyl-octene-(2)...(syn.)	C.S.T.:48°
35.	4,5-Dimethyl-heptene-(2)...(syn.)	C.S.T.:46°
36.	4,6-Dimethyl-heptene-(2)...(syn.)	C.S.T.:48°
37.	4,5,5-Trimethyl-hexene-(2)...(syn.)	C.S.T.:41°
38.	Decene-(1)...(syn.)	C.S.T.:49°
39.	4-Butyl-octene-(2)...(syn.)	C.S.T.:70°

40.	Cetene... (W.E.Messer)	Insol. at 100°
41.	2-Methyl-nonadecene-(1)... (Messer)	C.S.T.:112°
42.	Allyl-cyclohexane... (syn.)	C.S.T.:16°
43.	4-Cyclohexyl-pentene-(2)... (syn.)	C.S.T.:57°
44.	Pinene... (stock, tech.)	Sol. at -15°
45.	Pentadiene-(1,3)... (syn.)	Sol. at -20°
46.	Heptadiene-(2,4)... (syn.)	Sol. at -18°
47.	Octadiene-(2,4)... (syn.)	Sol. at -20°
48.	Octine-(1)... (syn.)	Sol. at -20°
49.	2,2-Dimethyl-hexadiene-(3,4)... (syn.)	Sol. at -18°
50.	4-Methyl-heptadiene-(1,5)... (syn.)	C.S.T.: -5°
51.	4,5-Dimethyl-octadiene-(2,6)... (syn.)	C.S.T.:31°
52.	4-Allyl-octene-(2)... (syn.)	C.S.T.:34°
53.	4-Propyl-heptadiene-(1,5)... (syn.)	C.S.T.:26°
54.	4,5-di-n-Butyl-octadiene-(2,6)---(syn.)	C.S.T.:78°
55.	Hexadecine-(1)... (syn.)	C.S.T.:79°

Ethers:

56.	n-Butyl ether... (E.)	Sol. at -15°
57.	Isoamyl ether... (E.)	C.S.T.: -11°

SOLUBILITIES OF HYDROCARBONS IN BENZYL ALCOHOL

Paraffins:

1. Petroleum ether...(stock)	Insol. at 20°
2. n-Hexane...(syn.)	C.S.T.:57°
3. n-Heptane...(E.)	Insol. at 20° ^{xx}
4. n-Octane...(syn.)	C.S.T.:54.5°
5. 2,2,4-Trimethyl-pentane...(A.D.Little)	C.S.T.:73.6°
6. n-Decane...(E.)	Insol. at 50° ^x
7. Diisocamyl...(syn.)	C.S.T.:71.9°
8. n-Tetradecane...(E.)	Insol. at 70° ^x

Naphthenes:

9. Cyclohexane...(H.W.Underwood,Jr.)	Sol. at 20°
10. Hexahydrotoluene...(E.)	Sol. at 30° ^x
11. p-Menthane...(E.)	Sol. at 50° ^x
12. Decahydronaphthalene...(H.W.Underwood,Jr.)	Sol. at 20°

x Results by Gilbert C. Toone in the course of work in his course in Advanced Qualitative Organic Analysis in 1929-30

xx H. R. Batchelder, in the course of Advanced Organic Qualitative Analysis, 1928-29.

For the solubility of hydrocarbons in sulfuric acid and their color reactions with it, see the specific characterization section. In general it may be said that acetylenes are completely miscible with sulfuric acid (d_4^{20} :1.84) and aromatics are not thus miscible when the acid is slowly added to the hydrocarbon surrounded with an ice bath, then shaken and allowed to warm up to room temperature. The tests made with sulfuric acid were carried out in the small test tube with 0.1 cc. of each reactant, as previously described for other solubility tests. See paragraph 132.

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The solubility tests here described appear to make it possible to divide the hydrocarbons into groups, largely on the basis of their miscibility with equal volumes of various solvents at various temperatures, according to their molecular weights. Solvent properties alone, however, are not sufficient to make a clean-cut division. For example, in general, aromatic hydrocarbons are soluble in nitromethane at far lower temperatures than non-aromatic, but heptane - (1), ethyl-isoamyl ether, and similar compounds are soluble at -17° in this solvent. These compounds can of course be readily distinguished from the aromatics because of their far lower gravity.

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Thinking first, then, of a method of distinguishing liquid aromatic hydrocarbons from other liquid species, it may be said that if the gravity of the compound is less than 0.84 it is not aromatic, if above, it may be aromatic, but not necessarily. The lowest densities reported in the tables for aromatic hydrocarbons are : mesitylene, d_4^{20} : 0.856; p-cymene, d_4^{20} : 0.857; 4-benzyl-heptane, d_4^{20} : 0.854;

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n-octyl-benzene, d_{15}^{15} : 0.849, except for 1-phenyl-hexene-(5), reported d_{4}^{20} : 0.844, which is probably considerably in error. This gives ample opportunity for errors in the determination of density without affecting the decision as to the type of compound. If the density lies between 0.84 and 0.87 the hydrocarbon may be an aromatic with a saturated side chain. It cannot be any other type of aromatic, as a glance through the tables will show. It might equally well be a terpene, or some other cyclic compound. The terpenes are not soluble in nitromethane below 25° , as the critical solution temperatures show. If the way in which solubilities change with structure be studied from the tables just given, it will be evident that a conjugated diolefine should be more soluble in nitromethane than an isomer whose unsaturations lie farther apart. Phellandene is a conjugated terpene. It has a critical solution temperature in nitromethane of 37° . This being the case, it may be said that if the hydrocarbon boils below 200° and has a density lying between 0.84 and 0.87 and is not soluble in nitromethane below 25° it is not aromatic (it may be a terpene). The cyclohexyl acetylenes have densities between 0.84 and 0.87. These compounds have not been handled in this work but it is probable, by comparisons, that their solubilities approximate those of some aromatics of similar boiling points. Apparently all acetylenes are completely soluble in concentrated sulfuric acid ($d:1.84$) at room temperature, whereas aromatic hydrocarbons are not miscible with sulfuric acid under these conditions, provided the side chains are saturated. The development of color or partial

reaction in either case has nothing to do with the miscibility in this description. It is thus possible to remove the cyclohexyl acetylenes (and allenes, if they were found soluble in nitromethane) from the aromatic group. Other groups are removed from the aromatics by similar properties. Thus, perhydrophenanthrene, $d_{20}^{20} : 0.933$, is placed apart from the aromatics because of its insolubility in nitromethane. The sesquiterpenes are removed from their isomeric aromatic hydrocarbons on the same basis.

Of the liquid hydrocarbons remaining, the ethers (included in this Genus because of their failure to respond to the tests of the first eight geni of Mulliken, "Identification of Pure Organic Compounds") are soluble in two volumes of hydrobromic acid (d:1.48) or one volume of sulfuric acid (d:1.84) at 0°, and in general are thrown out of solution from the latter solvent in their original state by dilution with water. The behavior of these compounds toward hydrobromic acid is a reaction with which the writer is unfamiliar as far as actual laboratory experience is concerned, but which has been described to him by George W. Rigby. For details his Ph.D. thesis should be consulted. (47) The desirability of a phase including solubility in hydrobromic acid lies in the fact that the ethers of tertiary butyl alcohol (and probably of other alcohols) dissolve in sulfuric acid, but at once react to give diisobutylene etc. as a separate layer, making this test unsuitable as a means of distinguishing ethers of tertiary alcohols. (47) Hydrobromic acid, on the other hand, under the conditions here described holds these ethers in complete solution at 0°; no second layer being formed. (47) The ethers of secondary and primary alcohols are provided for on the basis of their

customary solubility in sulfuric acid and rejuvenation on dilution, the exceptions being provided for by the hydrobromic acid procedure. The ethers may thus be removed from the other hydrocarbons.

Of the compounds remaining, the terpenes, the acetylenes, the cyclic olefins, and the diolefines (whose unsaturation is no more widely separated than in the diallyl configuration) are considerably more soluble in aniline than the olefines, paraffins, and naphthenes. The difference in solution temperatures which exists here may be employed in dividing the former group from the latter. In the diolefines whose unsaturation is separated in the molecules more widely than in diallyl, each double bond must necessarily act independently toward bromine. Since the bromide-bromide titrations previously described showed no olefine which substituted and added a total of more than 2.6 atoms of bromine, and no diallyl homologue which added less than 3.2 atoms of bromine, and since the latter type diolefine undoubtedly adds less than those in which the double bonds are farther apart, the remaining diolefines may be separated from the olefins^c by the addition of more than 3.25 atoms of bromine per molecule. The impossibility of using a bromine titration, alone, as a means of dividing these two groups is readily seen by noticing the divergence, in some cases, extreme, between the theoretical and the found bromide-bromate numbers. This is particularly true of the acetylenes and terpenes.

Of the groups remaining, i.e. the olefines, the naphthenes, and the paraffins, the first is distinguishable from the last two by addition of

bromine by its members. Bromide-bromate titrations made by H. R. Batchelder, V. Schneider, G.C. Toone, and the writer, on a number of paraffins and naphthenes has shown that in no case tested has sufficient substitution taken place to give values even approaching one half of the normal value for eicosane. The compounds tested include 3-methyl-pentane, n-hexane, n-heptane, n-octane, 2,2,4-trimethyl-pentane, n-decane, diisooamyl, n-tetradecane, hexahydro-benzene, hexahydrotoluene, and hexahydro-p-cymene.

There are thus left, the naphthenes and the paraffins. A glance at the rather inadequate table of solubilities in benzyl alcohol shows that this solvent can be used to distinguish between these two types of compounds. The naphthenes are much more soluble in this solvent. 73

This series of properties is, in brief, the basis of the procedure, given in the following pages, for the sectional division of the hydrocarbons. A detailed written description of the scheme would be altogether too involved to be of any value whatsoever. It is best presented in the form of the chart given later. It need only be said that the temperature limits both for boiling points and solubilities in various sections appear very arbitrary. They have been selected, however, in an attempt to give the greatest possible margin of safety. A single example will suffice, 74

Phellandrene boils at 171-2° and has a density of 0.852 at 20°. It is a conjugated diolefine, and therefore probably one of the most soluble of the terpenes. Tertiary amyl benzene boils at 189-90° and has a density of 0.866 at 22/4. It is probably one of the least soluble of the aromatics boiling around 190°. In nitromethane phellandrene gives a C.S.T. of 37°, tertiary-amyl-benzene a C.S.T. of 13.5°. Neither of these compounds were purified in this work, but used as obtained from the British Drug Houses and from Eastman, respectively. The pure compounds would probably 75

show no lower solution temperatures (see discussion in the specific characterization section concerning critical solution temperatures in nitrobenzene of the paraffins[^]). ^{Paragraph 138} A dividing line of 25° for the critical solution temperature in nitromethane of hydrocarbons boiling between 160° and 190° has therefore been set on this basis. Compounds in this boiling point range soluble in nitromethane at 25° are aromatics (or compounds otherwise provided for), those insoluble in nitromethane at 25° are not aromatics. This provides for the highest boiling members of this group. The lower boiling aromatics are still more soluble in nitromethane than the higher boiling, and of the lower boiling terpenes, probably sabinene, boiling at 163-4°, is one of the most soluble. This has a C.S.T. in nitromethane of 52°. This temperature therefore provides also for the lower boiling compounds of this boiling point range.

The specific manner of carrying out the solubility tests is that given in the foregoing[^] and ^(page 516) in Test 922, following the tables (except for the test with hydrobromic acid, in which 0.1 cc. of hydrocarbon and 0.2 cc. of acid are used). 76

The hydrocarbons are divided into three divisions, solid (at 0°), liquid (at 0°), gaseous (at 20°). 77

The solids are further subdivided into two groups, the first containing principally non-aromatics, the second containing principally aromatics. The liquids are further subdivided into six sections, the first containing principally aromatics, and their very close relations, as the fulvenes, the second containing the aliphatic ethers, the third containing the cyclic unsaturates and the acyclic unsaturates of a higher degree of unsaturation than the olefines. This last group therefore contains diolefines, acetylenes, terpenes, and cyclic olefines. The fourth section contains the acyclic olefines, the fifth the naphthenes, and the sixth the paraffins.

The method of using this sectional division chart, which follows at once, can best be shown by specific illustrations. In the illustrations reference is made to the specific characterization of the compounds. The discussion of this is in the next section of this work, and the procedure will be found in the tables. The illustrations are taken solely from a "Report on the Testing of a Tentative Scheme for the Qualitative Analysis of Hydrocarbons proposed by R.L. Wakeman" by Gilbert C. Toone, January 10, 1930, written for the advanced course in organic qualitative analysis.

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Slight changes have been made in the wording of these illustrations in order to make it conform to the final scheme given here. No change has been made which violates any experimental result or conclusion, the changes have been made merely for the sake of clarity. The illustrations follow the scheme.

Reagents

The solvents and reagents used have been prepared as follows:

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1. Nitromethane -- Eastman, pure.
2. Aniline -- Stock material allowed to stand with potassium hydroxide (stick) and then distilled, the first third of the distillate being discarded and the next, constant-boiling, third being used in these tests.
3. Benzyl alcohol -- Eastman, pure.
4. Hydrobromic acid -- colorless, d: 1.48.
5. Sulfuric acid -- colorless, d: 1.84.
6. 0.5 N. bromide-bromate solution -- 56 gms. of C.P. potassium bromate and 200 gms. of C.P. potassium bromide were dissolved in 4 liters of distilled water and thoroughly shaken. Standardized against 0.25 gms. of purified aniline.
7. 0.2 N. thiosulfate solution -- 100 gms. of C.P. $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ were dissolved in 2 liters of distilled water, thoroughly shaken and standardized against 0.3 gms. of C.P. potassium dichromate.
8. Dimethyl sulfate -- Eastman, tech., shaken with 10% sodium bicarbonate solution and dried with calcium chloride.

9. Diethyl sulfate -- purified as dimethyl sulfate.

Note: -- these last two solvents have been abandoned in the final procedure.

KEY TO THE CHART

The Tables of Properties are listed in three

Divisions, - A, B, and C.

Division A contains compounds which are solid at 0°.

See the chart on page 76.

Division B contains compounds which are liquid at 0° and boil above 20°. See the chart on page 81. The temperatures at which solubilities in various solvents should be determined are given for various boiling point ranges as follows:

Nitromethane	77
Aniline	78
Benzyl Alcohol	79

The density dividing line for determining whether an unsaturated is cyclic or acyclic is given for various boiling point ranges on page 80.

Division C contains compounds which are gaseous at 20°. It is not subdivided and therefore has no chart.

DIVISION A

Solid @ 0°

Melting Point

0° - 40°

40° - 110°

above 110°

Solubility in
(Test 922)
CH₃NO₂ @ 65°

Boiling Point

SECTION 2 !!

Insoluble
SECTION 1 !!

Soluble
SECTION 2 !!

< 200°

> 200°

Insoluble
SECTION 1 !!

Solubility
(Test 922)
in CH₃NO₂ @ 65°

Solubility in
(Test 922)
CH₃NO₂ @ 100°

Soluble
SECTION 2 !!

Soluble
SECTION 2 !!

Insoluble
SECTION 1 !!

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Temperatures for the Determination of Solubility
in Nitromethane.

If the Compound boils between	Determine its solubility in CH_3NO_2 at
20° and 40°	0°
40° and 70°	15°
70° and 130°	0°
130° and 160°	10°
160° and 190°	25°
190° and 220°	40°
220° and 250°	55°
250° and 280°	70°
above 280°	85°

Temperatures for the Determination of Solubility
in Aniline.

If the compound boils between	Determine its solubility in aniline at
70° and 130°	0°
130° and 190°	15°
190° and 240°	30°
240° and 285°	45°
above 285°	60°

Temperatures for the Determination of Solubility
in Benzyl Alcohol.

If the compound boils between	Determine its solubility in $C_6H_5CH_2OH$ at
20° and 70°	20°
70° and 130°	30°
130° and 190°	50°
above 190°	70°

The Density Dividing Line Between Unsaturated
Cyclics and Acyclics.

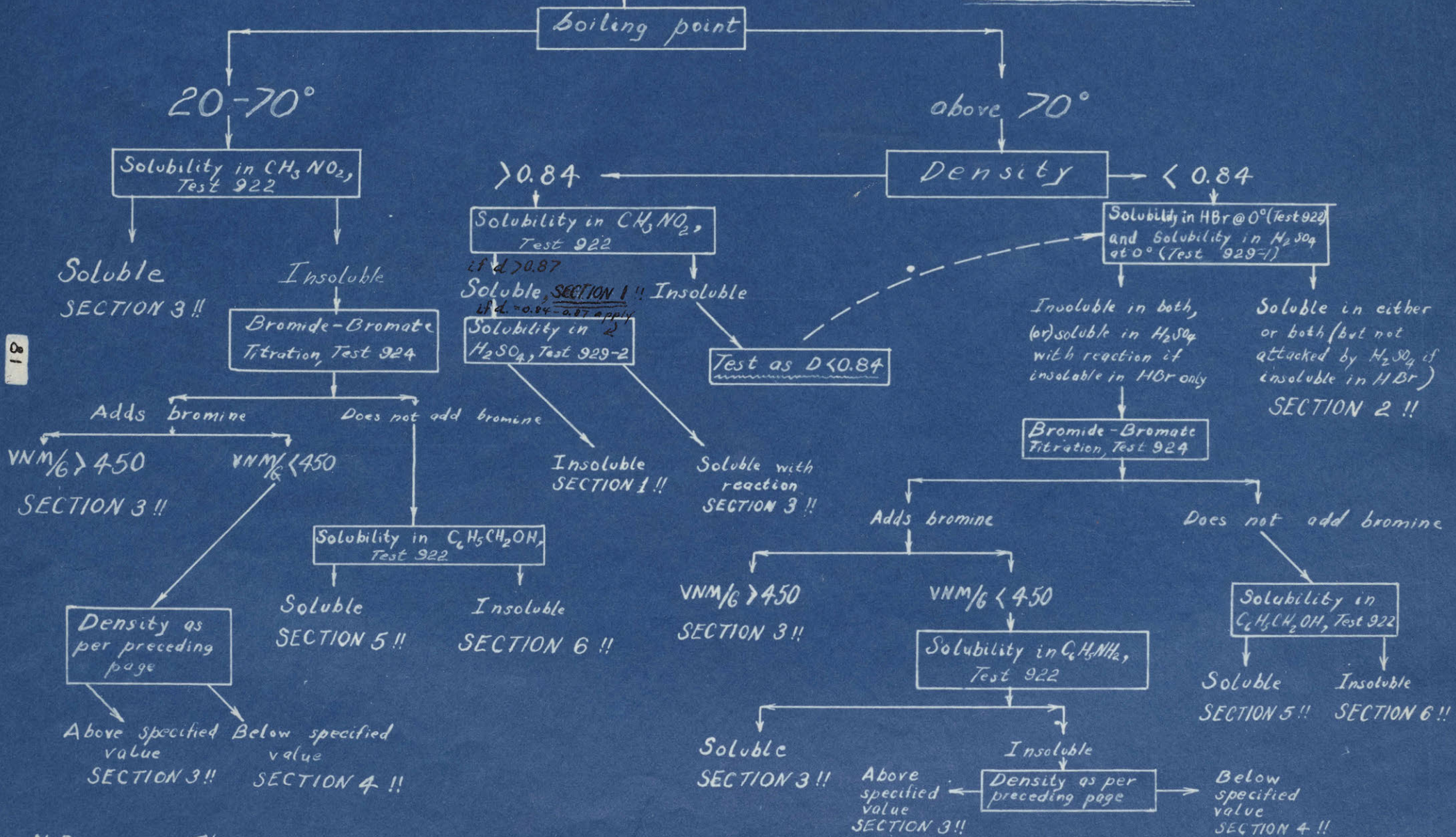
If the compound boils between	It belongs in Div. B, Sect. 4 (if it is unsaturated) if it has a lower density at 20°/4° than
----------------------------------	--

20° - 40°	0.70°
40° - 70°	0.73°
70° - 130°	0.78°
130° - 190°	0.80°
190° - 300°	0.83°

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Liquid @ 0°
(B.p. above 20°)

DIVISION B



N.B. → The temperatures at which to determine solubilities in various solvents are given in the immediately preceding pages!

EXAMPLES OF THE USE OF THE CHART

The following illustrations of the way the divisional chart is used are taken from work done by Gilbert C. Toone in his graduate course in Organic Qualitative Analysis. An abridged edition of the tables was used for the selection of the specific compounds.

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The illustrations are taken from Division B. This is the most difficult part of the chart, that part dealing with Division A being used similarly.

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Sectional tests and results

State at 0° liquid
 B.P. 102°
 S.G. 0.726
~~Sol. $C_2H_5NH_2$ at 0° soluble~~
 Sol. H_2SO_4 at 0° reacts violently
 Sol. HBr at 0° insoluble
 Add. Br₂
 Sol. $C_2H_5NH_2$ at 0° VNM/G < 450
 soluble

Possibilities of similar boiling point

	B.P.	S.G.
1. 3-ethyl-pentadiene-(1,2)	96.8	0.728
2. heptine-(1)	99.5-100.8	0.731
3. heptaine C_7H_{14}	103-4	0.831
4. ethyl-propyl-acetylene	105-6	0.740

Specific Tests

Ammoniacal $AgNO_3$ white precipitate
 Hg salt; test 926 M.P. 58.5-59.0

Test 926 is a specific test which identifies the unknown beyond doubt*

Sectional tests and results

State at 0° liquid
 B.P. 146°
 S.G. at 20° 0.911
 Sol. CH_2NO_2 at 10° soluble

Possibilities of similar boiling point

	B.P.	S.G.
1. p-xylene	138	0.860
2. m-xylene	139.2	0.866
3. phenyl acetylene	141.6	0.930
4. o-xylene	142	0.873
5. crotonyl ether	143-5	0.870
6. styrene	146	0.905

Specific tests

Test 902; H_2SO_4 polymerizes
 Test 903; HNO_3 soluble

All but styrene are eliminated by the specific gravity.

Sectional tests and results

State at 0° liquid
 B.P. 169-170
 S.G. at 20° 0.742
~~Sol. in H₂O + H₂SO₄ at 15°~~ ~~insoluble~~
 Add. Br₂ unsaturated; 3.08 cc.
 Formula $\frac{VNM}{G} > 450$ less than 450
 Sol. in C₆H₅NH₂ at 15° insoluble

Possibilities of similar boiling points

	B.P.	S.G.	P.I.
1. decene-(1)	172	0.743	1.4383
2. triisobutylene	177.8	0.754	

Specific tests

C.S.T. C₆H₅NH₂ 44°
 B.B. no. 129
 Hg salt; Test 927 M.P. 81-82°

The Hg salt is a specific test for decene-(1).

Sectional tests and results

State at 0° liquid
 B.P. 166°
 S.G. at 20° 0.798
~~Sol. in H₂O - H₂SO₄ at 15°~~ ~~insoluble~~
 Add. Br₂ saturated
 Sol. C₆H₅CH₂OH at 50° soluble

Possibilities of similar boiling point

	B.P.	S.G.
1. dekanaphthene	160-2	0.781
2. α terpene tetrahydride	160-2	0.783
3. β terpene tetrahydride	164	0.793
4. hexahydro-p-cymene	abt. 170	0.795

Specific Tests

None

1 and 2 eliminated by S.G.
 Not enough data to identify specifically.

Sectional tests and results

State at 0°	liquid
B.P.	250°
S.G. at 20°	0.763
<i>Sol. in</i> Sol. C₆H₅NH₂ at 45°	insoluble ← <i>insoluble</i>
<i>HBr-H₂SO₄</i> Add. Br ₂	saturated
Sol. C ₆ H ₅ CH ₂ OH at 70°	insoluble

Possibilities of similar boiling point

	B.P.	S.G.	R.I.	M.P.
1. tetradecane	252.5°	0.765	1.4459	5.5°

Specific Tests

M.P. 6.0°

Physical constants show that unknown is tetradecane.

Time 2 hours

Sectional tests and results

State at 0°	liquid
B.P.	253°
S.G. at 20°	0.819
<i>Sol. in</i> Sol. C₆H₅NH₂ at 45°	soluble ← <i>insoluble, but attacked</i>
<i>H₂SO₄ 0°</i> Sol. H₂SO₄ at 0°	insoluble, but attacked
Add. Br ₂	<i>v_{max}/λ > 4.50</i>

Possibilities of similar boiling point

	B.P.	S.G.
1. 4,5-di-n-propyl-octadiene-(2,6)	221	0.780 (25-4 vac.)
2. 4-cyclohexyl-heptene-(2)	230	0.836 (25-4 vac.)
3. sesquiterpenes	250-80	0.904-.922
4. 4,5-di-n-butyl-octadiene-(2,6)	252	0.795 (25-4 vac.)

Specific Tests

C.S.T. (C₆H₅NH₂) 54°
B.B. no. 145

3 is eliminated by the S.G.

4 is indicated by the B.P.

4 is indicated by the B.B. no.

The unknown is the di-butyl-diolefine:

Sectional tests and results

State at 0°	liquid
B.P.	131-136°
S.G. at 20°	0.778
Sol. C₆H₅NH₂ at 20°	soluble
Sol. H ₂ SO ₄ at 0°	soluble

Possibilities of similar boiling point

	B.P.	S.G.
1. ethyl hexyl ether	134.7	
2. butyl ether	141	0.769
3. trimethyl glycol dimethyl ether	140	0.840

Specific tests

None

3 is eliminated by the S.G.

The odor indicates butyl ether. A dinitrobenzoate derivative would prove this. It was considered unnecessary to make this derivative.

Time 1 3-4 hours

Sectional tests and results

State at 0°	liquid
B.P.	111.5-112°
S.G.	0.834
Add. Br ₂	unsaturated; 3.75 c.c. ^{VHM} / _G <450
Sol. C ₆ H ₅ NH ₂ at 0°	soluble
Sol. H ₂ SO ₄ at 0°	insoluble
Sol. HBr at 0°	insoluble

Possibilities of similar boiling point

	B.P.	S.G.
1. 4-methyl-heptadiene-(1,5)	110	0.728 (25-4 vac.)
2. heptene-(2)	111	0.743
3. 2,5-dimethyl-hexadiene-(1,5)	113-4	0.751 (20)
4. 1-methyl-cyclohexene-(1)	111-2	0.802

Specific Tests

Sol. H_2SO_4 at 25° insoluble
B.B. no. 120
C.S.T. ($C_6H_5NH_2$) below 0°

2 is eliminated by H_2SO_4 test at 25°
1 and 3 are eliminated by the specific gravity.

LIMONENE SECTION 3, DIVISION B Time 2 1-2 hours

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Sectional tests and results

State at 0° liquid
B.P. 179-80°
S.G. at 20° 0.866
Sol. CH_2NO_2 at 25° insoluble
Add. Br_2 unsaturated, *vnm/r > 450*
Insol. in HBr, H₂SO₄ *Insol. in HBr; Attacked by H₂SO₄.*

Possibilities of similar boiling point

	B.P.	S.G.	R.I.	S.R.P.
1. sylvestrene	176-7	0.851	1.4774	65°
2. α -limonene	176.5	0.843	1.4743	125°
3. terpinene	179-82	0.855	1.4846	
4. β -limonene	181-2	0.844	1.4727	
5. terpinolene	183-5	0.855	1.4823	

Specific tests

Br_2 derivative no results
R.I. 1.4771 (17)
S.R.P. 103.5°

The specific rotatory power shows that the unknown is limonene. The other constants seem to indicate sylvestrene.

C

Methods of Specific Characterization

METHODS OF SPECIFIC CHARACTERIZATION

A number of reactions for the specific identification of various types of hydrocarbons have been investigated. Of those tried, only a few have been thought really valuable and investigated beyond a preliminary test. The procedure followed in developing this part of the work was to give a cursory examination of a number of possible methods and then to investigate more carefully only those procedures which after a hurried examination offered hope of developing into quick methods capable of being applied by one who was not possessed of any high degree of specialized technique. This consideration is of course necessary in order to develop a procedure of any value whatsoever as a general qualitative scheme. The rather hasty discarding of a number of possibilities finds its explanation in these remarks. The writer feels confident, however, that certain of the procedures not here carefully investigated offer real opportunity for further investigation. It was felt wisest to develop, in this work, the most promising procedures. Unfortunately, the lack of time necessitated failure to investigate other valuable methods.

The use of benzoyl hydroperoxide to titrate unsaturated hydrocarbons has been referred to earlier. ^(See paragraph 37) It is reported that the glycols may be prepared from the oxides formed in this reaction. (1) From the reaction products of several titrations attempts were made to obtain the benzoyl or 2,5-dinitro-benzoyl esters of the glycols corresponding to the oxides formed. Hexene (1), amylene, heptene (1), and cetene were here used. Hydrations of the oxides were attempted by means of both dilute alkali and dilute acid, and esterification attempted by a Schotten-Baumann reaction with benzoyl chloride, and by refluxing with 3,5 dinitrobenzoyl chloride.

The titrations were run as earlier described; several runs were made in glacial acetic acid, some by evaporating nearly all of the chloroform, from a slightly warmed bath, by suction, and some by dilution of the chloroform with glacial acetic acid and water. In all of these attempts either the benzoate or the 3,5-dinitrobenzoate, ^{or the p-nitrobenzoate} was expected. Quantities varying from 0.5 to 1.0 cc. of hydrocarbon were used, and a large excess of benzoyl hydroperoxide. Universally no ester was obtained.

Following this, several attempts to prepare aromatic esters of the glycols by heating the dibromides of the olefines with the corresponding silver salts were made without success. (2,4) In these experiments silver benzoate, silver para nitro benzoate, and silver 3,5-dinitrobenzoate were prepared and added in excess to the dibromide of various olefines prepared from one cc. of hydrocarbon. Various solvents were used; ethyl alcohol, amyl alcohol, toluene, xylene, and glacial acetic acid. The time of refluxing was varied from two to ten hours. The various hydrocarbons used were amylene, hexene-(1), and heptene-(1). It was thought that perhaps the insolubility of the silver salts in the solvents chosen explained the failure of these

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attempts. It was therefore thought to try thallium salts, some of which have been reported as soluble in many organic solvents. (.). Thallic and thallic dinitrobenzoates were prepared from both the metal nitrates and the metal carbonates, and portions dissolved in a pyridine solution of 1,2-dibromopropane, (prepared by the absorption of propylene, from isopropylalcohol, in a carbon tetrachloride solution of bromine). A very slight precipitate was formed after a considerable period of boiling and no ester could be found. Benzene and ethyl alcohol did not dissolve either of the salts of thallium.

It is a remarkable fact that these esters are so much more difficult to prepare than the diacetates, which may be obtained

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see next page

with two and a half hours refluxing of the dibromide with silver acetate in acetic acid solution. (5)

Thus, cetene glycol was prepared by this latter process, followed by saponification of the resulting ester. The saponification required one and one half hours. This glycol, however, refused to give readily a crystalline dinitrobenzoate by the method of Tseng, (6) (although the dinitrobenzoate of ethylene glycol was readily prepared by this method. This latter compound melts at $170.3 - 170.6^{\circ}$ corrected, (6) and gives an α -naphthylamine addition product by the method of Reichstein (7) which, without purification, melts at $181-4^{\circ}$). Because the entire process of obtaining the dinitrobenzoate from the hydrocarbon requires about nine hours no further attempts were made to crystallize this ester of cetene glycol or to prepare other similar esters.

According to the literature nitrosochlorides of various olefines, diolefines, and terpenes have been prepared in a crystalline state. Most of the olefines give only unstable compounds. (8) In some cases what was once supposed to be the nitrosochloride has since been shown to be hydroxylamine hydrochloride. Schmidt (9) has prepared the nitrosochloride of trimethyl ethylene. A number of attempts to prepare this compound using technical amylene, amyl nitrite, and acetic acid, or nitrosylchloride from sodium nitrite and hydrochloric acid, failed. Likewise attempts to prepare the nitrosochlorides of diallyl and limonene gave only gums. Similarly several attempts to prepare crystalline nitrosates of diallyl, amylene, pinene, and limonene by means of liquid nitrogen tetroxide, prepared both from copper and nitric acid, and by heating lead nitrate, previously dried at 140° over a week-end, gave nothing but gums. In some of these experi-

ments a trace of crystalline material sometimes appeared, especially if the gum was extracted with carbon disulfide, in which it was not soluble, and the solution then evaporated. However, not enough material could in any case be crystallised to get a melting point. Undoubtedly the use of nitrosyl chloride as a reagent in the preparation of derivatives of cyclic olefines and diolefines of all kinds offers a valuable means of identification. The necessity of using ethyl nitrite as a reagent, however, instead of the rather unsatisfactory amyl nitrite tried here, coupled with the uncertainty of its reaction with olefines made it seem desirable to postpone further work with this reagent at this time. Unfortunately, time has not permitted a return to this investigation. The writer feels confident that a systematic investigation of its usefulness would be valuable.

A variety of experiments were made in attempts to prepare molecular compounds with olefines and mercury salts. Mixtures of one cc. of common glacial acetic acid, about one gram of mercuric acetate, and one cc. of hydrocarbon allowed to stand at room temperature for three hours gave the following results on neutralization with sodium hydroxide (neutral to phenolphthalein).

Cetene - liquid, solidifying to solid, M.P. 69-71°.

Technical Amylene - nothing.

Hexene (1) - heavy oil which would not solidify in a freezing mixture with scratching.

A number of other experiments varying the amounts of acetic acid and mercuric acetate, and either diluting with water or neutralising with caustic gave with amylene, hexene (1), and heptene (1) heavy oils, soluble in ether, but which could not be crystallized on cooling with a freezing

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mixture and scratching. In three cases, once with amylene and twice with hexene (1), after diluting the acetic acid solution with water and letting it stand for at least twelve hours, white, flaky crystals appeared which looked the same in all cases and did not melt but began to carbonize at 224°. In two of these cases, once with amylene and once with hexene (1), an oil also formed. Probably the crystals were of basic mercuric acetate. Diallyl gave neither an oil nor a crystalline precipitate, but considerable heat was evolved after adding it to the acetic acid solution of mercuric acetate. This reaction appeared unsuitable for specific characterization and was abandoned.

According to Henry (10) an acid solution of mercuric sulfate gives a yellow-orange precipitate with trimethyl ethylene. This is said also to apply to ethylene, propylene, and butylene. A few drops of amylene were added to a solution of mercuric sulfate in dilute sulfuric acid. A yellow precipitate formed at once, increasing in volume on standing a few minutes. It did not melt, but decomposed on heating, finally getting entirely black.

A few drops of diallyl were boiled in alcohol solution with 0.10 gms. of picric acid. On crystallization only picric acid resulted. Likewise T.N.T. and T.N.B. gave no molecular compounds with diallyl.

According to Bruson and Calvert (11) the addition of thiocyanogen to isoprene and dimethyl butadiene (using seven or more grams of hydrocarbon) gives crystalline products with definite melting points which may be used to identify these diolefines. To a solution of sodium thiocyanate in glacial acetic acid was added one cc. of hydrocarbon and the requisite amount of bromine in acetic acid. The solution was kept cold and in one case allowed to stand one and a quarter hours, in another, overnight.

After dilution the yellow precipitate was filtered, extracted with benzene, and precipitated with petroleum ether. Diallyl and hexadiene-(2,4) were used. In both cases only an insignificant amount of precipitate was obtained.

Chlorosulfonic acid added to amylene at room temperature or in freezing solution reacted vigorously, producing a black tar which gave no promise of yielding anything crystalline after treatment with phosphorous pentachloride and para toluidine. When hexene (1) was diluted with petroleum ether and treated in a freezing mixture with chlorosulfonic acid, reaction evidently occurred slowly because of darkening and increased viscosity of the acid, which was insoluble in the ether, but on evaporation of the ether, treatment of the residue with ice and ammonium hydroxide, extraction with ether, and evaporation of the latter, only a slight amount of gum remained. The solid insoluble in the ether is ammonium salt. Aniline similarly gives its hydrochloride (or sulphate).

Sulfuryl chloride does not react at once with technical amylene, but after mixing and standing with it for a minute it reacts explosively. The amylene reaction is similar. These reactions have not been further investigated.

The action of various polymerising agents on three isomeric hexadienes showed the following results, where + and - indicate respectively that polymerization did or did not ensue on addition of a few drops of hydrocarbon to a trace of the reagent.

Diallyl:

Cold ZnCl_2 -

Cold SnCl_4 -

Cold AlCl_3 + , with formation of brown-red solid polymer, yellow on addition of ethyl alcohol, same on heating with ethyl alcohol.

Hexadiene-(2,4):

Cold SnCl_4 + , with formation of red, viscous liquid; same on addition of ethyl alcohol; vigorous heating with alcohol gives light brown gum.

Cold ZnCl_2 - ,

+ when warmed gently by a free flame, with brown-red color. When warmed with ethyl alcohol leaves a heavy immiscible light yellow oil.

Cold PCl_3 + , with vigorous warming, red-yellow liquid.

Cold PCl_5 - ,

+ on heating, yellow to golden-red clear solution. Addition of ethyl alcohol gives a clear yellow solution.

Cold FeCl_3 + , with formation of green-brown gum, black on heating, same on boiling with ethyl alcohol. Cold addition causes heating.

Cold syrupy H_8PO_4 - ,

+ hot, nearly colorless on
addition of ethyl alcohol.

Repetition of this last experiment yielded
a light yellow-green color after shaking
15 minutes. No heat evolved.

2-Methyl-pentadiene-(2,4):

Cold $ZnCl_2$ - ,

+ when warmed by a free flame, brown
color. Ethyl alcohol on heating yields a
heavy, immiscible, light yellow oil.

Cold $SnCl_4$ + , with formation of brown limpid
liquid, slight amount of solid. On warming
turns brown-black. Addition of ethyl alcohol
leaves deep brown-black gum on heating.

Cold $FeCl_3$ + , with heating, light green-yellow
liquid.

Cold PCl_3 +, ?,

No colorizing, slight warming can be detected
by placing test tube against cheek. No color
on heating.

Cold PCl_5 - ,

+ on heating, red-black. Gives rust-
colored solution with ethyl alcohol.

Cold syrupy H_3PO_4 +,

Yellow-brown-red color slowly appears
on shaking for several minutes. Heating
develops after two or three minutes.

Repeated and checked.

The reactions here given indicate that it might be possible to use selective polymerizing agents, or agents which cause action of some sort with certain structures and not with others. It would be possible to distinguish the three hexadienes above described on this basis, for instance, as the table shows. But the chemistry of what happens here is so vague, and the presence of traces of impurities frequently makes such a vast difference, that it was felt wisest to try to develop some more certain means of characterization.

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In an attempt to characterize olefines, cetene was treated with hypobromous acid and the reaction product then treated with dinitrobenzoyl chloride, but no solid ester was obtained. A portion of the bromhydrin was treated with boiling aqueous-alcoholic ammonia and then picric acid, but no picrate of any nitrogen compound was obtained. It had been hoped that in the one case the 3,5-dinitrobenzoate of the bromhydrin, in the other case, the picrate of the bromo-amine or a substitution product might be obtained as crystalline derivatives. Cetene was used as probably the most unfavorable case. Further work was

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neglected, not because the reactions offered no hope, but for lack of time. It is felt that the preparation of compounds corresponding to triethanol amine is entirely possible, and these latter can undoubtedly be characterized through their picrates. Further study of this reaction is recommended.

(25) The action of sodium sulfide and sodium thiophenolate on olefine dibromides has been investigated, in the hope that sulfides or thiophenyl ethers might be obtained which might be further identified. Sodium sulfide offers no promise, but thiophenol, although no positive results have as yet been obtained, gives an immediate precipitate of sodium bromide in an alcoholic solution of sodium with propylene dibromide. It is recommended that this reaction be further investigated.

The preparation of mercury derivatives of certain monosubstituted acetylenes has been described by John R. Johnson.

(12) These derivatives give definite melting points without exploding. A list of some of the acetylenes from which he prepared such derivatives, together with the melting points of the derivatives follows:

<u>Acetylene</u>	<u>M.P. Mercury Salt</u>
Chloroethine	185° (dec. at 195°)
Bromoethine	dec. 153-5°
Propine-(1)	203-4°
Butine-(1)	162-3°

<u>Acetylene (cont.)</u>	<u>M.P. Mercury Salt (cont.)</u>
3,3-dimethyl-butine (1)	91-2 ^o
Heptine (1)	61 ^o
Decine (1)	83-4 ^o
Phenyl-ethine	124.5-125 ^o
3-Phenyl-propine (1)	106.5-107.5 ^o
4-Phenyl-butine (1)	84.5-85 ^o
4 ¹ -Methyl-phenyl-ethine	199-202 ^o (with previous darkening)
4 ¹ -Methoxy-phenyl-ethine	207-9 ^o (with previous darkening)
3-Cyclohexyl-propine (1)	104 ^o
3-Phenoxy-propine-(1)	120.5-121 ^o
Furyl-(2 ¹)-ethine	118-118.5 ^o
5-Bromofuryl-(2 ¹)-ethine	175-7 ^o

To an ice cooled solution of 3 cc. of Nessler's reagent (prepared by Melcher's assistant by dissolving 115 grams red, pure, HgI₂ and 80 grams pure KI in 500 cc. distilled water, adding 500 cc. 6 N. NaOH, and filtering after standing) were slowly added, with stirring, three drops of hexadecine (1) dissolved in 1 cc. of reagent alcohol. The precipitate was filtered immediately and after washing dissolved in about 5 cc. of hot benzene, boiled with decolorizing carbon and filtered. Transparent crystals separated, M.P. 94-5^o. Another experiment dissolving a little hexadecine (1) in three times its volume of xylene and using a larger volume of alcohol showed that the precipitation

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can take place in the presence of this hydrocarbon. Pentine-(1) similarly gave its mercury salt, M.P. 118.5-9^o.

Because the preparation of acetylenes from dibromides and sodamide appears to be a valuable reaction (13) it seemed advisable to seek to apply it to the specific identification of olefines with the double bond in position (1) which do not have side chains on the second carbon atom. This group comprises about one fourth of all of the possible (known and unknown) olefines.

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A bromide-bromate titration was run with 0.5 cc. of olefine according to the procedure given in paragraph 50. About ten cc. of petroleum ether were then added to the titration bottle, thoroughly shaken, then separated and dried with sodium sulfate. The petroleum ether solution of the dibromide was poured away from the sulfate, the sulfate washed twice with petroleum ether, the ether evaporated off by an air blast, and the dibromide heated under a reflux in a small flask with about two grams of finely powdered, fresh sodamide. An oil bath held at 150-160^o was used as the source of heat. The heating was continued for fifteen minutes. At the end of this time, the flask

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was immersed in a freezing mixture and slowly and cautiously treated with a solution of 5 cc. of HCl (1.19) in 20 cc. of water. The flask was shaken until no solid remained undissolved. The bottom, aqueous layer was withdrawn by a pipette (connected to an aspirator), the acetylene washed twice with 20-25 cc. of water, and then precipitated as its mercury salt, as in paragraph 113. Benzene, ethyl acetate, and petroleum ether were variously used as solvents for the recrystallization of the precipitate.

This procedure is the one finally adopted, and described later on page 524 as Test 927. It was developed by Charles W. Schroeder, M.I.T., 1931, after preliminary work on the part of the writer. The melting points of the derivatives thus far prepared by this procedure are listed on page 104, paragraph 121. Of those listed, the melting points of all but the derivatives of pentene-(1) and heptene-(1) were determined by Schroeder. 117

It should be mentioned that since sodamide causes rearrangement of disubstituted acetylenes to true acetylenes, (13) it may be that the procedure given would also give derivatives of olefines with the double bond in position two. Two other factors enter into the likelihood of this latter reaction, however. The one is that sodamide shows a marked tendency to react with dibromides of olefines with the unsaturation in on the chain as does the zinc-copper couple; i.e., it splits out two atoms of bromine to regenerate the olefine. G. C. Toone has applied the first procedure given (using xylene) to heptene-(3) and failed to get any precipitate. It is probable that the rearrangement of triple bonds in position three to position one is so slow, that even if enough acetylene (as heptene-(3)) should be formed to give a derivative on rearrangement it will not so rearrange under the time limit of this test. The action of olefines with the double bond in position two is a question. There is, however, sufficient difference between the boiling points and other constants of olefines of the same carbon skeleton containing the double bonds in positions one and two to prevent confusion if the same derivative is formed by the same test.

It had been hoped to apply this test to olefines with the double bond in position two by treating the dibromides of these olefines with slightly in excess of the theoretical amount of alcoholic potassium hydroxide necessary to remove the first

molecule of hydrobromic acid, and refluxing the solution for a few minutes. After this first molecule of acid had been removed, the bromo-olefine obtained by dilution with water was to be treated as usual with sodamide, the procedure from this point continuing as for olefines with unsaturation at the end of the chain. This procedure was to be applied in case the simple procedure above, without the use of alcoholic caustic, failed to give derivatives. Lack of time precluded the possibility of trying this on a small scale, although in the preparative work later described, exactly this procedure was run, according to the methods of Bourguel. (13)

It was thought well to discover whether this procedure might be applicable to diolefines with unsaturation at the end of the chain. Accordingly about 15 grams of diallyl were brominated and refluxed in xylene solution for four or five hours with two and a half times the theoretical amount of sodamide. The resulting solution after treatment with water did not give anything boiling at the boiling point of hexadiene-(1,5) (87°). Accordingly the first ten cc. of distillate were collected and precipitated with cold Nessler's reagent in ethyl alcohol. A white precipitate, insoluble in benzene and ethyl alcohol was obtained. It did not melt at 270° , but began to decompose at 197° . On rapid heating it flashed. So little precipitate was obtained that the attempt to use this procedure for diolefines was abandoned.

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by Test 927, page 524,

The derivatives thus far prepared, are given in the

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following table:

MELTING POINTS OF MERCURY DERIVATIVES OF OLEFINS

<u>Olefine</u>	<u>M.P. of Hg Salt</u>
Pentene-(1)	118-9° x
Hexene-(1)	93-4°
Heptene-(1)	60-1°
4-Methyl-hexene-(1)	87.5-8.5°
5-Methyl-hexene-(1)	88-9°
4,4-Dimethyl-pentene-(1)	108.5-12°
Octene-(1)	79-80° x
Nonene-(1)	68-9°
Decane-(1)	81-2°
Hexadecene-(1)	95-6°

x Prepared only from the acetylenes.

These compounds were recrystallized until their melting points remained constant; in some cases several solvents were used, no change in melting point being noted, according to the solvent used.

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An alternative procedure which can probably be applied not only to mono but to disubstituted ethylenes and acetylenes and also to allenes depends upon the hydration of either the acetylene or the allene, by means of an acid solution of mercuric salt, to the corresponding ketone, and the condensation of this ketone with 2,4 dinitrophenyl hydrazine to give a crystalline

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hydrazone. It is hoped that the hydration of acetylenes will occur so largely in one direction that the hydrazone may be freed by crystallization from any isomer that may be present. This reaction has been applied to hexadecine (1). Its hydration occurred readily in two or three minutes and the condensation with dinitrophenyl hydrazine, immediately. The resulting compound, which is probably the dinitrophenylhydrazone of methyltetradecylketone, melted at 88° after two recrystallizations. The exact procedure applied follows:

Three drops of hexadecine (1) were added to a round bottom long neck flask and shaken for five minutes with three ccs. of sulfuric acid (3:1 by vol.) which had been warmed with mercuric oxide. After shaking, an equal volume of water was added and the fatty, solid, precipitate extracted with two 3 cc. portions of ether. The ether was evaporated from the ketone in a small beaker, five ccs. of boiling ethyl alcohol containing 0.10 gms. of dinitrophenylhydrazine were added, and the solution boiled for a few seconds and cooled. After addition of a few drops of water a yellow precipitate separated which was filtered off, and after drying and recrystallizing twice from ethyl alcohol melted at $87.5-88.5^{\circ}$. After one more recrystallization it separated in fluffy needles melting at $88.2-88.7^{\circ}$, and after another it crystallized in tufts of needles melting at $88.1-88.3^{\circ}$.

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The procedure adopted for the preparation of a similar derivative from olefines was the same as this, after the preparation of the acetylene by the use of sodamide according to the method already described for mercury derivatives, ^(Paragraph 115.) That is, the dibromide prepared from one-half to one cc. of olefine was treated with sodamide, then acid, and then sulfuric acid (3:1) containing mercuric sulfate, and after shaking and rinsing with water, with an ethyl alcohol solution of 2,4 dinitrophenyl hydrazine. In these treatments the acetylene was not removed from the flask in which it was formed. A long stemmed dropper was plunged beneath its surface and used to remove the aqueous layer. This made practicable the treatment of smaller quantities (0.5 cc.) of olefines than would otherwise have been possible. Unfortunately, the amount of precipitate of 2,4 dinitrophenyl hydrazone formed was sufficient to recrystallize only in the case of heptene (1). The melting point of the precipitate thus obtained from heptene (1) was 70.5-71.5°, from 5 methyl hexene (1), 86-7°.

This procedure has not been further developed because of lack of time. It warrants considerable investigation. Because of the inadequacy of the experimental work it has not been included among the tests at the end of the tables.

For many of the tetrasubstituted ethylenes, and it may prove applicable to other types as well, the pinacolone rearrange-

ment gives a means of derivative formation. The glycol can be prepared from the dibromide of the olefine by the intermediate preparation of its diacetate by the use of silver acetate in acetic acid solution. The glycols then undergo the pinacoline rearrangement when refluxed with dilute sulfuric acid, and the corresponding pinacolines may be identified through the dinitro phenyl hydrazone.

No tetramethyl ethylene was at hand, but to test the promise of such a procedure, 0.25 gms. of pinacone (which could probably be obtained from 0.5 cc. of olefine) was refluxed for fifteen minutes with ten ccs. of 6 N. hydrochloric acid, extracted with ether, washed, and added in ether solution to 5 ccs. of boiling alcohol containing 0.10 gms. of 2,4-dinitrophenyl hydrazine. After boiling for five minutes this was cooled, treated with a few drops of water, and the precipitate obtained recrystallized from alcohol, M.P. 122-3°. While this particular glycol was not prepared from the olefines, it has been earlier shown that the glycol of cetene can readily be prepared in good yield by the given procedure. ^(Paragraph 95.) Undoubtedly pinacone could be similarly prepared, especially as R. L. Berry has shown in the course of his B. S. Thesis, 1930, that tetramethyl ethylene readily gives its solid dibromide. ^(12.) The data just mentioned, ^{paragraphs 124-128,} may be set down in the form of the following table:

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Olefine	B.P.	Ketone	M.P. of 2,4-dinitro-phenyl hydrazone
$(H_3C)_2C:C(CH_3)_2$	71-2 ⁰	$(H_3C)_3C.(CO).CH_3$	122-3 ⁰
$(H_3C)_2C.(CH_2)_2.CH:CH_2$	85-6	$(H_3C)_2C^H(CH_2)_2.(CO).CH_3$	86-7
$H_3C.(CH_2)_4.CH:CH_2$	94-5	$H_3C.(CH_2)_4(CO).CH_3$	70.5-1.5
$H_3C.(CH_2)_{13}.CH:CH_2$	274-5	$H_3C.(CH_2)_{13}.(CO).CH_3$	88.0-8.5

In the characterization of unsaturated hydrocarbons the bromide-bromate titration offers a valuable means of determining the molecular size of an unknown compound, as well as its degree of unsaturation. The method of running this titration adopted in this work has been given earlier, in the part dealing with methods of group division. ^(Paragraph 50.) Tables of results obtained on several compounds are given there, also. ^(Page 38.) Unfortunately, lack of time, and in some cases, sample, prohibited running but one titration with certain compounds. From the degree of reproducibility of results in other cases, however, it is felt that these single results are very little in error, especially as they are very close to the expected values. This titration is of particular value in the series of acyclic olefines where it is only occasionally the case that the olefine does not add very close to the calculated amount of bromine. ^{analytical} The tables list the "bromide-bromate numbers" reported in this work. It might be observed that conjugated diolefines added the theoretical amount of bromine by this titration, whereas diolefines of the diallyl configuration (except diallyl itself, which was not.

titrated in this work, but which, titrated by Cortese, gave practically the theoretical value) added from 0.77 to 0.89 of the theoretical amount of bromine. Whether this is a rule cannot be said from the limited number of titrations here made, but it is at least an interesting observation that should be borne in mind while seeking to identify diolefines.

It may be mentioned in this connection, that after several months of standing most of the diolefines prepared in this work were redistilled before having their constants determined. Judging from the distillation curves, the odor of various fractions of the distillate, and the amount of residue left behind, of the samples which had been standing in glass-stoppered bottles the diallyl-type diolefines had oxidized far more than the conjugated-type.

The colors developed by the action of sulfuric acid on highly unsaturated acyclic hydrocarbons appear to be quite indicative of the type of compound. ^(See Test 929-2&3) The test is carried out as other solubility tests, in the small tube earlier described. The tube is kept ice cold and 0.1 cc. of cold concentrated (1.84) sulfuric acid is added to 0.1 cc. of the hydrocarbon. The tube is shaken, removed of the ice bath, and allowed to warm up to room temperature. The results obtained are given in the following table, page 111. Acetylenes go completely into solution in the acid giving a clear solution with a deep-red color and are reprecipitated on dilution with water in the form of a pleasant, ketone-odored oil. Allenes

are violently attacked in a similar manner but give pasty non-transparent solutions. Conjugated diolefines are vigorously attacked in the cold giving a red intersurface; on warming they form a thick red paste which on dilution with water leaves a heavy red oil in suspension. Diallyl type diolefines are much less attacked than those previously mentioned; they frequently form only unstable, colored emulsions with the acid and on dilution with water they leave a light yellow oil on top of the aqueous layer. The tables of reactions obtained in this work follow on the next page.

TABLE OF THE COLOR REACTIONS OF 0.1 cc. OF COLD HYDROCARBON WITH 0.1 cc. OF
SULFURIC ACID (d.:1.84) See par. 132

No.	Hydrocarbon	Color at the Intersurface	Appearance after Shaking	Appearance after Dilution	Remarks
1.	Allyl-cyclohexane	Very slight yellow	Yellow-golden emulsion which quickly breaks	Light yellow oil thrown out	SO ₂ formed
2.	4-Cyclohexyl-pentene -(2)	Yellow-brown ring	Immiscible. Yellow oil on top. H ₂ SO ₄ colored golden amber	Light yellow oil thrown out. Color in H ₂ SO ₄ destroyed	SO ₂ formed
3.	4-Methyl-heptadiene- (1,5)	Red ring	Red, viscous emulsion formed with difficulty, separates on standing	Amber oil thrown out	
4.	4,5-Dimethyl-octadiene- (2,6)	Same as No. 3	Same as No. 3	Same as No. 3	
5.	4-Propyl-heptadiene-(1,5)	Yellow-red	Pasty, yellow-red emulsion	Light yellow oil thrown out	
6.	4-Allyl-octene-(2)	Yellow-red	Red emulsion formed with difficulty	Light brown oil thrown out	

No.	Hydrocarbon	Color at the Intersurface	Appearance after shaking	Appearance after Dilution	Remarks
7.	4,5-Di-n-butyl-octadiene-(2,6)	Slight yellow	Immiscible, H ₂ SO ₄ becomes yellow-brown.	Light pink Liquid thrown out	
8.	Pentadiene-(1,3)	Red	Slight shaking gave deep red paste	Not much affected. Same light pink solid formed	Reacted vigorously on addition of H ₂ SO ₄
9.	Heptadiene-(2,4)	Red	Viscous red paste (on cooling)	Red oil thrown out	
10.	Octadiene-(2,4)	Red	Red paste	Red oil thrown out	
11.	2,2-Dimethyl-hexadiene-(3,4)	Formed a solid red mass very rapidly		Vaseline-like grease and solid in tube. No oil thrown out	
12.	Octine-(1)	Red	Heavy, deep red, perfectly clear solution. Odor like heptyl alcohol.	Red oil thrown to top of H ₂ SO ₄ , with odor like heptyl alcohol and aldehyde	The oil formed did not cling to the tube.
13.	Octine-(2)	Orange	Deep red, perfectly clear solution	Light oil of pleasant, ketone-like odor thrown to top of H ₂ SO ₄	
14.	Nonine-(4)	Same as No. 13	Same as No. 13	Same as No. 13	

No.	Hydrocarbon	Color at the Intersurface	Appearance after Shaking	Appearance after Dilution	Remarks
15	Hexadecine-(1)	Same as No. 13	Same as No. 13	Solid w. pleasant odor, precipitated, filling the tube	

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As a means of identification of the paraffins, their critical solution temperatures in aniline have frequently been determined. These values have been determined for all of the pentanes, hexanes, and heptanes. However, it must be observed that the solution temperatures in aniline are considerably above room temperature (cf. the tables, Div. B, Sect. 6, for their exact values), and frequently they are above the boiling points of the paraffins, as is the case with all of the hexanes, except n-hexane, whose boiling point (69°) and critical solution temperature in aniline are coincident. This introduces the difficulty of requiring an apparatus for this determination which will hold in the paraffin at a temperature above its boiling point. While this is not necessarily a severe obstacle, it is a drawback in trying to use the very simple solubility tube earlier described. Also, duplication of results at temperatures around 70° is more difficult than when the

temperature is near that of the room and when there are only a few degrees between the solution temperatures of isomers, a small error in determination of these temperatures is more serious than at first appears. (14,15) Some work has been done replacing aniline by nitrobenzene. Timmermanns in studying a series of solution temperature curves has determined the values for n-hexane and nitrobenzene. (16) These are given in the following table and shown graphically in the plot.

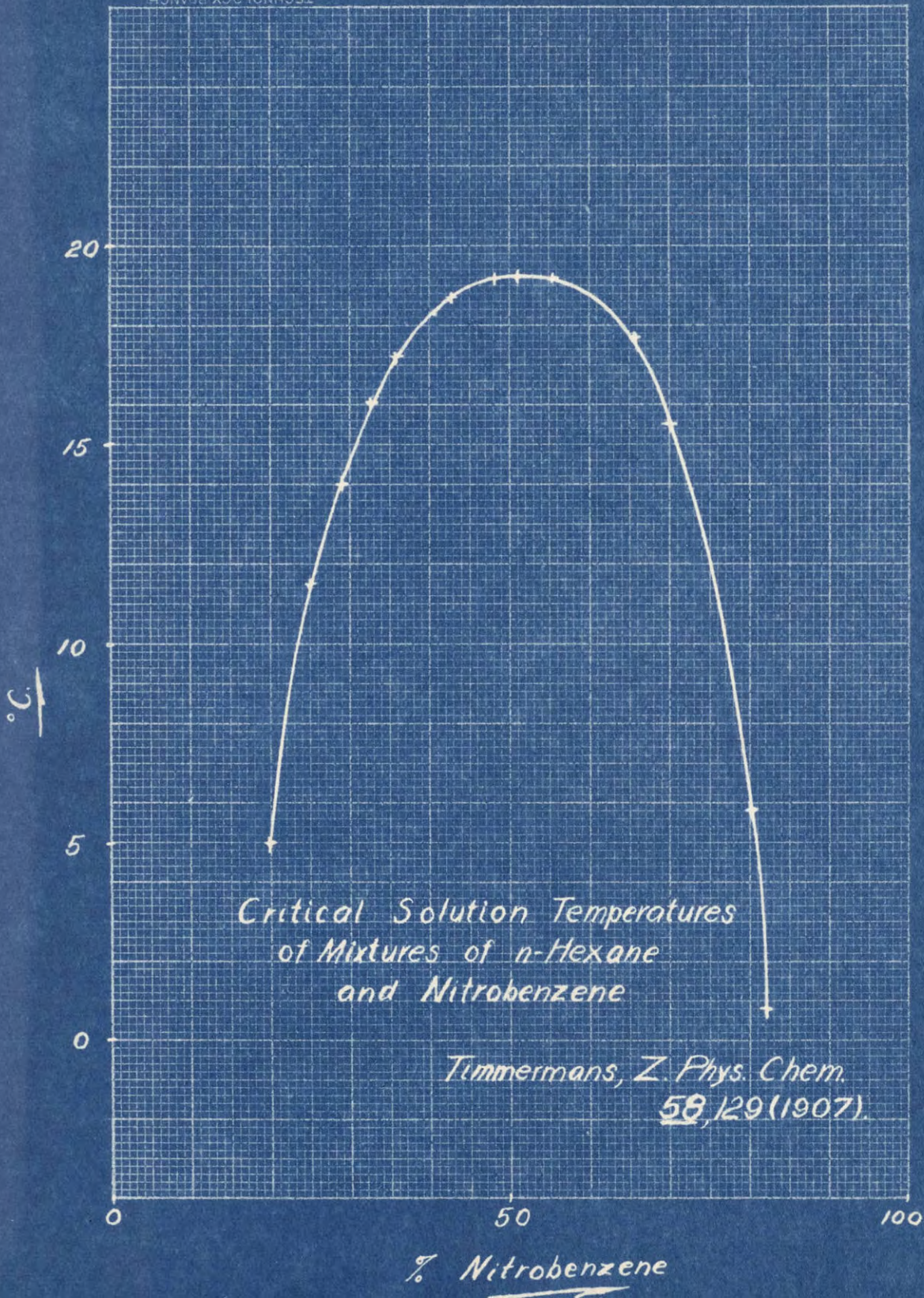
<u>% Nitrobenzene</u>	19.2	25.13	29.06	32.79	35.65	42.71	48.13	51.03	55.74
<u>C.S.T.</u>	5.0°	11.5°	14.0°	16.1°	17.2°	18.7°	19.2°	19.2°	19.2°

<u>% Nitrobenzene</u>	65.79	70.28	72.27	80.53	82.14	84.14	
<u>C.S.T.</u>	17.7°	15.5°	13.9°	5.8°	-0.2°	homogeneously crystallizes at -2.0°	

(24) Erskine has determined the critical solution temperatures ^{at approximately equal weights} of several paraffins fractionated out of petroleum. His values are given in the following table:

<u>Compound</u>	<u>C.S.T. in Nitrobenzene (Erskine) (24)</u>
Isopentane	31.25°
n-pentane	24.5°
Isohexane	24.05°
n-hexane	14.8°
Isoheptane	18.05°
n-heptane	11.5°

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While the value for n-hexane is obviously wrong, being much lower than that given by Timmermanns, yet the results indicate that there is a possibility of differences in solution temperatures between isomers - especially when similar differences in aniline are remarked. These temperatures are far easier to duplicate than those obtained by using aniline, for they are near 20° . It has been found possible in this work to check Timmermanns to within a few tenths of a degree, using the test tube previously described, 0.15 cc. of paraffin, and 0.1 cc. of nitrobenzene. (The value 19.8° checks the value 20.0° of Kohnstamm and Timmermanns (17) even more closely than the original value of Timmermanns (16). From the curve obtained by Timmermanns (16) it can be seen that a considerable error in measurement of the liquids, in the flat portion of the curve, would cause hardly any change in the temperature found. This permits measuring them into the tube either from a graduated one cc. pipette or from a graduated capillary dropper.

The critical solution temperatures of a few paraffins in nitrobenzene have been determined in the course of this work and are given in the table on the following page.

Critical Solution Temperatures of Various Paraffins in Nitrobenzene
(see Paragraph 136)

<u>Paraffin</u>	<u>B.P.</u>	<u>D.</u>	<u>n</u>	<u>C/S.T. in C₆H₅NO₂</u>
n-Heptane	98-9°	0.684 (20/4)	1.388 (20)	18-9°
2,2,4-Trimethyl pentane	98-9	0.692 (20/4)	1.392 (20)	29-30 32° (tech. C ₆ H ₅ NO ₂)

2,7-Dimethyl octane	159-60°	0.728 (18/4)	1.409 (18)	25-6° 27.5 (tech. C ₆ H ₅ NO ₂)
4,5-Dimethyl octane	161.5-2.5			19-20

2,2,4-Trimethyl pentane	98-9°	0.692 (20/4)	1.392 (20)	29-30°
3,4-Dimethyl hexane	118-9	0.717 (25/4)	1.404 (25)	13-4
n-Octane	124-5	0.705 (18/4)	1.399 (18)	14-5
<i>n Hexane</i>				<i>19.8°</i>

These values are all valuable as means of specific characterization, especially in such cases as n-heptane and 2,2,4-trimethyl-pentane, where the boiling points are identical, but where the solution temperatures differ by 10 degrees. These values were determined using nitrobenzene which had been steam distilled, dried with calcium chloride, and distilled from an ordinary distilling flask, the constant boiling portion being collected. The steam distillation is unnecessary. 0.15 cc. of hydrocarbon and 0.1 cc. of nitrobenzene were used. This ratio gives points on the flat portions of the curves (like that on page 115).

The critical solution temperatures of paraffins are, of course, considerably raised by the presence of small amounts of water. It has been observed by H. S. Davis (in a private communication) that the amount of moisture in nitrobenzene saturated with water is only a very small fraction of a percent and that therefore the determinations of critical solution temperatures in this solvent should be easier to duplicate than those in aniline which is hygroscopic.

Richard L. Berry has prepared the isomeric hexanes and determined their critical solution temperatures in nitrobenzene in his bachelor's thesis, 1930. (18) Reference should be made to this work for a very satisfactory review of these solubilities. He has shown marked differences between some isomers, and has been able by a combination of this property, with speed of reaction with permanganate

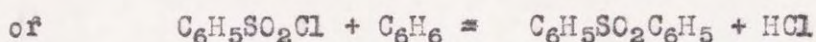
and bromine, together with physical constants, to effect a sharp distinction between all of the five isomeric hexanes. Reference should also be had to his thesis for a consideration of the way in which solution temperatures change by admixture of various amounts of isomer. This is no place to review his work, but it may be said that he has shown that in general with a mixture of isomers, the critical solution temperature of the mixture can never be lower than that of the component having the lower critical solution temperature. If this be true of nitrobenzene, it is probably also true of other solvents. Therefore, in the scheme outlined for sectional division, the

presence of isomeric hydrocarbons of the same general type as the principal component of an impure compound will not affect the placement of this compound in the proper section. Only impurities of a different nature, such as oxygenated compounds, far easier to remove than isomeric hydrocarbons, can invalidate the solution tests as far as sectional classification is concerned. If this be true, as seems highly probable, it greatly increases the applicability of this procedure.

To consider other types of hydrocarbons, many of the condensed aromatics form picrates of definite melting points. Many are reported not to form them, but this may be on account of the conditions that were used, such as the solvent. Thus, most picrates have been formed in ethyl alcohol solution whereas anthracene picrate is decomposed by alcohol, and has to be made in benzene. Nitromethane appears to be an excellent solvent from which to crystallize all picrates. In this work it has been used for naphthalene, 1,6-dimethyl-naphthalene, acenaphthene, and anthracene, whose picrate is not decomposed by it, as well as by G. C. Toone for biphenylene oxide, whose picrate he was unable to obtain from other solvents. It is reported that the picric acid content of these picrates can be quantitatively titrated, thus providing a further method of identification. (19) Unfortunately, time has prevented an investigation of this titration.

For the characterization of liquid aromatics, resort has generally been had to oxidation or nitration. Both are valuable procedures, but the former sometimes requires an unpleasantly

long time, and a rather large quantity of material. Preliminary experiments indicate that their reaction with chlorosulphonic acid is capable of general application. This yields with benzene either diphenyl sulphone or benzenesulphonyl chloride, depending on the conditions. The chloride can, of course, be readily transformed to the sulphonamide. The mechanism of this reaction which is usually given is shown below:



This reaction is instantaneous and a fraction of a cc. of hydrocarbon yields enough product to purify for a melting point determination. Presumably it has the advantage over oxidation that saturated side chains are not affected. Unfortunately this reaction has not been investigated at all in the course of this work, except to notice that diphenyl ether, toluene, and benzene, all give solid reaction products when they are treated with an equal volume of chlorosulphonic acid. The procedure adopted was merely to add to a few drops of hydrocarbon, ^{to} a few drops of acid until no further reaction occurred, then, after cautiously adding ammonium hydroxide in excess, filtering off the precipitate. No attempt was made to obtain by acidification any sulfonamide, soluble in the ammonia. Only in the case of benzene was the sulfonamide of M.P. 156° recrystallized until pure. This method of characterization certainly bears study.

In addition to these chemical tests, the solubilities of certain aromatics in nitromethane are useful characteristics.

The table shows this:

<u>B.P.</u>	<u>Compound</u>	<u>Solubility in CH₃NO₂</u>
167-8 ^o	Methyl benzyl ether	Soluble at (-17 ^o)
169-70	Pseudo-cumene	" " "
171-2	o-cresyl methyl ether	" " "
172-3	Phenetole	" " "
173-5	sec.-Butyl benzene	Insoluble at (-17 ^o)(C.S.T.,-1 ^o)
175-6	p-Cymene	" " " (C.S.T.,-4 ^o)

Seven of the aromatic hydrocarbons boiling between 167^o and 176^o are listed. Of these, five are completely soluble in nitromethane at -17^o, two are insoluble. Thus, if we should have p-cymene, a number of possibilities can be immediately eliminated by its insolubility in nitromethane at -17^o.

H. W. Underwood, Jr., with several of his students, has for the past few years been investigating the catalytic reactions of ethers with acids, acid anhydrides, and acid chlorides. (20,21) Gilbert C. Toone, working with him, has developed a procedure for the identification of symmetrical aliphatic ethers by their reaction with 3,5 dinitrobenzoyl chloride in the presence of zinc chloride. (22) The crystalline ester is readily obtained from a half cc. of sample. The writer is taking the liberty of adopting this procedure for the identification of this type of ether, with their permission. The experimental details of the procedure, written by Toone, are included after the tables as Test 928 (page 527).

It has long been known that certain aromatic hydrocarbons, especially condensed ring systems, show fluorescence - sometimes very marked, while others appear to be entirely devoid of this property. Under the ultra violet light, fluorescence is especially marked, and varies very considerably in color. Thus, under a light loaned by E. H. Huntress, fl^orene shows a blue fluorescence, and 9-isoamyl-anthracene a dazzling blue which becomes purple in benzene solution. The fluorescence of many hydrocarbons is noted in the tables.

To summarize the work reviewed in this section the reactions described (referred to below by paragraph numbers) are listed in three groups; those abandoned, those offering promise but not developed, and those adopted as numbered Tests in the section following the Tables.

<u>Reactions abandoned:-</u>	Paragraph number
Esters of glycols	93, 94a, 94b, 95
Nitroschlorides, nitrosates	96
Molecular compounds with mercuric acetate	97, 98
Molecular compounds with mercuric sulfate	99
Molecular compounds with picric acid, T.N.T., T.N.B.	100
Addition of thiocyanogen	101
Chlorosulfonic acid and olefines	102
Sulfuryl chloride and olefines	103
Polymerization of diolefines	104-108
Derivatives of bromohydrins	109
<u>Reactions offering promise but not developed:-</u>	
Sodium thiophenolate and dibromides	110

Hydration of acetylenes and condensation of the resulting ketones with dinitrophenylhydrazine	123-126
Pinacolone rearrangement of glycols from tetrasubstituted olefines	127
Picrates of aromatics	139
Chlorosulfonic acid and aromatics	140
Solubilities of aromatics in nitromethane	141
Fluorescence of aromatics	143

Reactions adopted as numbered Tests:-

Mercury derivatives of acetylenes	111-115
Mercury derivatives of this type from olefines	114-121
Bromide-bromate titrations	130-131
Colors with sulfuric acid	132
Critical solution temperatures of paraffins in nitrobenzene	133-138
3,5-Dinitrobenzoates from ethers	142

D

Preparation and Purification

PREPARATION AND PURIFICATION

Introductory

In the description of preparative work in the following pages the writer has tried to be as concise as possible. A few general remarks, therefore, will be made at this time.

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In running the Grignard reactions in the course of this work, almost always the reaction mixture was cooled during addition of the halide to the magnesium. Ice-water was used very effectively. The time required for the preparation was very materially reduced in this way, although the yields of Grignard were probably lower than would otherwise be the case. It was felt that the economy in time warranted the sacrifice of a higher yield. The Grignard reactions were run as they are usually run, all chemicals and apparatus being kept bone dry. The ether used in these reactions was prepared from ordinary ether by the method given by Ashdown.⁽¹⁾ Ordinary ether was shaken two or three times with one fifth of its volume of sulfuric acid (1:1 by vol.), separated after standing for a few minutes, and dried by running fresh sodium wire into it on three or four successive days. It was not distilled except when

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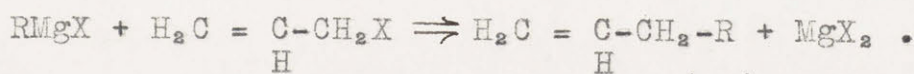
it became difficult to free it of sodium hydroxide by decanting or filtering. During the shaking with acid the apparatus was kept cold, both ether and acid having been ice cold at the beginning; and the pressure was relieved frequently. The magnesium used for most of these reactions was obtained from Eastman, that used in the majority of the final hydrocarbon preparations was obtained from Pfanstiehl Chemical Company.

In the preparation of olefines by the use of unsaturated halides and Grignard reagents it is frequently the case that the product is contaminated with halogen impurities which boil so near the boiling point of the hydrocarbon prepared that fractionation alone will not remove them. If the product be allowed to stand overnight, or longer, with alcoholic sodium ethylate or sodium methylate in methyl alcohol, a major portion of the halide is transformed into ether and a second treatment usually removes all traces. If the halide used in the preparation of the Grignard reagent has a secondary or tertiary halogen atom, then the reaction with the alcoholate takes place more slowly. In the following work, most of the olefines prepared were purified in this way, but the gravity of 4-methyl-pentene-(1), which was treated

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for only about two hours, indicates that it was not entirely freed from isopropyl bromide. The gravity of 4-methyl-hexene-(1), treated in the same way, indicates that it also was not entirely freed from sec.-butyl bromide. The olefines with the double band in position two were treated overnight with alcoholate. Some, at least, of the ethers thus formed may be washed out from the olefines by shaking with cold hydrochloric acid (1.19). The boiling points of the methyl and ethyl ethers of the halides used must be considered with respect to the boiling point of the olefine expected, and sodium methylate or sodium ethylate chosen accordingly, so that the resulting ether may be fractionated away from the hydrocarbon.

Allyl bromide has been used for some time in the preparation of olefines with the double bond in position one. (15,16). More recently this synthesis has been extended to homologues of allyl bromide (17), and to those homologues which undergo the so-called "allyl rearrangement". (18). The general equation is



The allyl rearrangement⁽¹⁹⁾, however, which has recently attracted a considerable amount of attention, may frequently cause a supposedly individual halide to give rise to two differ-

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ent olefines ⁽¹⁸⁾ The general equation for this rearrangement is



The question may be raised whether with the unsaturated halides used as intermediates in this work some similar rearrangement may not have taken place, giving rise, therefore, to a different end product from that expected. In considering this, 4-bromo-pentene-(2) may be discussed first. If 4-bromo-pentene-(2) undergoes a somewhat similar rearrangement, the result would be:



That is, if the rearrangement takes place over three carbon atoms, as this type of rearrangement does, there is no difference between the initial and final halide, so that treatment of this halide with methyl Guignard reagent will result in the preparation of 4-methyl-pentene-(2). If by some curious rearrangement the bromine should go to the end carbon atom, treatment with methyl Grignard reagent would give rise to hexene-(3). This latter compound has recently been prepared by Lespieau and Wiemann (20) and been found to have the following constants:

$$B.P. = 64^{\circ} \quad d^{19} : 0.6807 \quad n_D^{19} : 1.394$$

The compound prepared in the course of this work by the action of methyl Grignard reagent on

4-brom-pentene-(2) has the following constants:

$$\text{B.P.} = 58.6-9.0^{\circ} \quad d_4^{25}: 0.6685 \quad n_D^{25}: 1.3869 .$$

These two compounds cannot be identical. 151

Furthermore the boiling point $58.6-9.0^{\circ}$ agrees well with what might be expected for 4-methyl-pentene-(2). All of these constants check reasonably well the values given by Gorski for 4-methyl-pentene-(2) prepared by the action of alcoholic potassium hydroxide on 4-chlor-(or iodo)-2-methyl-pentane (₂₁):

$$\text{B.P.} = 57-8.5^{\circ} \quad d_4^{20}: 0.6706 \quad n_D^{20}: 1.3883 .$$

There is no question, therefore, concerning the structure of the olefines, prepared from 4-brom-pentene-(2). There remains to be considered the possibility of a rearrangement in the cases of 4-bromo-heptene-(2) and 4-bromo-octene-(2) in the following sense: 152



If the Grignard reagent of these two compounds could be prepared and hydrolyzed there would result from the one heptene-(2), b.p. = $98-9^{\circ}$, or heptene-(3), b.p. = $94-6^{\circ}$; from the other octene-(2), b.p. = $123-4^{\circ}$, or octene-(3), an unknown olefine, but which should boil about $121-2^{\circ}$, the boiling point of 153

octene-(1). In this work were prepared as later described, heptene-(2) (from 4-brom-heptene-(2)), b.p. = 98.6°-99.6° and octene-(2) (from 4-brom-octene-(2)), b.p. = 125.1°-126.1°. ^(See distillation curve, page 176) These preparations, together with the fact that the olefines herein synthesized for the first time boiled about where they would be expected to, coupled with the analyses of the bromolefines and hydrocarbons (given in the experimental part) leave little doubt concerning the structure of the olefines synthesized from 4-brom-heptene-(2) and 4-brom-octene-(2).

New hydrocarbons of other types which have been prepared in this work were all prepared by standard procedures, leaving little question concerning their structures. Probably nonine-(4) is the most questionable of these other hydrocarbons: $H_3C-CH_2-CH_2-CHBr-CHBr-CH_2-CH_2-CH_2-CH_3$ could be split out hydrobromic acid to give either nonine-(4) or nonadiene-(3,4) or nonadiene-(4,5). Bouis has shown that allenes with unsaturation at the end of the chain rearrange in the presence of sodamide to true acetylenes (1,). It is therefore probable that nonadiene-(3,4) would rearrange to nonine-(3) and that nonadiene-(4,5) would rearrange to nonine-(4). Sodamide

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also causes wandering of the triple bond toward the end of the chain, but in this case probably not much occurs, for the triple bond in position four is moved outward with far greater difficulty than when it is at the end. (14). It is conceivable, but from the usual action of sodamide improbable, that some nonadiene-(3,5) might be formed in this preparation. The reactions of this compound indicate that it is an acetylene.

A word should be said concerning the difficulty of determining carbon and hydrogen in these compounds. It was found that only after a very long time of burning did combustion become complete. Thus, in the case of nonine-(4), combustion was not complete until forty-five hours of burning. By weighing the absorption bulb from time to time it appeared that the combustion was approaching completion asymptotically. The difficulties encountered in the combustion work prevented the number of compounds analysed from including all those prepared for the first time. Analyses were run, however, on at least one compound representing each type of synthesis used to prepare a new species.

There remains only to be considered the

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the reliability of the constants given for compounds heretofore not prepared. The densities and refractive indices were kindly determined by John J. O'Brien, the accuracy of the determinations doubtless being better than one part in the fourth decimal. The purity of the compounds probably warrants reliance on the fourth decimal to within a few parts, except in the cases of 4-methyl-pentene-(1), 4-methyl-hexene-(1), and possibly 5-methyl-hexene-(1), where the gravities obtained indicate the presence of halide in the product. The one cc. specific gravity bottle used for the determination of the density of 4,4-dimethyl-pentene-(1) and the **probable** slight impurity of this compound probably make the constants for this compound reliable to about one in the third decimal. The boiling points were determined by running distillation curves, redistilling as many times as necessary to get a flat portion in the curve. The thermometers used were ^{260° and 360° stems,} calibrated against Bureau of Standards standard thermometers, and the temperatures read were corrected for steam exposure and barometric pressure (where specified as corrected). This probably warrants reliance on the boiling points reported to about 0.5°.

It may be well to give a list of the hydrocarbons described in the following pages which appear to have been prepared for the first time in the course of this work. These follow:
 4-methyl-hexene-(1), 4,4-dimethyl-pentene-(1), 4-methyl-heptene-(2), 4-methyl-octene-(2), 4,5-dimethyl-heptene-(2), 4,6-dimethyl-heptene-(2), 4,5,5-trimethyl-hexene-(2), 4-butyl-octene-(2), 4-cyclohexyl-pentene-(2), 4-cyclohexyl-heptene-(2), octadiene-(2,4), 4-methyl-heptadiene-(1,5), 2,2-dimethyl-hexadiene-(3,4), nonene-(4), 4,5-dimethyl-octadiene-(2,6), 4-propyl-heptadiene-(1,5), 4-allyl-octene-(2), 4,5-di-n-propyl-octadiene-(2,6), 4,5-di-n-butyl-octadiene-(2,6).

The writer wishes at this point to express his sincere appreciation of the excellent and painstaking work of Harold T. Gerry, with whose help the compounds described in this section were prepared.

PREPARATION AND PURIFICATION

Apparatus

The only apparatus used in the preparative work which needs to be especially mentioned is the fractionating columns. A number of Vigreux columns of various dimensions were very kindly built for the writer by W. E. Higbee. The side arms of most of these columns were sealed directly onto the column itself. The side arms of a few of them were sealed to larger, supplementary side arms sealed onto the columns and bent upward, as in a Claisen flask. The columns can best be described by the following tabulation, ^{page 136.} By "Claisen Arm" is meant the supplementary arm bent upwards, as distinct from the side arm. The columns were jacketed with glass tubing packed, if necessary, with magnesium oxide. The heads of the columns were cooled by a current of air or water when necessary. Column No. 1 was kindly built by A. A. Ashdown, No. 3 by R. T. Armstrong, and No. 4 by W. E. Higbee for G. C. Toone and loaned for this work by Mr. Toone. Except where otherwise specified, the fractionations described in the following pages were carried out with the use of these Vigreux columns. The particular column used was selected by taking into consideration the quantity of material to be fractionated and its boiling point. Column No. 1

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was most useful for the fractionation of five ccs., or less, of any compound, regardless of its boiling point. No. 2 was found useful for the fractionation of from ten to thirty ccs. of material boiling below 130°, although this column was not much used. No. 3 was found useful in distilling small amounts (less than five ccs.) of material boiling above 140°. No. 4 was used in the fractionation of less than 100 ccs. of material boiling below 170°. Nos. 5,6,7,8, and 9 were used for distillations of more than 50 ccs. of material, usually more than 100 ccs., boiling anywhere below 190°.

Column No. 1 was sealed directly to a flask of about **160** ten ccs. volume. Column No. 2 was sealed directly to a flask of about fifty ccs. volume. Column No. 3 was sealed directly to a flask of fifteen ccs. volume.

In addition to these columns, Mr. Higbee also built **161** one for vacuum distillations, designed by H. T. Gerry and the writer to overcome difficulties which they had encountered in the course of this work. This column proved to be most satisfactory for such distillations. It consisted of a flask of 200 ccs. capacity having a neck 18 mm. in internal diameter drawn down to 11 mm. internal diameter 7 cm. above the bulb, and at this point sealed to a tube of 11 mm. internal diameter 1 cm. long. 2.5 cm. above the bulb of the flask a large, upright side arm, 18 mm. in internal diameter and 6 cm. in vertical height was sealed to the neck of the flask.

A 200 cc. Claisen flask with both necks cut off was used for this part of the apparatus. This Claisen arm was sealed to a tube 31 cm. long and 15 mm. in internal diameter which was punched in at intervals of 10 mm., providing glass prongs on the inside, as in a Vigreux column. The punched section of the tube began 2 cm. above the joint with the Claisen arm of the flask and continued for 14 cm. - 14 cm. above the punched section of the tube, a long side arm of 5 mm. internal diameter was sealed on and bent downward to provide ample condensing surface for the distillate. The modified Claisen arm was drawn down to an internal diameter of 8 mm. at a point 2 cm. above the side arm, and joined to a short tube, 5 cm. long, of 8 mm. internal diameter. Condensers, jackets, and lagging were used in distillations as necessary, a glass jacket of 25 mm. internal diameter and 27 cm. length being always about the Claisen arm. This jacket was divided into three separate lengths of 14 cm., 6 cm., and 7 cm. The middle section was provided with an outlet and an inlet tube, as an ordinary condenser.

DESCRIPTION OF COLUMNS

<u>Column No.</u>	<u>Length of Column</u>	<u>Internal Diameter of Column</u>	<u>Vertical Height of Claisen Arm</u>	<u>Internal Diameter of Claisen Arm</u>	<u>Distance between top of Column + top of Claisen Arm</u>	<u>Distance between top of Column + top of Punched Section</u>	<u>Length of Punched Section</u>	<u>Average Distance between Punches</u>	<u>Distance between top of Column (or top of Claisen Arm) + Side Arm</u>	<u>Internal Diameter of Side Arm</u>
1	21cm.	11 mm.				7.5 cm.	10 cm.	10 mm.	3.5 cm.	2.5 mm.
2	24cm.	10 mm.				9 cm.	12.5cm.	8-9mm.	4.5 cm.	4 mm.
3	25cm.	12 mm.				7.5 cm.	14 cm.	20 mm.	3 cm.	4 mm.
4	35cm.	10 mm.	13 cm.	8 mm.	6 cm.	11 cm.	16 cm.	10 mm.	3.5 cm.	4 mm.
5	52cm.	10 mm.				25.5 cm.	23 cm.	9 mm.	3.5 cm.	3.5 mm.
6	60cm.	12 mm.				29 cm.	23 cm.	10 mm.	5 cm.	5 mm.
7	63cm.	12 mm.				28 cm.	26 cm.	10 mm.	7 cm.	5 mm.
8	62cm.	11 mm.	24 cm.	8 mm.	10 cm.	18 cm.	34 cm.	10 mm.	2.5 cm.	5 mm.
9	77cm.	10 mm.	17 cm.	9 mm.	6.5cm.	14.5cm.	50 cm.	12 mm.	2 cm.	5 mm.

PREPARATION AND PURIFICATION.

Part I. - Various Oxygenated Compounds.

1. - sec.- Butyl Alcohol

The sec.- butyl alcohol used in this work was manufactured 162
by the Standard Oil ^{of New Jersey} and purified by the method described by Ash-
(1) down. A representative run follows: 450 gms. of technical alcohol
were shaken with water until two layers formed and the constant
boiling mixture fractionated out. In this run the temperature at
which the mixture was collected was 86.4-87.4° corr. In later work
it was found that 87.2-87.7° corr. seemed to correspond to the
flattest portion of the distillation curve, the major part of the
alcohol used was prepared from constant boiling mixture of this
latter boiling point range. The constant boiling mixture was shak-
en with stick potassium hydroxide, the aqueous layer withdrawn, the
process repeated, and the alcohol refluxed with lime twice, four
hours each time, and after being finally distilled off from the
second lime treatment it was fractionated and collected from
99.4-100.4° corr. 140 gms. of alcohol thus purified were obtained,
correspond to 31% of the technical alcohol.

2.- Heptanol - (4).

740 gms. of n- propyl bromide were made into the Grignard reagent, 150 gms. of magnesium being used, and refluxing from a water bath effected for one-half hour after the last of the bromide had been added to the magnesium. 183 gms. of ethyl formate were slowly added, and the mixture refluxed over a water bath for two hours after the formate had been added. The reaction mixture was then decomposed by dilute sulphuric acid, the layers separated, and the ether distilled off from a water bath through a two-bulb column. The crude alcohol was dried by standing with potassium carbonate and then fractionated. The heptanol-(4) thus obtained was collected at 154.5-156.0° corr. The yield was 226 gms.- % yield 79, based on the ethyl formate used.

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The ethyl formate was prepared by letting Eastman's white label product stand 2-3 hours with sodium bicarbonate, then, after pouring off from this, letting it stand over night with calcium chloride, and then fractionating; the product boiling at 54.1-54.2° corr. being collected.

3. - 2,4- Dimethyl-pentanol-(3).

680 gms. of isopropyl bromide were made into the Grignard reagent as in the preparation of heptanol-(4), 140 gms. of magnesium being used. 157 gms. of ethyl formate were then added, and the preparation then conducted as in the preceding preparation of heptanol-(4). 160 gms. of 2,4- dimethyl-pentanol-(3) were obtained,

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collected from 138.6° to 140.1° corr. - 60% yield based on the ethyl formate.

The ethyl formate was purified as in the preceding preparation.

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4. - Nonanol - (5).

The preparation of this alcohol was carried out similarly to the two preceding, 540 gms. of n-butyl bromide, excess magnesium, and 145 gms. of ethyl formate being used. 185 gms. of nonanol - (5) were collected, boiling from 195-197° corr. - 66% yield based on the ethyl formate.

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5. - Pentene - (2) - ol - (4).

The preparation of this alcohol was carried out on a small scale first, in order to be certain of the success of the proposed syntheses. 297 gms. of methyl bromide in anhydrous ether solution were made into the Grignard reagent and treated with 191 gms. of crotonaldehyde. After standing over night the reaction mixture was refluxed for three hours and then decomposed with saturated ammonium chloride solution. After distilling off the ether and drying the alcohol with sodium sulfate 96 gms. of pentene-(2)-ol-(4) were obtained by fractionation, boiling from 120.2- 121.7° corr. - 41% yield based on the crotonaldehyde.

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Later a large preparation was carried out. Two runs of about two kilograms each of methyl bromide were treated in twelve liter flasks with about 1250 gms., each, of croton-

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aldehyde (after preparing the Grignard in smaller lots and then combining the solutions). The reaction mixture, after warming, was hydrolyzed with dilute sulphuric acid (1:10 by vol.). The ether solution was dried with potassium carbonate, the ether distilled off through a two bulb column, and the alcohol dried over night with sodium sulfate and fractionated.

The yield from this large run was poorer than from the preliminary attempt. This was probably due to the difficulty of controlling the temperature of the reaction mixture. According to Kyriakides,⁽²⁾ normal 1,2- addition to crotonaldehyde probably takes place in preference to 1,4-, the colder the solution. Other reactions also probably occur as the reaction mixture warms up to 0° and above. A yield of 918 gms. was obtained, boiling at 121.6-123.6° corr.- Yield- 30% based on the crotonaldehyde.

6. - Heptene - (2) - ol - (4).

Heptene-(2)-ol-(4) was prepared as pentene-(2)-ol-(4) in the second run described above, except that more thorough cooling was ensured by running only 350 gms. of halide at a time. Four runs were made; in each were used 350 gms. n-propyl bromide, 70 gms. magnesium, 210 gms. crotonaldehyde, 400ccs. ether. It was found that dilution of the crotonaldehyde with ether was of assistance in preserving a cool reaction mixture. The alcohol was fraction-

ated at 105mm. and collected at 99-101°. The product was redistilled at 75mm. and collected at 92.8 - 95.8°. To this was added more alcohol fractionated from the foreruns after they had been treated with sodium bisulphite. The total yield was 522gms., corresponding to 47% of the theoretical, based on the crotonaldehyde.

7. - Octene-(2)-ol-(4).

This alcohol was prepared as the preceding one in four lots, 300gms. of n-butyl bromide per lot. To each run were used 400ccs. of ether, 55gms. of magnesium, and 154gms. of crotonaldehyde. 567gms. of octene-(2)-ol-(4) were obtained, boiling at 93.9-95.9° at 40mm. and corresponding to a yield of 62% of the theoretical based on the crotonaldehyde. This yield was divided into two portions. One portion contained a few ccs. of distillate boiling slightly outside of the given range, probably due to fluctuations in the pressure. This product was later made into its bromide. The other portion was one boiling at 93.8-95°; this was later converted in part to octadiene-(2,4).

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8. - 2,2-Dimethyl-hexene-(4)-ol-(3).

This alcohol was prepared as the other alcohols of this type, 1000gms. of ter.-butyl chloride, 264gms. of magnesium, and 752gms. of crotonaldehyde being used. The yield was extremely poor. Most of the product was high boiling, but 47gms. of 2,2-dimethyl-hexene-(4)-ol-(3) were

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obtained, boiling at 73.4-76.4° at 35mm. - 4.2% of the theory, based on the crotonaldehyde.

9. - Crotonaldehyde.

The crotonaldehyde used in this work was bought from the Rubber Service Laboratories Co. Calcium chloride was added to this product and after standing variously from eighteen hours to a week it was poured off and fractionated. The pure crotonaldehyde was collected from 102.1-103.1° corr., mostly 102.5-102.6°. The fore-runs were again treated with calcium chloride and fractionated. From fifteen pounds of technical product ten and one-half pounds of pure crotonaldehyde were obtained, corresponding to 70% of the original material.

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10.- Heptaldehyde.

This aldehyde was prepared, in several runs, from castor oil. A considerable amount was obtained by vacuum distillation of crude heptaldehyde supplied to R. C. Elderfield by the Naugatuck Chemical Co. A representative run from castor oil follows. 900ccs. of oil were distilled at about 10mm. from a liter and a half distilling flask, using no column and heating the flask carefully with a free flame. Distillation, accompanied by much gas evolution at the start, was discontinued when the contents of the flask suddenly frothed up and filled the flask. The distillate, about 400ccs., was redistilled

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at 10mm. and collected in four fractions; up to 70°, 70-148°, 148-160°, 160-180°. The fraction boiling below 70° was distilled at atmospheric pressure from a distilling flask, 77gms. boiling at 149-152° u.c. being collected. The decomposition of the castor oil should be stopped short of the point at which the flask fills with foam for this residue, consisting partly of polymerized undecylenic acid, forms on cooling a transparent rubber-like mass which is very difficultly soluble in all of the common solvents (most soluble in ethyl acetate) and which is difficultly attacked by both acids and alkalies. The liquid in the flask becomes very viscous and the quantity of distillate decreases markedly prior to the curious foaming phenomenon. The heptaldehyde used in this work was fractionated and collected at 151-154° corr.

11.- Undecylenic Acid.

Undecylenic acid was prepared also from castor oil exactly as the heptaldehyde above. From the particular run described the three upper cuts were redistilled at 10mm. and 74.5gms. of undecylenic acid collected boiling at 148-153° u.c. at this pressure. The melting point of this preparation was not determined. A preparation similarly made and collected in two portions, one at 159-163° at 12-14mm. and the other at 163-5° at the same pressure, melted at 13-18° and 18-22° resp.; on redistillation and

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collection of the distillate boiling up to 170° the product softened at 18° and melted at 20-22°.

PREPARATION AND PURIFICATION.

Part II. - Various Halides.

1.- Hydrobromic Acid.

The hydrobromic acid used in this work^(d.:148) was prepared by the method given in Norris' laboratory manual⁽³⁾. A representative run made in a five liter flask follows. 2060gms. of sodium bromide, 1700gms. of water, and 1960gms. of sulfuric acid were mixed together and the hydrobromic acid rapidly distilled off with a free flame until the distillation became very slow and the thermometer registered 126° or had dropped below that value. By collecting the distillate in two portions and dipping the tip of an adapter connected to the condenser beneath the surface of the higher boiling portion from a previous run so that the gaseous acid might not escape, acid of density very closely approaching 1.48 was obtained to some 90% of the theoretical amount.

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2.- Methyl Bromide.

The procedure used for this preparation was that described by W. E. Messer⁽⁴⁾. The best of several runs was as follows. - 1100ccs. of sulfuric acid were slowly added to 750ccs. of methyl alcohol cooled by ice water, and

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this mixture was then poured slowly onto 750gms. of ice and cooled to about 30°. This solution and 865gms. of sodium bromide were then mixed in a five liter ring-neck flask closed with a rubber stopper bearing a thermometer, a small straight tube of about four feet in length to act as a pressure outlet, and a bulb-type reflux condenser. The condenser was in turn connected to a three foot tube of about 14mm. diameter filled with alternate layers of coarse and semi-fine calcium chloride and a short soda lime layer. This tube was connected with a delivery tube leading beneath the surface of anhydrous ether in a suction flask cooled by ice water and connected to a second similar flask closed with a calcium chloride tube. The reaction flask was warmed until evolution of methyl bromide apparently ceased. 762gms. of methyl bromide were obtained. This was dissolved in 879gms. of anhydrous ether. This corresponds to a 95% yield based on the sodium bromide. The yields from other similar preparations were not greatly lower than this.

It is unnecessary to cool the reaction mixture below 30° before adding the sodium bromide as the evolution of methyl bromide begins at about 50° and takes place with greatest vigor between 50 and 75°.

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3. - n - Propyl Bromide.

This halide was prepared from Eastman white label alcohol by the sodium bromide method of "Organic Syntheses."⁽⁵⁾ Thus, 1400gms. of sodium bromide, 1880ccs. of water, 2780gms. of sulfuric acid, and 1000gms. of n-propyl alcohol were refluxed for two hours, the halide distilled off, washed, cold, with about 250gms. of cold conc. sulfuric acid, water, 10% sodium carbonate solution, and dried over calcium chloride. About 1400gms. were collected, boiling upon fractionation from 70.5° to 71.5° corr., corresponding to 68% of the theory.

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4. - Isopropyl-Bromide.

98% isopropyl alcohol was dried with lime, fractionated, collecting between 82.4° and 83.2° corr., and the product treated as in the preceding preparation. From two and one-half kilograms of 98% alcohol about two kilograms of isopropyl bromide boiling at 59.5-61.0° corr. were obtained, about 40% of the theory.

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5. - n-Butyl Bromide.

The n-butyl bromide used in this work was prepared according to the sodium bromide method of "Organic Syntheses,"⁽⁵⁾ fractionated and collected at 101-102° corr.

181

6.- Isobutyl Bromide.

This halide was prepared several times, varying

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the conditions each time. It is difficult to obtain in good yield due to the ease of dehydration of its alcohol and the readiness with which it rearranges to the tertiary bromide.⁽⁶⁾ The use of sodium bromide and sulfuric acid is entirely unsatisfactory. The best procedure found follows.- 300gms. of Eastman's white label isobutyl alcohol were fractionated, giving 260gms. boiling at 106.9-107.9° corr. This was treated, according to the hydrobromic acid procedure of "Organic Syntheses",⁽⁵⁾ with 704gms. of hydrobromic acid(d:1.48) and 395gms. of concentrated sulfuric acid, refluxed an hour and a half, let stand over night, again refluxed an hour and a half, and finally distilled, washed as in the preceding preparations, dried, and fractionated. 124gms. of isobutyl bromide boiling at 91.2-92.2° were collected. Yield-22% of the theory.

Note: - The second period of refluxing is unnecessary.

7. - sec.- Butyl Bromide.

The sec.-butyl bromide used in this work was prepared by the sodium bromide method of "Organic Syntheses",⁽⁵⁾ less sulfuric acid being used than there called for. The alcohol was purified as described earlier,^{paragraph 162.} A representative preparation is one in which about two liters of purified alcohol (from seven kilograms of technical alcohol) were treated with three quarters of the customary amount of

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sulfuric acid, the remainder of the preparation being as usual. About 1200gms. of sec.-butyl bromide was collected, boiling at 90.8-91.8° corr., yield, about 40% of the theory, based on the purified alcohol.

8. - ter. - Butyl Chloride.

This halide was prepared by the method described for it in "Organic Syntheses"⁽⁷⁾ 1900ccs. of technical alcohol were shaken with five liters of hydrochloric acid (d.:1.19), in several portions, and let stand 15-20 minutes. The chloride was separated, washed with 200ccs. of hydrochloric acid (d.:1.19) and quickly with water and 10% sodium bicarbonate solution, then dried over calcium chloride. About 1500ccs. of ter.-butyl chloride were obtained, boiling at 51-2° corr.- 68% of the theoretical yield.

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9. - iso - Amyl Bromide.

This halide was prepared from technical (i.e., not synthetic) alcohol, which therefore contained some active alcohol. By the sodium bromide procedure, in the poorest run, 100gms. of iso-amyl bromide, b.p. 117-9°, were obtained from 200gms. of alcohol. This halide was not fractionated, but collected only from a distilling flask. - 29% of the theoretical yield.

185

10. - n - Heptyl Bromide.

85gms. of a mixture of heptyl bromide and alcohol obtained from R.C. Elderfield were refluxed for two

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and a half hours with 324gms. of hydrobromic acid (d.:1.48) and 84gms. of sulfuric acid. The top layer was separated the next day and steam distilled. It was then washed as usual with sulfuric acid, sodium carbonate, and dried with calcium chloride. Fractionation gave 66gms. of n-heptyl bromide boiling at 179-81° corr.

The alcohol had been prepared by Elderfield from heptaldehyde distilled from a crude product supplied by the Naugatuck Chemical Co.

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11.- Cyclohexyl Bromide.

This halide was prepared from Eastman white label cyclohexanol. In one case 530gms. of alcohol were gently warmed for two and a half hours with 2200gms. of 48% hydrobromic acid. After cooling, the top layer was separated, washed with sulfuric acid, water, sodium carbonate solution, and dried with calcium chloride. It was then distilled at atmospheric pressure, that boiling at 160-172° u.c. being collected. It appeared to decompose partially upon distillation, giving hydrobromic acid. This distillate was therefore washed again with water, sodium carbonate solution, and dried with calcium chloride, and then vacuum distilled. 485gms. of cyclohexyl bromide were collected, boiling at 85.7-86.7° at 55mm., 562% of the theoretical yield. A second run was carried out reducing the amount of hydrobromic acid. To one kilogram of alcohol,

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1700ccs. of hydrobromic acid and 500gms. of sulfuric acid were used. This latter product was collected from 83.7 to 85.7° at 55mm.

12.- 1,1- Dichlorheptane.

This dichloride was prepared from oenanthol obtained as described earlier. In one run 100gms. of heptaldehyde, b.p. 151.8-152.8° corr., were placed in a separatory funnel which in turn was thrust through a cork closing the mouth of a distilling flask. In the flask was placed the theoretical amount of phosphorous pentachloride, according to the equation, $C_6H_{13}CHO + PCl_5 = C_6H_{13}CHCl_2 + POCl_3$. The arm of the flask was connected to a calcium chloride tube and the flask cooled in ice water. The aldehyde was slowly run into the chloride with shaking until practically complete solution had occurred, and the contents allowed to warm up to room temperature. The oxychloride was drawn off with a water pump, keeping the flask surrounded with a pan of water at 75°. The remainder in the flask was then poured onto ice water, stirred vigorously, and allowed to warm up to room temperature, until all reaction was over. This required some time. The slightly brown product was then extracted with ether, thoroughly washed with sodium bicarbonate solution, and water, and then dried with calcium chloride. After distilling off the ether the mixture was vacuum distilled at about

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15mm., distillate being collected until the temperature began to rise rapidly and reached 150°. The distillate was then fractionated at atmospheric pressure. 32gms. boiling at 184 -5° u.c. were collected or 22% of the theoretical. This product was the best prepared. For the preparation of heptene -(1) the mixture of this dichloride with isomeric straight chain dichlorides and chlorheptenes which is obtained in this reaction can be used equally as well as the pure 1,1- dichlorheptane. Hence, later preparations were run similarly to that described, various factors being varied, the ether treatment being omitted, and the product boiling up to 86° at 15 mm. being collected. The major portion of this boiled at 84-5° at 15mm.

The yield in this preparation is very low. In order to keep it even up to this low level several precautions are necessary. The decomposition of the oxychloride with ice water must be kept cold. The chloride does not react with ice; on the other hand it reacts violently with water at room temperature, once reaction begins. Careful control is necessary.

It is unwise to make large preparations. All of the reactions are violent and cannot be controlled satisfactorily with large amounts of reactants. The preparation described in detail was the most satisfactory run.

13. - Allyl Bromide.

This halide was prepared both by the method of Cortese and McCollough^(a), i.e. several days standing of the alcohol with an excess of hydrobromic acid, and by the method of "Organic Syntheses"^(b), using stirring and both hydrobromic and sulfuric acids. A better yield was obtained by the latter procedure. Technical alcohol from Eastman, and redistilled student preparations were used, together with hydrobromic acid prepared as earlier described.

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The preparations were conducted as described in "Organic Syntheses", Vol. I, pg. 3. The product was fractionated and collected over a degree range, or less, between 70° and 72°.

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14. - 4 - Bromopentene - (2).

After experimentation to discover the best method of preparing this halide from its alcohol, and after a preliminary run, 744gms., or 53% of the theoretical, of 4-bromo pentene-(2) boiling at 70.2- 72.2° corr. at 145mm. were obtained by shaking 800gms. of pentene-(2)-ol-(4), prepared as described, with 1650gms. of hydrobromic acid (d.:1.48) for twenty minutes, then with two successive lots of 400gms. of hydrobromic acid for eight minutes each. The bromide thus prepared was washed with water, sodium bicarbonate solution and then water, and finally dried with calcium chloride. The distillation curve obtain-

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ed from this preparation indicated that considerable alcohol was in the halide. It was accordingly shaken with successive portions of hydrobromic acid until the gravity of the latter no longer changed by shaking with the halide. It was then very quickly washed with water, and sodium bicarbonate solution, and dried with calcium chloride. The product was obtained as stated.

Hydrolysis of this halide takes place rapidly when it is washed. In this respect, and in the ease with which it forms from the solution of its alcohol in hydrobromic acid it is very similar to ter. butyl chloride. It loses hydrobromic acid spontaneously on standing, and hydrolyzes by the moisture of the air. It cannot be distilled at atmospheric pressure (b.p. 116.7-119.2° corr.) without decomposing to give hydrobromic acid.

Parr bomb analyses gave 52.3% and 52.5% bromine. Analyses made after the material had stood several days longer gave still lower results. The theoretical is 53.7%.

15. - 4 - Bromoheptene - (2).

422 gms. of heptene-(2)-ol-(4) were treated, ^{similarly} as in the preceding preparation. Two consecutive lots of 500ccs. each of hydrobromic acid were used, then three lots of 150ccs. each, and finally several small portions until the gravity of the acid no longer changed. This very

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last treatment was given it after rapid washing had shown some hydrolysis. Air was then bubbled through for one half hour, in an attempt to remove excess acid, and the bromide dried with calcium chloride. 325gms. of 4-bromoheptene(-2) were collected at 71.9-73.7° u.c. at 25mm.-49% of the theory.

16.- 4 - Bromooctene-(2).

407gms. of octene-(2)-ol-(4) were treated with 500ccs. of hydrobromic acid (d.:1.48) twice, ^{similarly} as above, and the halide washed with water, 10% sodium bicarbonate solution, and water, quickly, and dried with calcium chloride. 422gms. of 4-bromooctene-(2) were obtained, boiling at 84-6° at 15mm., 69% of the theoretical yield.

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Parr bomb analyses gave 42.2% and 41.4% bromine, Theoretical, 41.8%.

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$n_D^{28.2} : 1.4677$ $d_4^{24.3} : 1.1151$ (Westphal balance)

PREPARATION AND PURIFICATION.

Part III. - Paraffin Hydrocarbons.

1. - Hexane.

About 65gms. of n-propyl-bromide were added to 19.5gms. of sodium cut in cubes (about 2-3mm. on an edge) under a reflux condenser, and a few drops of acetonitrile added. The reaction began at once upon addition of the nitrile. After standing over night it was refluxed for sixteen hours and distilled from the sodium. An element test on the distillate showed a trace of bromine, hence it was refluxed for ten hours with an equal volume of 20% alcoholic sodium hydroxide. After dilution with water and washing with water, it was dried with calcium chloride and fractionated from a distilling flask by taking several cuts and redistilling these. 13.3gms. of n-hexane, boiling from 68.5-70.5°, or 59% of the theoretical, were obtained. There was practically no low nor high boiling. This compound was later fractionated and found to boil almost entirely from 69-70°. 200

2. - 3 - Methyl-Pentane.

It was thought that the direct synthesis of this compound, as well as its isomers, from ethyl sulfate and the Grignard product of the proper butyl halide might 201

afford a simple way of obtaining the compound free from isomers. The normal compound has been reported synthesized by the action of ethyl sulfate upon n-butyl magnesium bromide. The major portion of this work by Gilman and Hoyle⁽⁹⁾ deals, however, with the action of ethyl sulfate upon other types of Grignard compounds, generally those formed by the action of some other compound, as an aldehyde, upon the magnesium organic halide. The preparation of n-hexane is the only paraffin heretofore reported synthesized by this procedure, as far as we have been able to find. The use of ethyl sulfate in the preparation of other types of hydrocarbons has been studied, and applied especially to the synthesis of n-propyl benzene.⁽¹⁰⁾

The very poor yields¹ obtained in two successful runs in which 3-methyl pentane was obtained, together with a considerable low boiling distillate, apparently consisting of ethyl bromide and butene, led to a desire to further investigate this reaction to see whether it might be at all practicable in general in the preparation of paraffins. This investigation has been undertaken by Richard L. Berry, and his results will be found in his bachelor's thesis, M.I.T., Course V, 1930.

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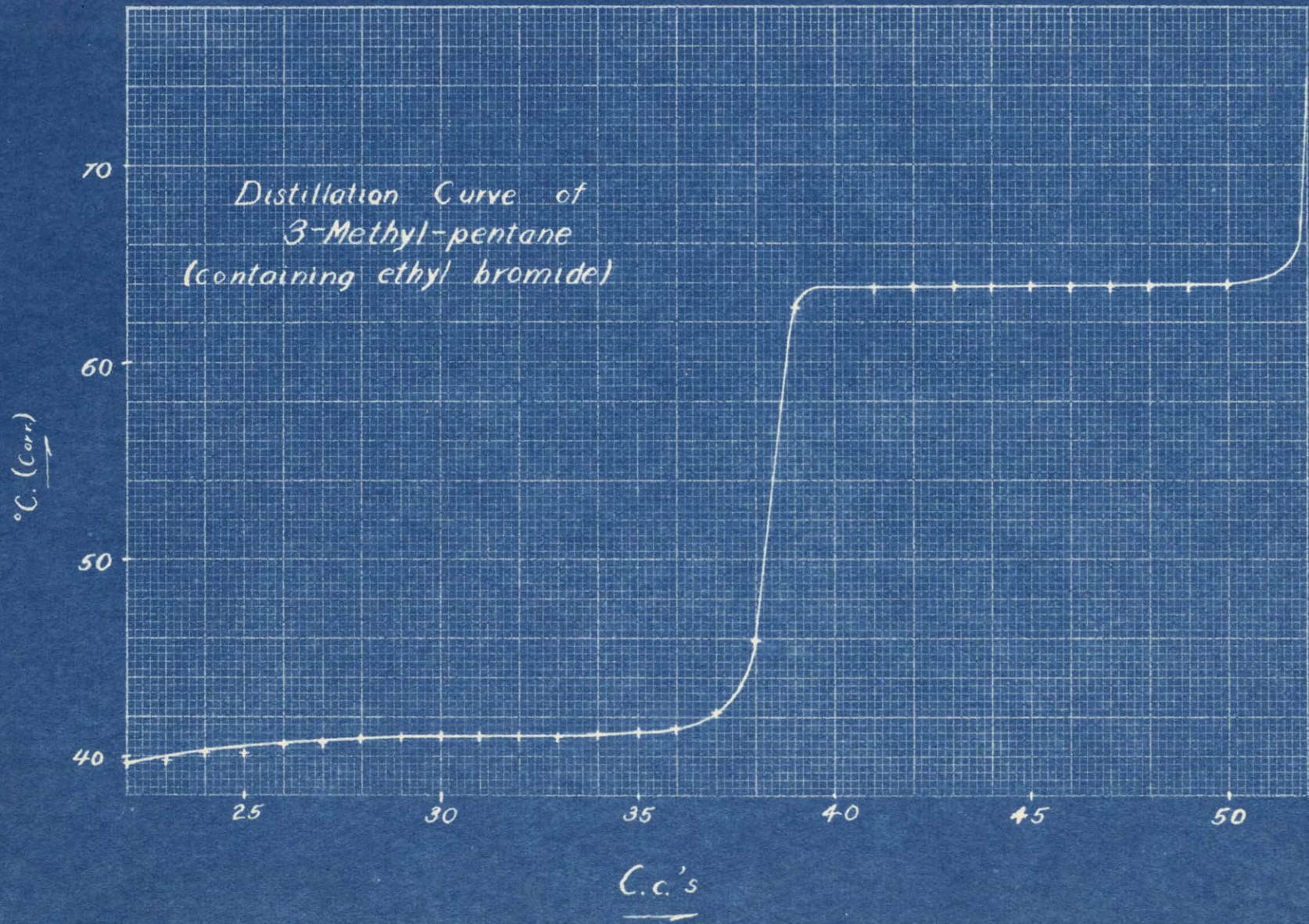
One of the successful runs[^] will be described.

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The ethyl sulfate used was obtained from Eastman. 200gms. were used. Of this, the last 40gms. added were not purified,

the first 160gms. were purified by shaking thoroughly with sodium carbonate solution, washing with water, and drying over night with sodium sulfate. 137gms. of sec.-butyl bromide were made into the Grignard reagent and this was then diluted to about one liter and the ethyl sulfate, diluted with ether, slowly added after the reagent had been poured from excess magnesium through a filter plate. A special stirrer, constructed from metal so that its wings might fold up when it was thrust through the neck of the flask and open out (when it was once inside) to a total stirring width of some three inches, was used in this last reaction. The ethyl sulfate was dripped onto the stirrer so that it was thrown out into the solution and well mixed. The reaction mixture became a very pasty mass, extremely difficult to stir well. It was decomposed by a solution of 100ccs. of conc. HCl in 500ccs. of water and 500gms. of ice. The ether was removed from the product by shaking it with ice cold hydrochloric acid (d.:1.19) until no further increase in volume of the acid occurred. It was then shaken with water, bicarbonate solution, and dried with sodium sulfate. The distillate boiling from 25° to 125° was collected from a distilling flask, dried over night with sodium in a flask provided with a Bunsen valve (placed in an ice chest) and fractionated.

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See page 158.

The curve shown was obtained. The product boiling at 63.6-64.1° corr. was collected. It was found to be extremely volatile, and best condensed by using the Gerry cooling device⁽¹¹⁾ to circulate brine through the condenser. 4.5gms. of 3-methyl-pentane were collected, - 5.2% of the theory.

3. - Octane.

40gms. of n-butyl bromide, 11gms. of sodium, and 300ccs. of dry ether were allowed to stand over night under a reflux condenser and then distilled off from the sodium with a smoky flame. The product was collected from a distilling flask taking several cuts and redistilling each of these. 8.5gms. of n-octane, b.p.:124.5-126.8° corr. were collected.- 51% of the theory.

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4. - 3,4-Dimethyl-hexane.

(as by-product)

This hydrocarbon was obtained from the high boiling products from two Grignard preparations in which sec.-butyl bromide had been used. 229gms. of bromide had been used all together, the 3,4-dimethyl-hexane obtained amounted to 3.9gms., boiling at 118.1-118.6°, corr., after after it had been fractionated through a small column. This corresponds to 4.1% of the halide used.

205

5. - 2,2,3,3-Tetramethyl-butane.

This compound was obtained from the high boiling residues from several Grignard preparations in which ter.-

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butyl chloride had been used. It was found that it sublimed out of these residues to give a clear crystalline product on the top of the flask when the residues were allowed to stand at room temperature in a tightly stoppered flask.

6. - 2,7-Dimethyl-octane.

This hydrocarbon was obtained by a Wurtz synthesis from isoamyl bromide. 5.4gms. were prepared, boiling from 157.7 to 160.7°. This product was later fractionated and collected over a degree range, from 159 to 160°.- Yield: 38% of the theory.

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7. - 4,5- Dimethyl-octane.

151gms. of sec.- amyl bromide, b.p.:117.5-119°, corr., prepared from sec.-amyl alcohol, b.p.:118.5-119.8°, corr., fractionated out of dried technical alcohol, were used in a Grignard reaction. From this about 1.5gms. of 4,5-dimethyl-octane were obtained, which was dried with and fractionated from sodium, and collected at 161.2° to 161.9° corr. - This corresponds to 2.1% of the halide used in the Grignard.

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8. - Dodecane.

The high boiling residue from the preparation of nonene-(1) was fractionated through a small column. From the action of magnesium upon the n-hexyl bromide (b.p.:153.3-155.3°) in the sense of the Wurtz reaction

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about one cc. of n-dodecane was collected, boiling from 213 to 214° corr. - This corresponds to 6.8% of the halide used.

9. - Tetradecane.

As in the preceding preparation, the high boiling residue from the preparation of decene-(1), in which n-butyl bromide was used, was distilled from a 15cc. distilling flask. About 10cc. boiling over a ten degree range from 240 to 250° corr. was collected. The melting point of this n-tetradecane was 4.5-6.0°. - This corresponds to 21.6% of the halide used.

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PREPARATION AND PURIFICATION

Part IV. - Olefines.

1. - Pentene - (1).

This olefine was prepared by the action of ethyl magnesium bromide on allyl bromide. The Grignard reagent was prepared in four lots, 200gms. of ethyl bromide, 400ccs. of ether, and 47gms. of magnesium each time. The time required for the addition of the ethyl bromide was about an hour and a half each time. The solution was refluxed for one half hour further and then poured through a large filter plate into a stock bottle, glass stoppered. If the reagent separated on cooling, more ether was added to this solution before it was used, until solution was again practically complete. The reagent was added to about 800gms. of allyl bromide dissolved in 1500ccs. of dry ether in a twelve liter flask, mechanical stirring being used, and a reflux condenser cooled with brine being connected to the flask. Addition required eight hours. The flask was cooled during the addition sufficiently to prevent undue speed of reaction. The mixture was permitted to stand for four days and then decomposed, cold, with a large volume of water. The ether solution was shaken with hydrochloric acid (d.:1.19) until there was scarcely any volume change, and then after being washed with water, it was

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1893

°C. (Corr.)

32
31
30
29
28
27
26

*Distillation Curve of
Pentene-(1)*

0 50 100 150 200 250 300 350

C.c.'s

added to a solution of 25gms. of sodium in 500ccs. of alcohol. After standing over night it was diluted with water and again treated with alcoholic sodium ethylate, standing, this time, five days. At the end of this time practically no sodium bromide had precipitated. The alcohol was washed out and the product shaken with hydrochloric acid until the change in volume became very slight. After washing with water, sodium carbonate solution, and water, it was dried with calcium chloride. It was placed in an ordinary distilling flask and fifteenccs. forerun rapidly distilled off and discarded. It was then fractionated, the curve shown being obtained. Yield, 160gms., b.p.: 30.4-30.6°.

2. - Hexene - (1).

This olefine was prepared similarly to pentene-(1). 123gms. of n-propyl bromide, 25gms. of magnesium, and 104gms. of allyl bromide were used. After the allyl bromide had been added the reaction mixture was allowed to stand for thirty-six hours before it was worked up. The product was fractionated and collected from 63.6° to 64.1° corr. before being treated with sodium methylate. It was then stirred for two hours with an equal volume of alcoholic sodium methylate solution, as it gave a slight test for halogen. It was then washed twice with hydrochloric acid (d.:1.19), twice with water, then with bicarbonate solution, water, and dried. It was collected at

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63.6-64.1° corr. The distillate gave no test for halogen. Yield, 22.8gms., 31.6% of the theoretical.

3. - 4 - Methyl-pentene-(1).

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This olefine was prepared as described for hexene-(1) from 123gms. of isopropyl bromide, 25gms. of magnesium, and 105gms. of allyl bromide. A similar sodium ethylate treatment was given it as for hexene-(1). The distillation curve showed a considerable flat space of 24ccs. boiling from 55.2° to 55.3°, but beyond this a rise in temperature which was indicative of the presence of some isopropyl bromide, which reacts with alcoholates very slowly. 23.6gms. of 4-methyl-pentene-(1) boiling from 55.0 to 55.5° corr. were collected. This corresponds to about 32% of the theory. That this contains some bromide is obvious from the constants which are:

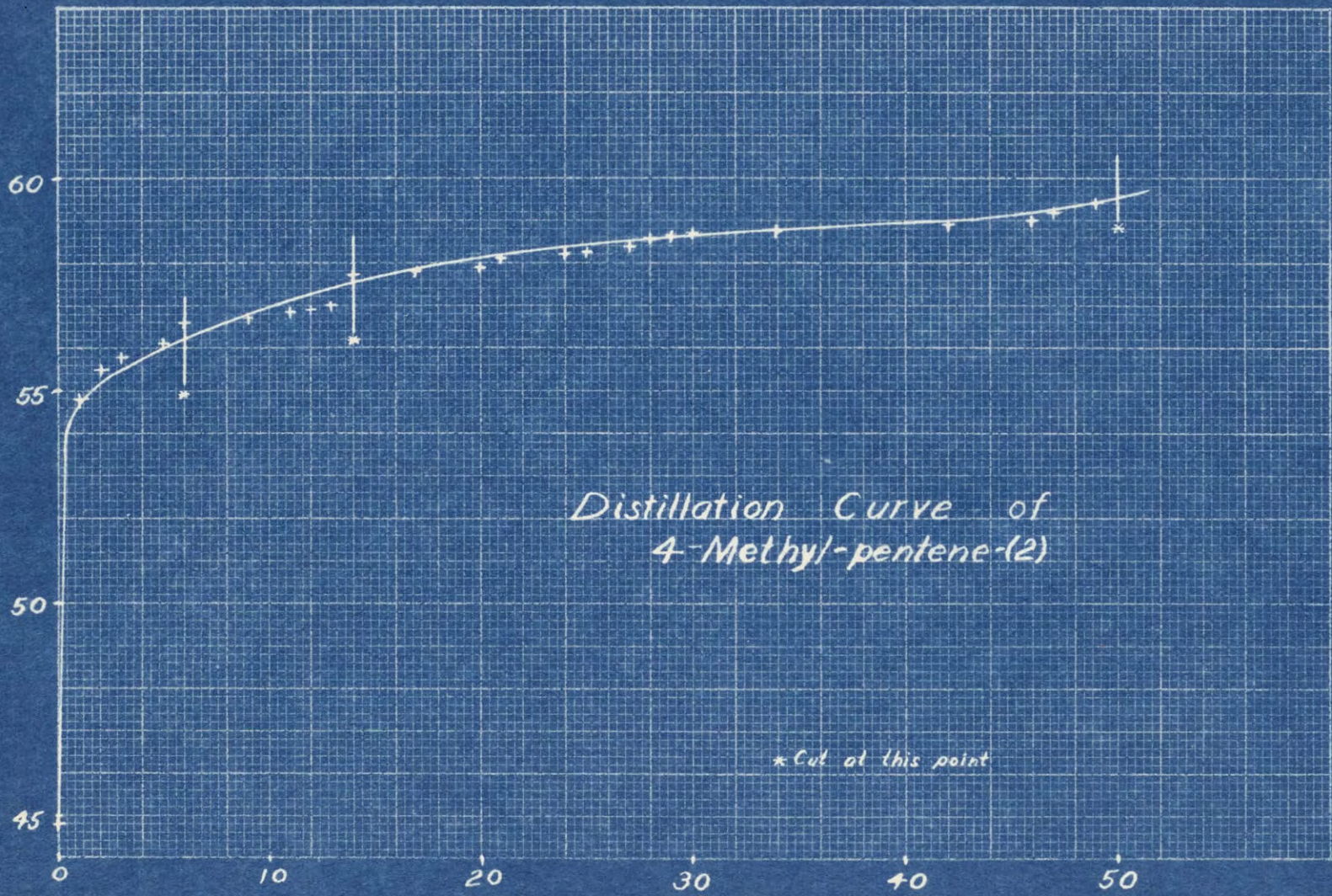
$$d_{4}^{25}(\text{vac}): 0.7072 \qquad n_{D}^{25} : 1.3850$$

4. - 4 - Methyl-pentene-(2).

This olefine was prepared similarly to those already described. 96gms. of methyl bromide in 90gms. of ether were added to an excess of magnesium covered with ether. After the halide had all been added and the reaction mixture warmed for a half hour it was poured through a filter plate and 80gms. of 4-brom-pentene-(2) were added and the solution heated for three hours. This was then decom-

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°C. (Corn)



*Distillation Curve of
4-Methyl-pentene-(2)*

* Cut at this point

C.c.'s

posed with ammonium chloride solution, the ether washed out with hydrochloric acid, and the product dried with calcium chloride and fractionated. The curve shown was obtained, the product being cut at the points indicated. Upon refractionation of these three cuts, 25.6gms. of 4-methyl-pentene-(2), b.p. 58.6-59.0° corr., were obtained. Constants determined upon this material follow:

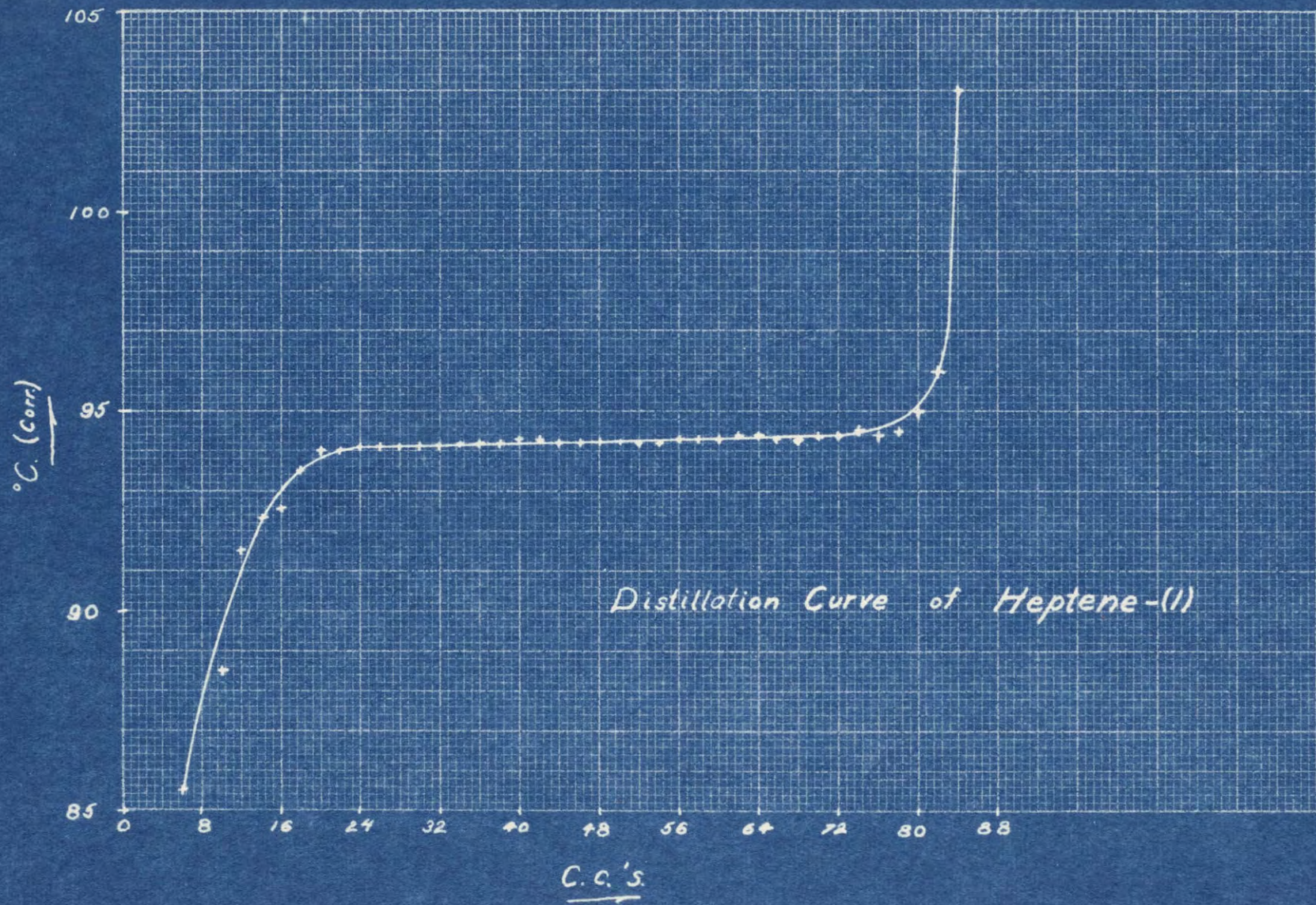
$$d_{4}^{25}(\text{vac}): 0.6685 \quad n_{D}^{25.3}: 1.3869$$

5. - Heptene - (1).

This olefine was prepared similarly to 215
hexene-(1). 137gms. of n-butyl bromide, 25gms. of magnesium, and 104gms. of allyl bromide were used. The reaction product was worked up after it had been refluxed, with stirring, for eight hours after the last of the allyl bromide had been added. 13gms. of crude heptene-(1) similarly prepared from an earlier run were combined with this product, the combination treated twice with alcoholic sodium ethylate, washed, dried, and fractionated. The curve shown was obtained, ^{page 168.-} 39.4gms. of heptene-(1), boiling at 94.0-94.4° were collected.

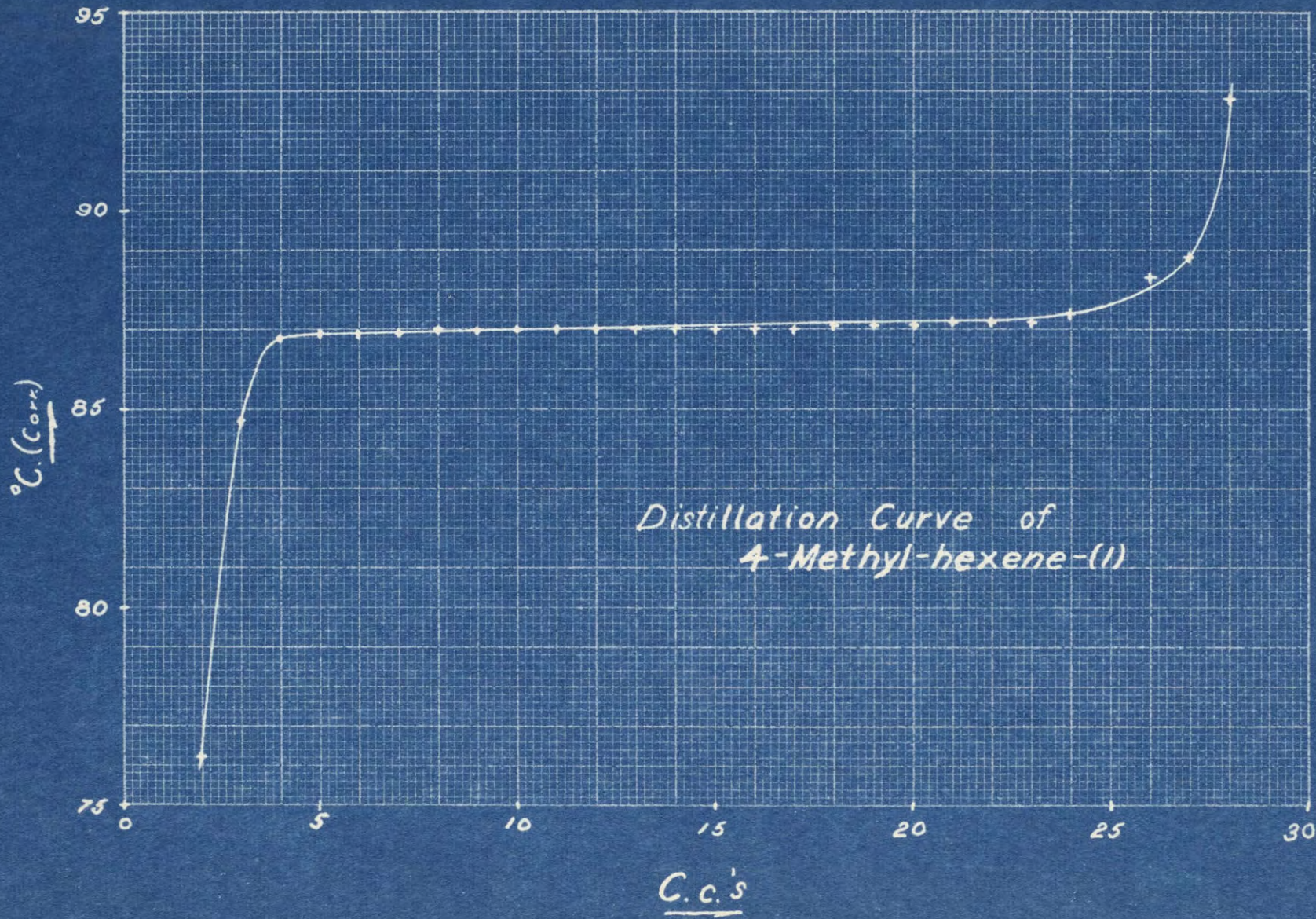
6. - 4 - Methyl-hexene - (1).

This compound was prepared as the others. 216
92gms. of sec.-butyl bromide were used and the reaction mixture refluxed for twelve hours before water was added.



Distillation Curve of Heptene-(1)

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It was twice treated with sodium ethylate. After the usual procedures it gave the distillation curve shown, *page 169*. 14.5gms. of 4-methyl-hexene-(1), b.p.:86.8-87.3° corr., were collected. The constants follow:

$$d_{4}^{25} : 0.7110 \quad n_{D}^{24.9} : 1.3940$$

The density indicates that this compound may not be entirely free from sec.-butyl bromide. 217

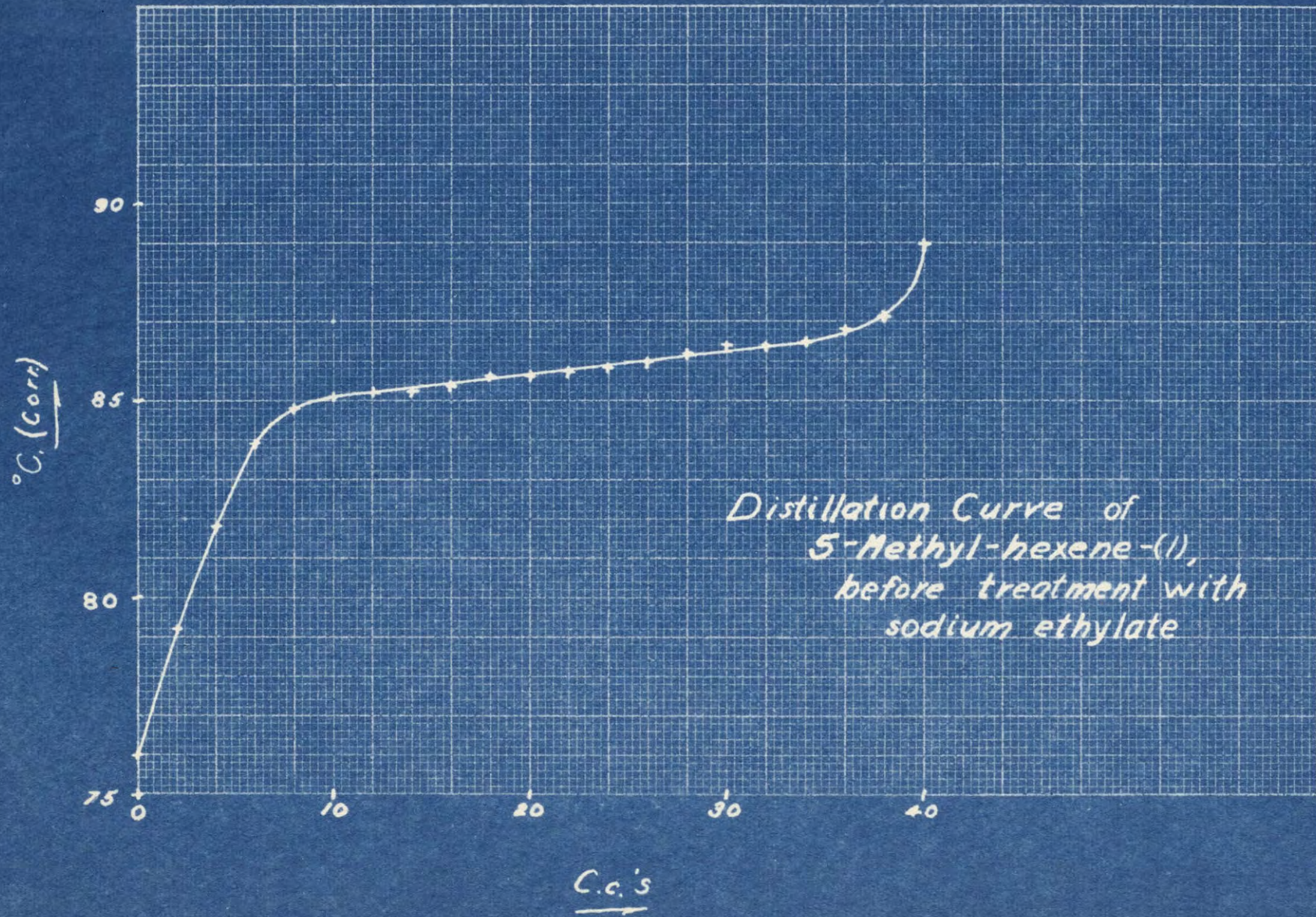
7. - 5 - Methyl-hexene -(1).

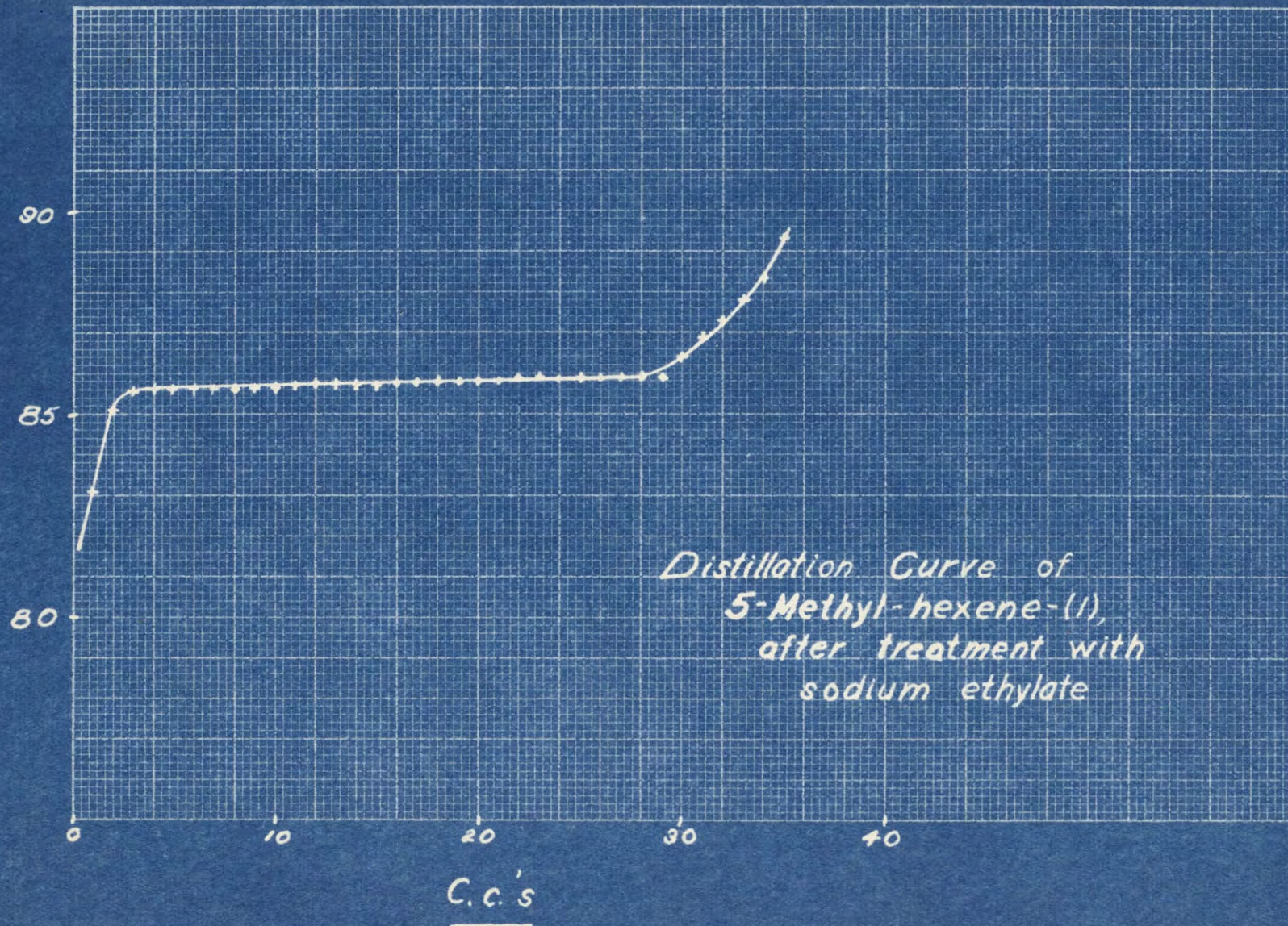
This heptene was prepared from 137gms. of isobutyl bromide and was worked up after eight hours of refluxing. Before being treated with alcoholate it was distilled, the first of the curves shown being obtained, *page 171*. The product boiling above 76° corr. was collected and treated with alcoholic sodium ethylate. After this treatment the second curve shown was obtained, *page 172*, 18gms. of 5-methyl-hexene-(1) were obtained, boiling from 85.5° to 86.0°. The constants follow:

$$d_{4}^{25} (\text{vac}): 0.6895 \quad n_{D}^{24.9} : 1.3940$$

8. - 4,4 - Dimethyl-pentene-(1).

This hydrocarbon was prepared as the others, from 92gms. of tertiary butyl chloride. It was found necessary to treat the crude olefine three times with sodium ethylate, the second time letting it stand for about two weeks. Finally about 6ccs. of 4,4-dimethyl-pentene-(1) 219





*Distillation Curve of
5-Methyl-hexene-(1),
after treatment with
sodium ethylate*

b.p.: 71.2- 72.4° corr. were collected. In order to have a sufficient volume to determine the density, to this were added a few ccs. of olefine prepared in a similar manner by H.D. Addison as a portion of his special problem in junior organic chemistry laboratory in 1929, boiling at 72.3° corr.

d_{25}^4 (vac): 0.6883 (determined quickly in a one cc. specific gravity bottle, its rough gravity having been already determined.)

$n_D^{25.2}$: 1.3909

9. - Heptene -(2).

This olefine was obtained as a by-product in the preparation of 4,5-di-n-propyl-octadiene-(2,6) by regulating the amount of ether used so that some magnesium compound of the unsaturated halide might form as in the independent preparation of allyl magnesium bromide. 70gms. of 4-brom-heptene-(2) were treated as described under ^{the} diolefine, and upon hydrolysis of the Grignard compound, after washing out the ether with hydrochloric acid a distillate of 10ccs. were collected by raising the temperature of the oil bath to 215°. From this were fractionated 2ccs. of heptene -(2), b.p.: 98.6-99.6° corr., and 0.5cc. from 99.6 - 101.1° corr. The distillation curve showed this cut to be distinctly an individual compound.

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10. - 2,4 - Dimethyl-pentene-(2).

140gms. of 2,4-dimethyl-pentanol-(3) were heated on a sand bath with 100gms. of oxalic acid ($C_2H_2O_4 \cdot 2H_2O$) under a short column. The distillate was washed with sodium hydroxide, dried with potassium carbonate and distilled under a column until the distillate came over at 105° . The high-boiling was put back with the oxalic acid and treated as before. The combination of the two lots of low boiling distillate was fractionated and 54gms. of 2,4-dimethyl-pentene-(2) b.p.: $82.9-83.4^\circ$ corr. were collected. The fraction boiling from $83.4-83.9^\circ$ corr. amounted to 24gms. The tests described were run upon the former fraction.

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11. - Heptene -(3).

200gms. of heptanol-(4) were heated on a sand bath under a short Vigreux with 115gms. of oxalic acid ($C_2H_2O_4 \cdot 2H_2O$). The distillate was washed with alkali, dried with potassium carbonate, and distilled up to 110° . The residue was put back with the oxalic acid and the treatment repeated. The combined distillates were fractionated giving 73gms. of heptene-(3), b.p.: $95.8 - 96.3^\circ$ corr. and 31.5gms., boiling from 96.3 to 96.8° corr.

222

12. - Octene - (2).

This olefine was prepared in small yield in a manner similar to that by which heptene -(2) was prepared,

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except that in this case the procedure whereby allyl magnesium bromide can be prepared was followed more closely. 81gms. of 4-bromooctene-(2) dissolved in 300ccs. of ether were dropped on six times the theoretical amount of magnesium (61gms.) covered with ether (about 400ccs.). The Grignard solution was refluxed for an hour and decomposed with ammonium chloride solution. The ether solution was dried with calcium chloride and the ether removed by distilling it off through a spiral column loaned by H.S. Davis. The resulting liquid residue was heated in a distilling flask on an oil bath up to about 200°. The distillate thus collected was treated with sodium ethylate, dried with calcium chloride after dilution with water, and distilled from a small column. The curve shown was obtained, ^{page 176.} The first drop came over at 124.6° corr. 3.4cc. of octene -(2) boiling at 125.1-126.1° were collected. The residues of high boiling material were combined with the preparation of 5,6 - dipropene - (5¹,6¹)-yl-decane.

13. - 4 - Methyl - heptene - (2).

This olefine was prepared by the general procedure described under the preparation of 4-methyl-pentene -(2), number four, of this part. From 24gms. of n-propyl bromide and 24gms. of 4-bromopentene-(2), 4.8gms. of 4-methyl-heptene -(2) were collected, b.p.: 113.8 - 114.1° corr.

$d_{4}^{25}(\text{vac}): 0.7188$

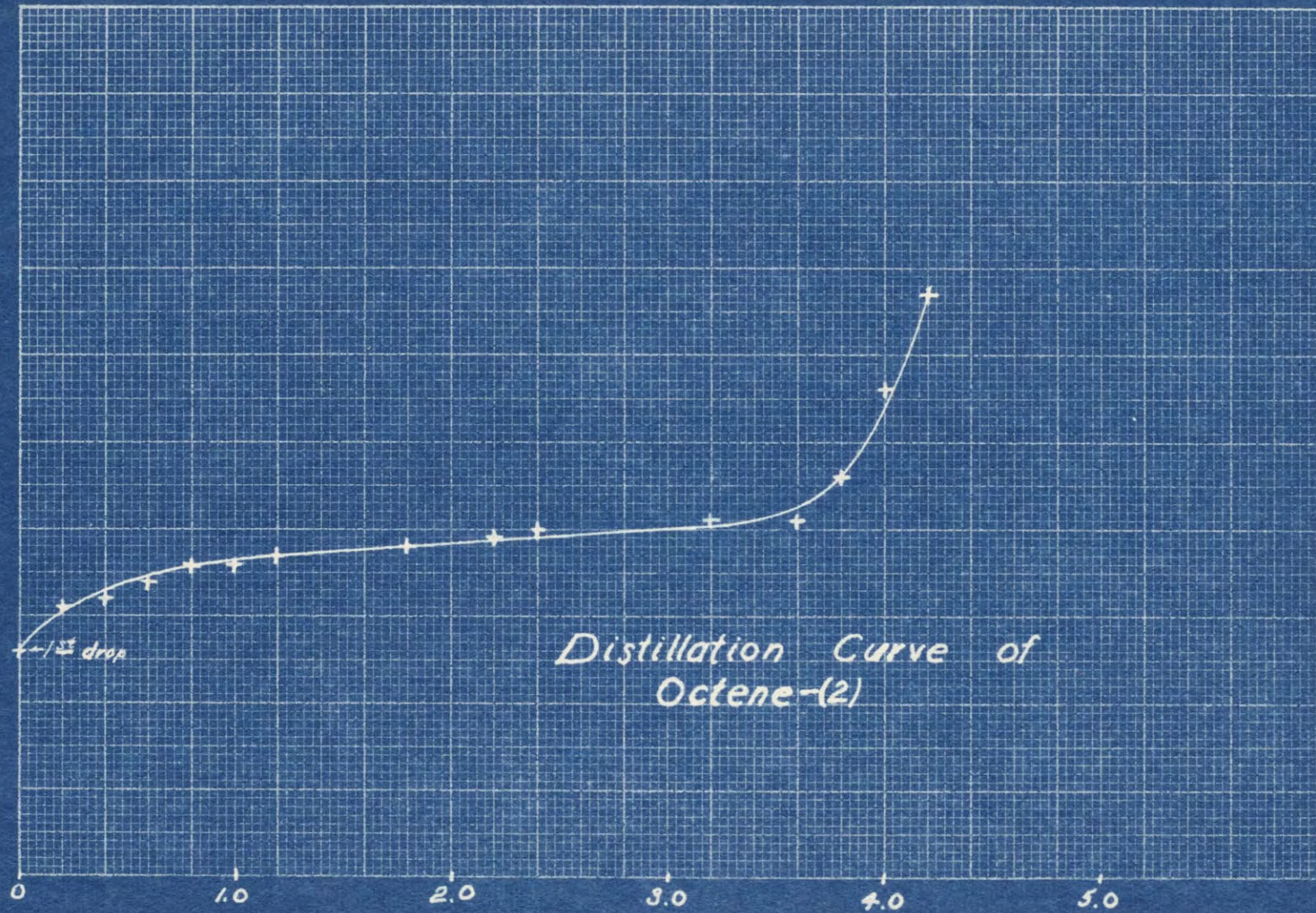
$n_D^{25}: 1.4100$

224a

126

°C. (corr)

129
128
127
126
125
124



Distillation Curve of
Octene-(2)

C.c.'s

14. - Nonene - (1).

This compound was prepared by the general procedure for olefines of this type. 21.6gms. of n-hexyl bromide, prepared by G.C.Toone from alcohol synthesized by him from n-butyl bromide and ethylene oxide, and 20gms. of allyl bromide were used. The hexyl bromide boiled at 153.3 - 155.3° corr. After refluxing the reaction mixture for three hours it was worked up as usual and gave upon fractionation 6.1gms. of nonene -(1) boiling from 145.3° to 145.8° corr. The high boiling gave dodecane, Part III, Number eight.

2246

15. - Nonene - (4).

This compound was prepared from nonanol -(5) as heptene -(3) (number eleven) from its alcohol. 165gms. of alcohol and 100gms. of oxalic acid, after two treatments in which the distillates boiling up to 175° were collected and redistilled, gave 77.5gms. of nonene -(4), b.p.: 147.5-148.1 ° corr.

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16. - 4 - Methyl - octene - (2).

This compound was prepared as 4-methyl-pentene -(2). The product was treated with sodium ethylate. 83gms. of n-butyl bromide with 18gms. of magnesium and 80gms. of 4-brom pentene -(2) gave 19gms. of 4-methyl octene -(2), boiling from 137.3° to 137.9° corr.

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$d_{4}^{25}(\text{vac}): 0.7286$ $n_{D}^{24.8}: 1.4158$

Analysis:	<u>Found</u>		<u>Theoretical</u>
	I	II	
wgt. sample	0.1522	0.1442	
wgt. CO ₂	0.4758	0.4491	
wgt. H ₂ O	0.1983	0.1863	
%C	85.28	84.95	85.63
%H	14.58	14.46	14.37

17. - 4,5 - Dimethyl-heptene -(2).

The procedure whereby 4-methyl-pentene-(2) was prepared was adapted to the preparation of this compound. 89gms. of sec.-butyl bromide, 20gms. of magnesium, and 80gms. of 4-brom-pentene-(2) were used. The product was treated with sodium ethylate solution and distilled. 26ccs. boiling from 95-136° were obtained. As some hydrobromic acid was given off on distillation, this product was treated with anhydrous potassium carbonate until clear, and redistilled. 5.5gms. of 4,5-dimethyl-heptene-(2) were collected, boiling from 135.0- 136.1° corr.

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$$d_4^{25}(\text{vac}) : 0.7431 \quad n_D^{24.8} : 1.4220$$

Analysis:	<u>Found</u>	<u>Theoretical</u>
	wgt. sample	0.1495
wgt. CO ₂	0.4712	
wgt. H ₂ O	0.1916	
% C	85.99	85.63
% H	14.34	14.37

18. - 4,6 - Dimethyl-heptene -(2).

This compound was prepared, ^{similarly} as the last, from 90gms. of isobutyl bromide, 17gms. of magnesium, and 80gms.

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of 4-bromopentene -(2). The reaction mixture was refluxed three hours, decomposed with water and worked up as usual, being treated with sodium methylate in methyl alcohol solution. 24gms. of 4,6-dimethyl-heptene-(2) boiling from 129.5- 130.1° corr. were collected.

$d_4^{25}(\text{vac}): 0.7239$	$n_D^{25}: 1.4135$	Analysis: Found	Calc.
		7.C { 85.71 (a)	85.63
		7.H { 85.69 (b)	
			{ 85.57 (c)
			{ 14.09 (a)
			{ 14.46 (b)
			{ 14.13 (c)

19. = 4,5,5-Trimethyl-hexene-(2).

This compound was prepared as the preceding two from 20gms. of ter.-butyl chloride, 23gms. of magnesium, and 80gms. of 4-brom-pentene-(2). The product was treated with alcoholic sodium ethylate. Five ccs. of 4,5,5-trimethyl-hexene-(2), boiling from 128.7° to 129.5° corr. were collected.

$d_4^{25}: 0.7382(\text{vac})$ $n_D^{25}: 1.4198$

20. = Decene - (1).

This olefine was prepared by the method used for pentene -(1). 66gms. of n-butyl bromide and 50gms. of allyl bromide gave, after the reaction mixture had been refluxed for three hours and worked up as usual (including treatment with sodium ethylate), 25gms. of decene -(1), b.p.: 170.4 - 170.8° corr. As a by-product, tetradecane, Part III, No. 9, was obtained.

A preparation of decene, mostly decene - (1), was made, earlier than the preceding preparation, from undecylenic acid. 74.5gms. of acid were dissolved in a hot solution of 80gms. of sodium in 1.5 liters of reagent alcohol, with vigorous shaking. The alcohol was distilled off, as much as

possible, at atmospheric pressure, and the remainder pumped off at 50mm. The pressure was then reduced to 15mm., the flask containing the mixture was surrounded with asbestos boards and a clay chimney, and vigorous heat applied. The distillate was redistilled at 46mm., that portion boiling at 87 - 90° being collected. This was fractionated at atmospheric pressure and 4gms. boiling at 172 - 172.5° corr. were collected. This procedure undoubtedly gave a far less pure product than the first procedure. The tests described herein were run on the first described preparation.

21. - 4 - Butyl-octene -(2).

This olefine was prepared as 4-methyl-pentene- (2). 64gms. of n-butyl bromide, 13gms. of magnesium, and 81gms. of 4-bromooctene-(2) gave, after refluxing for an hour and treatment with sodium ethylate in the usual purification process, 29gms. of 4-butyl-octene-(2) boiling at 115.2 - 117.2° corr. at 55 - 57mm. *Siwoloboff b.p.: 197-8° corr.*

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$$d_{4}^{25} \text{ (vac) : } 0.7558 \quad n_{D}^{25.0} : 1.4287$$

22. - Hexadecene -(1).

This olefine, or what corresponds very largely to hexadecene -(1), was prepared by the procedure customary for this compound. 1265gms. of spermaceti were heated in an iron still at about a half atmospheric pressure. Six

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burners, including one Meker, were required to heat it hot enough to get a stream of distillate. After 600ccs. of distillate had collected very little more appeared to be coming over. The still was allowed to cool, then the pump turned off, and the distillate which had collected was redistilled, that boiling from 150° to 185° at 44mm. being shaken with an equal volume of almost boiling concentrated ammonia. This last process was performed four times in all. The emulsions formed were cut by means of ether and ammonium sulphate. After washing and drying with calcium chloride it was distilled at 44mm. and cuts made at 180° corr., 185° corr., and 190° corr. These three portions were again treated with ammonia as before, washed, dried, and fractionated at 43mm. That boiling at 181.4 - 183.6° at this pressure was collected, a yield of 120gms. The product melted at 4-5°.

23. - 1 - Cyclohexyl-propene -(2).

This hydrocarbon was prepared similarly to pentene -(1). 206gms. of cyclohexyl-bromide, 35gms. of magnesium, and 121gms. of allyl bromide were used. The crude product was treated with alcoholic sodium ethylate, but as the distillate first obtained, boiling up to 151° u.r.c., contained some hydrobromic acid from decomposition of the residue in the flask it was treated with potassium carbonate and calcium chloride, and redistilled. 66gms. of 1-cyclohexyl-propene -(2) were obtained, b.p.: 154.0 - 154.4° corr.

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38.

24. - 4 - Cyclohexyl - pentene - (2).

This compound was prepared as 4-methyl pentene-235
(2). 115gms. of cyclohexyl-bromide and 80gms. of 4-bromo-
pentene -(2) were used. The reaction mixture was re-
fluxed for three hours and the crude product treated with
alcoholic sodium ethylate, 12.5gms. of 4-cyclohexyl-
pentene -(2) were collected, b.p.: 125.3 - 126.3° corr.,
at 95mm. *Siwoloboff b.p.: 195-6° corr. (slight dec).*

$$d_4^{25} \text{ (vac) : } 0.8322 \qquad n_D^{25} \text{ : } 1.4595$$

25. - 4 - Cyclohexyl - heptene - (2).

This compound was prepared similarly to the236
last. 84gms. of cyclohexyl bromide, 15gms. of magnesium,
150 ccs. of ether, and 70gms. of 4-brom - heptene -(2)
yielded 14.5gms. of 4- cyclohexyl - heptene -(2), b.p.:
135.6 - 136.6° corr. at 45mm.

$$d_4^{25} \text{ (vac.): } 0.8355 \qquad n_D^{25.3} \text{ : } 1.4630$$

A Siwoloboff determination indicated its boil-237
ing point to be 230 - 1° at 760mm. (*slight dec.*)

PREPARATION AND PURIFICATION

Part V - Diolefines.

1. - Pentadiene - (1,3).

This conjugated diolefine was made by the procedure of Reif⁽¹³⁾ for the preparation of this type of hydrocarbon from the corresponding alcohol, oxalic acid being here used as the dehydrating agent. 100gms. of pentene -(2)-ol-(4) were heated under a two foot Vigreux column in an oil bath, with 45gms. of crystalline oxalic acid. Ice-water was circulated through the condenser, and the receiver was surrounded with ice-water. The distillate was separated from the water, dried with calcium chloride, and fractionated. 14.4gms. of pentadiene -(1,3) were obtained, boiling from 42.0° to 42.3° corr. This corresponds to 14.5% of the theoretical yield. A larger yield could have been obtained by working up the residues again with oxalic acid, but it was not considered advisable to spend time in this way.

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2. - Diallyl.

The diallyl used in the course of this work was obtained from two sources. Frank Cortese contributed a sample of very pure diallyl⁽¹²⁾. The solubility data were obtained with this sample. The diallyl used in the specific characterization work was prepared by J.J.O'Brien as a special

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problem in the course of his junior organic chemistry laboratory experience. The procedure was that detailed by Cortese⁽¹²⁾, the product was not fractionated, but collected between 69° and 72° on distillation. In the coupling reaction, exactly the theoretical amount of magnesium was treated with 300gms. of allyl bromide. The mixture was kept cold during addition, and then allowed to stand two days, after which time it was worked up as any Grignard solution.

3. - Heptadiene -(2,4)

This diolefine was synthesized in a manner similar to pentadiene -(1,3). From 83gms. of heptene-(2) - cl - (4) and 35gms. of crystalline oxalic acid, 21.7gms. of heptadiene -(2,4), b.p.: 107.5- 108.0° were obtained, or 31% of the theory. 240

4. - Octadiene- (2,4).

From 100gms. of octene -(2)-cl-(4) and 40gms. of crystalline oxalic acid, by a procedure similar to that used in the synthesis of pentadiene -(1,3), 28.5gms. of octadiene -(2,4), boiling from 133.5° to 134.0° corr., were obtained, corresponding to a 33% yield. 241

$d_4^{25}(\text{vac}): 0.7427$ $n_D^{25}: 1.4542$

Analysis:	I	Found	Calc.
% C	87.02	86.89	87.19
% H	12.67	13.26	12.81

5. - 4 - Methyl-heptadiene -(1,5).

This diolefine was prepared by taking advantage of the procedure of Gilman and McGlumphy⁽¹³⁾ for the independent preparation of allyl magnesium bromide. 85gms. of allyl 242

bromide dissolved in 300ccs. of ether were dropped onto six times the theoretical quantity of magnesium covered with 150ccs. of ether. After the bromide had been added the solution was refluxed for a half hour, decanted from the excess magnesium, the latter rinsed with a little ether, and the rinsings combined with the reagent solution. 80gms. of 4-brom-pentene-(2) were then slowly added and the solution heated for three hours. The solution was then decomposed as usual, and, after having been freed from ether by the customary treatment with hydrochloric acid (d.:1.19), it was let stand over night with sodium ethylate solution in ethyl alcohol. The next day it was thrown out of solution with water, washed with water, dried with calcium chloride, and fractionated. 28.8gms. of 4-methyl-heptadiene-

(1,5) were obtained, boiling from 110.5° to 110.9° corr., 52% of the theoretical yield.

Analysis:		Found		Calc.
% C	86.8	I	87.09	87.19
% H	13.40	II	13.09	12.81

$d_4^{25}: 0.7284(\text{vac})$ $n_D^{25}: 1.4213$

6. - 2,2 - Dimethyl-hexadiene - (3,4)

This allene was prepared in a manner similar to that used for the conjugated diolefines, except that the alcohol used contained a tetra-substituted carbon atom in such a position that dehydration to form a conjugated diolefine was impossible. 42gms. of 2,2-dimethyl-hexene-(4)-ol-(3) were heated on an oil bath with 25gms. of crystalline oxalic acid, under a column, as before. 6.7 gms. of 2,2-dimethyl-hexadiene-

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(3,4) were obtained, b.p.: 107.4-108.0° corr. This corresponds to 18.5% of the theoretical yield.

$d_4^{25}(\text{vac}) : 0.7375$ $n_D^{25} : 1.4425$
7. - 4,5 - Dimethyl-octadiene - (2,6)

This diolefine was prepared by a Grignard procedure which was run somewhat in the Wurtz sense. 45gms. of 4-brom-pentene-(2) in 150ccs. of ether were added to about five times the theoretical quantity of magnesium. After addition of the bromide the solution was refluxed for a half hour, decanted from the magnesium, and treated with 35gms. more of 4-brom-pentene-(2). After heating an hour on the water bath, about two grams of magnesium were added and refluxing was continued for two hours. The reaction mixture was decomposed in the usual manner, the ether solution dried with calcium chloride, the resultant solution fractionated, and 14.1gms. of 4,5-dimethyl-octadiene-(2,6) finally obtained after redistillation. B.p.: 152.9 - 153.8°, 38% of the theoretical yield.

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$d_4^{25}(\text{vac}) : 0.7611$ $n_D^{25.1} : 1.43755$

Analysis:	<u>Found</u>		<u>Theoretical</u>
	I	II	
Wgt. sample	0.1946	0.1807	
Wgt. CO ₂	0.6226	0.5815	
Wgt. H ₂ O	0.2277	0.2060	
% C	87.28	87.79	86.87
% H	13.10	12.76	13.13

13.

8. - 4- Propyl-heptadiene-(1,5).

This diolefine was prepared in a manner similar to that used for 4-methyl-heptadiene -(1,5), number 5 of this part. 63gms. of allyl bromide in 250ccs. of ether, 60gms. of magnesium in 100ccs. of ether, and 70gms. of 4-brom-heptene-(2) gave 34.7gms. of 4-propyl-heptadiene-(1,5). Sodium methylate solution was used to free the product from halides. The diolefine was fractionated, and collected from 156.2 to 156.6° corr. 64% of the theoretical yield.

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$$d_4^{25}(\text{vac}): 0.7501 \qquad n_D^{25} : 1.4310$$

Analysis:

	<u>Found</u>		<u>Theoretical</u>
	I	II	
Wgt. sample	0.2085	0.1369	
Wgt. CO ₂	0.6598	0.4373	
Wgt. H ₂ O	0.2506	0.1648	
% C	86.34	87.14	86.87
% H	13.45	13.47	13.13

9. - 4- Allyl-octene-(2).

This diolefine was prepared similarly to the last. 68gms. of allyl bromide in 300ccs. of ether, 80gms. of magnesium in 100ccs. of ether, and 81gms. of 4-brom-octene-(2) gave, after proper sodium ethylate treatment, 27gms. of 4-allyl-octene-(2), b.p.: 110.1 - 111.1° corr. at 85mm. A Siwoloboff determination indicated its boiling

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point to be $178-9^{\circ}$, with slight decomposition.

$$d_4^{25}(\text{vac}): 0.7565 \quad n_D^{25}: 1.4342$$

10. - 4,5-Dipropyl-octadiene-(2,6).

This diolefine was prepared in a manner quite similar to that used for 4,5-dimethyl-octadiene-(2,6), number 7 of this part. 70gms. of 4-brom-heptene-(2) in 250ccs. of ether were added to 60gms. of magnesium in 100ccs. of ether. Refluxing, after addition of the halide, was effected for a half hour, the solution was decanted from the magnesium and decomposed as usual. Beside the heptene-(2) obtained from this procedure (and mentioned earlier) 19gms. of 4,5-dipropyl-octadiene-(2,6) were prepared after sodium ethylate treatment. This was fractionated in vacuo and collected from 117.1° to 118.1° corr., at 25mm. A Siwoloboff determination indicated its boiling point to be $221-2^{\circ}(\text{dec.})$ at 760 mm. The yield was 49.5% of the theory.

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$$d_4^{25}(\text{vac}) : 0.7804 \quad n_D^{25.0} : 1.4472$$

11. - 4,5- Di-n-butyl-octadiene-(2,6)

81gms. of 4-brom-octene-(2) were added to 5.1gms. of magnesium and 20ccs. of ether, after which 30ccs. of ether were added and refluxed for three hours. 2gms. of magnesium were then added and refluxing continued for three hours longer. The resulting solution, after decom-

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position, was dried with calcium chloride and distilled from an oil bath until all of the ether had been distilled off. The high-boiling from the octene-(2) (earlier described) was added to the residue in the flask, and the whole vacuum distilled. 18.5gms. of 4,5-di-n-butyl-octadiene-(2,6) were collected, boiling from 148.7° to 150.7° corr., at 25mm. A Siwoloboff determination gave 252.3°(1cc) as its boiling point at 760mm.

$d_4^{25}(\text{vac})$: 0.7949

$n_D^{24.9}$: 1.4528

PREPARATION AND PURIFICATION

Part VI, - Acetylenes.

1. - Pentine -(1)

This acetylene was prepared by H.D. Addison 249
in the course of a special problem in his junior organic
chemistry laboratory in 1929. The procedure adopted was
that described by Bourguel at great length⁽¹⁴⁾. The experi-
mental details given by him were found entirely adequate.
2-bromo pentene-(1) was prepared from 2,3-dibromo-propene-
(1) and ethyl Grignard reagent, and from this one mole-
cule of hydrobromic acid was removed by sodamide. Two
ccs. distilled out of xylene between 39.5-44.5° corr.
(principally 39.5 - 40.5°) and the tests here described
were run upon this product, which probably was more pure
than its boiling point range would indicate, because un-
doubtedly the vapor was superheated by the heat required
to vaporize it out of the xylene. No impurity could be
present which would interfere, at any rate, with the
formation of the mercury derivative described earlier (par. 121).

2. - Heptine -(1).

325gms. of dichlorheptane (prepared as previous- 250
ly described and therefore containing some isomers and
some chlorheptenes) were slowly dripped onto 257gms. of
finely powdered fresh sodamide which was suspended in

600ccs. of decahydronaphthalene and 900ccs. of commercial xylene. Three flasks were used, to which were added 200, 100, and 25gms. of dichlorheptane, respectively. The flasks were heated in an oil bath at 160-70° before addition of the chloride, and addition was continued so as to maintain a gentle reflux under the water condensers. After all the halide was added, the flasks were heated for two hours further at the same temperature, making the total period of heating three hours. The next day the condensers were turned over, and the major portion of the xylene distilled off - until the total volume of distillate measured 950ccs. The flasks were then surrounded with ice water and their contents slowly hydrolyzed, first with ice water, then with dilute hydrochloric acid until the contents became slightly acid. The top layer was separated, washed with water, sodium carbonate solution, water, and finally dried with calcium chloride. Upon fractionation 71gms. of heptene-(1), b.p.: 100.4 - 100.8° corr. and 27gms., b.p.: 99.4 - 100.4° and 100.8 - 101.9° corr. were collected. The tests herein described were made upon the fraction collected over four tenths of a degree. The total weight of heptene boiling from 99.9° to 101.9° was 98gms., 53% of the theoretical yield.

3. - Heptene -(2).

50gms. of heptene-(3), including the higher boiling portion collected (31.5gms., b.p.: 96.3°-96.8°) in the previously described preparation were shaken with 85gms. of bromine in an aqueous solution of sodium bromide, acidified with sulfuric acid (to prevent the formation of bromhydrin). Shaking was continued for twenty minutes. The product was separated and washed with sulfurous acid and water. The dibromide was treated with a little more than an equimolecular amount of potassium hydroxide in alcoholic solution, and refluxed for two hours. The alcoholic solution was diluted with water, the bromheptene washed with water, quickly dried with calcium chloride and dropped onto 30gms. of sodamide in an oil bath at 150°, under a reflux condenser. The flask was heated at 150 - 165° for two hours and the reaction product then distilled from the flask, without any hydrolysis, a sand bath being used to support the flask. The distillate was washed with water, dilute acid, water, dilute sodium carbonate solution, water, and then dried with calcium chloride. Upon fractionation, and refractionation of the cuts from 108-120°, 3ccs. of heptene-(2) were collected, b.p.: 111.5 - 113.0°, and upon refractionation of the lower cut,

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4. - Heptene -(3)

was obtained - 16.5gms. boiling from 105.3 - 106.7° corr.

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This corresponds to 33.7% of the theoretical yield.

Between 107° and 111.5°, about one cc. of a mixture of heptine -(2) and -(3) was obtained. This appears to indicate that the three cc. cut taken from 111.5 - 113.0° represents heptine -(2), rearrangement having occurred as would be expected from the work of Bourguel.⁽¹⁴⁾

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5. - Octine - (1)

15gms. of octine -(2) prepared as specified in the immediately following section were added all at once to a suspension of 8gms. of finely powdered sodamide in about 40ccs. of Eastman's "terpene-free p-cymene (No. 83)" held in a flask in an oil bath at 160 - 170°. The bath was held at this temperature for two and a half hours, until the refluxing, at first rapid, had practically ceased and the mass had become a paste. The condenser was then turned over and about 30ccs. of the solvent were boiled off from a sand bath. The flask was then suspended in an ice bath, and ice-water slowly added, followed by dilute hydrochloric acid until the reaction mixture was acid. The top layer was separated, washed with water, sodium carbonate solution, water, then dried with calcium chloride and fractionated. 7.3gms. of octine-(1), boiling from 127.6° to 128.0°, corr., were obtained. This corresponds to 48.5% of the theory.

254

6. - Octine -(2).

33gms. of sodamide (finely pulverised, fresh product) were suspended in about 900ccs. of anhydrous ether in a two liter three-neck flask, and 58gms. of heptine-(1) were added to it dropwise. Part of the heptine used included the 27gms. previously described as consisting of cuts boiling from 99.4 - 100.4° and 100.8-101.9° corr. After a few drops of heptine-(1) had been added, the flask was warmed on a water-bath. Heating was discontinued when gelatinous material began to form. The heptine was added during an hour, with stirring, and stirring was then continued for a half hour longer, during which time the flask was heated on a water bath. 110gms. of dimethyl sulfate were then slowly added over a period of about one hour. Ether was added from time to time in an effort to keep the mass semi-plastic. The mixture was stirred and warmed for an hour after all of the methyl sulfate had been added, and then let stand over night. The next day cold water was added, the ether layer separated and refluxed for a half hour with water, then a half hour with concentrated ammonium hydroxide (to saponify the poisonous methyl sulfate), then washed with water, with dilute hydrochloric acid, water, sodium carbonate solution, water, and then dried with calcium chloride. The ether was distilled off through a fractionating column and the remainder fraction-

255

ated, 39.3gms. of octine -(2), b.p.: 138.0 - 138.4°, corr., being collected. 23.7gms. of heptine-(1), boiling from 99.5 to 100.2° were recovered from this preparation. This corresponds to a 59% over-all yield, or to a 100% yield based on the heptine-(1) actually used.

The methyl sulfate here used was Eastman's technical grade, purified by shaking with water, then twice with sodium bicarbonate solution, and then drying with calcium chloride over night.

256

7. - Nonine -(4)

This acetylene was prepared in a manner similar to that by which heptine -(3), number 4 of this part, was prepared. 39gms. of nonene-(4) and 55gms. of bromine gave a dibromide which after two and a half hours of refluxing with slightly more than the theoretical amount of alcoholic potassium hydroxide gave a brom-olefine which was diluted with about 100ccs. of toluene (boiling below 117°) and dried with calcium chloride. This solution and 55gms. of pulverised sodamide (moistioned with toluene before the solution was added to it) gave a distillate after two hours of heating which was collected between 117° and 175°. 23gms. of high-boiling material remained behind. This ^{latter} was dropped onto 17gms. of powdered sodamide held in an oil bath at 150° - 165° for three hours. The distillate

257

52

from this second treatment was combined with that first obtained. Upon fractionation, 10.5gms. of nonine-(4) were obtained, b.p.: 155.1 - 156.1°. This corresponds to 27.4% of the theoretical yield.

$$d_{4}^{25}(\text{vac}): 0.7716 \quad n_{D}^{24.9}: 1.4330$$

Analysis:	<u>Found</u>	<u>Theoretical</u>
Wgt. sample	0.1537	
Wgt. CO ₂	0.4895	
Wgt. H ₂ O	0.1743	
% C	86.91	86.96
% H	12.69	13.04

8. - Hexadecine -(1).

20gms. of cetene were shaken for five minutes with slightly more than the theoretical amount of bromine in an aqueous solution of sodium bromide in sulfuric acid. The excess bromine was destroyed with sulfurous acid, the dibromide extracted with ether, dried with sodium sulfate, and the ether evaporated. 20gms. of dibromide thus formed were dissolved in an equal volume of xylene and dried with sodium sulfate. The solution was added to a suspension of two and a half times the theoretical amount of sodamide in xylene in a liter flask clamped in an oil bath at 100°. Frothing began at once and continued until the entire flask

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filled with foam. The temperature of the oil bath was gradually raised until it reached 160° and the xylene began to reflux vigorously. The flask was heated over a period of about two and a half hours. After cooling, the reaction mixture was poured onto ice, extracted with ether, and dried with calcium chloride over night. The next day the ether and xylene were distilled off at ordinary pressure, and the residue distilled at 15mm. 7.5gms. of crude hexadecine-(1) were collected, boiling at 145 to 170° u.c. at 15mm. This corresponds to 65% of the theoretical yield. This was dissolved in 22ccs. of alcohol and treated with 11.5gms. of silver nitrate dissolved in 12ccs. of water and 95ccs. of alcohol. The precipitate was filtered with suction, washed twice with 20cc. portions of ether, and the acetylene liberated from the salt by treatment with dilute (about 8 N.) hydrochloric acid. The hexadecine layer was extracted with ether, dried with sodium sulfate, and distilled at 15mm. Two separate cuts were taken separately at 156-7° corr. and 158 - 160° corr. These two cuts were probably the same, boiling differently due to rather wide fluctuations in the pressure. The former cut was taken as the sample tested in this work. It melted at 15°, according to H. R. Batchelder, who identified it as hexadecine-(1) by the use of a preliminary outline of this procedure in the course of his advanced

course in qualitative organic analysis, in 1928.

The acetylene apparently decomposes on distillation even at this pressure, for the flask contained a brown residue that darkened as the distillation continued.

259

PREPARATION AND PURIFICATION

Part VII. - Aromatics.

1. - 9 - Isoamyl - anthracene.

This hydrocarbon was the only aromatic prepared in the course of this work. It was made by H.T. Gerry as a special problem in the course of his junior organic chemistry laboratory experience in 1928. Through his courtesy his detailed directions for its preparation are included here. The procedure of Liebermann⁽²⁴⁾ was followed in a general way. 260

200gms. of anthraquinone were moistened with about 50cc. of alcohol and to this were added 417gms. of zinc dust and 283gms. of sodium hydroxide dissolved in 2850ccs. of water. This was refluxed for an hour and a half on a sand bath and then 150gms. of isoamyl bromide were added in portions through the reflux condenser over a period of half an hour. The mixture was then boiled eight hours longer. 261

The resulting solution was filtered, the residue being returned to the flask and boiled with about two liters of water without the use of a reflux condenser. The liquid was again filtered and the filtrate discarded (boiling without the reflux condenser ridding the reaction product of any isoamyl bromide or alcohol remaining). 262

The solid residue was then treated with two liters of alcohol and heated to boiling. The hot solution was filtered and set aside over night. In the morning the anthracene and anthraquinone that had precipitated was filtered off and the solution was diluted with water to about nine liters. A heavy sticky oil separated which was filtered off.

263

This oil was dissolved in a liter and a half of cold alcohol and the white precipitate of anthracene filtered off. Sixty-five grams of picric acid were added to this alcoholic solution, which immediately turned a deep red, the color of the hydrocarbon picrate. This solution was refluxed for two hours and then allowed to cool. The precipitate of picrate came down only on shaking, as supersaturated solutions of it in alcohol are readily formed. After filtering off this yield of the picrate the solution was evaporated to about 500cc. and another yield of the picrate obtained.

264

The picrate was then taken up in slightly more boiling alcohol than that required to dissolve it. Then concentrated ammonia was added to the hot solution until the red color of the solution decreased and became no lighter on further addition. The hot solution was filtered and cooled. The hydrocarbon precipitated, although there was a great tendency to form supersaturated solutions.

265

57.

The hydrocarbon was then filtered off and washed first with a small portion of alcohol and then with water until the water began to come through colorless. This product was recrystallized twice from alcohol.

266

By this procedure 27 grams of 9-isoamyl anthracene were obtained. The product melted at 58-59°C. It formed long thin needlelike crystals that greatly resemble silk fibers in shape, luster, and feeling. The color is white with a strong blue fluorescence.

267

On standing, the solid, white hydrocarbon develops a slight yellow color, deepening on long standing and exposure to the sunlight to a golden-orange shade. At this latter stage in change of the compound, the odor of isovaleric acid in the stoppered bottle is very pronounced. The tests herein described were made upon this hydrocarbon before this oxidation had begun.

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CHAPTER III

SUMMARY

SUMMARY

A new procedure has been developed for the identification of pure hydrocarbons. It classifies these compounds primarily as solids, liquids, and gases. It further divides the solids into (1) non-aromatics and (2) aromatics, and the liquids into (1) aromatics, (2) ethers, (3) cyclic unsaturates together with acyclic acetylenes and polyolefines, (4) acyclic olefines, (5) naphthenes and (6) acyclic paraffins. These divisions are based upon miscibility with various selective solvents, boiling point, gravity, and addition of bromine.

269

To test this procedure some two hundred compounds have been obtained from various sources, of which number about fifty have been synthesized.

270

Tables including the properties and reactions of the most important hydrocarbons have been completed from abstract journals.

271

CHAPTER IV

THE PROCEDURE FOR THE IDENTIFICATION
OF HYDROCARBONS

A

Preliminary Considerations

Outline of the Method

Reliability of the Tables

System of Nomenclature of the Tables

List of Abbreviations

THE PROCEDURE FOR THE IDENTIFICATION OF HYDROCARBONS

The procedure for sectional division has been discussed in the body of this thesis. Reference should be had to the chapter dealing with methods of group division for a detailed consideration of it. (Cf. par. 69).

272

In general it may be said that the method of locating a compound in the proper section consists of the following steps, not all of which are always necessary.

273

1. Determination of state at 0° or 20°.

In this determination the compound should always be cooled to (at least) fifteen degrees below either 0° or 20° respectively, held at this temperature for several minutes, and scratched if liquid. If a change in state has occurred due to this cooling, the compound should be allowed to warm up slowly, the state at 0° or 20° then being noted.

274

2. Determination of boiling point.

Methods for this determination are given in Mulliken, "Identification of Pure Organic Compounds", Vol. I, p. 221 et seq.

275

3. Determination of density.

Methods for this determination are given in Mulliken, "Identification of Pure Organic Compounds",

276

Vol. II, pp. 227 et seq.

4. Determination of solubility in nitromethane.

The method for this determination is given at the end of these tables, Test 922.

277

5. Determination of solubility in sulfuric and hydrobromic acids.

The methods for these determinations are given at the end of these tables, Tests 922 and 929.

278

6. Determination of solubility in aniline.

The method for this determination is given at the end of these tables, Test 922.

279

7. Determination of unsaturation by the bromide-bromate titration.

The method for this determination is given at the end of these tables, Tests 924 and 925.

280

8. Determination of solubility in benzyl alcohol.

The method for this determination is given at the end of these tables, Test 922.

281

To locate a compound in the proper section, its Division is first determined, Then the chart given on the following pages (pages 220 -226) should be consulted and the compound placed by follow-

282

ing through the scheme as directed by the "flow sheet".

Sectional tests, semi-specific tests, and specific tests, are given at the end of the tables, page

283

Illustrations of the way to use the procedure are given on page 82.

284

Bibliographical references have been given to the properties and reactions listed. Not all of the possible references have been included, by any means, but only those references in which the properties actually chosen are given. The reference numbers are found above the properties in the property columns, and in parentheses following the properties and reactions in the descriptive columns. This variation has been adopted to conserve space and preserve clarity. The numbers refer to the same numbers in the corresponding Divisions and Sections of Appendix II.

285

Stars have been placed against the names of those compounds upon which the writer or one of his colleagues has done sufficient work to feel that it is properly placed, although this does not mean that all of the sectional tests have necessarily been applied to it. The exact tests applied are given in

286

various places in the body of this thesis. Stars (*)
have also been placed against those properties and
reactions which have been obtained in this laboratory.

THE RELIABILITY OF THE TABLES

A statement is necessary concerning the reliability of the tables given in the following section. All hydrocarbons of known structure, many of known carbon skeleton but unknown or doubtful location of unsaturation, and the most important of unknown structure but known properties are included in these tables, subject to the following qualifications.

These compounds and their descriptions, except in a few cases where original sources were consulted, were all collected by the use of the 1910 Beilstein, Vols. I and V, the Supplement to Vol. I, and the subject indices (~~decennial~~ and annual, as necessary) of those "Chemical Abstracts" published since the "Literatur-Schlussstermin" of these volumes of Beilstein. Undoubtedly the writer missed a good many important compounds and properties, but the indices were carefully read through, all of the references to hydrocarbons looked up, and the data selected as seemed judicious. In collecting the data on acyclic paraffins, acyclic olefines, acyclic diolefines, and acyclic acetylenes, the abstract of the London Chemical Society and the Chemische Zentralblatt were also used in a similar manner. Abstracts

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288

published up to January 1, 1928, were consulted for all of the non-aromatic hydrocarbons. (The London Abstracts were not consulted beyond 1925.) Abstracts published up to January first, 1914, were consulted for all of the aromatic hydrocarbons. The writer has abstract references for the aromatic hydrocarbons reported from 1914 to 1927, inclusive, but lack of time prevented him from looking them up and listing any valuable information obtained from them. These statements are all subject to the exception that no literature work has been done for the gaseous compounds of Div. C of these tables beyond listing the names and boiling points of those given in Beilstein and the recent literature to January first, 1928.

The writer has used his own discretion in selecting data and reactions for the tables. He has taken into consideration the methods used for various preparations of the same compound, the care which seemed to have been exercised by the original worker, the reputation of the original worker and the frequency with which constants for one compound have been duplicated, where a given compound has been described several times.

In considering the more common terpenes, the

289

290a

writer has found the section dealing with "Die Terpene" of Abt. I, Chemische Methoden, Teil 11, Heft 5" of Abderhalden's "Handbuch der biologischen Arbeitsmethoden", written by Konrad Bournot, of great assistance in confirming the reliability and value of the constants and data selected and in supplying additional information concerning the compounds discussed therein.

According to the scheme of Mulliken's "Identification of Pure Organic Compounds", certain more or less unreactive oxygenated compounds, such as ethers, fall in the hydrocarbon section. This research deals with hydrocarbons only, but provides for the removal of acyclic ethers from them. In order to show this, and to remind the reader that other oxygenated compounds may fall in this section, those oxygenated compounds at present in Genus IX of Vol. I of Mulliken¹ have been included here. Except as noted in the tables, the descriptions of these compounds have not been brought up to date. The constants, only, of a few other ethers which were handled in this research are also included.

290b

For a discussion of the way in which the reported constants have been abbreviated and extrapolated for inclusion in the tables, see paragraphs 290o and 290q.

290c

THE USEFULNESS OF PHYSICAL CONSTANTS
IN HYDROCARBON IDENTIFICATION.

The writer has been requested to make a few general statements concerning the physical constants of hydrocarbons, in particular, concerning their dependence on structure, and the methods of calculating them. This discussion is written with some hesitance for it is feared that these statements will be taken too literally. In seeking to apply the information given, therefore, it must be remembered always that all sorts of complications enter into these relations which have to be considered in each particular case. No general rules can be universally applied!

290d

In general, corresponding (in structure) members of an homologous series have boiling points which, plotted against the number of carbon atoms in the molecule give a smooth curve for any one series. This appears to be especially true of non-aromatics. Thus, in the diagram on page 212j the boiling points of the members of several series are plotted in this way. The values are taken from the tables. Curve I is for pentene-(2), hexene-(2), heptene-(2), octene-(2), nonene-(2), and undecene-(2). Curve II is for pentene-(1), hexene-(1), heptene-(1), octene-(1), nonene-(1), decene-(1) (these values, except for octene-(1) were obtained experimentally in this research). Curve III is for 2-methyl-butene-(3), 2-methyl-pentene-(4),

290e

2-methyl-hexene-(5), and 2-methyl-heptene-(6). Curve IV is for n-pentane, n-hexane, n-heptane, n-octane, n-nonane, n-decane, n-dodecane. Curve V is for methyl-cyclohexane, ethyl-cyclohexane, propyl-cyclohexane, butyl-cyclohexane. Curve VI is for cyclohexane, cycloheptane, cyclooctane, cyclononane. Curve VII is for toluene, ethyl-benzene, propyl-benzene, butyl-benzene, amyl-benzene. In general the curvature of the lines for different members of the same series is the same, cf. lines I, II, and III. The curvature for other lines of other series may be nearly the same; this is especially true of the paraffins and olefines. It is apparent, from the Diagram, where these statements may be helpful, as well as where they cannot be applied.

The members of curve III represent well what is meant by "members of an homologous series corresponding in structure". In this case as the main chain lengthens, the double bond at one end remains in position 1, the methyl group at the other remains on the second carbon from the other end. It must be remembered that these compounds are not strictly corresponding for the relation of the side chain to the double bond does not remain constant. This must be taken into consideration especially when the side chains join directly an unsaturated carbon atom. In general, it

290f

is the distance from the end of the chain which is the most important influencing factor in the change of a constant upon introduction of a side chain or an unsaturation.

Furthermore, in general, the entrance of a side chain or an unsaturation in position 2 causes an abnormal change in the constants of a compound. The introduction of a double bond in position 2 gives an olefine a higher density (5 to 10 parts in the third decimal for the lower members) and a higher boiling point (in general about four degrees) than it would have were the unsaturation in position 1, and causes the boiling point to be almost identical with the paraffin of the same carbon skeleton, cf. curves I and IV. The introduction of a methyl group in position two causes a marked lowering in boiling point and density from those of the straight chain compound of the same carbon skeleton. This decrease in boiling point is usually about nine degrees for the first methyl group and even more than as much again for the second methyl group on the same carbon atom, in the olefine series, - somewhat less in the paraffin series. 290g

The absolute values of differences between constants of hydrocarbons of different structures decrease with increase of molecular weight. The boiling points of olefines with the double bond in position three are nearly 290h

the same as those for their isomers with the double bond in position one, their densities and refractive indices are slightly greater than those of the latter group of olefines, but less than those of olefines with the double bond in position two. The introduction of a methyl group in position three raises the boiling point, in general, above that of the isomer having its methyl group in position two and increases the density and refractive index even slightly above the straight chain isomer; this is especially true in the paraffin series, e.g.

	B.P.	d	n_D
n-hexane	69-70	0.660(20/4)	1.375(20)
2-methyl-pentane	60-1	0.654(20/4)	1.375(15)
3-methyl-pentane	63-4	0.665(20/4)	1.379(15)
n-octane	125-6	0.705(18/4)	1.399(18)
2,2,4-trimethyl-pentane	98.5-9.5	0.692(20/4)	1.392
2,2,3-trimethyl-pentane	110.5-111.5	0.722(15/15)	1.416(25)

The difference in boiling points between corresponding members of a homologous series is approximately constant and tapers off uniformly with increase in molecular weight, cf. the diagram on page 212k. This same statement applies to boiling points at reduced pressure, see, for 290i

instance, Bourguel, Ann. Chim., 10 3, 366 (1925). It must always be remembered that differences in all physical constants, of the kinds thus far mentioned, decrease with increase of molecular weight.

In general, in the non-aromatic series, the densities and refractive indices increase with increase in molecular weight. 290j

In general, acetylenes, conjugated diolefines and allenes have higher boiling points than the corresponding olefines of similar structure, the order of ascending boiling points being the order in which these compounds are named. The same statements apply to these compounds as have already been given for olefines and paraffins. 290k

In general, monocyclic, bicyclic, and tricyclic systems may be distinguished from one another by increasing density and refractive index in the order named. (This, as other statements, must take on life to the reader by a study of the constants in the tables.) This fact has frequently been used in determining the structure of sesquiterpenes. It must always be remembered, however, that in a family of a given empirical formula, these two constants are subject to change not only due to previously mentioned causes, as well as to the number of rings present, but also to the state of saturation, so that an increase in density 290l

due to formation of a second ring may be partly or wholly offset by decrease in density due to increased saturation.

In general, the density and refractive index decreases with increasing saturation.

290m

In general, in the aromatic series, increase in molecular weight due to increase in length of the side chain causes decrease in the density. This is especially noticeable after the molecule contains over nine carbon atoms.

290n

Frequently, because of the approximate validity of the approximate Clausius-Clapeyron equation, it is possible to obtain an approximation to the boiling point of a hydrocarbon whose boiling point is reported at only a single reduced pressure by plotting this point on semi log paper on which the ordinates are pressures in mms., and the abscissae are reciprocals of the absolute boiling points. A straight line is then drawn through this point parallel to a similar line drawn between points plotted for a compound in the same series of similar structure whose boiling points at at least two different pressures are recorded. Those compounds listed in the tables as boiling at "about" a certain temperature have had their boiling points computed by the writer in this way. The boiling points at reduced pressures are listed, together with the references to the articles in which they are given.

290o

Recently a valuable recommendation has been made of the use, for the purpose described, of a paper ruled in such a way that while it is semi log, the temperatures may be plotted directly, instead of their reciprocals. See D. S. Davis, Ind. Eng. Chem., Analytical Edition, 2, 306 (1930).

In general it may be said that if a density 290p is determined at a temperature within a few degrees of that at which it is desired to have it, it may be computed approximately, for qualitative work, by using a factor of $dd/dt =$ one part in the third decimal per degree. The density, of course, decreases with increase in temperature. The refractive index may similarly be computed approximately by applying a factor of five parts in the fourth decimal per degree. In general the density and the refractive index vary in the same way.

For the reasons set forth on pages 12 to 17 290q the writer does not feel justified in encouraging confidence on the part of the qualitative analyst, in the reliability of physical constants beyond the limits there set (pages 13, 14). Accordingly, in the tables of properties, the boiling points have been listed as degree ranges, and the fourth decimal has been struck from the densities and refractive indices. Moreover, the temperatures at which densities and refractive indices have been determined are reported to the nearest whole numbers, and boiling points reported close to 760 mm.

have been corrected to that pressure by applying an approximate correction factor of 0.1° per 2.7 mm. This correction factor has not been applied when it would cause a correction of more than one degree, except when the boiling point which has been corrected is also listed at the experimental pressure. The exact constants are given in the references.

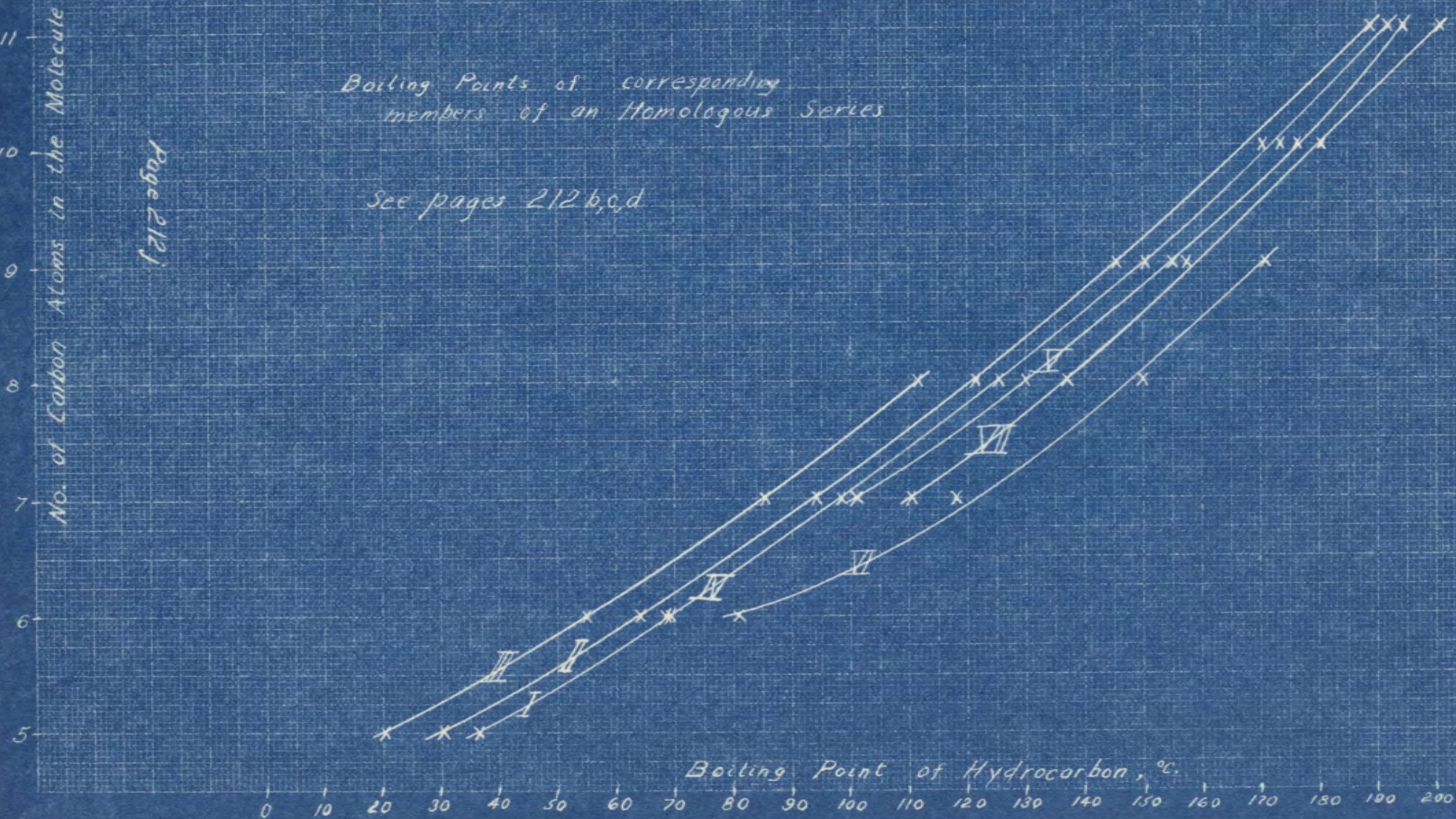
These statements are not intended in any sense 290r as a literature review. With respect to boiling points, however, the reader may find of interest an article by Boris Nekrasov, *Z. physik. Chem. Abt. A*, 141, 378-86 (1929) where a method is given for calculating the boiling points of hydrocarbons from their structure which has been successfully applied to 260 out of 316 hydrocarbons (the calculated boiling point being within five degrees of the actual one). The boiling point of pinene was calculated to within one degree. See also Nekrasov, *Z. physik. Chem., Abt. A*, 140, 342-54 (1929).

The calculation of molecular refraction and 290s the uses to which it may be put are too well known and too frequently alluded to to warrant detailed consideration here. A brief review of this subject may be found in Brooks "The Non-Benzenoid Hydrocarbons", pages 550-562. Reference may also be had to the entire sixteenth chapter of this volume for a general review of physical properties of the hydrocarbons. See also; Bourguet, *Bull. soc. chim.* 41, 1475 (1927); Edgar, co-workers, *J. Am. Chem. Soc.* 51, 1485, 1540 (1929); Dykstra, Lewis, Bond, *J. Am. Chem. Soc.* 52, 3396 (1930).

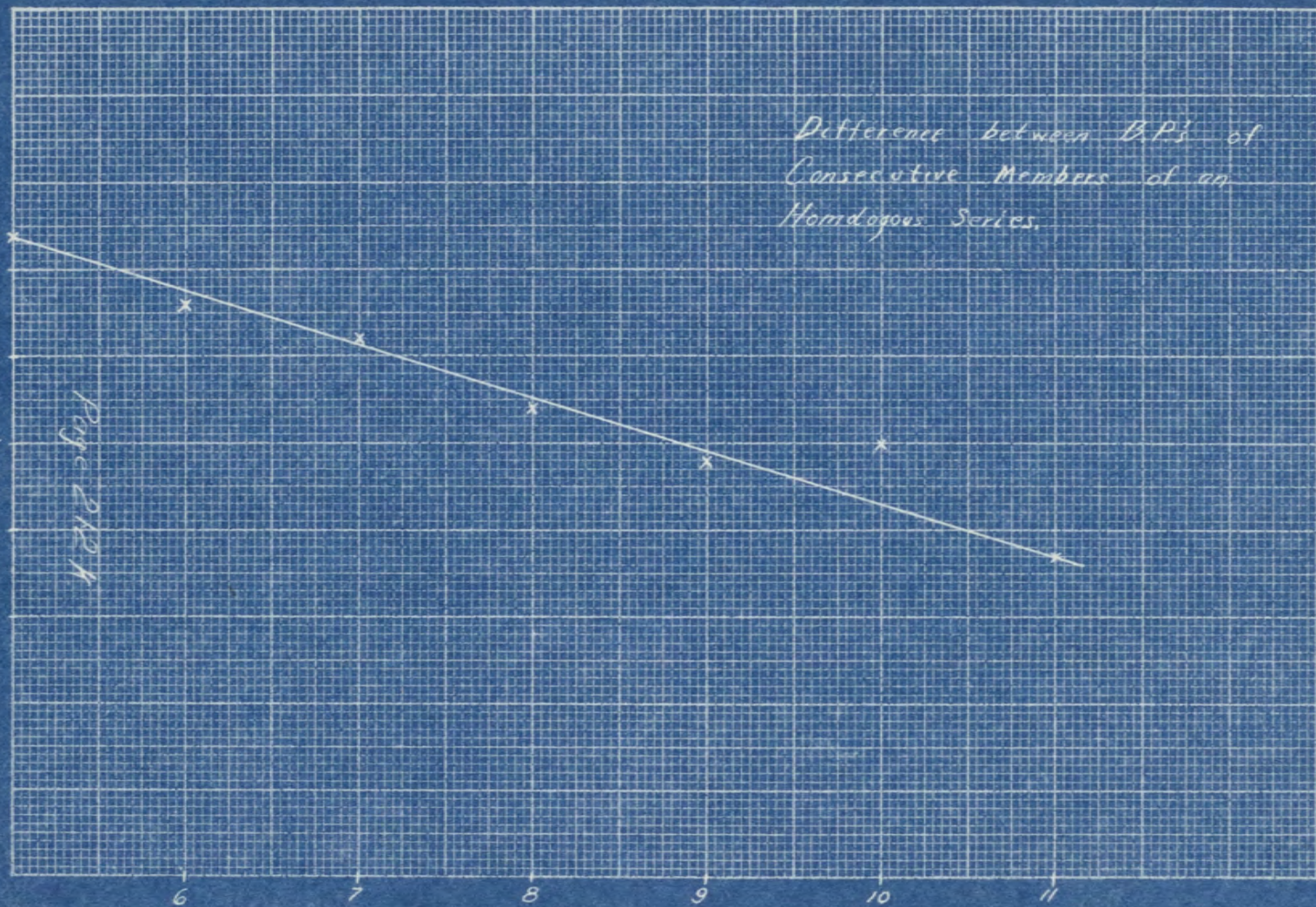
Page 212

Boiling Points of corresponding members of an Homologous Series

See pages 212 b, c, d



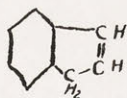
$^{\circ}\text{C}$ Difference between Boiling Points of Consecutive Members of the Series, plotted against the B.P. of the higher of the two whose B.P. difference is being considered.



No. of Carbon Atoms in Normal, Monosubstituted Ethylenes

THE SYSTEM OF NOMENCLATURE
OF THE TABLES

Probably the best statement that can be made concerning the nomenclature in the tables is that in general it conforms to the rules given in the 1910 edition of Beilstein. Strict consistency has been sacrificed to clarity, thus $\text{Ph}\cdot\text{CH}_2\cdot\text{CH}_2(\text{CH}_2)_2$ has been called isobutyl benzene, whereas $\text{Ph}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{Ph}$ has not been called (1st-phenylpentyl)-benzene, but 1,5-diphenyl-pentane. The terms isopropyl, isobutyl, sec.-butyl, ter.-butyl, n-amyl, and isoamyl have been used for these groups instead of the more systematic, but less clear metho-ethyl, etc. The word "benzolo" frequently used in Beilstein has been shortened here to the more familiar "benzo", indicating a benzene ring condensed in the ortho positions with another ring, as benzo-cyclopentadiene:



An attempt has been made to include the most important trivial names.

291

292

List of Abbreviations used in the Tables

In addition to the customary chemical abbreviations the following abbreviations are also used in the Tables.

293

+ = and

abt. = about

ac. = acid

Ac. = MeCO-

acid. = acidify

addn. = addition

alc. = alcohol (or alcoholic)

alk. = alkaline

Am. = isoamyl

ammon. = ammoniacal

amt. = amount

amts. = amounts

anhyd. = anhydrous

aq. = aqueous (or water)

assym. = assymmetrical

atm. = atmospheric

b.b.no. = "bromide bromate number"

Boil. = boiling

Calcd. = calculated

chem. = chemical

conc. = concentrated

corr. = corrected

corresp. = corresponding

cpd. = compound

C.S.T. = critical solution temperature

dec. = decomposes (or decomposing, or decomposition)

decomp. = decomposes at (or decomposition)

depolym. = depolymerizes

deriv. = derivative

detn. = determination

d-form = dextro-rotary form

dil. = dilute

diln. = dilution

distd. = distilled

distn. = distillation

div. = division

Et = H_5C_2-

exp. = exploding

extn. = extraction

filtn. = filtration

fm. = from

frm. = from

fum. = fuming

g. = gives

g. = gram (whether g. means "gives" or "grams" is
always clear from the context of the sentence.)

gluc. = glucose

hr. = hour

glac. = glacial
hr. = hour
hrs. = hours
ht. = heat
htd. = heated
htg. = heating
hy. = hydrocarbon
i. = insoluble
ident. = identical
Ident. = identification
insol. = insoluble
l-form = laevo-rotatory form
m. = melting
Me = $\text{H}_3\text{C}-$
min. = minutes
mixt. = mixture
ord. = ordinary
org. = organic
orig. = original
oxid. = oxidizes (or oxidizing, or oxidation)
part. = partial
pet. = petroleum
Ph = C_6H_5-
polym. = polymerizes
ppt. = precipitate

pptn. = precipitation
ppts. = precipitates
Pr = H_7C_3-
prepns. = preparations
press. = pressure
prim. = primary
pt. = part
pts. = parts
quant. = quantitatively
resp. = respectively
rextald. (or rextalld.) = recrystallized
rextaln. = recrystallization
rm. = room
s. = soluble
sapn. = saponification
satd. = saturated
sect. = section
sepn. separation
septd. = separated
shak. = shaking
sol. = soluble
soln. = solution
stg. = standing
syst. = system

temp. = temperature

theor. = theoretical

unoxid. = unoxidized

unsatd. = unsaturated

unsatn. = unsaturation

$VNM/G = (\text{volume of standard bromide-bromate solution}) \times (\text{normality}) \times (\text{m. w. of hydrocarbon}) / (\text{gravity}$

vol. = volume

of hydrocarbon)

vols. = volumes

[cf. page 519,

w. = with

Test 924]

wash. = washing

wgt. = weight

wh. = which

xs. = excess

xtalize = crystallize

staln. (or xtaln.) = crystallization

xtaln. = crystalline

xtals. = crystals

B

The Chart

KEY TO THE CHART

The Tables of Properties are listed in three Divisions, - A, B, and C.

Division A contains compounds which are solid at 0°. See the chart on page 221.

Division B contains compounds which are liquid at 0° and boil above 20°. See the chart on page 226. The temperatures at which solubilities in various solvents should be determined are given for various boiling point ranges as follows:

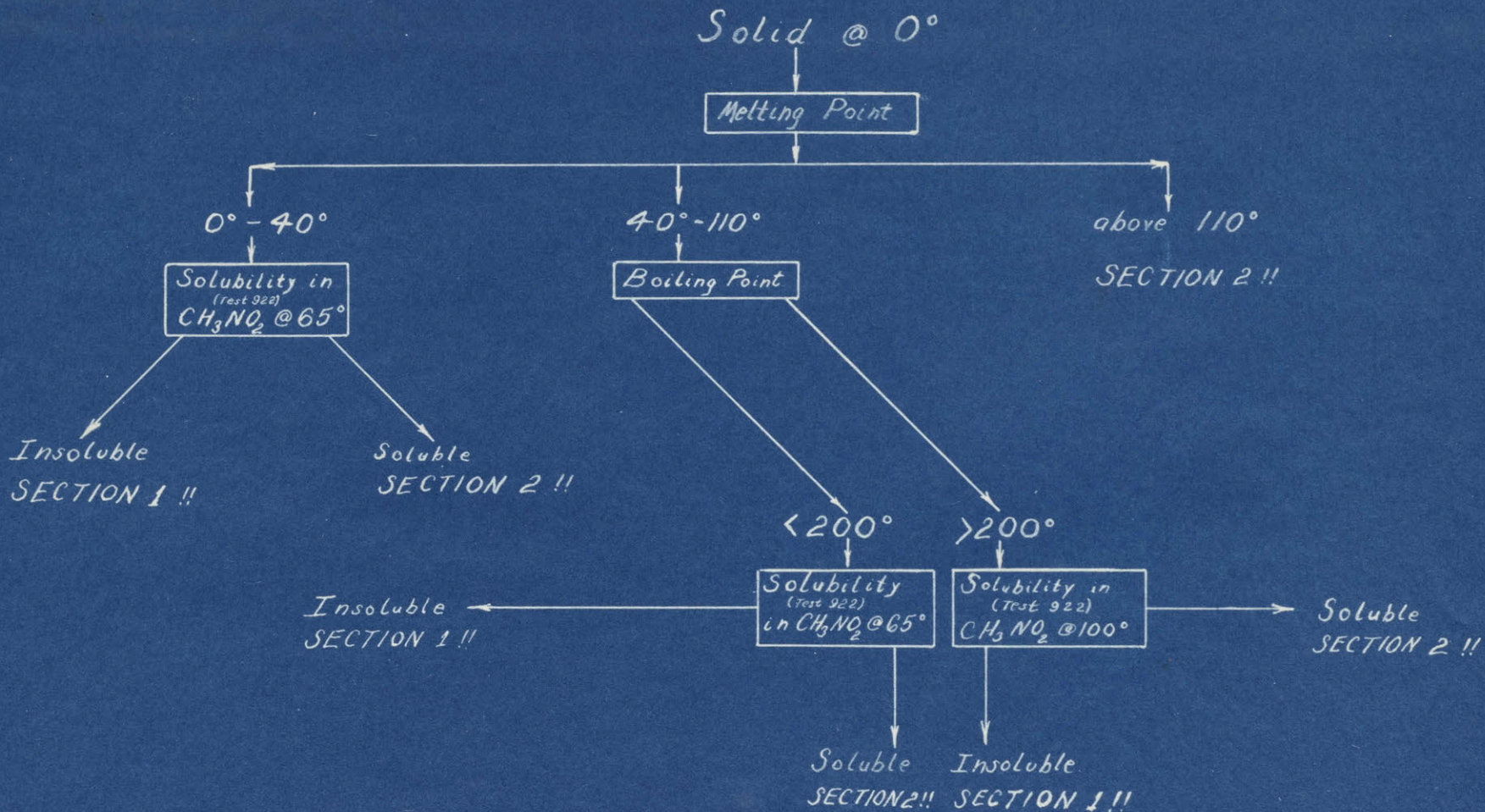
Nitromethane	222
Aniline	223
Benzyl Alcohol	224

The density dividing line for determining whether an unsaturated is cyclic or acyclic is given for various boiling point ranges on page 225.

Division C contains compounds which are gaseous at 20°. It is not subdivided and therefore has no chart.

DIVISION A

221



Temperatures for the Determination of Solubility
in Nitromethane,

If the compound boils between	Determine its solubility in CH_3NO_2 at
20° and 40°	0°
40° and 70°	15°
70° and 130°	0°
130° and 160°	10°
160° and 190°	25°
190° and 220°	40°
220° and 250°	55°
250° and 280°	70°
above 280°	85°

Temperatures for the Determination of Solubility
in Aniline.

If the compound boils between	Determine its solubility in aniline at
70° and 130°	0°
130° and 190°	15°
190° and 240°	30°
240° and 285°	45°
above 285	60°

Temperatures for the Determination of Solubility
in Benzyl Alcohol.

If the compound boils between	Determine its solubility in $\text{C}_6\text{H}_5\text{CH}_2\text{OH}$ at
20° and 70°	20°
70° and 130°	30°
130° and 190°	50°
above 190°	70°

The Density Dividing Line Between Unsaturated
Cyclics and Acyclics.

If the compound boils between	It belongs in Div. B, Sect. 4 (if it is unsaturated) if it has a lower density at 20°/4° than
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20° - 40°	0.70°
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40° - 70°	0.73°
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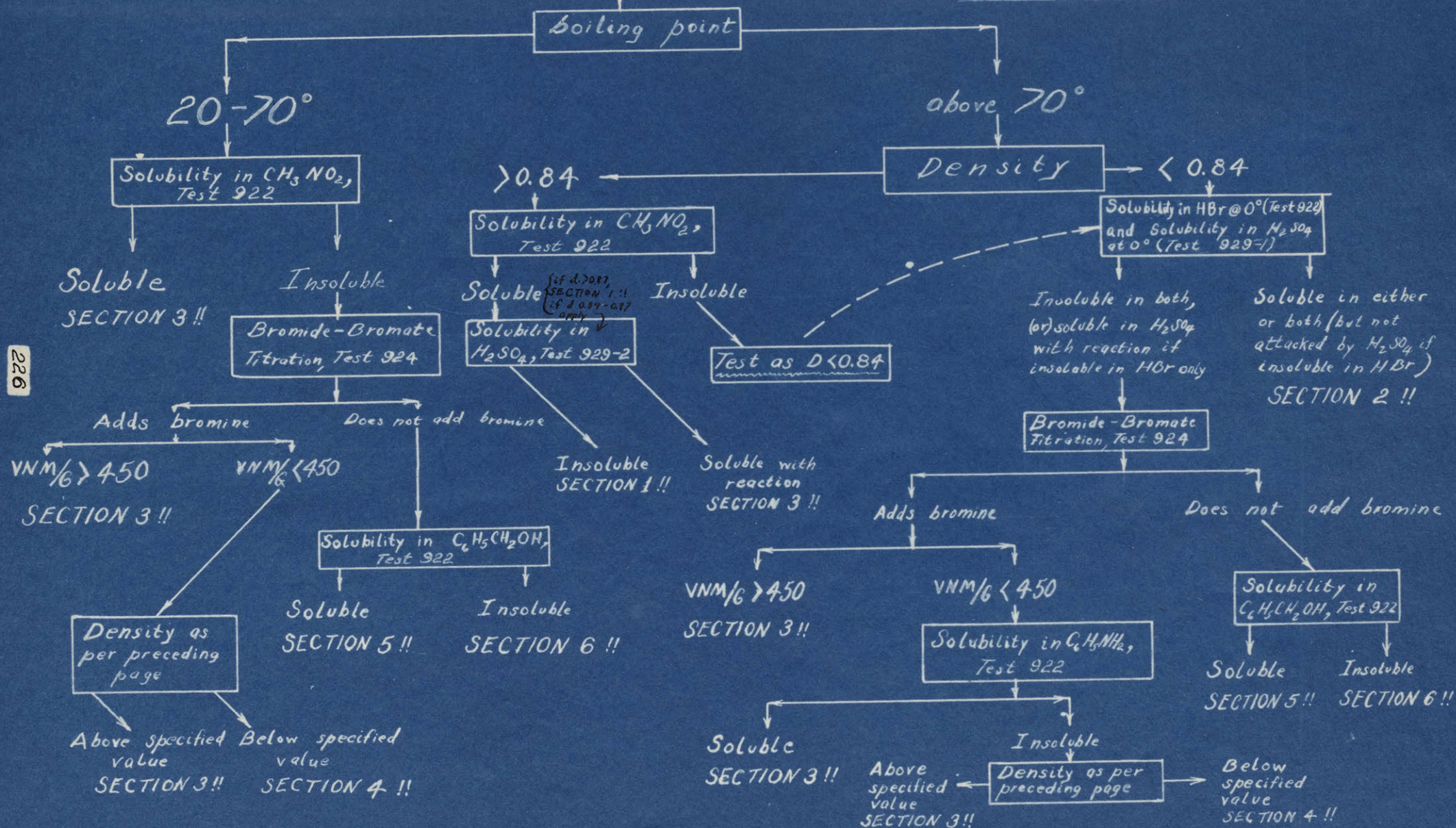
70° - 130°	0.78°
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130° - 190°	0.80°
-------------	-------

190° - 300°	0.83°
-------------	-------

Liquid @ 0°
(B.p. above 20°)

DIVISION B



N.B. → The temperatures at which to determine solubilities in various solvents are given in the immediately preceding pages!

DIVISION A

.....
Solid Hydrocarbons

Section 1

.....
Non-Aromatics

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
1	2	Abt. 1 190 (15mm)	<u>cis-Octadecene-(9)</u> , $C_{18}H_{36}$. - $D^{19} = 0.792$; $n_D^{19} = 1.448$. (1) - Cf. Div. B, Sect. 4, No.
3	Abt. 3 ³	201-2 ² (11mm) (ther- mome- ter immer- sed to 110°)	<u>Heneicosene-(9)</u> , $C_{21}H_{42}$. - $D^{20} = 0.802$. (2). - Identity doubtful. (4).
5	4 ⁵ *	274 ⁷	<u>Hexadecene-(1)</u> * "Cetene", $C_{16}H_{32}$. - $D^{15} = 0.784$. (5). - $n_D^{19} = 1.442$. (6). - Br g. dibromide. (5,8). - B.b. no. = 71* (Test 925). - Hg deriv., M.P. = 95-6°* (Test 927).
7	4	234	<u>Dicyclohexyl</u> , $C_{12}H_{22}$. - Cf. Div. B, Sect. 5, No. 277.
9	5.5 ¹⁰⁷ *	252.5 ¹⁰³ *	<u>Tetradecane</u> * $C_{14}H_{30}$. - $D_4^{20} = 0.765$. (103, 107).
11	6.5	80.8	<u>Cyclohexane</u> * C_6H_{12} . - Cf. Div. B, Sect. 5, No. 23.
13	6.5 ⁸	134 ⁸ (15mm)	<u>Tetradecine-(2)</u> , $C_{14}H_{26}$. - $D^{15} = 0.800$. (8). - Does not g. Test 906. (9) 4
15	9 ¹⁰²	304-6 ¹⁰²	<u>1,4-Dicyclohexyl-butane</u> , $C_{16}H_{30}$. - Cf. Div. B, Sect. 5, No. 337.
17	10 ¹⁰³	271 ¹⁰³	<u>Pentadecane</u> , $C_{15}H_{32}$. - $D^{20} = 0.769$. (103).
18	11	134-5	<u>2,5-Dimethyl-hexadiene-(2,4)</u> , C_8H_{14} . - Cf. Div. B, Sect. 3, No. 267.
19	14.2	149.6 -50.6	<u>Cyclooctane</u> , C_8H_{16} . - Cf. Div. B, Sect. 5, No. 143.
21	14.5	133.5 -4.5	<u>2,5-Dimethyl-hexadiene-(2,4)</u> , C_8H_{14} . - Cf. Div. B, Sect. 3, No. 281.

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
23	15 ^{9*}	155 ⁹ (15mm)	Hexadecene-(1), [*] <u>C₁₆H₃₀</u> . - $D^{20} = 0.797$. (9). - G. test 906. (105). - Hg salt, Test 926, M.P.= 95-6°. - B.b. no.=31 (Test 925).
25	15-6	143	<u>2,2-Dimethyl-bicyclo-[1,2,2]-heptane, Camphen- ilane, C₉H₁₆</u> . - Cf. Div. B, Sect. 5, No. 113.
27	17-7.5 ¹⁰⁸	143.5 ¹⁰⁸	<u>1-Methyl-3-isopropyl-cyclo-pentane, "β-Apo- fenchane", β-Fenchocamphorane", C₉H₁₄</u> . - Cf. Div. B, Sect. 5, No. 115.
29	17-8 ¹⁰	175 ¹⁰ (1mm)	<u>cis+trans-TrikoseneO(11), C₂₃H₄₆</u> . - Mixt. of both. (10). - Oxid. g. undecenic and lauric acs. (10). - Dibromide, M.P.=35.5° (Corr.) (10).
31	18 ⁵	179 ⁵ (15mm)	<u>Octadecene-(1), C₁₈H₃₆</u> . - $D^{18} = 0.791$. (5).
33	20 ⁹	160 ⁹ (15mm)	<u>Hexadecene-(2), C₁₆H₃₀</u> . - D^{20} (Liquid):0.804. (9). - Na at 200° g. hexadecene-(1), (9) No. 23. - Does not g. Test 906. (9).
35	11+12 20	287.5 ¹³	<u>Hexadecane, "Cetane", C₁₆H₃₄</u> . - Shining, mother-of-pearl-like plates. (14). - $D^{25} = 0.771$. (13).
37	20.3 ¹⁵	Abt. ¹⁵ 175 ¹⁵ (25mm)	<u>Eikosadiene-(1,19), C₂₀H₃₈</u> . - Adds 4 atoms of Br. (15).
39	22.5 ¹⁵		<u>Tetratriakontadiene-(9,25), C₃₄H₆₆</u> .
41	15,16 22.5	303 ¹⁵	<u>Heptadecane, C₁₇H₃₆</u> . - $D^{22.5} = 0.777$. (15,16). - $n_D = 1.441$. (16).
43	26 ⁹	180 ⁹ (15mm)	<u>Octadecene-(1), C₁₈H₃₄</u> . - $D^{26} = 0.798$. (9). - G. test 906. (9).
45	30 ¹⁷	305-7 ¹⁷	<u>Octadecane, C₁₈H₃₈</u> . - $D^{28} = 0.777$ (liquid). (13).
47	30 ⁹	184 ⁹ (15mm)	<u>Octadecene-(2), C₁₈H₃₄</u> . - Leaflets. (9). - $D^{30} =$ (liquid) = 0.802. (9). - Does not g. Test 906. (9).

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
49	32 ¹⁸	330 ¹⁸	<u>Nonadecane, C₁₉H₄₀.</u> - $D_4^{32} = 0.777$ (liquid). (18,19).
51	34.2 ¹⁰		<u>cis-(?) - Heptakosene-(13), C₂₇H₅₄.</u> - Dibromide, xtaln., M.P.=38.5-9°. (10). - Oxid. g. mixt. of tridecanoic and tetradecanoic acids. (10).
53	35.5-6 ²⁰	136-7 ²⁰	<u>7,7-Dimethyl-bicyclo-[1,2,2]-heptene-(2), "Apobornylene", C₉H₁₄.</u> - KMnO ₄ g. cis-apo-camphoric ac. (20). - Ordinary apobornylene is a mixture of No. 53 + No. 57. (26).
55	38	205 ²¹ (15mm) 148 ¹² (0.6 mm.) 121 ¹⁸ (0mm)	<u>Eikosane, C₂₀H₄₂.</u> - $D_4^{37} = 0.778$. (liquid). (21).
57	42.5-3 ²²	138-9 ^{22,23}	<u>3,6-Endodimethylmethylene-bicyclo-[0,1,3]-hexane, "Apocyclene", C₉H₁₄.</u> - Readily volatile, xtaln. cpd. (26). - Sweet, irritating odor. (26). - Cf. No. 53. - $D_4^{45} = 0.869$. (23). - $n_D^{45} = 1.449$. (23). - Very stable to KMnO ₄ . (22). - Boil. w. AcOH+50% H ₂ SO ₄ for 3 hrs. g. an acetate, B.P. _s =81-2°, $d_4^{20} = 0.997$, $n_D^{20} = 1.462$, (22,26) wh. by sapon. + oxid. of resulting alc. g. apofencho-camphoric ac., C ₉ H ₁₄ O ₄ , monoclinic prisms, M.P.=144-5°, + dl-β-fenchocamphorone whose semicarbazone, needle-like prisms, M.P.=200-1°(?). (22). - HCl salt = B.P.=76-7°, M.P.=44-6°, w. Ca(OH) ₂ g. a terpene alc., B.P.=196-8°, M.P.=85.5-6.5° wh. on oxid. w. alk. KMnO ₄ + dehydration w. acetyl chloride g. apocamphoric anhydride, M.P.=174.5-5°. (24). - Unattacked by O ₃ . (26).
59	40.4 ²¹	215 ²¹ (15mm) 129 ¹⁸ (0 mm)	<u>Heneikosane, C₂₁H₄₄.</u> - $D_4^{40} = 0.778$ (liquid). (21)

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
61	41-3 ²⁵	170.5- 1.0 ²⁵	<u>α-Methyl-camphene, $C_{11}H_{18}$.</u>
63	44-5 ²⁷	152-5 (12mm) ²⁸	<u>Hexadecadiene-(1,15), $C_{16}H_{32}$.</u> - G. Test 906. (27,28).
65	45.2-5.4 ¹⁰	245-6 (1mm) ¹⁰	<u>cis(?) - Hentriakontene-(15), $C_{31}H_{62}$.</u> - Dibromide M.P.=43-3.5° (Corr.) (10) - Br reacts only slowly. (10).
67	47 ²¹	224.5 (15mm) ²¹	<u>Dokosane, $C_{22}H_{46}$.</u> - $D_4^{44} = 0.778$ (liquid). (21).
69	47.7 ¹³	234 (15mm) ¹³	<u>Trikosane, $C_{23}H_{48}$.</u> - $D_4^{48} = 0.779$ (liquid). (13) - B.p. 142.5° (Omm). (13).
71	48-8.5 ²⁹	322-9 ²⁹	<u>Tricyclohexyl-methane, $C_{19}H_{34}$.</u> - $D_0^{50} = 0.926$;, $n_D^{50} = 1.487$. (29).
73	51-2 ³⁴	160-1 ³⁵	<u>2,2-Dimethyl-3-methylene-bicyclo-[1,2,2]- heptane, "Camphene", d-Camphene was earlier known as "Austracamphene", l-Camphene was earlier known as "Terecamphene", $C_{10}H_{16}$.</u> - L- form occurs in American turpentine oil, (30), oil of citronella, (31), oil of bergamot. (32). D-form occurs in cypress oil, fennel oil. (33). $D_4^{54} = 0.842$, $n_D^{54} = 1.455$. (34). - D-form shows $[\alpha]_D^{17} = +103.89^\circ$ (in ether, $p=9.67$). (35). - L- form shows $[\alpha]_D^{50} = -89.29^\circ$. (36). - D-l-form g. M.P.=50°, (31). - $d_4^{73} = 0.822$. (37). - Slight htg. w. organic acs. + $ZnCl_2$ g. esters of isoborneol. (38). - Soln. of 22 g. hy. in 50 cc. $HCCl_3$ + addn. (w. thorough cooling), slowly, to 65 g. HNO_3 anhydride in 250 cc. $HCCl_3$ g. $C_9H_{14}(ONO_2)$ (CO_2H), prisms fm. aq.-alc., M.P.=140-1°, colors yellow by htg. above M.P., completely dec. at 165-70°. (39). - Shak. pet. ether soln. of hy. w. conc. aq. $HgAc_2$ g. $C_{14}H_{22}O_5Hg$, white plates, M.P.=188-9°. (40). - N_2O_3 + l-camphene g. l-nitro-camphene, M.P.=84-5°, $[\alpha]_D^{20} = -146.4^\circ$ (41). - 100 g. hy. in light pet. agitated w. $HClO$ (1 l. per 5 g.) till no further absorp- tion, repeatedly distilled w. steam g. chlor- hydrin, M.P.=93°, whose p-nitro-benzoate fm. MeOH, M.P.=111°, + wh. w. CrO_3 g. chloro- ketone, $C_9H_{15}Cl:CO$, M.P.=132° (fm. MeOH) wh.

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
			latter g. oxime, M.P.=142-3°, +semicarbazone, M.P.=220-1°. (42). - Dry HCl in MeOH g. hydrochloride (fm. l-form), xtals. fm. C ₆ H ₆ , M.P.=149-50°. (38). - Soln. in 4 times its wgt. of glac. AcOH, addn. of 30% H ₂ O ₂ +htg. at 60° g. chiefly camphenanic ac., C ₉ H ₁₅ CO ₂ H (fm. H ₂ O+light pet.), M.P.=95°. (43).
75	54 ¹²	237-40 ¹² (15mm)	<u>Tetrakosane</u> , C ₂₄ H ₅₀ . - D ₄ ⁵¹ =0.779 (liquid). (21). n _D ⁶⁵ = 1.430. (44).
77	54-4.5 ^{45,46}		<u>Pentakosane</u> , C ₂₅ H ₅₂ .
79	54-6 ⁴⁷		<u>Eicosadiene-(1,19)</u> , C ₂₀ H ₃₄ . - G. Test 906. (47).
81	55 ¹⁰⁸	300 ¹⁰⁸	<u>Cetyl ether</u> , C ₃₂ H ₆₆ O.
83	55-6 ⁴⁸		<u>Pinakonene</u> , C ₂₀ H ₃₀ . - D ₄ ⁶¹ =0.930. (48). - n _D ⁶¹ = 1.502. (48). - Difficultly sol. in MeOH+EtOH, easily in C ₆ H ₆ , pet. ether, + Me ₂ CO. (48). - Boil. w. CrO ₃ mixt. g. cpd., C ₂₀ H ₃₀ O, xtals. fm. pet. ether, M.P.=abt. 70°. (48). - Nitrochloride, M.P. = 150° dec. (48)
85	56 ⁵¹		<u>Isopentacosane</u> , C ₂₅ H ₅₂ .
87	56.6 ^{44,49,50}	262 ⁴⁴ (15mm)	<u>Hexakosane</u> , C ₂₆ H ₅₄ . - Lamellas. (49). - n _D ⁶⁵ = 1.433. (44).
89	57-8 ⁵²		<u>Cerotene</u> , C ₂₆ H ₅₂ . - From Chinese wax. (52). - Paraffin-like mass. (52).
91	58 ¹⁰⁸		<u>Dinonyl ketone</u> , C ₁₉ H ₃₈ O. - Cf. Mulliken, Ident. etc., Vol. I, VII, A, pg. 137.
93	59 ⁵³		<u>β-Cholesterylene</u> , C ₂₇ H ₄₈ . - [α] _D = -76.68° in PhMe. (53).

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
95	21,44,45 48,49,54 59.5	21,44 270 (15mm)	Heptakosane, $C_{27}H_{58}$. - In beeswax. (45). - In Carnuba wax. (54). - $D_4^{60} = 0.780$ (liquid). (21). $n_D^{65} = 1.435$. (44).
97	55 60-1		Cyclopentadecane, $C_{15}H_{30}$. - Unchanged by htg. w HI at 250° for 7 hrs. (55). - $D_4^{62} = 0.836$; $n_D^{62} = 1.459$. (55).
99	12,44 61-2	12 224 (1.1mm)	Octakosane, $C_{28}H_{58}$. - $n_D^{65} = 1.435$. (44).
101	56,58 62-3	60 218 (0.5mm)	Melene, $C_{30}H_{60}$. - In beeswax. - Dec. by distn. at 15 mm. (60). - $D^{65} = 0.791$, (56,60) $n_D^{90} = 1.423$. (56). - Not attacked by cold $KMnO_4$. (59). Attacked by Br in boil. CCl_4 . (59). - The nature of this substance is somewhat in doubt; according to Funcke, Arch. Pharm. 259, 93(1921) it is a mixture of satd.+unsatd. hys. w. abt. 30 C's; according to Marcusson + Bottger, Ber. 57, 633; Chem. Zentr. 1924 I, 2579, it is a paraffin.
103	108 63-4	108 440	Anthemene, $C_{18}H_{38}$. - $D^{15} = 0.942$. (108).
105	61 63-4.5	61 164	2,2,3-Trimethyl-bicyclo-[1,2,2]-heptane, d 1-Iso-camphane, $C_{10}H_{18}$. - $D_4^{67} = 0.828$, $n_D^{67} = 1.442$. (61).
107	12,45,46 63.5	12 286 (Abt. 15mm.)	Nonakosane, $C_{29}H_{60}$. - $n_D^{65} = 1.436$. (12). - In beeswax. (46).
109	62,63 64	62,63 129-30	Hexadiene-(2,4), C_8H_{16} . - H_2SO_4 g. hexine-(2)-one-(5)+other products. (63). - Br in CS_2 or $HCCl_3$ g. a tetrabromide. (63).
111	55 64-5		Cycloheptadecane, $C_{17}H_{34}$. - Unchanged by htg. w. HI at 250° for 7 hrs. (55). - $D_4^{74} = 0.824$, $n_D^{74} = 1.454$. (55).

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
113	⁸⁹ 66-7	⁸⁹ Abt. 192- 6(?) (12 m.m.)	<u>1,3-Dicyclohexyl-cyclohexane, C₁₈H₃₂.</u> - Long needles. (89). - $D = \text{abt. } 0.934(?)$. (89). - (B.P. + d. are for an isomeric liquid 1,3-dicyclo-hexyl-cyclohexane.) (89).
115	¹⁰⁶ 66-7	¹⁰⁶ 146-8	<u>1,7-Dimethyl-bicyclo-[1,2,2]-heptane(?), "Sautenane", C₉H₁₆.</u>
117	⁵⁰ 66.1	¹² 235 (1 mm.)	<u>Triakontane, C₃₀H₆₂.</u> - Occurs on apple skins. (64). - Brilliant scales. (49).
119	¹⁰ 66.5		<u>cis?-Pentatriakontene-(17), C₃₅H₇₀.</u> - Dichloride by Cl ₂ in CCl ₄ , M.P.= 47°(10). - Dibromide, M.P.= 63-3.5°. (Corr.). (10).
121	^{65,66} 67.5-8	[87 ^{65,66} 152-3	<u>1,2,2-Trimethyl-3,6-endomethylene-bicyclo-[0,1,5]-hexene, "Tricyclene", "Cyclene", C₁₀H₁₆.</u> - $D_{20} = 0.827$. (87). - $n_{D}^{20} = 1.430$. (87). - Un-attacked by long boil. w. KMnO ₄ . (88). - 57 g. + 285 cc. HNO ₃ (d=1.075) htd. in 12 tubes for 3 hrs. at 125-30° g. 18 g. unchanged hy. + 20 g. sec.-α-nitro-camphene, B.P. ₁₂ 119-9.5°, $d_4^{20} = 1.069$, $n_{20} = 1.494$, sol. in alk. w. yellow color, instantly decolorizes Br in CHCl ₃ +KMnO ₄ , wh. w. HNO ₂ g. the pseudonitrole, M.P.=99°, dec., wh. by red. w. Zn+AcOH g. α-amino-camphane, B.P. ₇₅₀ = 197-8°, $d_4^{20} = 0.937$, $n_{20} = 1.494$; Ac. layer fm. the nitration ² contains iso-camphoric ac., M.P.=166-7°. (104).
123	⁷¹ 68-9		<u>Mesembrene, C₂₈H₅₆.</u> - In wax fm. Mesembrianthenum expansum+M-tortuosum. (71). - Colorless plates fm. ether. (71) ₉ - Decolorizes KMnO ₄ . (71). - Ac ₂ O+NaOAc g. C ₃₀ H ₅₀ O ₂ , M.P.=66-7°. (71)
125	^{45,46,49} 68.4-9.0	¹⁸ 302 (15mm)	<u>Hentriakontane, C₃₁H₆₄.</u> - $D_4^{68} = 0.781$ (liquid). (18).
127	¹⁰⁸ 69		<u>Laurone[*], C₂₃H₄₆O.</u>
129	⁷² 69		<u>Neocholestene, C₂₇H₄₄.</u> - Dibromide, M.P.=125°. (72).

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
131	69 ⁷³	400 ⁷³	<u>Bryonane</u> , <u>C₂₀H₄₂</u> . - In the leaves of <i>Bryonia dioica</i> . (73).
133	71 ^{74,75}	310 ⁷⁴ (15mm)	<u>Dotriakontane</u> , " <u>Dicetyl</u> ", <u>C₃₂H₆₄</u> . - $D^{79}=0.775$. (37). - $n_D^{72}=1.433$. (76). - Occurs in candelilla wax to 76%. (75).
135	71-3 ⁷⁷	172-5 ⁷⁷	<u>1,6-Dimethyl-3,6-endodimethylmethylene-bicyclo-[0,1,3]-hexane</u> , " <u>α-peri-Cyclohomocamphane</u> ", <u>C₁₁H₁₈</u> .
137	73-4 ⁷⁸		<u>Decahydroanthracene</u> , <u>C₁₄H₂₀</u> .
139	73.2 ⁴⁹		<u>Tetratriakontane</u> , <u>C₃₄H₇₀</u> .
141	74.7 ²¹	331 ²¹ (15mm)	<u>Pentatriakontane</u> , <u>C₃₅H₇₂</u> . - $D^{75}=0.782$. (21). 4
143	49;79;80 76-6.5		<u>Hexatriakontane</u> , <u>C₃₆H₇₄</u> . - Brilliant lamellas. (49). - Cannot be distilled without dec. (12).
145	76.3 ¹⁰⁸		<u>Myristone</u> [*] , <u>C₂₇H₅₄O</u> . - Mulliken, Ident. etc., Vol. I, VII, A, pg. 138.
147	77 ⁸¹	192.5	<u>Tetrahydro-dicyclopentadiene</u> , <u>C₁₀H₁₆</u> . - Depolymerized only by passing the vapors thru a red-hot tube. (81). - $D^{79}=0.913$ (83), $n_D^{79}=1.473$. (84). - Unattacked by conc. H ₂ SO ₄ + a little pyro-sulfuric ac., slightly above its M.P., g. isomer wh. m. p.=abt. 9° (83), B.P.=191°, $d^{21}=0.949$, $n_D^{21}=1.494$. (84).
149	77 ⁵³		<u>α-Cholesterylene</u> , <u>C₂₇H₄₈</u> . - $[\alpha]_D = -109.30^\circ$ in PhMe. (53). - 12 g. finely powdered, stirred vigorously 1 hr. w. 80 cc. AcOH+20 cc. red fum. HNO ₃ , $d=1.52$, + let stand in ice g. 75% of nitrocholestene, M.P.=117-8°. (32).
151	79 ⁵³		<u>Cholestane</u> , <u>C₂₇H₄₄</u> . - $[\alpha]_D=24.59$ in CHCl ₃ . (53).
153	82.8 ¹⁰⁸		<u>Palmitone</u> [*] , <u>C₃₁H₆₂O</u> . - Cf. Mulliken, Ident. etc., Vol. I, VII, A, pg. 138.

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
155	85,86 85-7	86 162	<u>2,2,3-Trimethyl-bicyclo-[1,2,2]-heptane?</u> , " <u>Iso-hydrocamphene</u> ", " <u>Isocamphane</u> ", <u>C₁₀H₁₈</u> .
157	86 ⁸⁷		<u>Diphellandrene</u> , <u>C₂₀H₃₂</u> . - From d-β-phellandrene by long boil. or 20 hrs. htg. at 140-50° in sealed tube. (87). - Amorphous. (87). - Dec. at abt. 300°. (87). - L-rotating. (87).
159	86 ⁸⁸	88 148	<u>Apocamphane</u> , " <u>1-Fencho-camphorane</u> ", <u>C₉H₁₆</u> .
161	87.8 ¹⁰⁸		<u>Stearone</u> , <u>C₃₅H₇₀O</u> . - Cf. Mulliken, Ident. etc., Vol. I, VII, A, pg. 138.
163	88 ^{89, 90}	89,90 270	<u>Tetradecahydroanthracene</u> , " <u>Anthracene per-hydride</u> ", <u>C₁₄H₂₄</u> . - Plates or leaves fm. EtOH. (89). - Unattacked by HNO ₃ -H ₂ SO ₄ . (90). - Scarcely attacked by Br in CS ₂ . (89) - Completely oxid. by CrO ₃ . (89).
165	94 ^{91, 101}	91 321- 3.6	<u>Di-(1,7,7-trimethyl-bicyclo-[1,2,2]-heptyl-2)</u> ?, " <u>Dicamphanyl-2,2'</u> ", <u>C₂₀H₃₄</u> . - D ₁₅ :1.001. (92). - Hardly attacked by fum. H ₂ SO ₄ or conc. HNO ₃ . (92). - Unattacked by Br + only slightly by CrO ₃ mixt. (91).
167	95 ⁴⁹		<u>Tetrapentacontane</u> , " <u>Diceryl</u> ", <u>C₅₄H₁₁₀</u> .
169	98 ⁹³		<u>Pinakonane</u> , <u>C₂₀H₃₂</u> . - Br in CCl ₄ g. dibrom-pinakonane, M.P.=157°. (93).
171	Indist- inctly below 100° ⁹⁶		<u>"Tetraterpene"</u> , <u>C₄₀H₆₄</u> . - D ₀ =0.977. (96). - Does not vaporize at 350°. (96). - Conchoidal fracture. (96). - Completely insol. in abs. alc.; sol. in ether, CS ₂ , C ₆ H ₆ , ligroin, turpentine oil. (96). - Soln. in ether-alc. is d-rotatory. (96). - Quickly oxid. in air. (96). - Prepared by shak. 1-turpentine oil w. SbCl ₃ keeping the temp. below 50°. (96).
173	94,95 100-1	94,95 170- 0.5	<u>2,2,6-Trimethyl-3-methylene-bicyclo-[1,2,2]-heptane</u> , " <u>β-Methyl-camphene</u> ", <u>C₁₁H₁₈</u> . - Treat-ment w. oxides of N in pet. ether + subsequent warming w. alc. KOH g. β-methyl-camphenilone,

DIVISION A, SECTION I

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
			M.P.=141-2°, (94,95), whose semicarbazone, M.P. = 231-2° w. dec., whose oxime, M.P.=172°, whose hydrazone, M.P.=85-7°, whose azine, M.P.=163-4° (95) + wh. (the ketone) w. NaNH ₂ g. β-methyl-camphenylamide, C ₁₀ H ₁₉ ON, M.P.=124-5°. (94).
175	49,97 100.5		<u>Dohexacontane, "Dimyricyl", C₆₂H₁₂₆.</u>
177	97 102		<u>Tetrahexacontane, "Dilacceryl", C₆₄H₁₃₀.</u>
179	98 102-3		<u>Colophonene, C₄₀H₆₄.</u> - Fm. pinene + I + AlCl ₃ , beside other products. (98). - Amber colored. (98). - Brittle. (98). - Rosin fracture. (98). Insol. in alkalis. (98). - Stable. - (98). - Slowly oxid. by HNO ₃ . (98). - Unattacked by free Cl. (98). - Nascent Cl (fm. HCl+KMnO ₄) after several days g. the tetrachloride, (C ₁₀ H ₁₅ Cl) ₄ , M.P. = 119-21°. - SO ₂ Cl ₂ in CHCl ₃ g. white, flocculent powder, M.P. = 99-102°, (C ₁₀ H ₁₅ Cl) ₄ . (98).
181	99 103-4	99 106-7	<u>2,2,3,3,-Tetramethyl-butane, C₈H₁₈.</u>
183.	100 105.5- 6	100 185-6 (21mm)	<u>Di-(1-Methyl-4-isopropyl-cyclohexyl-5), "Dimenthyl", C₂₀H₃₈.</u> - M. w. part. sublimation. (100) - Noticeably volatile at 100°. (100). - Easily sol. in ether, C ₆ H ₆ , sol. in hot alc. (100). - [α] _D ¹⁸ = -51°18' in 19.4% C ₆ H ₆ soln. (100).

DIVISION A

.....
Solid Hydrocarbons

Section 2

.....
Chiefly Aromatics

Division A, Section 2.

No.	Melting Point (C°)	Boil. Pt. (C°)	Hydrocarbon
2	²⁵⁶ above 0	²⁵⁶ 187.5- 88 (12mm)	<u>cis-1,4-Diphenyl-butene-(1)-ine-(3), C₁₆H₁₂.</u> -- Yellow oil wh. solidifies in freezing mixture + melts above 0° (²⁵⁶).--Light changes to trans form, m.p.: 96.5-7°, No. 404 (²⁵⁶).-- Br in CS ₂ g. 3 tetrabromides, m.p.: 197°, 157-8°, + 135-6° (²⁵⁶).
4	1	172.5	<u>Cineole, C₁₀H₁₈O.</u> -- Cf. Div. B, Sect. 1, No. 82.
6	4.5	abt. 208 w. polym	<u>1-Phenyl-butadiene-(1,3), C₁₀H₁₀.</u> -- Cf. Div. B, Sect. 1, No. 318.
8	5-7	286 (716mm)	<u>3,3'-Dimethyl-diphenyl, C₁₄H₁₄.</u> -- Cf. Div. B, Sect. 1, No. 778.
10	5.5	80	<u>Benzene, C₆H₆.</u> *-- Cf. Div. B, Sect. 1, No. 2.
12	7	240	<u>Phenyl-cyclohexane, C₁₂H₁₈.</u> *-- Cf. Div. B, Sect. 1, No. 498
14	8-9	277	<u>1,1-Diphenyl-ethene, C₁₄H₁₂.</u> -- Solidifies in freezing mixture + then melts at 8-9° (634).-- Cf. Div. B, Sect. 1, No. 704.
16	8-9	238.5- 9 (15 mm)	<u>1-Methyl-2-hexadecyl-benzene, C₂₃H₄₀.</u> -- Cf. Div. B, Sect. 1, No. 1010.
18	²³ 11	²³ 233	<u>Safrole, C₁₀H₁₀O₂.</u> *-- Odor like sassafras (23).-- Cf. Div. B, Sect. 1, No. 474.
20	11-2	236.5- 7 (15 mm)	<u>1-Methyl-3-hexadecyl-benzene, C₂₃H₄₀.</u> -- Cf. Div. B, Sect. 1, No. 1008.
22	11-2	281-3	<u>1-Methyl-4-octyl-benzene, C₁₅H₂₄.</u> -- Cf. Div. B, Sect. 1, No. 728.
24	13	250	<u>1,2,4,5-Tetraethyl-benzene, C₁₄H₂₂.</u> -- Cf. Div. B, Sect. 1, No. 556.
26	14	190-2 (13mm)	<u>1,1-Diphenyl-heptane, C₁₉H₂₄.</u> -- Cf. Div. B, Sect. 1, No. 922.
28	16	138	<u>1,4-Dimethyl-benzene, C₈H₁₀.</u> -- Cf. Div. B, Sect. 1, No. 16.

DIVISION A, SECTION 2.

No.	Melt- ing. Point (C°)	Boil- ing Point (C°)	Hydrocarbon
30	16-17		<u>C₁₆H₂₀</u> fm. phenyl-fenchol. - Cf. Div. B, Sect. 1, No. 788.
32	16-17 ¹	234 ¹ (12mm)	<u>1,10-Diphenyl-decane, C₂₂H₃₀</u> . - HNO ₃ (d.=1.52)+ H ₂ SO ₄ on H ₂ O bath g. yellowish-white needles of 1,10-bis-2,4-dinitrophenyl decane, M.P.=63°. (622). (623).
34	622,623 16.7	623 295	<u>1,2,3,4,5,6,7,8-Octahydrophenanthrene</u> , "Octanthrene", <u>C₁₄H₁₈</u> . - D ²⁰ =1.026. (622,623). - S at 180-220° g. H ₂ SO ₄ + phenanthrene (622), No. 428. - S at 220° g. tetranthrene, M.P.=33-4°, (622), No. 80. - Sulfonates. (623).
36	17.8 ²	259	<u>2,2'-Dimethyl-diphenyl, C₁₄H₁₄</u> . - KMnO ₄ g. diphenic ac. (2). - Xtals. fm. EtOH. (2).
38	19.5 ³⁰	68-70 ³⁰ (12mm.)	<u>β-Dicyclopentadiene, C₁₀H₁₂</u> . - Htd. at 170-80° g. part. depolym. to cyclopentadiene (30), cf. Div. B, Sect. 3, No. 19. - H g. dihydro cpd., B.P. ₁₂ =103-5°, M.P.=57°, (30), No. 186. - Cf. No. 78.
39	21.5-2.0 ²³	267.5-85 ²³	<u>o-Tolyl phenyl ether, C₁₃H₁₂O</u> . - Cf. Div. B, Sect. 1, No. 656.
40	21.6	233c.	<u>Anethole, C₁₀H₁₂O</u> . - Odor +taste of anise oil in wh. it occurs. (23). - D ²² =0.9855. (23). - n _D ¹⁸ =1.5615. (23). - Shak. w. little conc. H ₂ SO ₄ g. anisoin, M.P.=140-5°, (23), cf. No. 604. - Htd. w. solid KOH at 200-30° g. p-oxybenzoic ac. + anol. (23).
42	23 ³	120-1 ³ (20 mm.)	<u>1-Phenyl-cyclopentene-(1), C₁₁H₁₂</u> . - D ²⁵ _D =0.962, n ²⁵ _D =1.573. (3). - Picrate, M.P.=64.5°. (3).
44	23 ^{4,5}	168-70 ^{4,5}	<u>1-Methyl-4-ethinyl-benzene, C₉H₈</u> . - Odor of anise + fennel. (5). - D ¹⁸ =0.912. (5). - G. yellow-green Cu salt. (4).
46	24.5-4.8 ⁶	211-2	<u>1,4-Dihydronaphthalene, C₁₀H₁₀</u> . - Cf. Div. B, Sect. 1, No. 338.
48	25 ⁷	120-2 ⁷ (10mm)	<u>1-Phenyl-cyclobutene-(2?), C₁₀H₁₀</u> . - Plates fm. ether. (7). - Does not decolorize Br in CHCl ₃ . (8).
50	26-7 ⁹	261-2 ⁹	<u>Diphenyl-methane, "Ditan", C₁₃H₁₂</u> . - D ²⁶ =1.001. (10). - n _D ¹⁶ =1.570. (11). - Odor resembling orange peel. (614). - Ptcharcoal at 300° g.

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
			fluorene. (12). - 20 g. hy. slowly added w. cooling to 100-120 g. HNO ₃ (d=1.53), htd. slightly till in soln., after short stg. poured into 500 cc. H ₂ O, ppt. washed w. ether + diss. in little hot C ₆ H ₆ (tetranitro cpd. left undiss.) g. 4,4'-dinitro-ditan fm. C ₆ H ₆ soln., M.P.=183°. (13). - CrO ₃ mixt. g. benzophenone. (9). - Cpd.: 1 Hy..2 SbCl ₃ , M.P.=100°; cpd.=1 Hy..2 Sb Br ₃ , M.P.=90°; each system also shows 2 eutectic pts. (17). - Cf. color reaction, Test 904. (23). - Benzal-chloride+H ₂ SO ₄ g. brick-red color. (e14) SbCl ₅ in CCl ₄ g. green color. (e13).
52	14 27	14 250 (15 mm.)	<u>n-Hexadecyl-benzene, C₂₂H₃₄</u> . - D ₂₇ ²⁷ =0.857. (15). Nitration g. <u>eso</u> nitro cpd., xtaln. powder, M.P.=35-6°. (18). - 20 g. hy., 20 g. I, 4 g. HI (in 15 cc. H ₂ O), + 75 cc. AcOH boiled 12 hrs. g. colorless plates (fm. pet. ether) of 4-iodo-1-cetyl-benzene, M.P.= 38°, B.P. ₂₀ =260-5°. (16).
54	Around 27 "Summer temp."		<u>Dodecahydro-1,3,5-triphenyl-benzene, C₂₄H₃₀</u> . - Oil wh. after long time solidifies. (18). - CrO ₃ in AcOH g. benzoic ac. (18).
56	19 27	19 115 (18 mm.)	<u>2-Methyl-4-phenyl-butadiene-(1,3), C₁₁H₁₂</u> . - Polymerizes very easily. (19). - Na+EtOH g. 2-methyl-4-phenyl-butene-(2), (20), Div. B, Sect. 1, No. 286.
58	21 27	21 266-7	<u>1,1-Diphenyl-butane, C₁₆H₁₈</u> . - D ₄ ¹⁶ =1.006. (21). - n _D ¹⁶ =1.577. (21).
60	22 27	22 286	<u>1-Phenyl-2-[p-tolyl]-ethane, C₁₅H₁₆</u> .
62	15 27.5	15 239.5- 40 (15 mm)	<u>1-Methyl-4-hexadecyl-benzene, C₂₃H₄₀</u> . - D ₄ ^{27.5} =0.850. (15). - Dil. HNO ₃ oxid. g. p-toluic ac. (15).
64	23 28	23 252-3	<u>Diphenyl ether, C₁₂H₁₀O</u> . - Odor like geranium. (23). - Unchanged by CrO ₃ in AcOH, ignition w. Zn dust, or HI at 200°. (23). - Xs. Br in CS ₂ ctg. little I g. quantitative yield of tetrabrom cpd., colorless xtals., turning brown on exposure to light, M.P.=83-4°, B.P. ₂₅ =280-90°.

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
66	28 ²⁵	289- 91 ²⁶	B.P.=410-25°. (24). <u>Di-[p-tolyl]-methane, C₁₅H₁₆.</u> - G. no picrate. (26). - Br at ord. temp. g. dibrom cpd., needles fm. EtOH, M.P.=115°. (26). - Soln. in cold fum. HNO ₃ g. dinitro cpd., M.P.=164°. (26).
68	28.5-9 ²⁷	260 ²⁷ (23mm)	<u>1,3,5-Trimethyl-2-[hexadecene-(2¹)-yl]-benzene, C₂₅H₄₂.</u>
70	29 ²⁸	282-3 ²⁸	<u>2,2-Diphenyl-propane, C₁₅H₁₆.</u> - D ₂₀ ²⁵ (super-cooled liquid):0.996; n _D ²⁰ =1.570. (28).
72	30 ²⁵²	315-6 ²⁵²	<u>Dihydro-1-methyl-anthracene, C₁₅H₁₄.</u>
74	30 ²³	294 ²³	<u>Apiole, C₁₂H₁₄O₄.</u> - Sol. in H ₂ SO ₄ w. blood-red color. (23). - Volatile w. steam. (23).
76	31 ²⁹	168 ²⁹ (18 mm.)	<u>2-Phenyl-5-methyl-coumarone, C₁₅H₁₂O.</u> - Opaque needles. (29). - Sweet odor. (29). - Conc. H ₂ SO ₄ g. intense orange-red color wh. on warming becomes lighter. (29). - Br in CS ₂ in sunlight g. 1-brom-2-phenyl-5-methyl-coumarone, M.P.=95° wh. in conc. H ₂ SO ₄ g. pale red color. - AcOH, HNO ₃ +NaNO ₂ g. 1-nitro-2-phenyl-5-methyl-coumarone, yellow xtals., M.P.=119-20°. (29).
78	30,31 32.9	30,32 Abt. 170 w. part depol- ym.	<u>α-Dicyclopentadiene, C₁₀H₁₂.</u> - D ₂₀ ³² =0.977. (32). - Part. depolym. at 170-80° g. cyclopentadiene, (30,32), cf. Div. B, Sect. 3, No. 19. - H g. dihydro cpd. B.P. ₁₂ =103-5°, M.P.=57° (30), No. 186. - Exp. attacked by conc. H ₂ SO ₄ . (33). - Resinified by dil. acs. (33). - Slow addn. of 10% alc. HCl to hy. + C ₅ H ₁₁ ONO (1 mol:1 mol) + equal vol. AcOH in cold g. bis-nitrochloride, M.P.=182° (32), wh. by PhNEt ₂ at 140° g. plates fm. EtOH of monomer, M.P.=160°(dec.), wh. latter w. alc. KOH g. flakes of C ₁₀ H ₁₁ ON, M.P.=205°(dec.). (34). - Hy.+C ₅ H ₁₁ ONO(1 mol=1 mol) +AcOH+50% HBr g. bis-nitroso-bromide, dec. at 157° + wh. g. tetrabromide, plates fm. C ₅ H ₁₁ OH, M.P.=211°. (35). - Pseudonitrosite (by dry nitrous gases fm. HNO ₃ +As ₂ O ₃ +ether soln. of hy.), M.P.=144-6°, dec. (36). - Cpd., by N ₂ O ₄ +ether soln. of hy., ice cold, M.P.=122°. (36). - Cf. Div. B, Sect. 3, No. 565. - Cf. Div. A, Sect. 2, No. 38.

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
80	⁶²² 33-4	⁶²² 174-8 (11mm)	<u>Tetrahydrophenanthrene, "Tetranthrene", C₁₄H₁₄.</u> - Picrate, red, M.P.=170-8°. (622).
82	³⁷ 33-4	³⁷ Abt. 180 (11mm)	<u>1 or 3(?) - Benzyl-indene, C₁₆H₁₄.</u> - Yellow prisms. (37).
84	¹⁵ 33.5	¹⁵ 249.5 50 (15mm)	<u>1,5-Dimethyl-2-hexadecyl-benzene, C₂₄H₄₂.</u> - D ^{33.5} ₄ = 0.850. (15).
86	³⁹ 35.5	³⁹ 350	<u>2-Benzyl-naphthalene, C₁₇H₁₄.</u> - D°=1.176. (39). - Dil. HNO ₃ g. phenyl β-naphthyl ketone. (39). - CrO ₃ mixt. g. benzoic ac. (39). - Picrate, golden-yellow needles fm. alc., M.P.=93°. (39).
88	¹⁴ 36	¹⁴ 249 (15mm)	<u>n-Octadecyl-benzene, C₂₄H₄₂.</u> - Silvery leaf- lets. (23).
90	⁴⁰ 36-7	¹¹ 300-3	<u>1,3,5-Trimethyl-2-benzyl-benzene, C₁₆H₁₈.</u> - CrO ₃ g. benzoyl-mesitylene. (40). - 15 g. hy. + 45 cc. ice cold HNO ₃ (d=1.5) g. trinitro cpd., yellow prisms fm. EtOH, M.P.=185°. (40).
92	⁴¹ 36		<u>2-Ethynyl-naphthalene, C₁₂H₈.</u> - H ₂ SO ₄ g. methyl β-naphthyl ketone. (41). - G. colorless Ag salt. (41).
94	²³ 37	²³ 274-5	<u>Ethyl-β-naphthyl Ether, C₁₂H₁₂O.</u> - Odor like anise. (23).
96	⁴² 37-8	^{42,44} 241-2	<u>2-Methyl-naphthalene, C₁₁H₁₀.</u> - Picrate, orange-yellow needles fm. alc., M.P.=116-7°. (42).
98	²³ 38-9	²³ 286-7	<u>Phenyl benzyl ether, C₁₃H₁₂O.</u> - Htd. at 100° w. conc. HCl g. phenol+benzyl chloride. (23).
100	¹⁵ Abt. 40	¹⁵ 258- 8.5 (15mm)	<u>1,3,5-Trimethyl-2-hexadecyl-benzene, C₂₅H₃₂.</u> - D ⁴⁰ ₄ = 0.8452. (15).
102	⁴⁵ 40		<u>C₂₀H₂₄, dimer of 2-p-tolyl-propene.</u> - By cold conc. H ₂ SO ₄ on the monomer. (45).

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Boil- ing Point (C°)	Hydrocarbon
104	41 ⁴⁶	288 ⁴⁶ (722mm)	<u>2,4,2',4'-Tetramethyl-diphenyl, C₁₆H₁₈.</u>
106	41-2 ²³	241 ²³	<u>Methyl-phenyl-furane, C₁₁H₁₀O.</u> - Needles fm. cold alc. (23). - Volatile w. steam. (23). - Long stg. changes to yellow oil. (23). - Alk. KMnO ₄ easily oxid. to benzoic ac. (23). - Br g. brown-colored leaflets, M.P.=208-10. (23)
108	43 ²³		<u>Phloroglucinol-triethyl ether, C₁₂H₁₈O₃.</u> - Vol. w. steam. (23).
110	43 ⁴⁷		<u>Tetrahydrotricyclopentadiene, C₁₅H₂₂.</u> -
112	43-4 ⁴⁸		<u>1-Methyl-3-benzylidene-indene, C₁₇H₁₄.</u> - H ₂ SO ₄ g. red-violet color. (48).
114	Abt. 45 ⁴⁹	336-7 ⁵⁰	<u>1-Phenyl-naphthalene, C₁₆H₁₂.</u> - Melts in- distinctly; completely liquid at 45°. (49). - Faint-blue fluorescence. (51). - Alk. KMnO ₄ (51) or CrO ₃ in AcOH (50) g. o-benzoyl- benzoic ac. (50,51).
116	45 ⁶⁵⁴		<u>Pyrocatechol diethyl ether, C₁₀H₁₄O₂.</u>
118	45-5.5 ⁵²		<u>1,4-Diphenyl-butene-(2), C₁₆H₁₆.</u> - HNO ₃ g. cpd., M.P.=191°. (53).
120	46-7 ⁵⁴	Above ⁵⁴ 320	<u>9-Methyl-fluorene, C₁₄H₁₂.</u>
122	46-7 ⁵⁵	138- 40 (7 mm)	<u>2-Methoethenyl-naphthalene, C₁₃H₁₂.</u> - Picrate, orange-yellow needles, M.P.=88°. (55).
124	46-7 ⁵⁶	•	<u>9,9-Di-n-propyl-9,10-dihydro-anthracene, C₂₀H₂₄.</u> - Fluorescent in soln. (56).
126	47-8 ⁵⁷		<u>4-Methyl-diphenyl, C₁₃H₁₂.</u> - Plates fm. ligroin. (57). - Oxid. w. dil. HNO ₃ g. p- phenyl benzoic ac. (58). - CrO ₃ g. tere- phthalic ac. (58). - 15.5 g. hy. + 14.8 g. Br in CS ₂ htd. on H ₂ O bath, CS ₂ distilled off, residue washed w. NaOH+xtalized fm. boil. EtOH g. 2 or 3-brom-4-methyl-diphenyl, M.P.= 127-9°, as 1st ppt. (59). - 5 pts. hy., 1 pt. H ₂ SO ₄ , + 6 pts. HNO ₃ (d=1.45), stg., g. di- nitro cpd., needles fm EtOH, M.P.= 153-7°. (58)

DIVISION A, SECTION 2

No.	Melting Point (C°)	Hydrocarbon
128	47 ⁶⁰	<u>1,2-Diphenyl-cyclopentane, C₁₇H₁₈.</u> - CrO ₃ in AcOH g. <u>1,3-dibenzoyl-propane, benzoic ac., + an ac. of M.P.=133.5°. (61).</u> - B.P.=305° dec. (60), 189 (12mm.) (61).
130	47 ²³	<u>Pyrogallol trimethyl ether, C₉H₁₂O₃.</u> - B.P.=235°. (23)
132	48-50 ⁶²	<u>9,9-Diethyl-9,10-dihydro-anthracene, C₁₈H₂₀.</u> - CrO ₃ in cold AcOH g. <u>diethyl-anthrone (62), M.P.=136°. (23)</u>
134	49 ⁶³	<u>4,4'-Diisopropyl-diphenyl, C₁₈H₂₂.</u>
136	50 ⁶⁴	<u>2,5,2',5'-Tetramethyl-diphenyl, C₁₆H₁₈.</u> - B.P.=285°. (64).
138	50 ⁶⁵	<u>1-Phenyl-1,4-dihydronaphthalene, C₁₆H₁₄.</u> - Decolorizes alk. KMnO ₄ . (65). - Adds Br in HCCl ₃ . (65).
140	50 ²³	<u>p-Cresyl ether, C₁₄H₁₄O.</u> - Distils. (23).
142	51 ⁶⁶	<u>1,1,1-Triphenyl-propane, C₂₁H₂₀.</u> - Nitration g. <u>1,1,1-tris-[4-nitro-phenyl]-propane, light yellow flakes fm. AcOH, M.P.=194-5°, wh. g. fuchsine reaction of Gomberg and Cone, Ber. 39, 2962. (66).</u>
144	51.5-2 ^{67,69}	<u>1,1-Diphenyl-propene-(1)*, C₁₅H₁₄.</u> - D ₂₀ ²³ superfused: 1.008, n _D ²⁰ superfused: 1.593. (67). - D ₂₀ ⁶⁰ = 0.984, n _D ⁶⁰ = 1.582. (21). - Br g. 2-brom-1,1-diphenyl-propene-(1), needles fm. EtOH, M.P.= 48-9°, B.P. ₁₂ = 169-70°, unchanged by xs. Br or NaOEt. (70).
146	52-3 ⁶⁸	<u>2-Methyl-phenanthrene, C₁₅H₁₂.</u>
148	52 ⁶⁴	<u>2,4,5,2',4',5'-Hexamethyl-diphenyl, C₁₈H₂₂.</u> - B.P.= 321°. (64).
150	52 ⁵³	<u>1,4-Diphenyl-butane, C₁₆H₁₈.</u> - Xtals. fm. EtOH. (53). - Blue fluorescence in both liquid and dissolved states. (71).
152	52 ^{73,74}	<u>1,2-Diphenyl-ethane, "Dibenzyl", C₁₄H₁₄.</u> - B.P.= 284°. (74). - D ₂₀ ⁵³ = 1.014. (75). - CrO ₃ in AcOH or H ₂ SO ₄ or alk. KMnO ₄ g. benzoic ac. (76). - Soln. in HNO ₃ (d=1.52), filtn. of ppt. of 4,4'-dinitro-

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Hydrocarbon
		dibenzyl (yellow needles fm. EtOH or C ₆ H ₆ , M.P.=179-80°) (78,77) + pptn. of filtrate w. H ₂ O g. 2,4'-dinitro-dibenzyl, needles, M.P.=74-5°. (78,78).
154	52 ⁸³	<u>Phloroglucinol trimethyl ether, C₉H₁₂O₃.</u> - B.P.=255.5 (Corr.). (83). - Sol. in conc. HNO ₃ w. deep-blue color. (83).
156	79,80 52-3	<u>1,2-Dimethyl-1,2-diphenyl-cyclobutane, C₁₈H₂₀.</u> - B.P.= 299-300°. (80).
158	53 ⁸¹	<u>Pentamethyl-benzene*</u> , C ₁₁ H ₁₆ . - B.P.= 231°. (82). - D ¹⁰⁷ =0.847. (83). - Slowly oxid. by KMnO ₄ g. benzene- pentacarbonic ac. (82). - In CHCl ₃ , HNO ₃ -H ₂ SO ₄ g. 5,6-dinitro-1,2,3,4-tetramethyl benzene, M.P.=178°. (84). - Conc. H ₂ SO ₄ g. hexamethyl benzene (83), M.P.=166°, No. 698.
160	53 ¹	<u>1,10-Diphenyl-decadiene-(1,9), C₂₂H₂₂.</u> - Long needles fm. glac. AcOH (1). - Tetrabromide, xtaln. powder fm. EtOH, M.P.=164-5°. (18).
162	21,87 54	<u>1,1,2-Triphenyl-ethane, C₂₀H₁₈.</u> - B.P.=348.5-9.5. (21).
164	54 ⁸⁸	<u>2(?)-Benzyl-diphenyl, C₁₉H₁₈.</u> - B.P.=283-7°(abt.110 mm). (88). - Sol. in hot conc. H ₂ SO ₄ w. evolution of SO ₂ , g. brown-red color. (88). - G. no picrate. (88).
166	55-6 ⁸⁹	<u>2,3-Dimethyl-2,3-diphenyl-butane?, C₁₈H₂₂.</u> - B.P.=138-40°. (89).
168	55-6 ⁹⁰	<u>Phenyl-di-[p-tolyl]-methane, C₂₁H₂₀.</u> - Insol. in conc. H ₂ SO ₄ . (91). - Needles fm. MeOH. (90).
170	55-6 ⁹²	<u>1,2-Di-[m-tolyl]-ethene, C₁₈H₁₈.</u> - Xtals. fm. EtOH or MeOH. (92). - G. solid dibromide. (92). - Picrate, red xtals., M.P.= 96.5-7.5°, very sol. (188).
172	55-6 ⁹³	<u>Hydroquinone dimethyl ether*</u> , C ₈ H ₁₀ O ₂ .
174	56 ⁹³	<u>2,3-Diphenyl-bicyclo-[0,2,2]-hexane(?)</u> , C ₁₈ H ₁₈ . - Needles fm. dil. EtOH. (93). - Indifferent to Br. (93). - B.p. ₁₂ : 212-5°. (93).

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Hydrocarbon
176	56 ⁹⁴	<u>9,9-Dimethyl-9,10-dihydroanthracene, C₁₆H₁₆.</u>
178	56.5 ⁹⁵	<u>Cyclohexyl-diphenyl-methane, C₁₉H₂₂.</u> -
180	57.8 ⁹⁶	<u>1,4-Diphenyl-2-benzyl-butene-(2), C₂₃H₂₂.</u> - B.P.= 245-7° (22mm.) (96). - 0.3 g. benzaldehyde. (96).
182	57 ⁹⁷	<u>9-Isobutyl-anthracene, C₁₈H₁₈.</u> - Fluorescing needles fm. EtOH. (97). - Picrate, brown-red needles. (97).
184	57 ^{30,47}	<u>Dihydrodicyclopentadiene, C₁₀H₁₄.</u> - B.P.=103-5°.(30). (12mm.) - Br titration shows 1 double bond. (30). - More stable than dicyclopentadiene.(30). - Htg. part. dec. to cyclopentene, Div. B., Sect. 3, No. 27 + cyclo- pentadiene (30), Div. B, Sect. 3, No. 19.
186	57 ⁸³²	<u>1,3-Diphenyl-propene, C₁₅H₁₄.</u> - B.P.= Abt. 276°.(832) - Cf. Div. B, Sect. 1, No. 838.
188	57 ⁸³³	<u>1,2-Diphenyl-butene-(1), C₁₆H₁₆.</u> - B.P.= 296-7°.(833) - KMnO ₄ in H ₂ SO ₄ g. benzoic ac. + EtPhCO. (833). - Cf. Div. B, Sect. 1, No. 820.
190	58 ⁹⁸	<u>Cyclobutylidene-diphenyl-methane, C₁₇H₁₆.</u> - Yellow soln. in H ₂ SO ₄ . (98). - Violet ring w. layer of H ₂ SO ₄ . (98). - CrO ₃ oxid. g. Ph ₂ CO. (98). - HNO ₃ oxid. g. Ph ₂ CO+succinic ac. (98). - Br g. dibromide, M.P.=91-2°, wh. loses HBr on boil. w. MeOH g. di- phenyl-bromo-cyclobutyl-carbinyl methyl ether, M.P.= 81-1.5°. (98).
192	58.5-9 ^{99, 100,101}	<u>1-Benzyl-naphthalene, C₁₇H₁₄.</u> - B.P.=350°. (101). - D ₁₇ =1.166. (100). - CrO ₃ mixt. g. benzoic ac. (101). Dil. HNO ₃ g. phenyl α-naphthyl ketone. (101). - Picrate, yellow needles, M.P.=100-1°.(101) - Sol. in 30 pts. hot. alc., 2 pts. ether. (23).
194	59 ⁹⁷	<u>9-Isoamyl-anthracene*</u> , C ₁₉ H ₂₀ . - Light yellow needles fm. EtOH w. blue-green fluorescence. (97). - Solns. fluoresce blue. (97). - Hy in u. v. light g. beautiful rich blue fluorescence. - On long stg. g. odor of isovaleric ac. + turns yellow-orange. - Sol. in conc. H ₂ SO ₄ w. green color wh. by htg. g. dirty-red. (97). - CrO ₃ +AcOH g. isoamyl oxanthrol. (97). - 1 Mol Br in CS ₂ (20-30 pts.) g. 10-brom-9- isoamyl anthracene, orange-yellow needles, M.P.=76°.

DIVISION A, SECTION 2

No.	Melt- ing Point (C°)	Hydrocarbon
196	30, 47 60	whose picrate, orange-colored needles, M.P.=110°. (97) - Picrate, brown-red needles fm. EtOH, M.P.=115°. (97) <u>Tricyclopentadiene, C₁₅H₁₂</u> . - B.P.=90-2°(0.06mm.). (30). - Adds 4 atoms of Br in CS ₂ . (47). - Readily oxid. by HNO ₃ . (47). - G. tetrahydro deriv., M.P.= 43° (47), No. 110.
198	60-1 ⁹⁷	<u>9-Ethyl-anthracene, C₁₈H₁₄</u> . - Leaflets fm. alc. (23). Picrate, M.P.=120°. (97).
200	60 ¹⁰²	<u>Diphenyl-ethine, "Tolane", C₁₄H₁₀</u> . - Distills without dec. (102). - CrO ₃ mixt. g. benzoic ac. (102). - Htg. w. conc. H ₂ SO ₄ at 60°, then steam distn. of dil. reaction mixt. g. desoxybenzoin. (104). - For cpd. w. picryl chloride cf. Bruni, Ch. Z. 30, 568.- CHCl ₃ soln. satd. w. Cl g. tetrachloride, M.P.=163°. (23). - G. Test 901. (23). - Picrate, light yellow tabular xtals., M.P.=111°, exp. at higher temp. (23).
202	60 ¹⁰⁵	<u>1-Phenyl-1-[α-naphthyl]-ethene, C₁₈H₁₄</u> . - B.P.=350-5° (105). - Sol. in conc. H ₂ SO ₄ w. deep red color. (105). - Br in CS ₂ g. oil wh. by distn. at reduced press. and subtraction of HBr g. mixt. of both stereoisomers of 2-brom-1-phenyl-1-[α-naphthyl]-ethene, wh. latter can be separated by fractional xtaln. fm. EtOH, M.P. =71-2° (less sol.), + 54°. (105).
204	60.5 ¹⁰⁷	<u>1,2,4,5-Tetramethyl-3-benzyl-benzene(23), C₁₇H₂₀</u> . - B.P.= 310°. (716 _{m.m.})(107). - Needles fm. EtOH. (107).
206	108, 111 61-3	<u>9-Ethyl-phenanthrene, C₁₈H₁₄</u> . - B.P.=198-200° (11mm). (108). - CrO ₃ g. phenanthraquinone. (108). - Picrate, orange needles fm. MeOH, M.P.= 124°. (108).
208	61 ¹¹⁰	<u>1,1-Di-[p-tolyl]-ethene, C₁₈H₁₆</u> . - B.P.=304-5°. (109). - Plates fm. EtOH. (110). - CrO ₃ mixt. g. di-p- tolyl ketone. (109).
210	61-2 ¹¹²	<u>1,8-Diphenyl-octadiene, (1,7), C₂₀H₂₂</u> . - B.P.=210-20° (11 mm.) (112). - Leaflets. (112). - Br in CS ₂ g. tetrabromide, leaflets, M.P.= 196°. (112).
212	61.5 ¹¹³	<u>Diphenyl-[2,4-dimethophenyl]-methane, C₂₁H₂₀</u> . - B.P.= Above 360°. (113). - CrO ₃ mixt. g. 6-methyl- 3,3-diphenyl-phthalide+ 3,3-diphenyl-phthalide- carbonic acid-(6). (113).

DIVISION A, SECTION 2

No.	Melting Point (C°)	Hydrocarbon
214	114 62	<u>1-Methyl-3-phenyl-cyclopentadiene-(2,4)(?)</u> , $C_{12}H_{12}$. - B.P.=151 (12mm) (114). - $FeCl_3$ in ether g. 1st violet, then dark blue xtaln. ppt. (114). - HCl g. red-violet color. (114). - Cherry-red soln. in conc. H_2SO_4 treated w. ice H_2O shows no change on pptn. (114).
216	48 62-3	<u>1,3-Dibenzyl-indene</u> , $C_{23}H_{20}$. - W. calcd. amt. Br in $CHCl_3$ g. 1,2-dibrom-1,3-dibenzyl-indane. (48).
218	115 62	<u>Diphenyl-[m-tolyl]-methane</u> , "3-Methyl-tritan", $C_{20}H_{18}$. - B.P.=354° (706 mm). (116). - Shows triboluminescence w. blue color. (115). - Dil. solns. show strong blue fluorescence. (115). - Careful oxid. w. CrO_3 in $AcOH$ g. diphenyl-m-tolyl-carbinol, (116) and an oil of B.P.=310-20°. (117). - Energetic oxid. w. $K_2Cr_2O_7 + H_2SO_4$ g. α -oxy-tritan-carbonic ac.-(3). (118). - Fum. HNO_3 g., beside other products, trinitro deriv., wh. by $Zn+HCl$ g. leucaniline wh. latter g. ros-aniline. (118,119). - In pure state g. no color to H_2SO_4 . (118). - G. no picrate. (115).
220	120 62-3	<u>1-Methyl-2,3-diphenyl-cyclopentane</u> , $C_{18}H_{20}$. - Needles fm. $Et_2O+MeOH$. (120).
222	23 63	<u>2,5-Dimethyl-3,4-diacetyl-furane</u> , $C_{10}H_{12}O_3$. - Cf. Mulliken, Ident. etc., Vol. I, VII, A, pg. 137.
224	121 63	<u>1-Methyl-4,10-endomethylene-anthracene(?)</u> , "p-Dimethyl-anthracylene", $C_{16}H_{12}$. - Light yellow leaflets. (121). - Picrate, dark-red needles, M.P.=129°. (121).
226	122 63	<u>1,2,3,4,9,10-Hexahydroanthracene</u> , " γ -Hexahydroanthracene", $C_{14}H_{16}$. - B.P.=290°. (122).
228	85 63.5	Tri-[p-tolyl]-methane, $C_{22}H_{22}$. - Insol. in conc. H_2SO_4 . (85). - Tris-[x,x-dinitro-4-methylphenyl]-methane by 12 hrs. action of fum. HNO_3 , yellow prisms, M.P.=280°, wh. exp. on higher htg. (85).
230	123 64	<u>1, 2 or 3-Dimethyl-4,10-endomethylene-anthracene(?)</u> , "Trimethyl-anthracylene", $C_{17}H_{14}$. - Br in CS_2 g. dibrom deriv., $C_{17}H_{12}Br_2$, M.P.=105° dec. (123). - Picrate, dark-red needles, M.P.=134°. (123).
232	207 65	<u>3-Methyl-phenanthrene</u> , $C_{15}H_{12}$. - Little rods fm. dil. $EtOH$. (207). - Br in $HCCl_3$ g. a dibromide,

DIVISION A, SECTION 2

No.	Melting Point (C°)	Hydrocarbon
		M.P.=86-7° (207). - Picrate, yellow-red needles, M.P.=141° (Corr.) (207).
234	66.5 ¹²⁴	<u>1,2,3,4,11,12-Hexahydroanthracene, C₁₄H₁₈</u> . - B.P.=303-6° (124). - Plates (124). - Sol. in hot EtOH, AcOH, + C ₆ H ₆ w. blue fluorescence (124). - CrO ₃ g. 9,10-dioxy-anthracene-dihydride (124). - Br in AcOH or HCCl ₃ g. 9,10-dibrom-octahydroanthracene (124).
236	66.5 ¹²⁵	<u>1,2-Di[O-tolyl]-ethane, C₁₆H₁₈</u> . - B.P.=177-8 (20mm) (125).
238	67-8 ¹²⁷	<u>[3,5-Dimethophenyl]-[2,4,6-trimethophenyl]-methane, C₁₈H₂₂</u> . - B.P.=329° (127). - Boil. KMnO ₄ g. benzophenone-pentacarbonic ac. (127). - Br+I, cold, g. colorless plates fm. C ₆ H ₆ of tetrabrom cpd., M.P.=230-2° (127). - Fum. HNO ₃ in AcOH g. tetranitro cpd. plates fm. Me ₂ CO+EtOH, M.P.=233° (128).
240	67 ²³	<u>Asarone, C₁₂H₁₆O₃</u> . - Occurs in root of Asarum Europaeum. (23).
242	68-9 ¹³¹	<u>1,8-Endotrimethylene-naphthalene, "Peritrimethylene-naphthalene", "Perinaphthindan", C₁₂H₁₂</u> . - White, silvery, lustrous plates (131). - Unstable in air (131). - Picrate, red, silky, lustrous needles fm. EtOH, darkens at 80°, M.P.=127° (131).
244	68.5 ¹¹³	<u>Diphenyl-[3,4-dimethophenyl]-methane, "3,4-Dimethyl-tritan", C₂₁H₂₀</u> . - B.P.= Above 360° (113). - CrO ₃ mixt. g. α-oxy-tritan-dicarbonic ac.-(3,4), benzophenone, + another cpd. (113).
246	69 ¹³²	<u>Diphenyl, C₁₂H₁₀</u> . - B.P.= 255° (132). - $D_4^{25} = 0.992$. (133). - CrO ₃ in AcOH g. benzoic ac. (134). For cpd. w. picryl chloride cf. Brunl, Ch. Z. 30, 568. - Br in CS ₂ soln. g. 4-brom-diphenyl, lamellas fm. EtOH, M.P.=89°, B.P.=310° (134), +4,4'-dibrom-diphenyl, M.P.=164°, B.P.= 355-60° (134,135). - 5 pts. hy. + 10 pts. AcOH+4 pts. HNO ₃ (d=1.45) g., on boil., 4-nitro-diphenyl(2-nitro cpd. left in soln.), needles fm. EtOH, M.P.=114-4.5°, B.P.=340° (134,135) - 20 g. hy. + 20 cc. fum. HNO ₃ boiled for short time, cooled, xtals. washed w. EtOH+xtalized. fm. EtOH g. 4,4'-dinitrodiphenyl, M.P.=234-5° (137,138). - Cpd. =2 SbCl ₃ .Ph ₂ , non-hygroscopic needles, M.P.= 71° (17). - Cpd.= 2 SbBr ₂ .Ph ₂ , rhombic plates or pyramids, M.P.=60.5°(dec.). (17). - Cpd. 2 SbI ₃ .Ph ₂ ,

DIVISION A, SECTION 2

No.	Melt- ing Point (C°)	Hydrocarbon
		non-hygroscopic needles, red w. bluish tint, M.P.= 161°. (17). - All three immediately preceding systems also have 2 eutectic pts. (17). - Sol. in 10 pts. cold alc. (23). - Test 904 g. an intense + quite permanent blue (B) color. (23). - SbCl ₅ in CCl ₄ g. deeper yellow color than w. C ₆ H ₆ . (s13). - No color w. SbCl ₃ . (s14). - No color w. BrCl ₃ . (s14).
248	⁶³⁰ 70-0.5	<u>cis-cis-1,4-Diphenyl-butadiene-(1,3), C₁₆H₁₄.</u> - B.P.= Abt. 350? (405). - Exposure to light g. trans-trans form, M.P.= 152-2.5°, (s30). No. 640.
250	¹⁴⁶ 72	<u>1,1,2-Triphenyl-ethene, * "α-Phenyl-stilbene", C₂₀H₁₄.</u> - B.P.= 220-1 (14 mm.) (130). - Br g. dibromide. (129,130).
252	¹⁴⁴ 72-3	<u>1,2-Di-[tribenzyl-methyl]-benzene, C₅₀H₄₆.</u> - Amorphous. (144). - Non-volatile. (144).
254	¹⁴⁵ 72	<u>1,4-Di-[p-ethophenyl]-butadiene-(1,3), C₂₀H₁₄.</u> - Needles. (145).
256	⁶⁵⁴ 72	<u>Hydroquinone diethyl ether, * C₁₀H₁₄O₂.</u>
258	¹⁴⁷ 72	<u>4-[p-Tolyl-methyl]-fluorene, "4-[p-Xylyl-fluorene", C₂₁H₁₈.</u>
260	¹⁴⁸ 72	<u>Diphenyl-[p-tolyl]-methane, "4-Methyl-tritan", C₂₀H₁₈.</u> - B.P.= Above 360°. (119). - Boils without dec. (119).
262	⁸³ 72	<u>2-Methoxy-naphthalene, * "Methyl-β-naphthyl ether", "Nerolin", C₁₁H₁₀O.</u> - B.P.= 274°. (23). - Odor like oil of neroli (orange blossoms). (23). - Leaflets fm. ether. (23).
264	¹⁴⁸ 73-4 ¹⁴⁰ Sub- limes above this temp.	<u>1,2,3,4,5,6,7,8-Octahydroanthracene, * "sym-Octahydro-anthracene", "Octracene", C₁₄H₁₂.</u> - B.P.=293-5°. U.C. (140). - Energetic oxid. w. CrO ₃ g. anthraquinone. (140). - Careful oxid. in AcOH at ord. temp. g. dihydro-oxanthranol+hexahydroanthrone. (140). - KMnO ₄ g. phthalic ac. (141). - Htg. w. conc. H ₂ SO ₄ g. anthracene-oxtahydride-sulfonic ac.-(9) (141). - Br in CS ₂ or HCCl ₃ g. 9,10-dibrom-anthracene-oxtahydride needles, M.P.=194°. (141,140). - Boil. w. KMnO ₄ g. pyromellitic ac., xtals. w. 2 H ₂ O, M.P.= 264° (loss of H ₂ O) wh. by subliming at 290° under 13 mm. g. pyromellitic anhydride, M.P.= 286°, wh. latter w. resorcinol+ ZnCl ₂ at 220° g. pyromellitein, red-yellow, very faintly fluorescent; wh. last is con-

DIVISION A, SECTION 2.

No.	Melt- ing Point (C°)	Hydrocarbon
		verted by Br in C ₅ H ₅ N into pyromelliteosin, blue-red, dyeing silk in dil. AcOH, ctg. NaOAc, a deep blue-red. (142). - Htd. w. S at 180-200° g. anthracene+H ₂ S. (143). - Distn. w. Zn dust g. anthracene. (143). - Htd. w. Cu at 550° in CO ₂ g. anthracene+H. (143). - Picrate, orange-yellow needles, M.P.=80°.(dec.) (141).
266	73 ¹⁴⁹	<u>Tri-[m-tolyl]-methane, C₂₂H₂₂.</u> - B.P.=376-7°. (149).
268	73-4 ¹⁵⁰	<u>1,1-Di-[1,3,4-dimethoxyphenyl]-ethene, C₁₈H₂₀O₂</u> - K ₂ Cr ₂ O ₇ +H ₂ SO ₄ g. 3,4,3',4'-tetramethyl-benzophenone. (150).
270	74.5-5 ¹⁵¹	<u>Dibenzo-1,2;3,4-cyclobutadiene-(1,3), "Diphenylene", C₁₂H₈.</u> - Flat prisms. (151). - Closely resembles diphenyl in appearance and odor. (151).
272	76 ¹⁵²	<u>1,4-Di-ter-butyl-benzene, C₁₄H₂₂.</u> - B.P.=236.5: (153) - Sublimes easily. (153). - Fum. HNO ₃ at ord. temp. g. 2,6-dinitro-1,4-di-ter-butyl benzene, white, nearly odorless needles fm. EtOH, M.P.=190-1°. (154).
274	76-7 ¹⁵⁵	<u>Benzyl-α-naphthyl ether, C₁₇H₁₄O.</u>
276	76-7 ¹⁵⁵	<u>3,4,3',4'-Tetramethyl-diphenyl, C₁₆H₁₈.</u> - Pale yellow needles. (155).
278	76 ^{156a}	<u>Dihydrofluoranthene, "Dihydro-idryl", C₁₅H₁₂.</u> - Picrate, yellow or red needles, M.P.=186°. (156a).
280	76 ^{156b}	<u>9-Benzylidene-fluorene, C₂₀H₁₄.</u> - In molten state (156b)+ in soln. (sol. in EtOH, C ₆ H ₆ , AcOH) (157) colored yellow. (156b,157). - Picrate, orange-yellow needles, M.P.=115-6°. (156).
282	77-8 ¹⁶⁰	<u>1,2-Di-[3,5-dimethoxyphenyl]-ethane, C₁₈H₂₂O₂.</u> - B.P.=332-2.5°. (160). - Boil. HNO ₃ + boil. KMnO ₄ g. trimelic ac. (160). Br in AcOH+I g. tetrabrom cpd., prisms fm. EtOH, M.P.=170-1°. (16). - Gradual addn. of 80 cc. fum. HNO ₃ to 0.4 gm. hy. in AcOH g. tetra-nitro cpd. in 2 forms, fm. Me ₂ CO+H ₂ O, M.P.=205-6°+158-60° wh. latter after solidifying remelts at 215°. (161).
284	77 ¹⁶²	<u>4-Benzyl-fluorene, C₂₀H₁₆.</u> - White leaflets fm. EtOH. (162).
286	78 ¹⁶³	<u>1,2-Dibenzyl-benzene, C₂₀H₁₆.</u> - Needles fm. EtOH. (163). - CrO ₃ mixt. or CrO ₃ in AcOH g. 1,2-dibenzoyl-benzene+some benzophenone carbonic ac.-(2). (164). - G. no picrate. (163).

DIVISION A, SECTION 2

No.	Melt- ing Point	Hydrocarbon
288	79-80 ¹⁶⁸	<u>2-[α-naphthyl]-naphthalene, "α, β-Dinaphthyl", $C_{20}H_{14}$.</u> - Picrate, golden-yellow needles, M.P.=155-6°.(168).
290	79 ⁶⁶	<u>1,1,1-Triphenyl-butane, $C_{22}H_{22}$.</u> - Nitration g. a tris-nitrophenyl cpd., M.P.= 191-2°, xtals. fm. AcOH, wh. does not g. the fuchsine reaction of Gomberg and Cone, Ber. 39, 2963. (66).
292	80 ¹⁶⁵	<u>1,2,4,5-Tetramethyl-benzene, "Durene", $C_{10}H_{14}$.</u> - B.P.=191-2°.(165). - $D_{40}^{20}=0.838$. (166). - $HNO_3-H_2SO_4$ g. 3,6-dinitro-1,2,4,5-tetramethyl benzene, colorless prisms fm. EtOH, M.P.=205°, (167), or 1 ² ,3,6-trinitro-durene, prisms, M.P.=139°. (625). - Br in $CHCl_3$ g. mono and dibrom cpds. fm. wh. mono cpd. can be separated by steam distn. and wh. w. 98% HNO_3 at 0° in $CHCl_3$ +conc. H_2SO_4 g. 3-nitro-6-bromo-durene, pale yellow prisms wh. soften at 177°+ M.P.=178-9°+ has odor only when htd. (626). - The mono bromo deriv. g. w. HNO_3 (d=1.52), 3,6-dinitro-duryl-bromide, colorless prisms, M.P.=121.5° wh. w. boil. alc. KOH g. di-nitro-durylic ac. (626). -
294	80-1 ⁶³¹	<u>1,3-Dimethyl-2,4,5-triphenyl-cyclopentane, $C_{25}H_{26}$.</u> - B.P.= Abt. 246-8°?(631). - Cf. Div. B, Sect. I, (25 mm.) No. 1020.
296	80 ⁶⁵³	<u>5,6-Diphenyl-decane, $C_{22}H_{30}$.</u> - Unaffected by Na-K in ether over 48 hrs. (653).
298	80 ¹⁶⁹	<u>Naphthalene, $C_{10}H_8$.</u> - B.P.=218°. (169). - $D_4^{25}=0.962$; $n_D^{25}=1.582$. (170). - Oxid. w. manganic salts g. naphtho-quinone-(1,4). (171). - $K_2Cr_2O_7+H_2SO_4$ g. phthalic ac. (172). - Dil. HNO_3 at 130° g. phthalic ac. (173). - $KMnO_4$ oxid. (Test 905-1) g. phthalic ac. (Small yield). (23). - Boil. 1 minute w. conc. H_2SO_4 + yellow HgO + and then htd. w. resorcinol, then soln. in aq.+addn. of alk. g. green fluorescence of fluorescein thus formed. (612). - $HCHO+H_2SO_4$ g., in the cold, blue color, w. slight htd., more intense blue, finally dark-violet to blue-black. (612). - $H_2O_2-H_2SO_4$ g. intense green color. (612). - $PhCHO+H_2SO_4$ g. red color, quickly, by htd. on H_2O bath. (612). - Benzal-chloride+ H_2SO_4 g. fuchsine-red color. (612). - Filter paper dipped in C_6H_6 soln. of hy. + chloramil, + warmed on H_2O bath colors brown red+on cooling loses its color. (612). - Dil. soln. in CCl_4 g. w. PCl_5 first yellow-brown color, after a few seconds a brownish-lilac ppt. wh. dissolves w. the same color in a little $HCCl_3$ +by

DIVISION A, SECTION 2

No.	Melting Point (C°)	Hydrocarbon
		considerable diln. is suddenly decolorized. (e ₁₉). - Micro-procedure: PhNO ₂ soln. g. w. α-dinitrophenanthraquinone yellow, w. chrysammic ac. red rhombic xtals. (e ₁₂). - No color w. pure hy. + BiCl ₃ ; during cooling, yellow transparent needles separate. (e ₁₄). - Completely dry AlCl ₃ added to HCCl ₃ soln. + htd. g. evolution of HCl + intense green-blue color (174), Test 904. (23). - Pure naphthalene g. no red color w. SbCl ₃ on porcelain tile, only impure does so. (175). - Cpd. w. dinitro-benzene (in C ₆ H ₆ soln., not EtOH), needles, M.P.=52-3°, soon loses hy. in air. (176). - Cpd. w. p-dinitro-benzene (EtOH soln.), white needles, M.P.=118-9°. (176). - Cpd. w. 1,3,5-trinitro-benzene (boil. EtOH), white xtals. fm. EtOH+HCCl ₃ , M.P.=152°, at ord. temp. + by rextaln. fm. EtOH loses hy. (176). - Cpd. w. picryl chloride (conc. EtOH soln.) yellow xtals. fm. Me ₂ CO, M.P.=95-6°. (177). - Cpd. w. T.N.T. fm. alc., xtals. fm. Me ₂ CO, M.P.=97-8°. (176). - Picrate, yellow prisms, M.P.=149.5° (e ₅₃), Test 915. (23). - Styphnate, M.P.=165.5° (1 mol: 1 mol), soln. g. 2 eutectics. (191). - Cpd. w. picramide: M.P.=169° (192).
300	80 ¹⁷⁹	<u>4,4'-Diethyl-diphenyl, C₁₈H₁₈</u> -
302	80.5-1 ¹⁸⁰	<u>1,2,3,4-Tetraphenyl-cyclopentane, C₂₈H₂₈</u> - Colorless needles fm. 90% EtOH. (180).
304	81 ¹⁸¹	<u>1,4-Diphenyl-2,2,3,3-tetrabenzyl-butane, C₄₄H₄₂</u> - B.P.= 353.5-8.5°. (181). - HNO ₃ (d=1.475) g. hexanitro cpd. wh. softens at 75° + dec. at abt. 115°, wh. w. alc. KOH g. violet color (as other high molecular wgt. nitro cpds.), + wh. by red. w. HI g. hexamine, light yellow powder, dec. at 105°. (181).
306	81 ^{159,182}	<u>Dihydrobenzanthrene (159), "Iso-chrysofluorene", (15a) C₁₇H₁₄</u> . (15a). - Yellow needles fm. EtOH. (182). - In conc. H ₂ SO ₄ fluoresces red to reddish-brown; soln. evolves SO ₂ . (182). - Picrate, (1 mol hy:1 mol ac.), orange-yellow needles, M.P.=125°. (182). - 2 mols Br in glac. AcOH g. C ₁₇ H ₁₃ Br, colorless, lustrous xtals. fm. dil. AcOH, M.P.=123° wh. fluoresce in warm conc. H ₂ SO ₄ reddish-brown to brown. (182). - 4 mols Br in glac. AcOH g. dibromodihydrobenzanthrene, needles, w. yellow shade, fm. AcOH, M.P.=157°. (182).
308	81-2 ¹⁸³	<u>9-[9³-phenyl-propylidene]-fluorene, or 9-[9³-phenyl-propene-(9²)-yl]-fluorene, C₂₈H₁₈</u> - White plates fm. EtOH, M.P.=81-2°. (183). - Not reduced by Al-Hg. (183) - Picrate, red-yellow needles fm. EtOH or AcOH, M.P.= 128-9°. (183).

DIVISION A, SECTION 2

No.	Melt- ing Point (C°)	Hydrocarbon
310	82 ⁶⁷	<u>1,2-Diphenyl-propene-(1)</u> , <u>C₁₅H₁₄</u> .
312	82-3 ¹¹⁶	<u>Diphenyl-[o-tolyl]-methane</u> , <u>C₂₀H₁₈</u> . - Insol. in and not colored by grinding w. conc. H ₂ SO ₄ . (116).
314	82 ¹⁸⁴	<u>Diphenylmethene-cyclopentadiene</u> , " <u>ω,ω</u> - <u>Diphenyl-fulvene</u> ", <u>C₁₈H₁₄</u> . - Deep red plates or prisms fm. pet. ether. (184). - Odor faintly like azobenzene. (184). - Reduces alk. KMnO ₄ . (184). - Trace of AcOH-H ₂ SO ₄ added to AcOH soln. g., by boiling, deep green color. (184).
316	82 ¹⁸⁵	<u>1,6-Diphenyl-hexadiene-(1,5)</u> , <u>C₁₈H₁₈</u> . - B.P.=211°. (185). - Thin, large, colorless plates w. reddish-blue fluorescence in solid state and in soln. (185). - Tetrabromide, feathery needles fm. Me ₂ CO, M.P.=194°. (185).
318	82-3 ⁶⁹	<u>1,2-Diphenyl-propene-(1)</u> , <u>C₁₅H₁₄</u> . - B.P.=285-6.(21). - $D_4^{17} = 0.986$, $n_D^{17} = 1.564$. (21). - CrO ₃ in AcOH g. acetophenone+benzoic ac. (69). - Soln. in conc. H ₂ SO ₄ is wine-red to reflected, yellow-red to transmitted light. (69). - Br g. α,α'-dibrom-α-methyl-dibenzyl. (186,187).
320	82.5-3 ¹⁸⁸	<u>1,2-Di-[o-tolyl]-ethene</u> , <u>C₁₆H₁₆</u> . - Needles fm. MeOH. (188). - Picrate, red needles, M.P.=102-3°. (188).
322	83 ¹⁸⁹	<u>Di-(isopropylidene-cyclopentadiene)</u> , " <u>dimolecular Dimethyl-fulvene</u> ", <u>C₁₈H₂₀</u> . - Htg. above M.P. g. part. dimethyl-fulvene, cf. Div. B, Sect. 3, No. 445 (189). In alc. soln. reduces KMnO ₄ . (189). - Adds 4 atoms of Br. (189). - Conc. H ₂ SO ₄ g. yellow-red color. (189).
324	84 ¹⁸²	<u>Benzanthrene</u> , <u>C₁₇H₁₂</u> . - Small, highly lustrous, pale yellow plates fm. EtOH. (182). - Solns. have green fluorescence. (182). - In conc. H ₂ SO ₄ color is red w. reddish-brown fluorescence, soln. evolves SO ₂ . (182). - Oxid. in air to benzanthrene. (182). - Picrate (1 mol hy.: 1 mol ac.), slender dark red needles fm. EtOH, M.P.=110-1°. (182). - Br in glac. AcOH g. 10,10-dibromobenzanthrene, yellow xtals. fm. EtOH, M.P.=174°. (182).

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No.	Melting Point (C°)	Hydrocarbon
326	84 ¹⁹⁰	<u>[Diphenyl-methylene]-cyclohexane, C₁₈H₂₀.</u> - B.P. ₁₂ = 210-20°. (190). - Prisms fm. MeOH. (190).
328	85-6 ¹⁹³	<u>1-Methyl-anthracene (422), C₁₅H₁₂.</u> - Long white needles. (193). - Much more sol. in most solvents than anthracene or 2-methyl-anthracene. (193). - Blue fluorescence in alc. soln. - Picrate, red needles, ^{m.p.: 113-5° (193).} M.P.=170-1°, rapidly reddens on exposure to light (difference fm. β cpd.), wh. w. dil. HNO ₃ at 160° g. anthraquinone 1 carboxylic ac. wh. develops rose coloration when htd. w. soda-lime (anthraquinone 2 carboxylic ac. turns blue under similar conditions). (193).
330	85 ¹⁹⁴	<u>3-Methyl-1,9-endomethylene-anthracene, "m-Dimethyl-anthracylene", C₁₆H₁₂.</u> - Leaflets. (194). - Br in CS ₂ g. C ₁₆ H ₁₀ Br ₂ , S-yellow prisms, M.P.=175°, dec. (194). - Picrate, brown-red, M.P.= 135°. (194).
332	85-6 ¹⁹⁵	<u>1,2-Di-[p-tolyl]-ethane, C₁₆H₁₄.</u> - B.P. ₁₈ =178°. (196)
334	85 ¹⁹⁷	<u>9-Isoamyl-9-phenyl-9,10-dihydro-anthracene, C₂₅H₃₄.</u> - Br in CS ₂ g. 10-brom-9-isoamyl-9-phenyl-9,10-dihydro-anthracene, colorless xtals., M.P.=134-7°. (197).
336	85 ¹⁹⁹	<u>4-Benzoyl-diphenyl, C₁₉H₁₆.</u> - B.P.=285-6°. (199). - ^{at} Abt. 110. Leaflets. (23). - CrO ₃ in AcOH g. 4-phenyl benzophenone. (199). - Sol. in hot conc. H ₂ SO ₄ w. evolution of SO ₂ and production of blue-red color. (199). - G. no picrate. (199).
338	85 ²⁰⁰	<u>3-Phenyl-diphenyl, "Isodiphenyl-benzene", C₁₈H₁₄.</u> - B.P.=363. (200). - CrO ₃ in AcOH g. benzoic ac. + some diphenyl carbonic ac.-(3). - (200,201). - G. no picrate. (201). - Stg. 1 day w. xs Br, then boil. 1/2 hr. g. 4-brom-1-[14-brom-phenyl]-3-[3 ³ ,3 ⁴ -dibrom-phenyl]-benzene, plates fm. AcOH, M.P.=181°. (202). - Htg. w. fum. HNO ₃ g. trinitro cpd., M.P.=200°, needles fm. AcOH, wh. w. Sn+HCl g. a base, M.P.=288°. (203).
340	85 ²⁰⁵	<u>1,10-Diphenoxy-decane, C₂₂H₃₀O₂.</u> - At 150° HI g. 1,10-diiodo-decane, lustrous xtals. fm. EtOH, M.P.=29-30, B.P. ₁₆ =212-5°. (205).
342	86 ²⁰⁸	<u>1,4-Dibenzyl-benzene, C₂₀H₁₄.</u> - Leaflets fm. EtOH. (208). - CrO ₃ mixt. or CrO ₃ in AcOH g. 1,4-dibenzoyl-benzene + some benzophenone-carbonic ac.-(4). (210). - G. no picrate. (208). - 2-3 hrs. boil. of 5.2 g. hy. in 20 cc. HCCl ₃ + 2 cc. Br g. 1,4-bis-[α-brom-

DIVISION A, SECTION 2

No.	Melt- ing Point (C°)	Hydrocarbon
		benzyl]-benzene, leaflets fm. pet. ether + little C_8H_8 , M.P.= 112.5°. (209).
344	86 ²⁰⁶	<u>x,x(1,8?)-Dimethyl-anthracene, $C_{16}H_{14}$.</u> - CrO_3 quickly oxid. to a dimethyl-anthraquinone, M.P.=130° + further to acids. (206).
346	86-7 ²³	Diphenylene oxide*, " <u>(α,β), (α',β')-Dibenzofurane</u> ", $C_{12}H_8O$. - B.P.= 287-8°. (23,204). - Small leaflets fm. alc. (23). - Picrate fm alc. (23). (or CH_3NO_2), M.P.=94°. (23). - Br in CS_2 g. xtaln. dibrom cpd., M.P.=185°, difficultly sol. in alc. (23).
348	86 ²¹¹	<u>1-Phenyl-2-[2⁴-isopropophenyl]-ethene, "4-Isopropylstilbene"</u> , $C_{17}H_{18}$. - Scales. (23). - Adds Br (Test 901). (23).
350	86 ²⁰⁵	<u>Diphenoxy-dodecane, $C_{24}H_{34}O_2$.</u> - HI in xs. at 130° g. 1,12-diiodododecane, M.P.=41°; can be distilled. (205).
352	87-9 ²¹²	<u>1,1,3-Triphenyl-propene-(1), $C_{21}H_{18}$.</u>
354	88 ²¹³	<u>3-Benzylidene-indene, $C_{16}H_{12}$.</u> - Yellow plates. (213). - Sol. in much H_2SO_4 w. yellowish-green color. (213). - Al-Hg in aq.-EtOH g. 1-benzyl-indene. (48).
356	88-9 ²¹⁴	<u>Pentaethyl-benzyl-benzene, $C_{23}H_{32}$.</u> - B.P. = above 360°. (214). - Needles fm. EtOH (214). - 100 pts. EtOH at 18° dissolve 0.9 pts. hy. (214).
358	88 ²¹⁵	<u>9-[9³-Phenyl-propene-(9')-yl]-fluorene, $C_{22}H_{18}$.</u> - In alc. soln. reduces alk.ammon.-aq. soln. (215). - PhCHO + much conc. H_2SO_4 g. red color wh. is destroyed by H_2O . (215). - Boil. w. NaOEt soln. or w. alc. piperidine g. the dihydro-cinnamylidene-fluorene of M.P.=81-2°. (215), No. 308.
360	88 ²¹⁶	<u>1,4-Diphenyl-butadiene-(1,3), $C_{16}H_{10}$.</u> - Needles fm. 50% EtOH. (216). - Carbonized by H_2SO_4 and gentle gt. (216). - Picrate, light yellow xtals., M.P.=108°. (216). - Adds Br (Test 901). (23). - Does not g. Test 906. (23).
362	89 ²¹⁷	<u>x,x,x,x-Tetrahydroanthracene, "β-Tetrahydroanthracene, $C_{14}H_{14}$.</u> - B.P.=309-13. (217). Colorless plates fm. EtOH. (217). - Sublimes above its M.P. (217). - Solns. fluoresce magnificent blue. (217). - CrO_3 in AcOH g. anthraquinone. (217). - Br g. 9,10-dibrom-anthracene. (217). - Picric ac. g. red color. (217).

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No.	Melt- ing Point (C°)	Hydrocarbon
364	89 ²¹⁸	<u>1,3,5-Trimethyl-2,4-di-benzyl-benzene, C₂₃H₂₄.</u> - B.P. ₂₀ = 280°. (218).
366	89-90 ²¹⁹	<u>1-Phenyl-2-[2⁴-ethophenyl]-ethene, C₁₆H₁₆.</u>
368	90 ²²⁰	<u>2,6-Diisopropyl-9,10-dihydro-anthracene(?)</u> , C ₂₀ H ₂₄ . - B.P. = above 300°. (220). - Dirty-yellow amorphous powder. (220). - Insol. in EtOH. (220). - Easily sol. in EtOH. (220). - Easily sol. in Et ₂ O, CHCl ₃ , C ₆ H ₆ . (220). - Solns. are red and fluoresce green. (220).
370	90-1 ²²¹	<u>9,10-Diethyl-phenanthrene, C₁₈H₁₈.</u>
372	91 ²²³	<u>Diphenyl-furane, C₁₆H₁₂O.</u> - B.P. = 343-5°. (223). - Sol. in conc. H ₂ SO ₄ w. green color. (223).
374	91-2 ²²¹	<u>9-Benzyl-phenanthrene, C₂₁H₁₆.</u>
376	92 ²²²	<u>3,4-Diphenyl-hexane, C₁₈H₂₂.</u> - B.P. ₂₀ = 175°. (222).
378	92 ²²³	<u>Di-[β-naphthyl]-methane, C₂₁H₁₆.</u> - Needles fm. EtOH. (223). - Dibrom deriv., M.P. = 164°. (223). - Tetra-nitro deriv., M.P. = 150-60°. (223).
380	92 ¹¹⁵	<u>Diphenyl-[2,5-dimethophenyl]-methane, C₂₁H₂₀.</u> - Un-attached by alk. KMnO ₄ . (115). - CrO ₃ mixt. g. 5-methyl-3,3-diphenyl-phthalide + 3,3-dimethyl-phthalide-carbonic ac.-(5). (115). -
382	92-3 ²²⁴	<u>Acenaphthylene, C₁₂H₈.</u> - B.P. = abt. 265-75 w. part. dec. (225,226). - Golden yellow tabular xtals. - D ₄ ¹⁶ = .899. (226). - CrO ₃ g. naphthalic ac. (224,225). - Picrate, M.P. = 201-2°. (224). - Cpd. w. picryl chloride (1mol=1mol), M.P. = 109.4°. (240).
384	92 ²²⁷	<u>1-[Diphenyl-methylene]-4,5-benzo-cycloheptatriene-(2,4,6), C₂₄H₁₈.</u> - Yellow xtals. (227).
386	92.5 ²²⁸	<u>Triphenyl-methane, "Tritan", C₁₉H₁₂.</u> - B.P. = 358-9°. (229). - D ₂₀ ⁹⁵ = 1.057. (230). - For cpd. of picryl chloride cf. Bruni, Ch. Z. 30, 568. - Soln. of at least 1 mg. in fum. HNO ₃ , pptn. w. H ₂ O, filtn., washing w. H ₂ O, soln. of resulting tris[4-nitrophenyl]-carbinol in much cold AcOH or some HCl ctg. EtOH, + addn. of a little Zn dust g. fuchsine color. (231, 232). - Better, nitrate 0.1 gm. by soln. in 2 cc. fum. HNO ₃ without htg., ppt. trinitro cpd. by diln.

DIVISION A, SECTION 2

No.	Melt- ing Point (C°)	Hydrocarbon
		<p>w. H₂O, dissolve ppt. in 10 cc. hot glac. AcOH + red. by successive addns. of small portions of Zn dust to the hot soln. until the strong red color that at first appears is nearly discharged, decant and add a few cg. PbO₂ to the soln.; a very intense fuchsine-red color (para-rosaniline) forms at once. (23). - For color reaction w. Al Cl₃ cf. Test 904. (23). - Htg. w. PCl₃ g. trityl-chloride. (233). M.P.=108-11° (234). - Slow addn. of hy. to well cooled HNO₃(d=1.5) g. tris-[4-nitro-phenyl]-methane, M.P.=206-7° (231). - Cpd. SbCl₃.Ph₃CH, rhombohedrons, M.P.=79.5° w. dec. no eutectics. (17). - SbCl₅ in CCl₄ g. green color. (613). - Benzal-chloride+H₂SO₄ g. faint yellow color. (614). - SbCl₃ g. no color, greenish color w. xs. (614).</p>
388	235 92.5	<p><u>Phenyl-di-[2,5-dimethyl-phenyl]-methane, "2,5,2',5'-Tetramethyl-tritan, C₂₂H₂₄.</u> - B.P.= Above 360°. (235). - Prisms fm. ligroin. (235). - Boils without dec. (235). - Easily sol. in ord. solvents w. blue fluorescence. (235).</p>
390	236 93-6	<p><u>2-Phenyl-9,10-dihydro-anthracene, C₂₀H₁₆.</u> - CrO₃ g. 2-phenyl-anthraquinone. (236).</p>
392	237 94-5	<p><u>9,10-Dihydrophenanthrene, C₁₄H₁₂.</u> - B.P.=313-5. (237). - Oxid. g. phenanthraquinone. (237). - Picrate, brick-red needles, M.P.=135-7°. (237). B.P.=279° (241,242). - Odor like naphthalene. (614). - Long needles fm. alc. (23).</p>
394	226, 241,242 95	<p><u>Acenaphthene, C₁₂H₁₀.</u> ^{D₉₅} = 1.069. (230). - ^Dn_D²⁰ = 1.520 (226). - Na₂Cr₂O₇+H₂SO₄ g. 40% yield of naphthalene-1-8-dicarboxylic ac. (220). - Htd. w. equal wgt. of conc. H₂SO₄ for 2 hrs. on H₂O bath, then 0.5 hr. at 115-20° g. sulfonic ac., needles, soften at 80°, M.P. = 87-9°; cannot be further sulfonated; K salt g. w. Me₂SO₄ the methyl ester, needles, M.P.=122-3°; ethyl ester, prismatic needles, M.P.=87-8°. (221). - 30 g. boiled 3 hrs. w. 600 cc. HNO₃(d=1.2) g. O₂N.C₁₀H₅O₂ ("nitro-α-naphthoquinone") + 4-nitro-naphthalic ac. (separated fm. former by NaOH; the former, yellow-red needles, M.P.=208° wh. by stg. w. PhNH₂ g. PhNH₂(O₂N)C₁₀H₄O₂, dark violet needles, M.P.=128° (dec.) (243). - Soln. of hy. in Et₂O (244) or boil. HCCl₃ (245) + Br g. 5-brom cpd., plates fm. EtOH, M.P.=52-3°, (244,245). - B.P.=335°. (245), whose picrate, needles, M.P.= 137°. (245). - 25 g. hy. in 250 cc. AcOH treated w. 50 cc. colorless HNO₃ (d=1.47-1.48) g. yellow needles fm. ligroin, M.P.=106°. (246). - Cpd. w. 2,4-dinitro-toluene(1:1), M.P.=60°</p>

DIVISION A, SECTION 2

No.	Melting Point	Hydrocarbon
		(247). - Cpd. w. T.N.T. (1:1), M.P.=109°. (247). - Cpd. w. picramide (1:1), M.P.=195.4°. (192). - Cpd. w. picryl chloride, M.P.=113.2° (1:1). (240). - Styphnate (1:1), M.P. =156° (g. 2 eutectics also) (191) - Picrate, M.P.=160.8°, (248), Test 911. - Benzal-chloride-H ₂ SO ₄ g. intense-dark blue color. (212).
396	95 ²⁴⁹	<u>9-Methylene-fluorene, "Diphenylene-ethylene", C₁₄H₁₀.</u> -Orange-red xtals. fm. AcOHEt. (249). - CrO ₃ in AcOH g. a yellow quinone(?), M.P.=103°, needles. (249). - Picrate, bright-red needles, M.P.=184°. (249).
398	250, 251 95	<u>1,1,1-Triphenyl-ethane, C₂₀H₁₄.</u> - Very stable to oxid. agts. (250). - 2 hrs. htg. w. PCl ₅ at 190-200° g. β-chlor-α,α,α-triphenyl-ethane, M.P.=118°. (257). Fum. HNO ₃ (red) at -3° g. α,α,α-tris-[4-nitro-phenyl]-ethane, needles fm. AcOH, M.P.=200-2°. (250).
400	253 96-7	<u>(1,2), (3,4), (5,6)-Triendotrimethylene-benzene, "Triscyclotrimethylene-benzene", C₁₅H₁₂.</u> - Colorless xtals. fm. MeOH. (253).
402	254 96	<u>[α-Naphthyl], [β-naphthyl]-methane, C₂₁H₁₆.</u>
404	256 96.5-7	<u>trans-1,4-Diphenyl-butene-(1)-ene-(3), C₁₆H₁₂.</u> - Colorless prisms. (256). - Br in HCl ₃ or CS ₂ g. two diastereoisomeric tetrabromides wh. dec. at 197° +157-8°. (256). - Soln. in AcOH w. conc. H ₂ SO ₄ g. 1st blue, then violet-red w. blue dichroism. (256). - Cf. No. 2.
406	258 96.5-7	<u>1-Methyl-7-isopropyl(or vice versa) fluorene, "Retene-fluorene", C₁₇H₁₄.</u> - In molten state + alc. soln. shows violet fluorescence. (258). - HNO ₃ (d=1.43)+hy. in AcOH soln. boiled few min. + pptd. w. H ₂ O g. straw-yellow needles of dinitro cpd. fm. AcOH wh. blacken below 200° + m. abt. 245°. (258). - Completely destroyed by CrO ₃ in AcOH. (23).
408	259 97	<u>1,2-Dimethyl-4,5-diphenyl-cyclohexane, C₂₀H₂₄.</u> - B.P.= Abt. 270°. (259). - /gm. Sol. in 12 cc. boil. EtOH + in 6 cc. boil. pet. ether. (259).
410	255 97-8	<u>2-Methyl-3-6-diphenyl-hexatriene-(1,3,5), C₁₉H₁₄.</u>
412	23 98.5	<u>Ethylene diphenyl ether, C₁₄H₁₄O₂.</u>
414	260 98.5-9	<u>1-Methyl,7-isopropyl-phenanthrene, "Retene", C₁₈H₁₄.</u> - B.P.=390°. (261). - Odorless. (214). - Micaceous leaflets. (23). - Sublime below B.P. (262). - KMnO ₄ in ac., alk. or neutral soln. attacks only

DIVISION A, SECTION 2

No.	Melting Point (C°)	Hydrocarbon
416	<p>see 99</p>	<p>when temp. reaches 150°. (253). - $K_2Cr_2O_7 + H_2SO_4$ g. retene-quinone, AcOH, phthalic ac. and other products. (252, 253, 254). - CrO_3 in AcOH g. best yield of retene-quinone; beside an ac., $C_{18}H_{18}O_2(?)$, M.P.=222°; + an ac. $C_{18}H_{18}O_3$, M.P.=139°. (252). - Htg. 100 g. retene w. 28 g. S at 240° g. $C_{18}H_{18}S$, M.P.=225-6°, plates fm. C_6H_6 wh. w. fum. H_2SO_4 g. indigo blue color. (250). - Covering hy. w. H_2O + slow addn. of 2 mols Br w. htg. on H_2O bath till HBr evolution stops + xtaln. fm. CS_2 g. plates of dibrom-retene, M.P.=180°. (252). - Xs. Br in open dish on H_2O bath g. tetrabrom cpd., M.P.=210-2°. (252). - Cpd. w. picramide, M.P.=125.1°. (192). - Cpd. w. picryl chloride, M.P.=53.5°. (240). Styphnate, M.P.=135.7° (2 eutectics formed also). - (191). - Picrate, M.P.=120.9°. (239). $HCHO + H_2SO_4$ g., in presence of $HCCl_3$, dirty blue-green ppt. (512).</p> <p><u>α-Dypnopinalkolene, $C_{25}H_{42}$</u>. - B.P.₄₀=292-5°. (257). - Changes slowly in light and g. off benzaldehyde-like odor. (257).</p>

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
418	99-100 ²⁶⁸	Di-(<i>L</i> -indenyl), $C_{18}H_{14}$, -- Br at 0° in $CHCl_3$ g. 2 tetrabromides, m.p.: 222-4° (dec.), insol. in $CHCl_3$, + m.p. = 138-9°, sol. in $CHCl_3$. (268).
420	100 ²⁶⁹	Dibenzo - 2,3;6,7 - bicyclo - (0,3,3) - octadiene - (2,6), "Diphensuccindene", $C_{16}H_{14}$. -- Difficultly volatile w. steam. (269).
422	100-1 ²⁵⁷	2,3 - Dimethyl-naphthalene, "Guajene", $C_{12}H_{12}$. Sublimes in plates wh. show blue fluorescence - (257). Sol. in conc. H_2SO_4 w. green color and not pptd. by H_2O (257). - CrO_3 in AcH g. guajene - quinone, $C_{12}H_{10}O_2$, sublimes in citron yellow needles, m.p.: 121-2°. (257) - Picrate, needles, m.p. = 123°. (659).
424	100.5 ²³	xanthene, $C_{13}H_{10}O$. - B.P.: 315° corr. - Leaflets fm. alc. (23) - Sol. in conc. H_2SO_4 w. yellow color + green fluorescence. (23).
426	100.5 ²⁷⁰	2,4,6,2',4',6' - Hexamethyl-diphenyl, $C_{18}H_{22}$. - B.P. = 270°. (270).
428	100.5 ²⁷²	Phenanthrene*, $C_{14}H_{10}$. - B.P.: 340°. (273). Sublimes less readily than anthracene. (614). Leaflets. (23). Xtals. show blue fluorescence, weaker than of anthracene. (273). - Solns. show blue fluorescence. - (274). CrO_3 in $AcOH$ or $K_2Cr_2O_7$ + dil. H_2SO_4 g. phenanthraquinone. - (273, 275). Fused w. KOH shows green luminescence. - (276). Cpd. w. 4 - chlor - 1,3 - dinitro-benzene, orange needles fm. $EtOH$, m.p.: = 44°. (277). Cpd. w. 1,3,5 - trinitro-benzene, m.p. = 125°. (278). Cpd. w. picryl chloride, citron yellow needles, m.p. = 88°. (177). Cpd. w. T.N.T., m.p. = 87.5°. (278). Picrate, golden-yellow needles, m.p. = 143°, (275), Test 916. Br in boil+ HCl_3 in diffused light g., beside other products, needles of dibrom cpd. fm. $EtOH$, m.p. = 146°. (279). - Styphnate, m.p. = 132.7°. (191). - Cpd. w. picramide, m.p. = 160.2°. (192). + Sol. in 10 pts. hot alc. (23). - $HCHO$ + H_2SO_4 g. blue-green color + blue ppt. (612). - Benzal-chloride + H_2SO_4 g. carmine-red. (612). - Hot $PhNO_2$ soln. g.w. <i>L</i> -dinitro-phenanthraquinone g., by cooling, brown prisms. (612). - 0.1 g. hy. in 1-2 ccs. CCl_4 + dropwise addn. of $SbCl_3$ in CCl_4 (1:2 vols.) g. brown color. (613). - $SbCl_3$ g. faint-green color. (614). - $BiCl_3$ g. brown or greenish-brown color. (614). - G. GB-B color w. $AlCl_3$ in Test 904. (23).

Division A, Section 2.

No.	Melting Point (C°)	Hydrocarbon
430	101 ²⁷¹	9, 10, X, X - Tetrahydroanthracene, " <u>γ-Tetrahydroanthracene</u> ", C ₁₄ H ₁₄ . -- Plates. (271). - Easily sol. in ord. solvents without fluorescence. (271). CrO ₃ g. 9, 10 - dioxy-anthracene-dihydride. (271). - Br at ord+ temp. g. dibrom cpd., faintly yellow needles, m.p. = 169°. (271).
432	101.5 ²⁸⁵	1,1,4 - Triphenyl-butadiene - (1,3), " <u>β-Phenylbisteryl</u> ", C ₂₂ H ₁₈ . -- Needles fm. EtOH. (285). - Br in HCCl ₃ g. a monobrom cpd., white xtals, m.p. = 146-8°. (285).
434	102-2.5 ²⁸⁰	2-Phenyl-naphthalene, C ₁₆ H ₁₂ . -- B.P. = 345-6°. (281, 282). - Sublimes. (283). - Xtals. show blue fluorescence. (283). - vapors show odor of oranges. (283). - CrO ₃ in AcOH g. 2-phenyl-naphthaquinone - (1,4). (281). - Volatile w. steam+ (23). - SbCl ₃ g. no color. (614).
436	102 ²⁸⁴	1-(1 ³ -Phenyl-propene-(1 ²)-ylidene)-cyclopentadiene-(2,4), " <u>Cinnamylidene-cyclopentadiene</u> ", " <u>ω-Styrylfulvene</u> ", C ₁₄ H ₁₂ . -- Blue-red needles fm. MeOH. - (284). - Sol. in conc. H ₂ SO ₄ w. violet color + blue fluorescence. (284).
438	103-8 ²⁸⁷	9,10-Diisoamylidene - 9, 10-dihydroanthracene, C ₂₄ H ₂₈ . -- Yellow prisms fm. EtOH. (287). - Sol. in solvents w. blue fluorescence. (287).
440	103.5 ²⁸⁹	9-(α -Naphthyl)-fluorene, C ₂₃ H ₁₆ . -- Insol. in H ₂ SO ₄ . (289).
442	104 ^{289, 290}	9-Ethylidene-fluorene, C ₁₅ H ₁₂ . -- Insol. in conc. H ₂ SO ₄ . (289). Dec. by distn. at ord+ Press+ (289).
444	104-6 ²⁸⁶	2-Benzyl-fluorene, C ₂₀ H ₁₆ . -- Plates fm. EtOH. (88).
446	105 ²³	<u>β-Naphthyl ether</u> , C ₂₀ H ₁₄ O. -- B.P. = abt. 360°. (23). - Picrate, orange leaflets fm. warm ether, m.p. = 122°. (23).
448	105 ²³	Sequoiene, C ₁₃ H ₁₀ . - Occurs in needles of Californian Sequoia gigantea. (23). - Odorless leaflets w. faint bluish fluorescence. (23). - Red picrate. (23). - B.P. = 290-300°. (23).
450	105-6 ²⁹¹	1,2-Di-(2,4-dimethophenyl)-ethene, " <u>2,4,2',4'-Tetramethyl-stilbene</u> ", C ₁₈ H ₂₀ . -- Distills without dec. (291). - Boil w. dil. HNO ₃ g. 2,4-dimethylbenzoic ac. (291). - Adds Br directly. (291).

DIVISION A, SECTION 2.

No†	Melting Point (C°)	Hydrocarbon
452	108 ²⁹² -9	2,3-Diphenyl-indene, C ₂₁ H ₁₆ . -- Columns. (292). H ₂ SO ₄ g. exceedingly intense dark green color. (292). - NaOEt+C ₅ H ₁₁ ONO g. 2,3-diphenyl-indone oxime, yellow-brown columns, m.p. = 253-5°, dec. (292).
454	108 ²⁹³	3-Ethyl-1,2,3-triphenyl-indene, C ₂₉ H ₂₄ . -- Plates fm. Me ₂ CO+EtOH. (293). - Careful oxid. w. CrO ₃ in cold AcOH g. Ph.COC ₆ H ₄ .C(Et)(Ph).CO.Ph. (293). - Oxid. in hot AcOH g. o-dibenzoyl-benzene. (293).
456	108.5 ²⁹⁵	9,10-Dihydroanthracene, C ₁₄ H ₁₂ . -- B.P. = 305°. (122). - D ₄ = 0.898. (295) - Solid shows no fluorescence, but solns. fluoresce blue. (122). - Sublimes in needles (122). at temp. of boil aq. (614). - CrO ₃ mixt. g. anthraquinone. (122). - Dry Br g. 9,10-dibrom-anthracene. (122). - G. no picrate. (122). - Htd. w. conc. H ₂ SO ₄ g. SO ₂ +anthracene. (Test 912.) (23).
458	109 ^{288,297}	Di-(α -naphthyl)-methane, C ₂₁ H ₁₆ . -- B.P. = 270-2°, (298); abt. 360 ²⁹⁰ (23). - Distills without dec. above 360°. (297). + very difficultly attacked by CrO ₃ mixt. (297). - Br at 135-45° g. di- α -naphthyl-brom-methane, prisms fm. C ₆ H ₆ , m.p. = 181-2°. (298). - Br in ether soln. w. cooling g. dibrom deriv., m.p. = 193°, needles fm. alc.-C ₆ H ₆ . (297). - Picrate, red-yellow prisms, m.p. = 142-3°. (297). - S+ in 15 pts. hot + 120 pts. cold alc. (23).
460	109-10 ²⁹⁴	" χ -Ethyl-phenanthrene", " α -Ethyl-phenanthrene", C ₁₆ H ₁₄ . -- Plates fm. MeOH. (294). - Picrate, light orange spears, m.p. = 138-40° corr.
462	110 ²³	α -Naphthyl ether, C ₂₀ H ₁₄ O. -- Picrate, m.p. = 115°. (23). - Solns. show pale bluish fluorescence. (23). - Distills. (23).
464	110-1 ²⁹⁹	2,6-Dimethyl-naphthalene, C ₁₂ H ₁₂ . -- Plates fm. EtOH. (299). - Faint odor of orange blossoms. (299). Very easily volatile w. steam. (299). - Picrate, orange-yellow needles, m.p. = 142-3°. (299).
466	110 ³⁰⁰	Fluoranthene, "Idryl", C ₁₅ H ₁₀ . -- B.P. = 250-1°. ((301)). - Sublimes in flat needles. (300). Sol. in warm conc. H ₂ SO ₄ w. green-blue color, (300), changing at higher temp. to blue, + finally brown. (614). Boil. w. CrO ₃ mixt. g. fluoranthene-quinone + fluorenone carbonic ac. - (1). (301,302). - Careful addn. of Br in CS ₂ soln. g. yellow-green needles of dibromfluoranthene, m.p. = 204-5°. (302). - Picrate, reddish-yellow needles, m.p. = 182-3°. (302).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
468	110-1 ³⁰³	9-Benzyl-9,10-dihydroanthracene, $C_{24}H_{18}$. - Needles fm. EtOH. (303). - Soln. in H_2SO_4 is dark green. (303).
470	110.5-11 ⁶⁴³	1,2,2-Tetramethyl-3,6-endomethylene-bicyclo-(0,1,3)-hexane, "Methyl-tricyclene", $C_{11}H_{18}$. -- B.P. = 168-8.5°. (643).
472	111 ²³	Benzhydryl ether, $C_{26}H_{22}O$. - B.P. = 315°. (23). - Monoclinic xtals. fm. C_6H_6 . (23). - Boil. w. glac. AcOH, Zn + little HCl g. tetraphenylethane, (23), No. 838.
474	111-2 ³⁰⁴	1-(Diphenyl-methylene)-2,3-benzo-cyclopentadiene - (2,4), "8,8-diphenyl-benzofulvene", $C_{22}H_{16}$. -- Orange-yellow scales. (304).
476	111 ³⁰⁵	1,3,5-Triphenyl-cyclohexadiene-(1,3), $C_{24}H_{20}$. -- Needles fm. EtOH. (305). - $KMnO_4$ oxid. g. 1,3,5-triphenyl-benzene, (305), Div. A, sect+ 2, No. 700.
478	112-3 ³⁰⁶	Diphenyl-(p-diphenyl)-methane, "p-Benzhydryl-diphenyl", "Diphenyl-p-xenyl-methane", "4-Phenyl-tritan", $C_{25}H_{20}$. -- Long needles. (306).
480	112-3 ³⁰⁷	4-Benzyl-acenaphthene, $C_{19}H_{16}$. -- B.P. = 340-5°. (307). - Needles fm. EtOH. (307). - Sol. in cold conc. H_2SO_4 w. yellow color. (307). - $Na_2Cr_2O_7$ in AcOH g. mixt. of anhydrides of 3-benzyl - + 3-benzoyl-naphthalene dicarboxic acs. - (1,8). (307).
482	113 ^{648,649}	1,7,7-Trimethyl-bicyclo-(1,2,2)-heptene-(2), "Bornylene", $C_{10}H_{16}$. -- B.P. = 146.50° (648,649). - (5) (2°) -22.27° (649). - 1% aq. $KMnO_4$ (666) or $KMnO_4$ in C_6H_6 (648) g. camphoric ac. (648,666). - Repeated shaking w. dil. aq. HOCl, drying, distn., + xtaln. fm. satd. MeOH at 0° g. bornylene chlorhydrin, m.p. = 99-101, odor of borneol, wh. g. p-nitrobenzoate, m.p. = 152°, + wh. w. Zn dust + ht. g. borneol, + wh. w. CrO_3 g. a chloroketone, $C_{10}H_{15}OCl$, m.p. = 131-2° whose semicarbazone, m.p. = 223° (probably identical w. that fm. camphene chlorhydrin). (665). - The chlorhydrin w. KOH g. glycol, $C_{10}H_{16}(OH)_2$ whose dip-nitrobenzoate m. 103-5°. (665). - $NaNO_2$ + AcOH g. bornylene-nitrosite, needles, m.p. = 163°, wh. w. $KMnO_4$ g. camphoric ac. = HNO_3 g. camphoric ac. + 1,2-dinitro-camphane, pale yellow needles, m.p. = 137° wh. w. alk- $KMnO_4$ g. camphoric ac. (667).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
484	113 ³⁰⁸	<u>4,4'-Dibenzyl-diphenyl, C₂₆H₂₂.</u> -- Plates fm. EtOH. (308).
486	114-5 ³⁰⁹	<u>1,1,4,4-Tetraphenyl-butane-(2), "Tetraphenyl-crotonylene", C₂₈H₂₂.</u> --
488	115 ³¹⁰	<u>9,9-Dibenzyl-9,10-dihydroanthracene, C₂₈H₂₄.</u>
490	115 ³¹¹	<u>Fluorene*</u> , C ₁₃ H ₁₀ . -- B.P. = 293-5° † (312). - Faint fluorescence. (311). - CrO ₃ in AcOH g. fluorenone. (317). - Br + cold HCCl ₃ soln. of hy. g. x-bromofluorene, m.p. = 101-2°. (314). - Insol. in cold conc. H ₂ SO ₄ ; sol. on hgt†, w. beautiful blue color. (315). - Soln. of 30 g. hy. in 250 cc. AcOH, addn. at 50°, w. shak. of 40 cc. HNO ₃ (D. :1.4) + hgt. to 80-5° g. 2-nitrofluorene, m.p. = 156° wh. w. CrO ₃ in AcOH g. 2-nitro-fluorenone. (316). - Hy. + equal vol. fum. HNO ₃ + AcOH g. 2,7-dinitro-fluorene, m.p. = 199-201°. (317). - Cpd. w. picryl chloride, orange-yellow needles, m.p.: 69-70° (172). ^{Unstable cpd. w. picramides} m.p. = 127-5° (dec.). (192). - Picrate, red or red-brown, m.p. = 79-80° † (317). - SbCl ₅ in CCl ₄ g. green color. (313). - Cpd. w. 1,3,6,8-β-tetranitronaphthalic ac. by 15 min. boil. w. xs. ac. in alc., dull, brown-yellow needles, m.p. = 154-5°. (318).
492	115 ³¹⁸	<u>Pseudophenanthrene, C₁₆H₁₂.</u> -- Glistening, non-fluorescing plates. (318). - CrO ₃ + AcOH g. yellow quinone, m.p. = 170°. (318). - Picrate, light-red needles, m.p. = 147°. (318).
494	115-6 ³¹⁹	<u>2-Methyl-1,6-diphenyl-hexatriene-(1,3,5), C₁₉H₁₈.</u> - Leaflets fm. EtOH w. faint blue fluorescence † (319).
496	115 ⁶³⁵	<u>Octodecahydrochrysene, "Chrysene-perhydride", C₁₈H₃₀.</u> -- Fine needles fm. EtOH. (635). - Unattacked by Br + HNO ₃ (d.:1.48). (635).
498	116-7 ²³	<u>Di-o-oxyhydrobenzoin-diesoanhydride, C₁₄H₁₀O₂.</u> -- Needles fm. alc. (23). - Unattacked by dil. HCl or NaOH. (23).
500	116-7 ^{636, 637}	<u>2,2,5-Trimethyl-bicyclo-(1,2,2)-heptane, 5-methyl-camphenilane, C₁₀H₁₈.</u> -- Volatile, camphor-like odor. (636).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
502	^{644, 645} 117-7.5	2,2,3-Trimethyl-3,6-endomethylene-bicyclo-(0,1,3)-hexane?, "isocyclene", " β -Bornylene", $C_{10}H_{16}$. -- Sublimes. (644). - B.P. = 150.5-1.5°. (645). - Unattacked by $KMnO_4$ at rm. temp. (644).
504	117 ³²⁰	3-Methyl-1,6-diphenyl-2-benzyl-hexatriene-(1,3,5), $C_{26}H_{24}$. -- Needles† (320).
506	117 ³²¹	Methanthrene, $C_{15}H_{12}$. - B.P. = above 360°. (321). - Colorless xtals. w. violet fluorescence. (321). - CrO_3 + AcOH g. methanthrene quinone. (321). - Picrate, orange-red xtals., m.p. = 117°, dec† by alc. (321).
508	117-7.5 ³²²	1,4-Dimethoethenyl-cyclo-hexadiene-(1,4), $C_{12}H_{20}$. - Plates fm. EtOH. (322). - Adds 4 atoms of Br in $CHCl_3$ g. an oil. (322). - In glac. AcOH calcd. amt. Br g. little cubical xtals. of tetrabromide, m.p. = 107-9°. (322).
510	118 ³²³	Phenyl-di-(2-methophenyl)-methane, $C_{21}H_{20}$.
512	118 ³²⁴	γ -Methylene-diphenylene, $C_{13}H_{10}$. -- B.P. = 295°. (324). - Plates fm. EtOH. (324). - Solns† have faint blue fluorescence. (324). - Picrate, blood-red needles, (324), m.P. = 79-81°. (324). - CrO_3 g. a golden-yellow quinone, m.p. = 280-1°. (324). - Br + ether soln. g. dibromide, m.p. = 162°. (324).
514	118-24 ⁶⁴⁶	Polly's Polymer, * ($C_{10}H_{10}$) ₈₋₉ . - Cycloized polymeride of 1-phenyl-butadiene-(1,3) (Cf. No. 6). (646). M.W.: 1070. (646). - Sol† in ether, C_6H_6 . (646). - Insol. in alc. (646). - White amorphous powder† (646). - O_3 g. benzoic ac. (646).
516	119 ¹¹⁵	2-Methyl-9-phenyl-anthracene, $C_{21}H_{16}$. -- Yellow crystalline clusters. (115). - Dil† alc. soln. fluoresces strongly green-blue. (115). - CrO_3 in AcOH easily g. methyl-phenyl-oxanthranol. (115).
518	119 ³²⁵	9-Benzyl-anthracene, $C_{21}H_{16}$. -- Needles fm. EtOH. (325). - Solns. fluoresce blue. (325). - Soln. in H_2SO_4 is green w. red fluorescence. (325). - Br + CS_2 soln. g. yellow prisms fm. C_6H_6 , dec† at 113-4° + show blue fluorescence in C_6H_6 . (325).
520	120 ³²⁶	1-Phenyl-2-(p-tolyl)-ethene, "4-Methyl-stilbene", $C_{15}H_{14}$. -- Plates w. blue fluorescence† (326). - Br g. addn. product, m.p. = 186-7°. (326). - Distils. (23).

Division A, Section 2.

No.	Melting Point (C°)	Hydrocarbon
522	120-0.5 ³²⁷	9-Phenyl-9,10-dihydro-anthracene, $C_{20}H_{16}$. -- Distills without dec. (327). - Solns. have blue fluorescence. (327). - CrO_3 in AcOH g. phenyl- oxanthranol. (327). - Htg. w. HI+P g. an xtaln. hy., m.p. = 86-8°. (327). - Picric ac. g. brown- red xtaln. cpd. (327).
524	121 ³³²	1,1,4,4-Tetraphenyl-butane, $C_{28}H_{26}$.
526	121-2 ⁶³¹	1-Methyl-2,3,5-triphenyl-cyclo-pentane, $C_{24}H_{24}$. -- Needles im. EtOH. (631). - (Cf. Div. B, Sect.1, No. 1018.
528	122 ^{328, 331}	4,4'-Dimethyl-diphenyl, $C_{14}H_{14}$. -- B.P. = 295°. (328). - Glassy prisms im. ether. (23). - CrO_3 in AcOH g. p-tolyl benzoic ac. + then diphenyl- dicarbonic ac. -(4,4'). (329, 330).
530	122 ³³³	4,4'-Di-ter-butyl-diphenyl, $C_{20}H_{26}$.
532	123 ³³⁴	1-Methyl-phenanthrene, $C_{15}H_{12}$. -- Plates fm. dil. EtOH+ (334). - Stable to $KMnO_4$. (334). - CrO_3 g. 1-methyl-phenanthraquinone. (334). - Picrate, yellow needles (fm. alc.), m.p. = 139°corr. (334).
534	124 ^{336, 345}	1,2-Diphenyl-ethene*, "Stilbene", "Toluylene", "Solid distyrene", $C_{14}H_{12}$. - B.P. = 306-7°. (335). - $D_{13} : O \dagger 970$. (337). CrO_3 mixt. g. benzaldehyde + benzoic ac. (338). - Na + EtOH g. dibenzyl. (339). -- Br in CS_2 g. 2 stereoisomeric dibromides, m.p. = 237° + m.p. = 110-10.5°. (338, 340). - Nitrosyl chloride, by $NOCl$, $CHCl_3$ + hy. at -10°, amorphous, m.p. = 138-9° (dec.). (341). - Nitrosite by NO_2 + cold ether soln. of hy., softens at 160°, m.p. = 195-7°. (342). - Dimer by long ex- posure to light in C_6H_6 soln., m.p. = 163°, not oxid. by $KMnO_4$. (343). - Pseudo-nitrosite by passing gases fm. As + HNO_3 (d:1:23) into 10 g. hy. in 350 cc. ether for 2-3 hrs. + extracting product w. 40 cc. Me_2CO fine needles, m.p. = 132°. (344). - Cpd. w. picrylchloride in alc. soln., m.p. = 70-1°, dark yellow needles+ (177). - At 40° slightest trace w+ $SbCl_3$ g. orange color, destroyed at higher temp. (614). - Benzal-chloride + H_2SO_4 g. bluish-green color. (614).
536	124 ⁵³⁵	1,8-Diphenyl-octatetraene-(1,3,5,7), $C_{20}H_{18}$. -- Shows blue fluorescence in solid state. (533). - Alk. $KMnO_4$ g. PhCHO + benzoic ac. (533). - Cf. No. 874.

DIVISION A, Section 2.

No.	Melting Point (C°)	Hydrocarbon
538	^{339, 348} 126	<u>2,3-Diphenyl-butane, C₁₆H₁₈.</u> -- Plates fm. EtOH w. bluish lustre. (339). - Oxid. g. acetophenone. (348). - Unaffected by Na-K in ether over 48 hrs. (653).
540	126-7 ²³	<u>Hydrobenzoin anhydride, C₂₈H₂₄O₂.</u> -- Htg. at 250-70° g. PhCHO + Stilbene, (23), No. 534. † Non-volatile w. steam. (23).
542	127-8 ³⁴⁹	<u>1,3-Dimethyl-2,4,5-triphenyl-cyclo-pentadiene-(3,5), C₂₅H₂₂.</u> -- Needles fm. EtOH. - HI+P g. 2 stereoisomers of dimethyl triphenyl cyclopentane†
544	127 ³⁵⁰	<u>Hexahydropyrene, C₁₆H₁₆.</u> -- G. no picrate in EtOH. (350).
546	127-8 ³⁵¹	<u>1,1,3,3-Tetraphenyl-propene-(1), C₂₇H₂₂.</u> -- Soln. in conc. H ₂ SO ₄ is yellow. (351). - Na + EtOH g. 1,1,3,3-tetraphenyl-propane. (351). - Br in HCCl ₃ g. 2- or 3-brom deriv., m.p. (fm. Me ₂ CO-alc.) = 124°, wh† w. alc. KOH g. tetraphenyl-allene, (351), No. 692.
548	127.5-8.5 ³⁵²	<u>2,2-Diphenyl-butane, C₁₆H₁₈†</u>
550	128 ³⁵³	<u>9-Tolyl-fluorene, C₂₀H₁₆.</u> -- Silky needles. (23, 353). - G. no picrate. (353).
552	128 ³⁵⁵	<u>Tri-ter-butyl-benzene, C₁₈H₃₀.</u> -- B.P. 292-3°. (354). - Scales fm. EtOH. (354).
554	128+5-9 ³⁵⁵	<u>1-(Diphenyl-methylene)-dimethyl-benzo-4,5-cyclo-heptatriene-(2,4,6), C₂₆H₂₂.</u>
556	129 ³⁵⁶	<u>Hexaethyl-benzene,* C₁₈H₃₀.</u> -- B.P. = 305°. (357). D ₄ ²⁰ : 0.831. (358). - Conc. H ₂ SO ₄ + fum. HNO ₃ g. 3,6-dinitro-1,2,4,5-tetraethyl-benzene. (359). - May be rextalized fm. warm fum. H ₂ SO ₄ . (23).
558	129-30 ³⁶⁰	<u>(1,8),(4,5)-Diendotrimethylene-naphthalene?, "Di-peri-trimethylene-naphthalene?", "Hexahydropyrene", C₁₆H₁₆.</u> -- Picrate fm. alc. soln., red unstable needles, m.p. = 119°. (360).
560	130-1 ³⁶²	<u>2,2,7,7-Tetramethyl-octadiene-(3,5), C₁₂H₁₈†</u>
562	130 ³⁶³	<u>Di-(2,4,6-trimethophenyl)-methane, "2,4,6,2',4',6'-Hexamethyl-ditan", C₁₉H₂₄.</u>

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
564	130-1 ¹⁸³	9-Benzyl-fluorene, C ₂₀ H ₁₆ . -- Benzaldehyde + conc. H ₂ SO ₄ g. violet color wh. w. a little H ₂ O g. blue color wh. w. much H ₂ O disappears. (183). - W. benzyl-chloride + KOH at 230° g. 9,9-dibenzyl-fluorene. (183).
566	131-2 ⁶⁵³	2,2,3,3-Tetramethyl-1,4-diphenyl-butane, C ₂₀ H ₂₆ . -- Unaffected by Na-K in dioxane at 100°. (653).
568	131-2 ³⁶⁴	1,2-Di-(4-isopropophenyl)-ethene, C ₂₀ H ₂₄ .
570	131.5 ³⁶⁵	Di-m-xylylene, C ₁₆ H ₁₆ . -- B.P. = 290°. (365). - Br in CS ₂ g. dibrom deriv., m. p. = 213-4°. (365).
572	132-3 ³⁶⁶	3,4,5,3',4',5'-Hexamethyl-diphenyl, C ₁₈ H ₂₂ .
574	132-7 ²⁸⁷	9,10-Diisocamyl-anthracene, C ₂₄ H ₃₀ . -- Yellow-green needles fm. EtOH. (287).
576	132 ²³	Metanthole, (C ₁₀ H ₁₂ O) _x . -- Sublimes at 115-20°. (23). - Non-volatile w. steam. (23). - Needles fm. ether. (23). - CrO ₃ oxid. g. AcOH. (23).
578	134-5 ³⁵¹	1,3,3-Triphenyl-indene, C ₂₇ H ₂₀ . -- Careful oxid. w. CrO ₃ in cold AcOH g. o-benzoyl-triphenyl-acetic ac. (293). - CrO ₃ + boil. AcOH g. o-dibenzoyl-benzene. (351). - HI+P g. hy. C ₂₇ H ₂₂ (?), needles fm. EtOH, m.p. = 113-4°. (351). - Br in HCCl ₃ g. brom deriv., m.p. = 167-8°, fm. aq. - Me ₂ CO or EtOH. (351).
580	134 ³⁶⁷	Diphenyl-(λ -naphthyl)-methane, C ₂₃ H ₁₈ . -- Xtalizes, according to solvent in two modifications, m.p. = 134 + m.p. = 149° (No. 630) wh. can be transformed into one another. (367).
582	134.5 ³⁸	1,2-Di-(p-ethophenyl)-ethene, "4,4'-Diethyl-stilbene", C ₁₈ H ₂₀ . -- Plates fm. EtOH. (38). - Boil. w. dil. HNO ₃ g. terephthalic ac. (38).
584	135 ²⁹³	1,2,3-Triphenyl-indene, C ₂₇ H ₂₀ . -- Colorless prisms fm. Me ₂ CO + ether. (293). - Very little sol. in cold conc. H ₂ SO ₄ . (293). - CrO ₃ in AcOH at ord. temp. g. a little o-dibenzoyl-benzene + mostly 3-oxy-1,2,3-triphenyl-indene; in boiling AcOH g. only o-dibenzoyl-benzene. (293). - Br vapor passed thru hy. at 150-60° g. 3-brom-1,2,3-triphenyl-indene, yellow plates, m.p. = 129°. (293).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
586	136-7 ³⁶⁹	9-Phenyl-9-benzyl-fluorene, $C_{26}H_{20}$. -- Prisms fm. AcOH or ligroin. (369). - Solns. fluoresce blue. (368).
588	136 ³⁷⁰	Di-(p-tolyl)-ethine, "4,4'-Dimethyl-tolane", $C_{16}H_{14}$.
590	136 ³⁷¹	1,1-Di-(α -naphthyl)-ethane, $C_{22}H_{18}$. -- Plates. (371). - Solns. fluoresce violet. (371). - Br in CS_2 w. sunlight g. dibrom deriv., needles fm. CS_2 , m.p. = 215°. (371).
592	137 ³⁷²	9-Phenyl-3,4-benzofluorene, $C_{23}H_{16}$. -- Leaflets fm. AcOH. (372).
594	137-7.5 ⁴⁸	1-Benzyl-3-benzylidene-indene, $C_{23}H_{18}$. -- Yellow plates fm. EtOH. (48). - Conc. H_2SO_4 g. violet color. (48).
596	138 ³⁷³	2,5-Diphenyl-hexadiene-(2,4), $C_{18}H_{18}$.
598	139 ³⁵¹	1,1,3,3-Tetraphenyl-propane, $C_{27}H_{24}$. -- Needles fm. AcOH. (351). - CrO_3 in AcOH g. Ph_2CO + a little benzoic ac. (351).
600	139 ³⁷⁴	9,10-Dimethyl-phenanthrene, $C_{16}H_{14}$. -- Prisms fm. dil. AcOH. (374). - Sublimes without dec. (374).
602	140-1 ³⁷⁵	2-Methyl-7-p-tolyl-naphthalene, $C_{18}H_{16}$. -- Colorless leaflets. (375).
604	140-5 ²³	Anisoin, $(C_{10}H_{12}O)_x$. (Not identical w. cpd. of same name in Genus VII of Mulliken, Ident. etc.). - Small needles fm. ether. (23). - Dec. on distn. to liquid metanethole. (23). - Unattacked by dil. ac. or alk. (23).
606	141 ³⁷⁶	Benzo-1,2-anthracene, "Naphthanthracene", $C_{18}H_{12}$. -- Saw-toothed plates fm. EtOH or AcOH. (376). - Intense yellow-green fluorescence. (376). - Sublimes in plates. - $Na_2Cr_2O_7$ in AcOH g. naphthanthraquinone. (377). Picrate, red needles, m.p. = 133°, sol. in C_6H_6 , dec. by alc. (376).
608	144 ³⁷⁹	1,1,1,2-Tetraphenyl-ethane, $C_{26}H_{22}$. -- B.P. ₂₁ = 277-80°. (368). - CrO_3 in AcOH g. triphenylcarbinol. (379). - Htg. w. PCl_3 at 170-80° g. tetraphenyl-ethylene. (380). - Br g. brom deriv., xtals. fm. EtOH, m.p. = 177°. (378). - Nitration g. $\alpha, \beta, \gamma, \delta$ -tetrakis-(4-nitro-phenyl)-ethane(?), beside an isomer wh. latter m. 258°; wh. former

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
610	145-6 ⁶³⁸	yellow plates fm. AcOH, m.p. = 269°, + w. Zn dust + AcOH g. fuchsine color. (368). <u>Bicyclo-(1,3,3)-nonane, C₉H₁₆.</u> -- B.P. = 168.5-70°. (638). - White plastic mass similar to camphane in appearance + odor. (638). - Sublimes readily. (638). - Xtals. fm. AcOH or MeOH. (638).
612	145 ³⁹²	<u>1,2,4,5(?) - Tetramethyl-3-benzyl-benzene, C₁₇H₂₀.</u> ← B.P. = 325-70. (392). - Plates fm. AcOH. (392).
614	145-7 ³⁸⁶	<u>"Triphenyl-methyl," "Trityl", C₃₈H₃₀ ⇌ C₁₉H₁₅.</u> -- -- Colorless xtals, blackening immediately before m. + m.w. development of red color. (386). - Dec. by distn. at 19 mm. g. triphenyl methane. (386). - Solns. absorb 0 w. great avidity g. very slightly sol. peroxide (387) + an O-rich oil. (388). - Htd. in C ₆ H ₆ soln. w. Zn powder + AcOH g. triphenyl-methane. (389). - In C ₆ H ₆ , CS ₂ , or ligroin can be titrated approximately ^{w.} g. triphenyl-iodo-methane. (387, 390). - Tetraphenyl hydrazine in PhMe at 90-5° in atm. free of O g. (triphenylmethyl)-diphenyl-amine, lustrous needles fm. toluene, m.p. = 172°, wh. in boil. xylene is resolved into its components, + wh. w. conc. H ₂ SO ₄ g. greenish-yellow color + dec. to triphenyl-carbinol + diphenyl-amine. (391). - CO ₂ bubbled thru liquid N ₂ O ₄ + passed thru a hot tube into hy. in ether g. Ph ₃ CNO ₂ , soft, lustrous, colorless, microscopic plates, m.p. = 147°. (616). - Phenyl hydrazine g. triphenyl-methane + PhC ₃ NHNHPh, m.p. = 140°. (616).
616	145.5 ³⁸¹	<u>9-Phenyl-fluorene, C₁₉H₁₄.</u> -- B.P. = abt. 360°. (23). - Needles. (23). - Boils without dec. (382). - Solns. in EtOH + C ₆ H ₆ show blue fluorescence. (383). - Na ₂ Cr ₂ O ₇ + AcOH g. 9-oxy-9-phenyl-fluorene. (384). - CrO ₃ mixt. g. o-benzoyl-benzoic ac. (381). - "G. no picrate." (385). - Br in CS ₂ in direct sunlight g. 9-brom-9-phenyl-fluorene, m.p. = 99°, faintly yellow needles fm. ligroin. (384). - AcOH suspension + fum. HNO ₃ (d=1.52) w. cooling g. 2-nitro-9-phenyl-fluorene, yellow xtals., m.p. = 135°. (384). - Br in hot AcOH g. dibromide, m.p. = 181-2°. (23).
618	146-8 ³⁹³	<u>1,1,2,4-Tetraphenyl-butadiene, C₂₈H₂₂.</u> -- Almost colorless prisms fm. Me ₂ CO. - Na ₂ Cr ₂ O ₇ in AcOH or KMnO ₄ in Me ₂ CO g. PhCHO + triphenyl-acrolein, m.p. = 175°. (393).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
620	146.5 ³⁹⁴	<u>Dibenzyl-naphthalene, C₂₄H₂₀.</u> -- Needles fm. hot alc. (394).
622	149-50 ³⁹⁵	<u>9,9-Dibenzyl-fluorene, C₂₇H₂₂.</u>
624	149 ³⁹⁶	<u>1,2,4-Triphenyl-cyclopentadiene-(2,5), C₂₃H₁₈.</u> -- Yellow needles fm. EtOH. (396). - Sol. in H ₂ SO ₄ w. yellow-green fluorescence. (396). - 4 atms. Br in CS ₂ g. 3,5-dibrom-1,2,4-triphenyl-cyclopentadiene-(2,5), m.p. = 157°, yellow needles fm. EtOH. (396).
626	149 ³⁹⁷	<u>1,2-Di-(3-ter-butophenyl)-ethane, C₂₂H₃₀.</u> -- Plates. (397).
628	149-50 ³⁹⁸	<u>Pyrene, C₁₆H₁₀.</u> -- Sublimes. (399). - B.P. = 371° (614). - Tabular xtals fm. hot alc. (23). - Boils without dec. (399). Solns. show blue fluorescence. (398). - CrO ₃ mixt. g. pyrene quinone, C ₁₆ H ₈ O ₂ ^{m.p.:abt. 282° w. dec.} + pyrene-ac., C ₁₃ H ₆ O(CO ₂ H) ₂ . (400). - Htg. w. mixt. of equal pts. by vol. HNO ₃ (d:1.2) + H ₂ O g. nitropyrene, m+p. = 149.5-50.5°. (401). - Picrate, red needles, m.p. = 222°. (401). - Benzal-chloride + H ₂ SO ₄ g. emerald green, on stg. deep blue. (612). - SbCl ₃ g. faint greenish color. (614).
630	149 ³⁶⁷	<u>Diphenyl-(\curvearrowright-naphthyl)-methane, "Benzhydryl-naphthalene", C₂₃H₁₈.</u> -- Xtalizes, according to solvent in two modifications, m.p. = 134° + m.p. = 149°. (367). (Cf. No. 580.)
632	150-0.5 ⁶⁵³	<u>2,5-Dimethyl-3,4-diphenyl-hexane, C₂₀H₂₆.</u> -- Unaffected in boil. dioxane (for 10 min.) by Na-K. (653).
634	150 ^{614, 615}	<u>Pseudophenanthrene, C₁₆H₁₂.</u> -- Large white glistening plates wh. do not fluoresce. (614, 615). - Picrate, light-red needles, m.p. = 147°, separates on mixing satd. cold alc. solns. (614, 615). - CrO ₃ g. a yellow quinone, m.p. = 170°, readily sol. in alc. + cold C ₆ H ₆ . (614, 615).
636	150-5 ⁴⁰²	<u>1,2,3-Triphenyl-benzene, C₂₄H₁₈.</u>
638	150-1 ⁴⁰³	<u>1,9-Diphenyl-5-(diphenyl-methylene)-nonatetraene-(1,3,6,8), C₃₄H₂₈.</u> -- Xtalizes fm. C ₆ H ₆ in yellow prisms ctg. 1+1-2 mols. C ₆ H ₆ of xtaln. (403). - Xtalizes fm. AcOH or EtOAc in golden-yellow needles of m.p. = 150-1. (403).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
640	152-2.5 ⁴⁰⁴	<u>trans-trans-1,4-Diphenyl-butadiene-(1,3), C₁₆H₁₄.</u> -- B.P. = abt. 350°. (405). - White plates w. faint blue fluorescence. (405). - Picrate, yellow-red needles (fm. AcOEt), m.p. = 152-3°. (183). -
642	152-3 ⁴⁰⁷	<u>9-Phenyl-anthracene, C₂₀H₁₄.</u> -- Leaflets fm. EtOH. (407). - B.P. = 417°. (406). - Solns. show blue fluorescence. (407). - CrO ₃ in AcOH g. w. boil. phenyl-oxanthrol. (407). - G. red picrate. (407).
644	153 ¹⁴⁶	<u>Triphenyl-(p-tolyl)-ethene, C₂₇H₂₂.</u>
646	153-4 ⁶⁴⁰	<u>1,7,7-Trimethyl-bicyclo-(1,2,2)-heptane, "Camphane", C₁₀H₁₈.</u> -- B.P. = abt. 160°. (640, 641). - Odor like borneol. (640). - Prisms fm. MeOH. (640). - 6-sided plates fm. 95% EtOH. (640). - Optically inactive. (640). - Unattacked by cold HNO ₃ , CrO ₃ , fum. H ₂ SO ₄ , + Br. (642). - Unattacked by fum. H ₂ SO ₄ at 180°. (642).
648	154-5 ^{408, 409}	<u>1-(L-Naphthyl)-naphthalene, "L, L-Dinaphthyl", C₂₀H₁₄.</u> -- Boils without dec. above 360°. (410). - Picrate, red-brown needles fm. C ₆ H ₆ , m.p. = 145°, dec. in the air. (657). - 10 g. hy. in 150 cc. AcOH + 20 g. HNO ₃ (d:1.3) g. orange-yellow leaflets, fm. C ₆ H ₆ , of nitro-dinaphthyl-(1,1'), m.p. = 188°; (411); or, warmed to 60° g. light yellow needles, fm. C ₆ H ₆ , of dinitro-dinaphthyl-(1,1'), m.p. = 188°. (411). - Calc. amt. Br 9. dibrom cpd., prisms fm. C ₆ H ₆ , m.p. = 215°. (405). - SbCl ₃ + BiCl ₃ g. no colors. (614).
650	154-6 ⁴¹²	<u>Pyrodydnopinalkolene, C₃₂H₂₂.</u> -- B.P. = 330-3°. (412). - HNO ₃ or several days boil. w. NaOEt g. dehydropyrodydnopinalcohol, C ₃₂ H ₂₂ O, m.p. = 203.5° wh. by AcCl g. its acetate, m.p. = 200° (fm. C ₆ H ₆). (412).
652	154.5 ¹⁸³	<u>9-(9³-Phenyl-propene-(9²)-ylidene)-fluorene, "9-Cinnamylidene-fluorene", C₂₂H₁₆.</u> -- Citron-yellow needles fm. AcOH. (183). - (C ₂₂ H ₁₇) _x by red. of hy. in moist ether w. Al-Hg, xtals. fm. PhNO ₂ , m.p. = 257° corr. (183). - Picrate, m.p. = 178-9° (fm. CHCl ₃). (183).
654	155-6 ¹⁹⁹	<u>9(?) -Benzyl-phenanthrene, C₂₁H₁₆.</u> -- Needles fm. C ₆ H ₆ . (199). - CrO ₃ in AcOH g. Phenanthraquinone + benzoic ac. (199).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
656	156 ⁶³⁹	<p>2,2-Dimethyl-bicyclo-(1,2,2)-heptane, "Camphenilane", C_9H_{16}. -- B.P. = 143°, d_4^{20}:0.855, n_{20}:1.456. (639). -- Dil. HNO_3 g. β-nitro-camphenylene, m.p. = 18-20°, B.P.₂₀ = 124-5°, d_4^{20}:1.027, n_{20}:1.484, + 6-nitro-camphenilone, m.p. = 89-91° wh. oxid. to β-camphenylene (isocamphenylene), m.p. = 59-65°, B.P. = 196.5 whose semicarbazone m.p. = 192-3°. (639,746).</p>
658	156 ⁴¹³	<p>1,3-Diphenyl-cyclopentadiene-(1,3), $C_{17}H_{14}$. -- Yellow needles, m.p. = 156°. (413). -- Solns. show strong blue fluorescence. (413). -- Red soln. in H_2SO_4 fluoresces intensely dark blue wh. on addn. of H_2O g. green color, finally pptg. hy. (413). -- Dry HCl colors first green, then brown, then disappears. (413). -- Ether soln. w. $FeCl_3$ g. black-brown ppt. (413).</p>
660	157 ³⁸	<p>1,2-Bis-(2,5-dimethoxyphenyl)-ethene, "Tetramethylstilbene", $C_{18}H_{20}$. -- Distills without dec. (38). -- Leaflets. (23).</p>
662	158-60 ³⁹³	<p>1,1,2,6-Tetraphenyl-hexatriene-(1,3,5), $C_{30}H_{24}$. -- Yellow prisms fm. Me_2CO. (393). -- Tetrabrom deriv. (1,2,3,4), m.p. = 148-50°, colorless xtals. (393).</p>
664	158 ⁶¹⁷ dec.	<p>Phenyl-(p-diphenyl)-(\mathcal{L}-naphthyl)-methyl-peroxide, $C_{58}H_{42}O_2$. -- Silky xtaln. powder, m. at 158°, turning brown. (617). -- Part. dec. by xtaln. fm. C_6H_6 or $CHCl_3$. (617). -- (Cf. No. 1020†).</p>
666	160-1 ¹⁸³	<p>Dimonohydro-9-(9³-phenyl-propane-(9²)-ylidene)-fluorene, "Dimonohydrocinnamylidene-fluorene", $C_{44}H_{34}$. -- Xtalizes fm. Me_2CO w. Me_2CO of xtaln., m.p. = 112-5°. (183). -- Xtalizes fm. AcOH w. AcOH of xtaln+, m.p. abt. 124°. (183).</p>
668	160 ⁴¹⁴	<p>1,2-Di-(\mathcal{L}-naphthyl)-ethane, $C_{22}H_{18}$. -- Green-yellow leaflets. (414). -- Soln. fluoresces green-blue. (414).</p>
670	161 ³⁰⁶	<p>Phenyl-di(p-diphenyl)-methane, "Phenyl-di-p-xenyl-methane", "4,4'-Diphenyl-tritan", $C_{31}H_{24}$. -- Leaflets w. mol. of C_6H_6 fm. C_6H_6. (306). -- Slender needles fm. glac. AcOH. (306).</p>
672	161 ^{418,416}	<p>1,2-Di-(\mathcal{L}-naphthyl)-ethene, $C_{22}H_{16}$. -- Yellow needles fm. EtOH. (415). -- Solns. fluoresce violet. (415). -- CrO_3 mixt. g. \mathcal{L}-naphthoic ac. (415). -- Picrate, brown-red needles, m.p. = 210. (415).</p>

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
674	161 ⁴¹⁵	1,2-Di-(2,4,5-trimethophenyl)-ethene, C ₂₀ H ₂₄ . -- Xtals. w. violet fluorescence. (415). - Br in CS ₂ g. <i>L</i> , <i>L'</i> -dibrom-2,4,5,2',4',5'-hexamethyl-dibenzyl + <i>L</i> -brom-2,4,5,2',4',5'-hexamethyl-dibenzyl. (415). - Picrate, m.p. = 123°. (415).
676	161 ⁶⁵⁰	Phenyl-di-(p-diphenyl)-methane, C ₃₁ H ₂₄ . -- Rhombic leaflets w. 1 C ₆ H ₆ fm. C ₆ H ₆ m.p. = 161°. (650). - Slender needles fm. glac. AcOH. (650).
678	161 ²³	2,6 (<i>β</i>)-Dinaphthylene oxide, C ₂₀ H ₁₂ O. -- Silvery leaflets. (23). - Sol. in conc. H ₂ SO ₄ w. red color (difference fm. <i>α</i>) wh. changes to violet + dark blue on htg. + on diln. w. aq. g. orange-red fluorescent soln. (23). - Picrate, vermilion needles, m.p. = 135°. (23). - Distils. (23).
680	162-3 ⁴⁹²	1,3,6,8-Tetramethyl-anthracene, C ₁₈ H ₁₈ . -- Oxid. g. a quinone C ₁₈ H ₁₆ O ₂ , m.p. = 206°. (492).
682	162-3 ⁴¹⁷	4,4'-Di-(diphenyl-methyl)-diphenyl, C ₃₈ H ₃₀ . -- Br in CS ₂ in direct sunlight g. 4,4'-bis-(<i>α</i> -brom-benzhydryl)-diphenyl, reddish powder, m.p. = 215-9°, dec. (417).
684	162-3 ⁴¹⁸	1-Methyl-2,3,5-triphenyl-cyclopentadiene-(1,3), C ₂₄ H ₂₀ . -- Yellow needles fm. Me ₂ CO. (418). - Sol. in conc. H ₂ SO ₄ w. yellow color + eosin like fluorescence. (418).
686	162 ⁴¹⁹	1-Methyl-2-isopropyl-5-benzylidene-cyclohexane, C ₁₇ H ₂₄ .
688	162 ⁴²⁰	Di-(p-diphenyl)-methane, "Di-p-xenyl-methane", "Diphenyl-ditan", C ₂₅ H ₂₀ . -- B.P. = 360°. (420, 421). - Sol. w. blue color in conc. H ₂ SO ₄ . (420). - CrO ₃ mixt. g. 4,4'-diphenyl-benzophenone. - "G ₂ no picrate." (420).
690	163-4 ⁴²²	1,3,5,7-Tetramethyl-anthracene, C ₁₈ H ₁₈ . -- Pale yellow plates. (422). - Picrate, m.p. = 189-90°. (422).
692	164-5 ³⁵¹	1,1,3,3-Tetraphenyl-propadiene-(1,2), "Tetraphenyl-allene", C ₂₇ H ₂₀ . -- Conc. H ₂ SO ₄ colors dark violet-brown + then goes into soln. w. green-brown-violet color, wh. on stg. turns red then orange, + by htg., colorless. (351). - Energetic oxid. w. CrO ₃ in AcOH g. benzophenone. (351). - Htg. w. acs. as AcOH or 20% HCl, or Br or I in CHCl ₃ g. 1,3,3-triphenyl-indene. (293). No. 578.

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
694	164.2 ⁴²³	9,10-Diphenyl-9,10-dihydro-anthracene(?) (424), <u>C₂₆H₂₀</u> . -- B.P. = 437°. (423). - Prisms fm. C ₆ H ₆ . (423). - Boil. 5 g. hy. w. 4 g. Br in 200 g. HCCl ₃ g. 9,10-di-brom-9,10-diphenyl-9,10-dihydro-anthracene (?). (423).
696	No ⁴²⁵ definite m.p. because of auto- oxid.	9,10-Diphenyl-9,10-dihydroanthracene, C ₂₆ H ₂₀ . -- <u>Needles fm. boil. toluene + EtOH.</u> (425). - Does not fluoresce in unchanged state. (425). - Oxid. in air to 9,10-diphenyl-anthracene, (425). - No. 900.
698	166 ⁴²⁶	Hexamethyl-benzene,* C ₁₂ H ₁₈ . -- B.P. = 265°. (426). = <u>Tabular xtals. fm. alc.</u> (23). - Cold KMnO ₄ slowly oxid. to mellitic ac. (427). - 40 g. hy. + 50 g. PCl ₅ at 100-140° g. chlorhexamethyl-benzene, leaves fm. ether-alc., m.p. = 99°, boils without dec. abt. 285°. (428). - Picrate, orange-yellow leaflets, m. p. = 170°, (650), <i>dec. by alc.</i>
700	170-1 ^{429,430}	1,3,5-Triphenyl-benzene, C ₂₄ H ₁₈ . -- Distils without dec. (430). Unattacked by boil. w. CrO ₃ mixt. (431). - CrO ₃ in AcOH g. benzoic ac. (431). - Br in CS ₂ g. brom deriv., m.p. = 104°. (430).
702	170-1 ⁴³²	<u>1,2-Diphenyl-cyclohexane, C₁₈H₂₀.</u> -- Flattened needles fm. EtOH* (432). - Sublimes. (432).
704	171 ²⁶⁶	<u>β-Isodypnopinakolene, C₃₂H₂₄.</u>
706	171 ⁴¹	<u>Di-(ℒ-naphthyl)-butadiene, "Di-(ℒ-naphthyl- diacetylene", C₂₄H₁₄.</u> -- Picrate, red needles, m.p. = 180°. (41).
708	171-1.5 ⁴³³	<u>2,6,9,10-Tetramethyl-9,10-dihydroanthracene,</u> C ₁₈ H ₂₀ . -- Tabular xtals fm. C ₆ H ₆ . (23). - CrO ₃ in AcOH g. 2,6-dimethyl-anthraquinone. (433). - M.p. = 236°. (23). - Htg. w. Zn dust g. 2,6-dimethyl- anthracene. (433). - Picrate, brown-red needles, m.p. = 165°. (433).
710	171 ⁴³⁴	<u>1,3,5-Tri-(p-tolyl)-benzene, C₂₇H₂₄.</u> -- Scarcely sol. in cold EtOH. (434). - Dil. HNO ₃ at 160-80° g. C ₆ H ₃ (C ₆ H ₄ CO ₂ H) ₃ . (434). - 3 mols. Br in CS ₂ g. tribrom deriv., m.p. = 212° u.c. (434).
712	172 ⁴³⁵	<u>1,4-Di-(diphenyl-methyl)-benzene, C₃₂H₂₆.</u> -- White needles fm. AcOH. (435). - Insol. in conc. H ₂ SO ₄ . (435).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
714	172-3 ⁴³⁶	<u>α-Ethyl-phenanthrene, "β-Ethyl-phenanthrene",</u> <u>$C_{16}H_{14}$.</u> --
716	173-6 ⁴³⁷	<u>1,5-Diphenyl-3-(diphenyl-methylene)-pentadiene-</u> <u>(1,4), $C_{30}H_{24}$.</u> -- S-yellow needles fm. AcOEt. (437). - Light yellow needles w. 1/2 mol. C_6H_6 of xtaln. fm. C_6H_6 , wh. at 130° lose their C_6H_6 g. the S - yellow modification. (437). - Solns. in hot AcOH, Me_2CO + AcOEt are yellow colored. (437). - Br in $HCCL_3$ g. tetrabromide. (437).
718	173-4 ⁴³⁸	<u>Lupeylene, $C_{30}H_{48}$.</u> -- A triterpene fm. lupeol. (438).
720	173 ⁴³⁹	<u>Phthalacene, $C_{21}H_{16}$.</u> -- $K_2Cr_2O_7$ in AcOH g. phthalacene oxide, $C_{21}H_{14}O$, or phthalacone, $C_{21}H_{12}O_2$, according to ratio of reactants. (439). - Equal pts. by wgt. of hy. + Br in AcOH g. brom deriv., m.p. = 184-4.5°. (440). - Htd. 5 hrs. at 200° w. 20 pts. HNO_3 (d.:1.035) + then 5 hrs. longer w. 10 pts. more of ac. g. 3,4,5,6-di-o-benzoylenebenzoic ac., reddish-brown xtals., m.p. = 299-300° wh. w. HI + P. g. 3,4,5,6-di-o-benzylenebenzoic ac., slightly yellow needles, m.p. = above 300°, wh. latter g. ethyl ester, m.p. = 136-7°, brown xtals. (440).
722	173-4 ²³	<u>o-Benzophenone oxide, "Xanthone", $C_{13}H_8O_2$.</u> -- B.P. = 350-10. (23). - Long needles fm. alc. (23). - Sol. in conc. H_2SO_4 w. yellow color + intense light-blue fluorescence. (23). - Fusion w. KOH g. salicylic ac. + phenol. (23). - CrO_3 mixt. g. CO_2 . (23).
724	175 ²³	<u>Tetraphenyl-furane, "Lepidene", $C_{28}H_{20}O$.</u> -- Scales. (23). Sol. in 170 pts. hot alc., 52 pts. cold ether, 85 pts. cold C_6H_6 . (23). - Br in AcOH g. dibrom deriv., leaflets, m.p. = 190. (23)
726	175-6 ⁴⁴²	<u>9,9'-Di-(p-diphenyl)-difluorenyl-(9,9')</u> , <u>$C_{50}H_{34}$.</u> -- Colorless prisms fm. C_6H_6 . (441). - G. no color reaction w. H_2SO_4 . (441).
728	175 ⁴⁴³	<u>Doeikosihdropicene, "Picene perhydride", $C_{22}H_{36}$.</u> -- BP. = above 360°. (443). - Needles fm. EtOH. (443).
730	175.5 ²⁶⁶ corr.	<u>α-Isodypnopinakolene, $C_{32}H_{24}$.</u> -- Plates or needles. (266). - CrO_3 oxid. to benzoic ac. (266). - Htg. w. molten KOH at 220° g. $C_{32}H_{20}O$ (?) fm. $HCCL_3$ + pet. ether, m.p. = 173°. (266). - Fum. HNO_3 g. $C_{32}H_{23}O_2N$, yellow xtals. fm. $PhNO_2$, m.p. = 272°. (266). - 2 atoms Br in $HCCL_3$ g. $C_{32}H_{23}Br$, fm. C_6H_6 , m.p. = 192°. (266). - 4 atoms Br in CS_2 in sunlight g. $C_{32}H_{22}Br_2$, xtals. fm. $HCCL_3$ + MeOH, m.p. = 182°. (266).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
732	176.4 ²³	Camphor*, $C_{10}H_{16}O$. -- B.P. = 205.3°. (23). - Cf. Mulliken, Ident. etc. Vol. I, VII, A, pg. 139 + test 71b.
734	177-8 ^{444, 445}	Tetraphenyl-cyclopentadiene, $C_{29}H_{22}$. -- Sol. in conc. H_2SO_4 w+ eosine color. (444). - Br in CS_2 g. $C_{29}H_{20}Br_2$, red plates, m.p. = 151.5-2°. (444).
736	178-9 ⁴⁴⁶	Tri-(β -naphthyl)-methane, $C_{31}H_{22}$. -- Xtals. fm. C_6H_6 ctg. indefinite amts. of C_6H_6 , efflorescent, dried at 100°, m.p. = 178-9°. (446).
738	178-9 ⁴⁴² dec. 379	Pentaphenyl-ethane, $C_{32}H_{26}$. -- Scarcely reacts w. Br. (379). - Htg. w. PCl_3 in C_6H_6 g. Ph_3CCl . (252).
740	179 ⁴⁴⁷	2-Methyl-9,10-diphenyl-9,10-dihydroanthracene, $C_{27}H_{22}$. -- Colorless needles fm. AcOH wh. quickly become green dichroic in the air. (447). - $K_2Cr_2O_7$ in AcOH g. 9,10-dioxy-2-methyl-9,10-diphenyl-9,10-dihydroanthracene. (447).
742	179-80 ⁴⁴⁸	1,2-Di-(p-tolyl)-ethene, $C_{16}H_{16}$. -- B.P. = 304-5°. (415). - Sublimes. (449). - Dil. HNO_3 g. p-toluic ac. (449). - CrO_3 mixt. g. terephthalic ac., (449). (Test 905-2). - Bromination in CS_2 at 0° g. unstable additive cpd. wh. loses HBr during precipitation, g. monobrom (ω) cpd., m.p. = 53-4°. (629).
744	180-1 ⁶⁵³	2,2,5,5-Tetramethyl-3,4-diphenyl-hexane, $C_{22}H_{30}$. -- Unaffected in boil. dioxane (for 10 min.) by Na-K. (653).
746	180-90 ⁴⁵¹	Di-(benzo-1,2-fluorenylidene-9), "Di-chryso-fluorenylidene", $C_{34}H_{20}$. -- Dark violet-red xtals. fm. $HCCl_3$ + pet. ether. (451). - Solns. are intense red. (451). - Br in $HCCl_3$ g. xtaln. bromide wh. by htg. in PhMe w. Na g. back orig. hy. (451).
748	180-2 ¹¹¹	9,10-Dibenzyl-phenanthrene, $C_{28}H_{22}$. -- Needles. (111).
750	180 ⁴⁵²	Diphenyl-diphenyl-methyl-peroxide, $C_{50}H_{38}O_2$.
752	abt. 180 ⁴⁵³	Phenyl-di-(α -naphthyl)-methane, $C_{27}H_{20}$. -- Powder. (453).
754	181-2 ¹⁴⁶	1-(Diphenyl-methylene)-diphenyl-benzo-4,5-cycloheptiene-(2,4,6), $C_{36}H_{28}$. -- White cubes fm. Me_2CO . (146).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
756	181-1.5 ⁴⁵⁴	9,10-Dimethyl-9,10-dihydroanthracene, C ₁₆ H ₁₆ . -- Yellow plates. (454). - Easily sublimes in light-yellow needles. CrO ₃ in AcOH g. anthraquinone + CO ₂ . (454). - Picrate, dark-red needles, m.p. 172-4°, dec., dec+ by alc. (454).
758	183 ¹⁴⁵	1,4-Di-(p-tolyl)-butadiene-(1,3), C ₁₈ H ₁₄ . -- Needles. (145). - Dibromide, needles, m. p. = 148° ⁽¹⁴⁵⁾ . ^{Tetrabromide, m.p.:} 163° (145). - Octabromide, needles, m. p. = 156-7° (145).
760	184 ⁴⁵⁵	Dimolecular dibenzylidene-anthracene, C ₅₆ H ₄₀ . -- G+ green color w. H ₂ SO ₄ . (455).
762	184 ²³	κ -Dinaphthylene oxide, C ₂₀ H ₁₂ O. -- Picrate, dark red needles, easily sol. in alc. or C ₆ H ₆ , m.p. = 173°. (23).
764	185 ⁴⁵⁶	2,2-Dimethyl-1,1,1-triphenyl-propane, C ₂₃ H ₂₄ . --
766	186 in sealed tube filled w. CO ₂ ⁴⁵²	Tris-(p-diphenyl)-methyl, C ₃₇ H ₂₇ . -- Green-black xtals. fm. C ₆ H ₆ + gasoline. (452). - In soln., in thick layer, deep violet; in very thin layers, green. (452).
768	186 ⁴⁵⁷ (190.5 corr.)	Dibenzo-2,3;6,7-fluorene, " β , β' -Dibenzofluorene", C ₂₁ H ₁₄ . -- Fearly lustrous plates fm. C ₆ H ₆ . (457). - CrO ₃ in AcOH g. β , β' -naphthofluorenone, orange-red needles, m.p. = 160-2° (163.5° corr.). (457). - In conc. H ₂ SO ₄ g. deep blue color quickly changing to brownish red in air. (457).
770	186 ⁴⁵⁸	Di-(2,2-dimethyl-bicyclo-(1,2,2)-heptylidene-6-methyl), "Dicamphenylidene-ethane", C ₂₀ H ₃₀ . -- Sol. in CHCl ₃ . (458). - Brilliant white xtals., slightly soluble in ether. (458). - Very hard to burn. (458). - Very stable. (458). - Cannot be oxid. to KMnO ₄ . (458).
772	187-8 ⁴⁵⁹ corr.	Difluorenylidene, "Dibiphenylene-ethene, C ₂₆ H ₁₆ . -- B.P. = above 360°. (460). - Red rhombohedral needles. (627, 459). - In alc. + ether soln. in air g. fluorenone. (461). - CrO ₃ mixt. g. fluorenone + (459) + 10-oxo-9-diphenylene-(9,10)-dihydro-phenanthrene. (462). - Adds 2 atoms of Br. (459). - HNO ₃ g. κ , β -dinitro- κ , β -bis-diphenylene-ethane. (463). - Htd. w. Zn dust g. fluorenone. (23). - Picrate, brown-red needles, m.p. = 177°. (459). -

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
		Br in CS ₂ g. β -dibrom- β -bis-diphenylene-ethane, leaflets fm. C ₆ H ₆ , m.p. = 235° dec. (459). - Boil. for 15 min. in AcOH w. 2 mols. conc. HNO ₃ g. yellow needles of β -dinitro- β -bis-diphenylene-ethane, m.p. = 184-5° dec., wh. by boil. w. alc. KOH regenerates the hy. (463).
774	187.8 ⁴⁶⁴	2-(β -naphthyl)-naphthalene, " β , β -Dinaphthyl", " <u>isodinaphthyl</u> ", C ₂₀ H ₁₄ . -- B.P.=452.5°. (464). - Plates w. faint blue fluorescence. (464). - Sublimes. (464,465). - Not volatile w. steam. (464). - G. fluorescing solns. (466). + CrO ₃ in AcOH g. 2-(β -Naphthyl)-naphthoquinone-(1,4) + a little dinaphthyl-(2,2')-diquinone-(1,4;1',4'). (464). - KMnO ₄ or dil. HNO ₃ at 160° g. phthalic ac. (467). - Slow addn. of 1 pt. hy. to 12 pts. HNO ₃ (d:1.50), finally warming, then pouring into much H ₂ O + rextaln. fm. EtOH g. yellow-brown amorphous powder, m.p.= 150° w. dec. (467). - Picrate, orange needles, m.p. = 184-5°. (661). - SbCl ₃ + BiCl ₃ g. no colors. (614).
776	188 ⁴⁵¹	Benzo-1,2-fluorene, "Phenylene-(naphthylene-(1,2))-methane", "Chrysofluorene", "Naphthofluorene", C ₁₇ H ₁₂ . -- B.P. = 413°. (451). - Na ₂ Cr ₂ O ₇ in boil. AcOH g. chrysoketone. (451). - Picrate, orange-red xtals, m.p. = 127.5°, dec. by alc. (451). -
778	188 ⁴⁵³	Tri-(2,5-dimethophenyl)-methane, C ₂₅ H ₂₈ . -- Grains. (23).
780	188-90 ^{30,47}	Tetracyclopentadiene, C ₂₀ H ₂₄ . -- B.P., = 160°. (47). - Adds 1 mol Br in CS ₂ . (47). - Easily oxid. by conc. HNO ₃ . (47). - G. tetrahydro deriv., m.p. = 200-2°. (30,47). - Dec. by htg. at 180-200° g. cyclopentadiene. (30). Div. B, Sect.3, No. 19.
782	189 ²³	Benzal- β -dinaphthyl-oxide, C ₂₇ H ₁₈ O.
784	189-90 ⁴⁶⁸	Triphenyl-(p-diphenyl)-ethene, C ₃₂ H ₂₄ . -- HNO ₃ (d: 1.40) g. a tetranitro deriv., yellow xtals. fm. Me ₂ CO, m.p. = 278-80°. (468) - SO ₂ Cl ₂ g. cpd., xtals. fm. AcOEt, m.p. = 179-81° w. evolution of HCl. (468).
786	190 ²¹³	3-(3 ³ -Phenyl-propene(3 ²)-ylidene)-indene, C ₁₈ H ₁₄ . -- Yellow-red needles fm. AcOEt. (213).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
788	191 ⁴⁶⁹	Tri-(<i>L</i> -naphthyl)-methane, C ₃₁ H ₂₂ . -- Short lustrous prisms fm. AcOEt w. blue tinge. (469). - Solid + C ₆ H ₆ soln. fluoresces intense blue. (469). - G. no color w. conc. H ₂ SO ₄ . (469). - Exposure to light changes to brownish-yellow color. (469).
790	191 ⁴⁷⁰	2-Methyl-10-(p-tolyl)-anthracene, C ₂₂ H ₁₈ . -- Yellow needles. (470). - Htg. w. CrO ₃ in AcOH g. methyl-p-tolyl-oxanthranol. (470).
792	191 ⁴⁷¹	C ₁₆ H ₁₂ , perhaps Dibenzo-(1,2;5,6)-cyclooctatetraene-(1,3,5,7). -- B.P. ₁₂ = 260°. (471). - Needles. (471).
794	192 ⁴⁷²	10-Phenyl-9-tolyl-anthracene, C ₂₇ H ₂₀ . -- Slightly yellow prisms. (472). - Easily oxid. to 10-phenyl-9-tolyl-9,10-diol-dihydroanthracene, needles w. EtOH wh. effloresce in air + then m. p. = 212°, sol. in conc. H ₂ SO ₄ w. intense indigo-blue color, has strong oxid. powers. (472).
796	193 ⁴⁷³	(p-Diphenyl)-fluorenyl-peroxide, C ₅₀ H ₃₄ O ₂ . -- Hexagonal leaflets. (473). - Dissolves in conc. H ₂ SO ₄ w. deep blue-green color. (473).
798	194 ⁴⁷⁴	1,6-Diphenyl-hexatriene-(1,3,5), C ₁₈ H ₁₆ . -- Yellow leaflets. (474). - Dil. solns. in HCCl ₃ or Me ₂ CO fluoresce blue. (474). - Adds 6 atoms of Br. (474).
800	195-6 ⁴⁷⁵	9,9-Diphenyl-9,10-dihydroanthracene, C ₂₆ H ₂₀ . -- Colorless needles fm. AcOH. (475). - CrO ₃ in AcOH g. 9,9-diphenyl-anthrone. (475). - Xs. Br in CS ₂ g. 10-brom-9,9-diphenyl-9,10-dihydroanthracene, needles fm. AcOH, m.p. = 214-6°, immediately again becoming solid w. HBr evolution + formation of tetraphenyl-heptacyclene, C ₅₂ H ₃₆ , (475). No. 1012.
802	195.5 ⁴⁷⁶	9-Phenyl-benzo-1,2-fluorene, "ms-Phenyl-Chrysofluorene", C ₂₃ H ₁₆ . -- Na ₂ Cr ₂ O ₇ in AcOH g. cpd. C ₂₃ H ₁₄ O ₃ , yellow prisms fm. AcOH, m.p. = 151° + o-benzoyl-benzoic ac. (476).
804	197 ⁴⁷⁹	Dimolecular dibenzenyl-anthracene, C ₅₆ H ₃₆ . -- Light yellow xtals. fm. Me ₂ CO. (479). - Easily sol. in CS ₂ , HCCl ₃ , C ₆ H ₆ . (479). - Slightly sol. in AcOH, ether, alc. (479). - Decolorizes KMnO ₄ . (479). - Adds 4 atoms of Br in CS ₂ , g. yellow xtals., m.p. = 215°. (479).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
806	197 ⁴⁸⁰	<u>1-(Diphenyl-methylene)-4-(phenyl-p-tolyl-methylene)-cyclohexadiene-(2,5), "p-Quinone-(diphenyl-methide)-(phenyl-p-tolyl-methide)", C₃₃H₂₆.</u> -- Orange-red xtls. fm. xylene. (480). - Sol. in conc. H ₂ SO ₄ w. orange color. (480).
808	198 ⁴⁸¹	<u>β-Tri-(p-diphenyl)-methyl-peroxide, C₇₄H₅₄O₂.</u> -- Sol. in AcOH-H ₂ SO ₄ w. same bluish-red color as the carbinol. (481). - (Cf. No. 1028 + No. 844.).
810	198-8 ^{476, 477} corr.	Triphenylene, C ₁₈ H ₁₂ . -- Sublimes. (476). - Solns. show faint blue fluorescence. (476). - Fum. HNO ₃ in sealed tube at 150° g. mellitic ac. (476). - CrO ₃ in AcOH g. quinone-like substance. (476). - Picrate, yellow needles. (476, 477).
812	200-10 ⁴⁸³	<u>2-Methyl-1,4-di-(diphenyl-methylene)-cyclohexadiene-(2,5), "Tetraphenyl-toluquino-dimethane", C₃₃H₂₆.</u> -- Chlor deriv., m.p. = 195-200°. (483). - m-Dichloroquinone deriv., gleaming deep red xtls., m.p. = 225°. (483).
814	200-0.5 ⁴⁸⁴	<u>1,2,4,7-Tetraphenyl-cyclo-octatetraene-(1,3,5,7)(?), "Dypnopinakolene", C₃₂H₂₄.</u> -- Golden yellow xtls. fm. EtOH. (484).
816	200 ⁴⁸⁵ dec.	<u>1,4-Dimethyl-3,6-di-(diphenyl-methylene)-cyclohexadiene-(1,4), C₃₄H₂₈.</u> -- Red-violet shimmering needles fm. xylene. (485).
818	200-2 ^{30, 486}	<u>Tetrahydro-tetracyclopentadiene, C₂₀H₂₈.</u>
820	202 ⁴⁸⁷	<u>1,1,4,4-Tetraphenyl-butadiene-(1,3), C₂₈H₂₂.</u> -- Violet reflecting needles fm. AcOH. (487). - Xtalizes fm. C ₆ H ₆ w. 1 mol. C ₆ H ₆ . (487). - Oxid. g. CO ₂ + benzophenone. (487).
822	202-3 ⁴⁸⁸	<u>1,3-Dimethyl-anthracene, C₁₆H₁₄.</u> -- Plates. (488).
824	204-5 ²³	<u>ℒ-Benzpinacolone, C₂₆H₂₀O.</u> -- Stable at 350°. (23). - Does not react w. KOH. (23). - CrO ₃ oxid. g. benzophenone. (23). (Cf. Tests 905-2 + 714 (Mulliken, Ident+ etc.)).
826	205 ⁴⁸⁹	<u>9-(9¹, 9¹, 9², 9²-Tetraphenyl-ethyl)-fluorene, C₃₉H₃₀.</u> -- Needles fm. AcOH. (489).
828	205 ⁴⁹⁰	<u>δ-Methylene-diphenylene, C₁₃H₁₀.</u> -- B.P. = 320°. (490). - Plates. (490). - CrO ₃ in AcOH g. a quinone, C ₁₃ H ₈ O ₂ , white powder, m.p. = 276-8° wh. sublimes in needles. (490).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
830	206-7 ⁴⁹¹	Benzo-6,7-9,10-dihydro-anthracene, "9,10-Di-hydronaphthacene", $C_{18}H_{14}$. -- Needles or leaflets fm. C_6H_6 . (491). - B.P. = abt. 400°. (491). - Sol. in warm H_2SO_4 w. evolution of SO_2 + w. dark moss-green color. (491). - CrO_3 g. naphthacene-quinone. (491). - Conc. HNO_3 g. a nitro-naphthacene-quinone, m.p. = abt. 240°. (491).
832	207 ²³	Photoanethole, $C_{10}H_{12}O$. -- Fm. Anethole by sunlight. (23). - Odorless, tasteless, pearly leaflets. (23). - Sublimes. (23).
834	493, 494 207 corr.	2-Methyl-anthracene, $C_{15}H_{12}$. -- White plates w. green-blue fluorescence by sublimation. (494). - Boil. w. xs. CrO_3 in AcOH g. anthraquinone carbonic ac. (495). - Ab. soln. + conc. HNO_3 g. 2-methyl-anthraquinone. (496). - G. bis-(2-methyl-anthracene) by sunlight on its C_6H_6 soln., m.p. = 228°. (497). - (No. 880.). - 2 atoms of Br in CS_2 g. 9,10-dibrom-2-methyl-anthracene, yellow needles fm. EtOH or AcOH, m.p. = 142-3° wh. oxid. to β -methyl-anthraquinone, wh. latter w. Br at 130-40° g. a colorless dibrom cpd., m.p. = 219-20°. (494). - Dark-red needles, g. no constant M.P. because it is very quickly split by moisture of the air. (494).
836	210 ⁴⁹⁸	9-(β -Naphthyl)-dibenzo-2,3;6,7-fluorene, $C_{31}H_{20}$. -- Turns yellow at 190°. (498).
838	211 ⁴⁹⁹ corr.	1,1,2,2-Tetraphenyl-ethane, $C_{26}H_{22}$. -- B.P. = 379-83° corr. (499). - Sublimes. (500). - D: 1.182. (501). - $K_2Cr_2O_7$ in AcOH- H_2SO_4 g. benzophenone. (500). - "G. no picrate." (502). - Fum. HNO_3 g. tetrakis-(4-nitro)-deriv. (499, 503), (advantageously at 30-40°), m.p. = 337.5-8.5° corr., 300° u.c. (499).
840	212-3 ⁴⁹⁰ corr.	1,4-Diphenyl-benzene, $C_{18}H_{14}$. -- Sublimes in spangles (490) or leaflets. (504). - B.P. between 404 + 427°. (490). - B.P. ₄₅ = 250°. (506). - Non-volatile w. steam. (505). - C_6H_6 soln. fluoresces blue. (505). - CrO_3 in AcOH g. diphenyl carbonic ac.-(4). (505). - "G. no picrate." (505). - Xs. Br g. tetrabrom deriv., needles fm. C_6H_6 , m.p. = 245°. (507). Htg. in AcOH w. 8 times its quantity of fum. HNO_3 on H_2O bath g. dinitro cpd., needles fm. $PhNO_2$, m.p. = 277°. (506). - Fum. HNO_3 g. trinitro cpd., m.p. \pm 195°. (506). - CrO_3 oxid. g., under some conditions, terephthalic ac., (Test 905-2). (23).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
842	213 ⁵⁰⁸	2-Methyl-9,10-diphenyl-anthracene, $C_{27}H_{20}$. -- Yellow-green, strongly dichroic xtals. fm. C_6H_6 + EtOH. (508). - Most of its solns. fluoresce magnificent blue-violet. (508). - $K_2Cr_2O_7$ in AcOH g. 9,10-dioxy-2-methyl-9,10-diphenyl-9,10- dihydroanthracene. (508).
844	213 ⁴⁸¹	\mathcal{L} -Tri-(p-diphenyl)-methyl-peroxide, $C_{74}H_{54}O_2$. -- Sol. in AcOH- H_2SO_4 w. bluish-red color of the carbinol. (481). - Cf. No. 1028 + No. 808.
846	213-3.5 ⁵⁰⁹	1,2-Diphenyl-1,1-di-(p-tolyl)-ethane, $C_{28}H_{26}$. -- Microscopic xtals. fm. EtOH. (509).
848	215 ⁵¹⁰	9,10-(1,1'-Diphenylene)-phenanthrene, $C_{26}H_{16}$. -- Needles. (510). - Oxid. g. $C_{26}H_{16}O_2$, colorless xtals., m.p. = 269°. (510). - Unattacked by Br in boil. CCl_4 . (510).
850	217-8 ^{511,512}	9-(Diphenyl-methyl)-fluorene, $C_{26}H_{20}$. -- Xtalizes fm. C_6H_6 w. 2 mol. C_6H_6 , in leaflets. (511,512). - Picrate, yellow needles, m.p. = 198°. (511).
852	217 ⁵¹³ corr.	Anthracene, "Paranaphthalene", $C_{14}H_{10}$. -- B.P. = 351°. (514). - D_4^{27} :1.25. (515). - Leaflets, usually yellowish. (23). - Pure xtals. are colorless + show violet fluorescence. (516). - Boil. alc. soln. w. $1\% K_2Cr_2O_7$ alone or w. H_2SO_4 g. anthra- quinone. (516,518). - Boil. 1 g. by. w. 45cc. AcOH + add dropwise for 2 hrs. 15 g. CrO_3 in 10 cc + AcOH + 10 cc. H_2O ; continue boil. for 2 hrs., let stand, pour into 400 cc. H_2O + filter off anthra- quinone, m.p. = 267°. (520). Test 912. - Cpd. w. picryl chloride, m.p. = 141.6° dec. (522). - Cpd. w. picranide, m.p. = 158.8°. (522). - Styphrate, m.p. = 176.3° (also g. 2 entectics). (521). - Picrate, ^{fm. nitromethane} ruby-red needles, m.p. = 138-9°. (552,553). - 4 atoms Br in CS_2 at rm. temp. g. 9,10 dibrom- anthracene, yellow needles, m.p. = 221°. (518,519). - $HCHO + H_2SO_4$ g. dirty-green to black. (512). - Benzal-chloride + H_2SO_4 g. malachite-green color. (512). - $SbCl_3$ in CCl_4 g. intense-green-colored ppt. (513). - Micro-procedure: warm $PhNO_2$ soln. g. w. α -dinitrophenanthraquinone, on cooling, gray-blue rhombic xtals. w. chrysmic ac. long flat pleochroic green-yellow needles. (512). - $SbCl_3$ g. yellowish-green color w. trace of hy. (514). - $BiCl_3$ g. very characteristic purple-black coloration. (514).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
854	218-9 ⁵²³	Dimethyl-anthracene, $C_{16}H_{14}$. -- Faintly yellow-green plates. (523). - Sublimes in colorless plates. (523). - CrO_3 g. a dimethyl-anthraquinone of m.p. = 170°. (523).
856	220 ⁵²⁴	9,9,10-Triphenyl-9,10-dihydroanthracene, $C_{32}H_{24}$. -- White xtals. fm. EtOH + C_6H_6 . (524). - Sol. in conc. H_2SO_4 without color. (524).
858	220 ⁵²⁵	9-(o-Xylylene)-fluorene, $C_{21}H_{16}$. -- Needles fm. ether. (525).
860	220 ⁴⁹²	1,2,4,5,6,8 or 1,2,4,5,7,8-Hexamethyl-anthracene, $C_{20}H_{22}$. -- Picrate, dark-brown needles, m.p. = 203°. (492).
862	222 ⁵²⁶ corr.	9,9-Diphenyl-fluorene, $C_{25}H_{18}$. -- B.P. above 400°. (526). - Insol. in conc. H_2SO_4 . (526).
864	222 ⁵²⁹ corr.	Isophthalacene, $C_{21}H_{16}$. -- Yellowish leaflets fm. (527). - AcOH or C_6H_6 . (527). - $K_2Cr_2O_7$ in AcOH g. isophthalacene oxide + isophthalacene, $C_{21}H_{12}O_2$. (527).
866	222 ⁵²⁸	1,3,6-Trimethyl-anthracene, $C_{17}H_{16}$. -- CrO_3 in AcOH g. 1,3,6-trimethyl-anthraquinone. (528). - HNO_3 (d:1.1) at 210-20° in sealed tube g. anthraquinone-tricarboxic ac. (528). - Br in CS_2 g. 9,10-dibrom deriv., yellow plates fm. Me_2CO , m.p. = 142°. (528).
868	223.5-4.5 ⁴⁹⁹ corr.	Tetraphenyl-ethylene, $C_{26}H_{20}$. -- B.P. = 415-25°. (529). - CrO_3 in AcOH g. α -benzpinacolin, then benzophenone. (530). - $KMnO_4$ in AcOH g. β -benzpinacolin. (529). - Does not add Br. (499). - Br in CCL_4 (499) or $HCCL_3$ (531) g. tetrakis-(p-brom-phenyl)-ethene, m.p. = 253-5° corr. (499, 531). - Fails to g. test 901 for unsatn. + is not attacked by alk. $KMnO_4$ in Test 304 of Mulliken. (23).
870	224-35 ⁵³²	3,6-Dimethyl-9-(p-tolyl)-fluorene(?), $C_{22}H_{20}$. -- Non-volatile. (532). - Htg. leads to complete dec. + carbonization. (532).
872	225 ⁵⁰⁸	9-Phenyl-10-(\mathcal{L} -naphthyl)-9,10-dihydroanthracene, $C_{30}H_{22}$. -- white needles wh. in air slowly assume a dichroic violet color. (508). - $K_2Cr_2O_7$ in AcOH g. 9,10-dioxy-9-phenyl-10-(\mathcal{L} -naphthyl)-9,10-dihydroanthracene. (508).

DIVISION A, SECTION 2.

No.	Melting Point (°C)	Hydrocarbon
874	225 ⁵³³ dec.	1,8-Diphenyl-octatetraene-(1,3,5,7), C ₂₀ H ₁₈ . -- Golden yellow leaflets fm. AcOH wh. show green fluorescence. (533). - Br in HCCl ₃ g. octabromide + 1,4,5,8 tetrabrom deriv. (534). - Alk. KMnO ₄ g. benzaldehyde + benzoic ac. (533). - Light in absence of air g. white modification, m.p. = 124°. (533). - No. 536.
876	225 ⁵³⁶	Di-(<i>R</i> -naphthyl)-acetylene, C ₂₂ H ₁₄ . -- Boils w. dec. above 360°. (536).
878	227 ⁵³⁷	1,4,6-Trimethyl-anthracene, C ₁₇ H ₁₆ . -- Blue-green fluorescence. (537). - Plates. (537). - Sublimes below 100°. (537). - CrO ₃ in AcOH g. 1,4,6-trimethyl-antraquinone. (537).
880	228-30 ⁴⁹⁷ dec.	Di-(2-methyl-anthracene), C ₃₀ H ₂₄ . -- Formed by sunlight + C ₆ H ₆ soln. or monomer. (497). - Xtals. fm. toluene. (497). - M ⁺ g. xylene, anisole, etc. (497). - Cf. No. 834.
882	229 ⁵⁰⁸	9-Phenyl-10-(<i>L</i> -naphthyl)-anthracene, C ₃₀ H ₂₀ . -- Yellow xtals. (508). - Easily sol. in C ₆ H ₆ . (508). - Slightly sol. in EtOH + AcOH w. magnificent violet fluorescence. (508).
884	229.5 ⁵³⁸	9,9-(Diphenyl-methylene)-fluorene, C ₂₆ H ₁₈ . -- Almost colorless in solid state. (538). - Solns. an intense yellow. (510). - Na-Hg w. C ₅ H ₁₁ OH g ⁺ 1,1-diphenyl-2-diphenylene-ethane. (538).
886	539, 540 230-1 corr.	Triphenyl-(<i>p</i> -(diphenyl-methyl)-phenyl)-methane, "4-Trityl-tritan", C ₃₈ H ₃₀ . -- Soln. in Conc. H ₂ SO ₄ is colorless. (541). - Unattacked by boil. Na ₂ Cr ₂ O ₇ in AcOH. (540). - Htg. 10 hrs. w. CrO ₃ in AcOH part. dec. (540). - Br in CS ₂ in sunlight g. x-brom-4-trityl-tritan, red-yellow xtaln. powder fm. C ₆ H ₆ + ligroin, m.p. = 240-2° (541, 542). - HNO ₃ g. hexanitro deriv ⁺ , M+P ⁺ = 265°. (540).
888	232 ⁵⁴³	Di-(benzo-2,3-fluorenylidene), "Bis-phenylene-bis- β , β -naphthylene-ethane", C ₃₄ H ₂₀ . -- Intensely red xtaln. powder, or dark red leaflets w. blue tinge. (543). - Softens before melting. (543).
890	232-3 ⁵⁴⁴ corr.	Dodecahydrotriphenylene, C ₁₈ H ₂₄ . -- Needles fm. hot AcOEt. (544). - HNO ₃ g. mellitic ac. (544).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
892	233 ⁵⁴⁵	Tri-(p-diphenyl)-methane, C ₃₇ H ₂₈ . -- Retains 1 C ₆ H ₆ . (545). - C ₆ H ₆ removed by htg. at m.p. in vacuum, then separates fm. CHCl ₃ in xtaln. granules, m.p. = 241-2°. (545). - No. 910†
894	234-40 ⁵⁴⁶	9,10-(Endo-1,2-diphenyl-ethylene)-anthracene, "Monomolecular dibenzal-anthracene", C ₂₈ H ₂₀ . -- Yellow xtals. fm. HCCl ₃ + EtOH. (546). - M.p.'s of various preps. vary between 234 + 240°. (546). - Sol. in hot HCCl ₃ w. blue fluorescence. (546). - Very slightly sol. in AcOH, ether, alc., CS ₂ . (546).
896	235-5.5 ^{547,549}	9,10-Diphenyl-phenanthrene, C ₂₆ H ₁₈ . -- Sublimes. (548). - CrO ₃ in AcOH g. 2,2'-dibenzoyl-diphenyl. (548).
898	238 ⁵⁵⁰ corr.	Dimethyl-anthracene, C ₁₆ H ₁₄ . -- Sublimes without dec. (550).
900	240 u.c. ⁵⁵¹	9,10-Diphenyl-anthracene, C ₂₆ H ₁₈ . -- Amber-yellow octahedral xtals. fm. CS ₂ , reminding one of S. (551). - Sublimes without dec. abt. 270°. (551). - G† no color w. conc. H ₂ SO ₄ ; easily sulfonated. (551). - W† exception of CS ₂ , all its solns. fluoresce a beautiful violet-blue. (551). - K ₂ Cr ₂ O ₇ in AcOH g. 9,10-dioxy-9,10-diphenyl-9,10-dihydro-anthracene. (551).
902	240-1 ⁵⁵²	Tetrahydro-dibenzo-2,3;6,7-anthracene, C ₂₂ H ₁₈ . -- Plates. (552). - Cf. No. 960.
904	240 ⁵⁵³	1,6 or 1,7-Dimethyl-anthracene, C ₁₆ H ₁₄ . -- Greenish plates fm. PhMe. (553). - CrO ₃ in AcOH g. a dimethyl-anthraquinone of m.p. = 169°. (554). - 2 atoms of Br in CS ₂ at 0° g. brom deriv., light yellow needles, m.p. = 200°, wh. w. CrO ₃ g. a dimethyl-anthraquinone of m.p. = 169°. (554).
906	abt. 240 ⁴⁸⁰	1,4-Di-(diphenyl-methylene)-cyclohexadiene-(2,5), "p-Quinone-bis-diphenyl-methide", C ₃₂ H ₂₄ . -- Orange-red xtals. fm. xylene wh. m. in sealed capillary tube filled w. CO ₂ at 268° u.c., + in open tube w. air oxid. at abt. 240°. (480). - Solns. are yellow-orange + fluoresce golden-yellow + color in the light. (555). - Soln. in conc. H ₂ SO ₄ is yellow. (555). - Br decolorizes soln. g. ω, ω, ω', ω'-tetraphenyl-p-xylylene-dibromide. (555). - Ppts. I fm. soln. of HI in CCl ₄ . (555).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
908	240-1 ⁴⁸⁰ in CO ₂ atm.	1-(Diphenyl-methylene)-4-(phenyl- \mathcal{L} -naphthyl-methylene)-cyclohexadiene-(2,5), C ₃₆ H ₂₆ . -- Orange-red xtals. fm. boil. xylene. (480). - Sol. in conc. H ₂ SO ₄ w. green, at high conc., dark violet red color. (480). - Stable in dry state. (480). - Insoln. oxid. in air. (480).
910	241-2 ⁵⁴⁵	Tri-(p-diphenyl)-methane, C ₃₇ H ₂₈ . -- Cf. No. 892..
912	241 ⁵⁴⁶	9,10-Dibenzyl-anthracene, C ₂₈ H ₂₂ . -- Needles fm. AcOH. (546). - Blue fluorescence. (546). - (664). - CrO ₃ g. anthraquinone + benzoic ac. (664). - Br vapor dilt. w. CO ₂ + passed into CS ₂ soln. of hy. g. 9-benzyl-10-(\mathcal{L} -brom-benzyl)-anthracene, yellow plates fm. CS ₂ , m.p., rapid htg. = 187° without dec. (664). - Same procedure also g. sym. dibrom cpd., yellow xtals fm. CS ₂ , m.p. = 212° wh. is more difficultly sol. in CS ₂ , more easily sol. in HCl ₃ than the monobrom cpd. (546).
914	242.5 ⁵⁵⁶ corr.	Dibenzo-1,2;7,8-fluorene, C ₂₁ H ₁₄ . -- C ₆ H ₆ soln. Fluoresces violet-red. (556). - CrO ₃ in AcOH g. \mathcal{L} , \mathcal{L} -dinaphtho-fluorenone, deep red, microscopic needles fm. C ₆ H ₆ , m.p. = 255°, wh. g. deep red soln. w. conc. H ₂ SO ₄ . (619).
916	243 ⁵⁵⁷	1,2,4-Trimethyl-anthracene, C ₁₇ H ₁₆ .
918	244 ⁵⁵⁸	Dianthracene, Paranthracene, C ₂₈ H ₂₀ . -- M+p. varies with rate of htg. (559). - Rapid htg. g. m.p. between 270-280°. (559). - D ₄ ²⁷ :1.265. (560). - Htg. at m.p. g. anthracene. (559). - Formed by action of light on various solns. of anthracene. (559). - Insol. in C ₆ H ₆ . (614). - Unattacked by ord. HNO ₃ + by Br. (614). - "G. no picrate." (614). - CrO ₃ or warm fum. HNO ₃ g. anthraquinone. (624).
920	244.5 ⁵⁶¹	2,6-Dimethyl-anthracene, C ₁₈ H ₁₄ . -- Ab. soln. fluoresces strong blue. (563). - CrO ₃ in AcOH g. 2,6-dimethyl-anthraquinone. (562).
922	246 ⁵⁶⁴	Di-(1-methyl-anthracene), "Para- \mathcal{L} -methyl-anthracene", C ₃₀ H ₂₄ . -- Colorless plates wh. readily lose C ₆ H ₆ of xtaln. in the air. (564). - Htg. w. conc. H ₂ SO ₄ g. dark yellow color wh. on cooling changes to yellowish-green w. blue fluorescence. (564). - Distn. g. orig. \mathcal{L} -methyl-anthracene. (564). No. 328.
924	246 ⁵⁶⁵ corr.	Difluorenyl, C ₂₆ H ₁₈ . -- Na ₂ Cr ₂ O ₇ in AcOH g. fluorenone. (565). - "G. no picrate." (565).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
926	246 ⁵⁶⁶	2,3-Dimethyl-anthracene, $C_{16}H_{14}$. -- Blue-green fluorescing plates. (566).
928	248-9 ⁵⁶⁷	9,10,9',10'-Tetrahydrodianthranyl-9,9', $C_{28}H_{22}$. -- Prismatic needles fm. C_6H_6 . (567). - Sublimes. (567). - Br g. 9,10-dibrom-anthracene. (567). -
930	250 ⁵⁶⁹	Benzo-1,2-phenanthrene*, "Chrysene", $C_{18}H_{12}$. -- B.P. = 448° (570) part. dec. (614). - Commonly obtained yellow, due to presence of chrysogene. (514). - Colorless plates fm C_6H_6 or AcOH w. red-violet fluorescence. (568). - CrO_3 in AcOH g. chrysoquinone, $C_{18}H_{10}O_2$ (569,571) wh. dissolves in conc. H_2SO_4 w. deep-blue color. (23). - Br in CS_2 g. dibrom cpd., needles fm. C_6H_6 , m.p. = 273°. (568). - Htg. 10 g. hy. (pulverized as fine as possible) w. 100 g. AcOH + 4.5 g. HNO_3 (d:1.415) for 1 hr. on H_2O bath g. nitro deriv., chrom-red xtals., m.p. = 209°. (568). - Cpd. w. 2,7-dinitro-anthraquinone, red needles, m.p. = 294°. (568). - Picrate, red needles. (569). - Traces g. golden-yellow color w. $SbCl_3$. (614). - $HCHO + H_2SO_4$ g., in presence of $HCCl_3$, a red-violet ppt. (612). - Benzal-chloride + H_2SO_4 g. yellow-green, then quickly light green + dark olive-green color. (612).
932	253 ⁵⁷²	1,2-Di-(β -naphthyl)-ethane, $C_{22}H_{18}$. -- Solns. fluoresce blue-violet. (572).
934	254-5 ⁴⁴⁸	1,2-Di-(β -naphthyl)-ethene, $C_{22}H_{16}$. -- Plates fm. C_6H_6 . (448). - C_6H_6 soln. fluoresces violet-blue. (448).
936	254-5 ²³	Hydroxy-lepidene, $C_{28}H_{22}O_2$. -- Sol. in 100 pts. hot glac. AcOH. (23). - Sol. after long boil. in alc., then melting at 260-1°. (23). - Conc. HCl at 130-40° g. lepidene. (23).
938	255 ⁵⁷³	1,2,3,4-Tetraphenyl-butane, $C_{28}H_{26}$.
940	259-60 ⁵⁷⁴	2,6-Diphenyl-dibenzo-3,4;7,8-bicyclo-(0,3,3)-octatetraene-(1,3,5,7), "9,12-diphenyl-diphen-succindanedrine-(9,11)", $C_{28}H_{18}$. -- Brown xtals. fm. AcOEt, C_6H_6 , or $CHCl_3$. (574). - Often almost black, fm. C_6H_6 . (574). - Becomes strongly electrified on rubbing. (574). - $KMnO_4$ or CrO_3 g. o-benzoyl-benzoic ac. (574).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
942	260-70 ⁵⁴³	Di-(benzo-2,3-fluorenyl), $C_{34}H_{22}$. -- Turns red at 250°. (543). - M.p. = 260-70° according to rapidity of htg. (543).
944	262-3 ⁴⁸⁵	2,5-Di-(diphenyl-methylene)-benzo-1,6-cyclohexadiene-(3,6), " <u>α-Naphthoquinone-bis-diphenyl-methide</u> ", $C_{36}H_{26}$. -- Yellow xtl'n. powder fm. C_6H_6 . (485). - Htg. to 200° turns it orange, on cooling, yellow. (485).
946	262-3 ⁵⁷⁵	9-(Methyl-fluorenyl-methyl)-fluorene, " <u>Ethylidene-bis-fluorene</u> ", $C_{28}H_{22}$. -- Colorless soln. in quinoline is oxid. by atm. O, almost instantly, to yellow dehydro-ethylidene-bis-fluorene. (575). - In C_6H_6 + AcOH a yellow color is quickly formed. (575).
948	264-5 ⁵⁷⁶	Perylene, $C_{20}H_{12}$. -- Plates w. bronze lustre fm. C_6H_6 . (576). - Sublimes + is deposited in yellow plates. (576). - Dil. solns. are yellow w. blue fluorescence; conc. solns. are reddish-yellow. (576). - In Conc. H_2SO_4 color is reddish-violet. (576).
950	266 ⁵⁷⁷ (273.5 corr.)	1,1,2,2-Tetra-(β -naphthyl)-ethane, $C_{42}H_{30}$. -- Small prisms fm. C_6H_6 . (577).
952	267.5 ⁵⁷⁸	Dibenzo-1,2,7,8-anthracene, " <u>Dinaphthantracene</u> ", $C_{22}H_{14}$. -- Orange colored plates fm. C_6H_6 . (578). - Insol. in ether, alc., AcOH, Pet. ether; very slightly sol. in cold C_6H_6 . (578). - Red+ w. HI + Pg. $C_{22}H_{20}$ or $C_{22}H_{22}$, white xtals., m.p. = 178.5°. (578). - Picrate, brown needles fm. C_6H_6 , m.p. = 223-4°. (578).
954	270 ⁵³⁶	Dinaphthyl-anthrylene, $C_{22}H_{12}$. -- G. xtaln. cpd. fm. $CHCl_3$ w. picric ac. (536).
956	270 ⁵⁷⁹	Tribenzyl-dekacyclene, $C_{57}H_{36}$. -- Light yellow needles fm. C_6H_6 or $PhNH_2$. (579). - Almost insol. in alc., ether, AcOH; sol. in C_6H_6 , PhMe; easily sol. in xylene, naphthalene, + $PhNH_2$. (579). - Soln. in conc. H_2SO_4 g. green color. (579). - Dil. solns. fluoresce strong green. (579).
958	270 ⁴⁸⁶	Pentacyclopentadiene, $C_{25}H_{30}$. -- Sublimes in a high vac. (486). - Separates as a gel fm. very dil. solns. (486). - Boil. HNO_3 oxid. (486). - Decolorizes Br. (486). - Dec. g. dicyclopentadiene on htg. (486).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
960	270 ⁵⁵²	<u>Tetrahydro-dibenzo-2,3;6,7-anthracene, C₂₀H₁₈.</u> -- Needles. (552). - Cf. No. 902.
962	270-80 ⁵⁵⁹	<u>Dianthracene, C₂₈H₂₀.</u> -- Cf. No. 918.
964	271-2 ⁵⁷⁴	<u>2,6-Di-(p-tolyl)-dibenzo-3,4;7,8-dicyclo-(0,3,3)-octatetraene-(1,3,5,7), "9,12-p,p'-Ditolyl-diphensuccindanediene-(9,11)", C₃₀H₂₂.</u> -- Dark brown fm. AcOEt. (574). - Almost black fm. C ₆ H ₆ . (574).
966	276-9 ⁵⁸⁰	<u>1,1,2,2-Tetra-(p-diphenyl)-ethane, C₅₀H₃₈.</u> -- Short prisms w. 1 mol. of C ₆ H ₆ or xylene. (580). - Does not develop color or combine w. I in boil. ethyl benzoate. (580). - Does not combine w. O in boil. xylene. (580).
968	227-8 ⁵⁸¹	<u>1,2,4,5-Tetraphenyl-benzene, C₃₀H₂₂.</u> -- Colorless needles fm. C ₆ H ₆ . (581).
970	280 ⁵⁸²	<u>1,3,5,7-Tetramethyl-anthracene, C₁₈H₁₈.</u> ++ Faintly yellow plates w. green fluorescence fm. AcOH. (582).
972	282-3 ⁵⁸³	<u>Dinaphthyl-naphthalene, C₃₀H₂₀.</u> -- Yellowish leaflets. (583).
974	(285 ⁵⁸⁵ corr.) ⁵⁸⁴	<u>Tetraphenyl-methane, C₂₅H₂₀.</u> -- B.P. = 431°. (584). - Colorless xtals. fm. hot C ₆ H ₆ . (584). - Sublimes in iridescent needles. (584). - Insol. in conc. H ₂ SO ₄ . (584). - Cold fum. HNO ₃ g. 4,4',4''-trinitro-tetraphenyl-methane, faintly yellow xtals. fm. C ₆ H ₆ + EtOAc, m.p. = 330° wh. by red. w. Zn dust g. fuchsine-like colored sdn. (585).
976	285-6 ⁵⁸⁶ corr.	<u>1,1,2,2-Tetra-(α-naphthyl)-ethane, C₄₂H₃₀.</u>
978	300-10 ⁵⁵²	<u>Dibenzo-2,3;6,7-9,10-dihydroanthracene, "6,13-Dihydrodinaphthantracene", C₂₂H₁₆.</u> -- Micro xtals. (552).
980	300 ⁵⁸⁶	<u>Dianthranyl-9,9', C₂₈H₁₈.</u> -- Leaflets fm. toluene. (23,586). - 1 pt. hy. suspended in 5 pts. AcOH slowly mixed w. 2 pts. of a mixt. of 1 vol. HNO ₃ (d:1.48) + 1 vol. AcOH g. 10,10'-dinitro-dianthranyl-(9,9'), sulfur yellow needles or prisms, m.p. = 337° w. dec. wh. w. CrO ₃ in AcOH easily g. anthraquinone. (587).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
982	485, 588 305	9,10-Di-(diphenyl-methylene)-9,10-dihydroanthracene, $C_{40}H_{28}$. -- Conc. H_2SO_4 g. green color. (588).
984	306-7 ⁵⁸⁹	Heptacyclene, "Dinaphthylene-cyclobutane", $C_{24}H_{16}$. -- Silky needles. (589). - "G. no picrate." (589). - Very resistant to chem. reagents. (589). - Br reacts w. substitution; after many hrs. in cold $CHCl_3$ g. $C_{24}H_{14}Br_2$, needles fm. $PhNO_2$, m.p. = 303-4°. (589). - Long htg. in high boil. solvents at its m.p. g. a hy. whose solns. have an intense violet-blue fluorescence. (589). - $K_2Cr_2O_7$ in glac. AcOH g. $C_{10}H_6(CO)_2O$. (589). - Formed by sunlight on 15-20% acenaphthylene soln. in C_6H_6 . (589).
986	306 ⁵⁹⁰	Rubicene, $C_{26}H_{14}$. -- Intensely red lancet xtals. (590).
988	306 ⁵⁹¹	Picylene-methane, $C_{21}H_{14}$. -- Microscopic xtals. fm. C_6H_6 . (591). - Hot conc. H_2SO_4 g. green soln. (591).
990	315 ²³	Dixanthylene, "Tetraphenyl-ethylene-dioxide", $C_{26}H_{16}O_2$. -- Sublimes. (23). - Needles fm. C_6H_6 . (23). - Sol. in conc. H_2SO_4 w. yellow color. (23). - Adds Br. (23). - Boil. w. dil. HNO_3 g. xanthone (23), No. 722. Solns. show bluish-green fluorescence. (23).
992	481, 592 317 corr.	4,4'-Diphenyl-diphenyl, "p,p-Dixenyl", "Benz-erythrene", $C_{24}H_{18}$. -- B.P. ₁₈ = abt. 428°. (592). - Plates fm. C_6H_6 or $PhNO_2$. (592). - Insol. in alc., ether, $HCCl_3$ (592), ligroin; easily sol. in boil. $PhNH_2$, $PhNO_2$. (593). - Insol. in conc. H_2SO_4 . (592).
994	330 ⁵⁸⁰	Tetradiphenyl-ethene, $C_{50}H_{36}$. -- Pale yellow needles fm. xylene or $PhNO_2$. (580).
996	abt. 335 ⁵⁹⁴	Benzo-2,3-anthracene, "Naphthacene", $C_{18}H_{12}$. -- Orange to reddish-yellow leaflets. (594). - Sublimes. (594). - Vapor is greenish-yellow. (594). - Sol. in conc. H_2SO_4 w. moss-green color. (594). - Insol. in C_6H_6 . (594). - Fum. HNO_3 oxid. to naphthacene quinone. (594). - Distn. w. Zn dust g. dihydronaphthacene. (594).

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
998	350 u.c. (364 corr.) ⁵⁹¹	<p><u>Dibenzo-1,2;7,8-phenanthrene, "Picene", "Parachrysenes", C₂₂H₁₄.</u> -- B.P. = 518-20°. (595). -- Colorless plates w. blue fluorescence. (591). -- Almost insol. in most solvents. (596). -- Slightly sol. in boil. C₆H₆, HCCl₃, AcOH. (596). -- Xylene soln. of pure cpd. does not fluoresce. (597). -- Sol. in conc. H₂SO₄ w. green color. (596,597). -- Boil. CrO₃ in AcOH g. picene quinone (596), picene quinone carbonic ac., C₂₃H₁₂O₄, + a little phthalic ac. (591). -- Br in HCCl₃ g. 13,14-dibromopicene, needles fm. xylene, m.p. = 294-6°. (595). -- Benzal-chloride + H₂SO₄ g. olive-green color after short time. (614).</p>
1000	365-8 ⁵⁹⁸	<p><u>Truxene, "Tribenzylene-benzene", C₂₇H₁₈.</u> -- Plates fm. boil. xylene. (598). -- Almost insol. in most ord. solvents. (599). -- Sol. in boil. HCCl₃, PhNH₂ (599), PhNO₂ (600), xylene (601). -- CrO₃ mixt. g. tribenzoyl-benzene. (601,602). -- Long boil. w. HNO₃ (d:1.5) g. 4-nitro-benzene-dicarboxylic ac.-(1,2). (601). -- Only slowly attacked by molten KOH. (601). -- Dropping soln. in conc. H₂SO₄ into conc. HNO₃ g. unstable green color. (603).</p>
1002	387 ⁶⁰⁴	<p><u>Trinaphthylene-benzene, "Dekacyclene", C₃₆H₁₈.</u> -- Yellow-golden glistening needles. (604). -- Insol. in boil. alc., ether, AcOH; very slightly sol. in boil. C₆H₆, PhMe; difficultly sol. in boil. PhOH or pyridine; sol. in cold PhNO₂ + molten naphthalene, easily sol. in boil. PhNO₂ + boil. naphthalene. (604). -- Dil. solns. fluoresce green. (604,605). -- Sol. in fum. H₂SO₄ g. dark brown-green color. (605). Br in CS₂ g. on boil. a cpd., light yellow needles fm. PhNO₂, m.p. = 397-400°. (606). -- Picrate, dark-violet xtals., split by ether. (604,605).</p>
1004	above 300 ⁶⁰⁷	<p><u>1,2,3,4-Tetraphenyl-cyclopentene-(x), C₂₉H₂₄.</u> -- White xtn. powder fm. ether. (607). -- Easily sol. in C₆H₆, difficultly in alc. (607).</p>
1006	above 300 ⁶⁰⁸	<p><u>9-(Fluorenylidene-methyl)-fluorene(?), C₂₇H₁₈.</u> -- Red needles. (608).</p>
1008	above 360 ⁵⁷⁵	<p><u>9-(Methyl-fluorenyl-methylene)-fluorene, "Dehydroethylidene-bis-fluorene", C₂₈H₂₀.</u> -- Yellow prisms wh. darken at 280°. (575). -- G. deep blue color w. fum. H₂SO₄. (575). -- M+ + boils, apparently without dec., at a high temp. (575).</p>
1010	above	

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
1010	above ⁵⁸⁸ 360	9,10-Di-(diphenyl-methyl)-anthracene, $C_{40}H_{30}$. -- White needles. (588). - Intense violet fluorescence. (588). - Soln. in cold H_2SO_4 is yellow w. strong violet fluorescence. (588).
1012	above ⁶⁰⁹ 360	Tetraphenyl-heptacyclene, $C_{52}H_{36}$. -- Yellow-white xtals. (609). - Insol. (609).
1014	m. in ⁶¹⁰ boil. paraffin	9-(Fluorenylidene-methylene)-fluorene, "Bis-diphenylene-allene", $C_{27}H_{16}$. -- Orange-yellow needles fm. AcOEt + $HOCl_3$. (610).
1016	above ⁶¹¹ 360 dec.(?)	7,10';9,3'-Diendomethylene-dianthranyl-8,4', "Pyranthrene", $C_{30}H_{18}$. -- Slender brown needles when rapidly deposited. (611). - Short yellow-green prisms w. green fluorescence when allowed to xtalize slowly fm. xylene. (611). - M† w. evolution of orange-red vapors. (611). - H_2SO_4 g. violet-blue color, changing to blue on htg. (611).
1018		Tri-(ter-butyl-ethynyl)-methyl, $C_{19}H_{27}$. -- Absorbs 50% of calculated amt. of O to form a peroxide. (668). - Fm. the corresponding chloromethane + Ag in PhMe at 100°. (668). Cf. Div. B, Sect. 3, No. 13.
1020		Phenyl-(p-diphenyl)-(L-naphthyl)-methyl, $C_{29}H_{21}$. -- Known only in C_6H_6 soln. (617). - Coffee-brown color in thick layers, dirty yellow in thin. (617). G. peroxide, m.p. = 158°? (617). No. 664.
1022		Phenyl-di-(p-diphenyl)-methyl, $C_{31}H_{23}$? -- Known only in soln. (306). - C_6H_6 soln. is red. (306). - Behaves toward air like Ph_3C . (306).
1024		(L-Naphthyl)-di-(p-diphenyl)-methyl, $C_{35}H_{25}$. -- Light gray-green xtaln. powder; after drying, slowly and difficultly sol. in C_6H_6 w. brown-red color. (624).
1026		Diphenyl-(p-diphenyl)-methyl, $C_{25}H_{19}$. -- Known only in orange-red soln. (306).
1028		Tri-(p-diphenyl)-methyl, $C_{37}H_{27}$. --- L-cpd. = dark-green xtaln. powder g. brownish-red solns. (481). - L-cpd. is intense violet-red to brownish-red in C_6H_6 ; in glac. AcOH diluted w. large xs. of conc. H_2SO_4 the alc. g. same bluish-red color + absorption spectrum as the β -carbinol. (481). - β -cpd. † dark green needles g. pure blue solns. (481). - β -cpd. is dec. by light in very dil. soln. more quickly than the L, but oxid. by air more

DIVISION A, SECTION 2.

No.	Melting Point (C°)	Hydrocarbon
1030		<p>slowly. (481). - Both forms g. peroxides, m.p. = 213° + 198°. (481). - Cf. Nos. 844 + 808.</p> <p>Tri-(^β-naphthyl)-methyl, C₃₁H₂₁. -- Extremely unstable, dark-violet xtals. wh. absorb 0 w. great avidity. (625).</p>

DIVISION B

.....
Liquid Hydrocarbons

Section 1

.....
Aromatics, fulvenes, etc.

DIVISION B, SECTION 1.

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
2	80-1 ¹	0.879(20/4) ^{2,3}	1.501(20) ⁴	Benzene*, C_6H_6 . -- M.P. 5.5°.(5). 2 vol. H_2SO_4 + 1 vol. fum. HNO_3 g. m-di-nitrobenzene(6), M.P.=89° (442), apply test 913. $ClSO_3H+NH_3$ g. benzene sulphonamide (7), M.P. = 156° (8)*, apply test 929. G. yellow color w. $SbCl_5$. (452).
4	93-4 ⁴⁴²	0.903(18/4) ⁴⁴²		2,5(α)-Dimethylfurfurane, $C_8H_{10}O$. -- Insol. in aq. (442). Misc. w. alc. (442). Conc. HCl g. resinous body. (442). Dil. HCl at 170° g. acetyl acetone. (442).
6	110-1 ⁹	0.866(20/4) ^{4,13}	1.496(20) ⁴	Methyl-benzene*, "Toluene", C_7H_8 . -- Fum. HNO_3 g. 2,4-dinitrotoluene, (10,11), apply test 918. $KMnO_4$ in H_2O at 95° g. benzoic ac. almost quant. (12), apply test 905-1. G. light brown-red color w. $SbCl_5$.
8	abt. 118 ⁴⁹¹ 116 (724mm.)	0.888(19/4) ⁴⁹¹		Cycloheptatriene(1,3,5), C_7H_8 . -- CrO_3 mixture g. benzoic ac. + benzaldehyde. (492). In alc. soln. + H_2SO_4 g. dark brown-red color. (491). $NaOEt$ g. dark brown color. (491).
10	122-7 ⁴⁹⁰			Cardene, C_8H_8 . -- Unchanged by conc. H_2SO_4 . (490).
12	abt. 123 ⁴⁹¹ 120-1 (724mm.)	0.868(18) ⁴⁹¹		Cycloheptadiene(1,3), "Hydrotropilidene", C_7H_{10} . -- Alc. soln. + conc. H_2SO_4 g. brown-yellow color. (491).
14	136.5- ¹³ 7.5	0.874(20/4) ¹³	1.496(20) ¹⁴	Ethyl-benzene*, C_8H_{10} . -- $HNO_3-H_2SO_4$ g. 2,4-dinitro-ethyl benzene + 2,4,6-tri-nitro-ethyl benzene. (15, 16). Apply test 905-1.

DIVISION B; SECTION I.

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
16	^{17,18} 137.5- 8.5	0.861(20/4) ¹⁷	1.499(14) ¹⁸	1,4-Dimethyl-benzene,* "p-Xylene", C_8H_{10} . -- M.P.= +16°. (19,20). Identify by test 920. G. red color w. $SbCl_5$. (452).
18	^{18,21,22} 138.5- 9.5	0.864(20/4) ¹⁷	1.500(16) ²³	1,3-Dimethyl-benzene,* "m-Xylene", C_8H_{10} . -- M.P.= -54°. (442). Warming w. $HNO_3-H_2SO_4$ g. 2,4,6-trinitro-1,3-dimethyl-benzene (24,25). Identify by test 919. G. red color w. $SbCl_5$. (452).
20	⁴⁷⁶ abt. 140 42.2-2.4 (17mm.)	0.923(20/4) ⁴⁷¹	1.539(20) ⁴⁷¹	Cyclooctatetraene, C_8H_8 . -- M.P.=abt. -27°. (471). Yellow liquid w. intensely sweet odor. (471). Vapor g. headache. (471). Readily reduces $KMnO_4$. (471). $HNO_3 + H_2SO_4$ g. resin, brown flocks on exposure to air. (476). In $CHCl_3$ at -20° forms the dibromide $C_8H_8Br_2$, needles fm. pet. ether or anhydrous formic ac., m.p.=70.1.5°, wh. can add more Br w. HBr evolution g. $C_8H_7Br_3$, ^{m.p.: 53-5°, wh. latter instantly} decolorizes $KMnO_4$ in AcOH. Hydrobromide fm. C_8H_8 in $CHCl_3 + AcOH - HBr$ at rm. temp., b.p. 12.5=85-7°, unstable to O(atm.) and to $KMnO_4$ in AcOH, but does not decolorize Br in $CHCl_3$, g. orange color w. H_2SO_4 . (471).
22	²⁶ 141-2	0.937(13/4) ²⁶	1.552(13/14) ²⁶	Phenyl-ethine,* "Phenyl acetylene", C_8H_6 . -- Gives test 906. (27). Cu cpd. by shak. w. alc. NH_3 g. diphenyl-butadiene. (27). Boil. w. Zn dust in AcOH g. styrene. (28). Shak. w. aq. H_2SO_4 g. $PhCOMe$. (29). $HOBr$ g. ω , ω' dibrom acetophenone. (30). Hg cpd., test 929, M.P.=125°. (31,32).

DIVISION B, SECTION 1.

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
24	⁴⁷⁷ 143-5	0.870(20/0) ⁴⁷⁷	1.491 ⁴⁷⁷	<u>1-Ethenyl-cyclohexene(1), C₈H₁₂.</u> -- Unaffected by htg. w. Na. (477).
26	^{18,21} 144-5	0.880(20/4) ³³	1.505(22) ³³	<u>1,2-Dimethyl-benzene,* "O-Xylene, C₈H₁₀.</u> -- M.P.= -27.1°. (34). Dil. HNO ₃ oxid. to o-toluic ac., CrO ₃ mixture completely destroys. (35). Shak. w. boil. soln. of KMnO ₄ g. phthalic ac. + o-toluic ac. (36). Identify by test 921. G. red color w. SbCl ₅ . (452).
28	^{37, 38, 39} 145-6	0.912(13/4) ³⁷	1.549(13) ³⁷	<u>Phenyl-ethene,* "Styrene", "Cinnamene", "Cinnamomane", C₈H₈.</u> -- Polymerized by light, (40) and by conc. H ₂ SO ₄ . (41). K ₂ Cr ₂ O ₇ + H ₂ SO ₄ g. benzoic ac. (40). Unsat'd. (40). Nitroso-chloride, needles, M.P.= 97°. (42). G. test 903.
30	⁴⁷⁸ 147-8	0.903(20/4) ⁴⁷¹	1.528(20) ⁴⁷¹	<u>Cyclo-octatriene, C₈H₁₀.</u> -- Unstable to KMnO ₄ . (475) Reacts w. Br ₂ w. evolution of HBr. (475). W. alc. H ₂ SO ₄ g. deep yellow color wh. after addn. of conc. H ₂ SO ₄ g. wine red soln. (475). Vapor g. head-ache. (478). Exposure to air g. amorphous brown solid. (478). Dibrom deriv., C ₈ H ₁₀ Br ₂ , b.p. ₉ = 129.5-30°, b.p. ₁₄ =136-7.5° d ₄ ²⁰ =1.762, wh. when dis-tilled w. Me ₂ NPh g. dimethyl amino-cyclooctatriene, C ₈ H ₉ NMe ₂ , b.p. ₁₀ =81-91°, d ₄ ²⁰ =0.934, whose methio-dide, fm. H ₂ O, m.p.=224-5° (dec.) + whose chloro-platinate, fm. H ₂ O, M.P.= 200°(dec.) (478).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
32	152-3 ⁴³	0.864(20/4) ⁴⁴	1.493(20) ⁴⁴	<u>Isopropyl-benzene, "Cumene", C₉H₁₂.</u> -- Hot nitration g. 2,4,6-trinitro-1-isopropyl-benzene. (45). Apply test 905-1.
34	156-6 ⁴⁶	0.988(21/4) ⁴⁷	1.515(22) ^{443, 48}	<u>Methyl phenyl ether, "Anisole", C₇H₈O.</u> -- Aromatic odor. (442). Insol. in aq. W. conc. HI at 130°-40° gives phenol +McI. (442).
36	abt. 156 ⁴¹⁸ 153-4 (717mm.) 46 (11mm.)	0.881(20/4) ⁴⁷¹	1.547(20) ⁴⁷¹	<u>[Dimethyl-methylene]-cyclopentadiene, "W,w-Dimethyl-fulvene", C₈H₁₀.</u> -- By stg. in absence of air g. bis-dimethyl-fulvene (C ₈ H ₁₀) ₂₁ , six-sided plates or needles fm. MeOH or dil. EtOH, M.P.=83° wh. w. conc. H ₂ SO ₄ g. yellow-red color + wh. by htg. above M.P. g. dimethyl fulvene. (473).
38	156-7 ⁴⁹	0.901(15) ⁴⁹	1.514 ⁴⁹	<u>1-Phenyl-propene(2), C₉H₁₀.</u> Apply test 905-1.
40	38-40 ⁴⁷⁹ (15mm.)			<u>1,1-Dimethyl-4-methylene-cyclohexadiene(2,4), C₉H₁₂.</u> -- A few drops of conc. HCl in AcOH g. pseudo-cumene. (479).
42	157-8 ^{45, 51}	0.862(20/4) ²³	1.494(16) ²³	<u>n-Propyl-benzene, C₉H₁₂.</u> -- Apply test 905-1.
44	158-9 ⁵²	0.869(20) ⁵²		<u>1-Methyl-3-ethyl-benzene, C₉H₁₂.</u> -- CrO ₃ oxid. g. isophthalic ac. (52). Fum. HNO ₃ +conc. H ₂ SO ₄ g. 2,4,6-trinitro-1-methyl-3-ethyl-benzene, nearly colorless xtals fm. EtOH, M.P.=86°. (53). Test 905-1 g. isophthalic ac. (442).
46	82-4 ⁵⁴ (19mm.)	0.907(17/4) ⁵⁴	1.486(20) ⁵⁴	<u>5,6-Dihydro-2,6-dimethyl-3-propene(3')-yl-1,2-pyrane, C₁₀H₁₆O.</u> -- Odor

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
48	⁴⁸⁹ 159-60	⁴⁸⁹ 0.882 (20)		<p><i>of mint-anise. (54). - Sweet anise taste. (54).</i></p> <p><u>2,7,7-Trimethyl-bicyclo-[1,1,3]heptene-(2),</u> "Verbenene", C₁₀H₁₄. -- 1.34 g. in cold CHCl₃ w. 1-N. Br until color persists (20.8 cc.) g. dibromide, prisms fm. pet. ether, M.P.=70-2° wh. liquify and give off HBr on stg. and wh. is un-attacked by KMnO₄, di-isomer, M.P.=50-2°. (488). 3 g. shaken at rm. temp. w. 18 g. KMnO₄ + 6 g. KOH in 600 cc. H₂O until KMnO₄ is decolorized (abt. 45 min.) g. <i>cis-norpinic ac.</i>, M.P.=175.5-6.5° (488). 3 g. verbenene boiled few min. w. 0.5 g. ZnCl₂+dist'd. w. steam g. 1.2 g. volatile produces wh., treated w. dil. KMnO₄ until the color persists + further oxid. w. boiling KMnO₄ g. 0.8 g. <i>p-Me₂C(OH)C₆H₄CO₂H</i>, M.P.=156-7°. (488). L-form shows [α]_D¹⁹=-74.90° (100mm) (489).</p>
50	^{44, 56} 161-2	⁵⁶ 0.909 (21)	1.533 (21)	<u>2-Phenyl-propene, C₉H₁₀.</u>
52	^{37, 57, 58} 161-2	³⁷ 0.869 (14/4)	³⁷ 1.494 (14)	<p><u>1-Methyl-4-ethyl-benzene, C₉H₁₂.</u> -- Oxid. w. dil. HNO₃ g. <i>p-toluic ac. (57)</i>. CrO₃ mixture g. <i>terephthalic ac. (59)</i>. Htg. w. HNO₃-H₂SO₄ g. <i>2,3,5(?)-trinitro-1-methyl-4-ethyl-toluene, (57, 60)</i>, M.P.=94°. (60). Test 905-1 g. <i>terephthalic ac. (442)</i>.</p>
54	⁴⁴⁴ 162-3	⁴⁴⁴ 0.882 (20/4)	⁴⁴⁴ 1.504 (20)	<u>1-Methyl-2-ethyl-benzene, C₉H₁₂.</u> -- Test 905-3 g. <i>phthalic ac. (442)</i> .
56	^{61, 62} 163.5- 4.5	⁴ 0.856 (20/4)	⁴ 1.491 (20)	<p><u>1,3,5-Trimethyl-benzene,</u> "Mesitylene", C₉H₁₂. -- Oxid. w. alk. KMnO₄ at 80° g. <i>uvitic ac.+trimesic ac. (63)</i>. HNO₃-H₂SO₄ in the</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
58	⁶⁷ 163.5- 4.5			<i>yellow plates. (cc).</i> <u>1-Methyl-3-ethenyl-benzene</u> <u>C₉H₁₀.</u>
60	⁷³ 167-70	0.908(15) ⁷³	1.543 ⁷³	<u>1-Phenyl-propene-(1),</u> <u>C₉H₁₀.</u> -- G. dibromide, M.P.=66-7°.(73).
62	^{442, cf 35} 167-8	0.938(18) ³⁵		<u>Methyl benzyl ether,</u> <u>C₈H₁₀O.</u> *
64	^{70, 71, 72} 167.5- 8.5	0.869(20/0) ⁷⁰	1.497(19) ⁷²	<u>ter-Butyl-benzene, C₁₀H₁₄.</u> *
66	⁷⁴ 168-70	0.903(18) ⁷⁴	1.548(17) ⁷⁴	<u>1-Methyl-4-ethinyl benzene</u> <u>C₉H₈.</u> -- G. test 906.(74). Dibromide B.P. ₁₃ =139-43, d ₁₇ =1.669. cf Cu salt bright yellow flocculent ppt. wh. w. alk. K ₃ Fe(CN) ₆ g. p- tolyl-diacetylene. (74).
68	^{13, 69} 168.5- 9.5	0.881(14/4) ⁶⁸	1.508(14) ⁶⁸	<u>1,2,4-Trimethyl-benzene,</u> <u>"Pseudo-cumene", C₉H₁₂.</u> -- CrO ₃ in HAc g. tri- mellitic ac. (76). For coloration w. AlCl ₃ cf. test 904. (442). Identify by test 917. (442).
70	^{18, 71} 169.5- 70.5	0.867(20/4) ²³	1.496(15) ²³	<u>Isobutyl-benzene, C₁₀H₁₄.</u> -- CrO ₃ mixt. g. benzoic ac. (75).
72	⁷⁷ 170-5	0.898(16/4) ³⁷	1.531(16) ³⁷	<u>1-Methyl-4-ethenyl-benzene</u> <u>C₉H₁₀.</u>
74	⁷⁸ 170.5- 1.5		1.494(17) ⁷⁸	<u>1-Methyl-2-ter-butyl-</u> <u>benzene, C₁₁H₁₆.</u> -- Gives a β-[methyl-ter-butyl- benzoyl]-acrylic ac. (Beilstein 1910, Syst. No. 1296) w. maleic an- hydride +AlCl ₃ . (78).
76	⁴⁴² 171-2	0.981(20/4) ⁴⁴⁴	1.505(20) ⁴⁴⁴	<u>o-Cresyl methyl ether,</u> <u>C₈H₁₀O.</u> *

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
78	172-3 ⁴⁴²	0.982(0) ⁴⁴²	1.508(21) ⁴⁴³	<u>Ethyl phenyl ether, "Phenetole",</u> $C_8H_{10}O$. -- Aromatic odor. (442). Insol. in aq. (442). At 400° g. phenol (test 414) + ethylene. (442).
80	172-3 ⁷⁹	0.908(0) ⁸⁰		<u>1-Methyl-2-methoethenyl-benzene, C₁₀H₁₂.</u>
82	172-3 ⁸¹	0.927(20) ^{443, 82}	1.461(20) ^{444, 83}	<u>Cineole, "Eucalyptol",</u> $C_{10}H_{18}O$. -- M.P.=+1°.(81). Cpd. w. α-naphthol (1 mol: 1 mol), M.P.=75°.(81). Cpd. w. o-cresol(1 mol: 1 mol), M.P. 50°.(81). Cpd. w. resorcinol (1 mol: 1 mol), M.P.=89°.(81). Shaken w. satd. soln. of I in satd. KI soln. g. ppt. of minute xtals. w. greenish lustre. Agreeable odor like cardamon + camphor. (442). Unsatd. (492). Dibromide very unstable. (442). Dry HCl conducted into mixture of equal vols. eucalyptol + ligroin g. xtaln. ppt. of unstable (C ₁₀ H ₁₈ O) ₂ .HCl. - Cpd. w. β-naphthol (1mol:1mol), m.p.: 48°(81). <u>3-Phenyl-pentene-(1),</u> $C_{11}H_{14}$. -- CrO ₃ in HAc g. benzoic ac. (82). G. test 901. (442). Continued boiling g. diamenyl benzene, B.P.=208-12°.(442). Test 905 g. benzoic ac. (442).
84	173-4 ⁸²	0.846(23) ⁸²		<u>3-Phenyl-pentene-(1),</u> $C_{11}H_{14}$. -- CrO ₃ in HAc g. benzoic ac. (82). G. test 901. (442). Continued boiling g. diamenyl benzene, B.P.=208-12°.(442). Test 905 g. benzoic ac. (442).
86	173-4 ⁸³	0.945(15/0) ⁸³	1.534(15) ⁸³	<u>Phenyl-cyclopropane, C₉H₁₀.</u> Odor like xylene. (83). H ₂ SO ₄ polymerizes to C ₁₈ H ₂₀ thick odorless oil, B.P.= 330-2°, d ₁₇ =1.002, n _D = 1.571. (83).
88	174-5 ⁸⁴	0.863(20/4) ⁸⁴	1.489(21) ⁸⁴	<u>sec-Butyl-benzene,</u> $C_{10}H_{14}$. CrO ₃ mixt. g. benzoic ac. (71).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
90	abt. ¹⁰¹ 174 64 (10 mm.)	0.883(20/4) ¹⁰¹	1.506(20) ¹⁰¹	<u>1-Phenyl-butene-(3), C₁₀H₁₂</u> --Sharp odor, unlike that of isomers. (101). In Me ₂ CO powdered KMnO ₄ g. hydrocinnamic ac. (101).
92	175-6 ⁹³	0.886 ⁹³		<u>1-Phenyl-butene(2), C₁₀H₁₂</u> -- Odor of mushrooms. (93)
94	175-6 ⁸⁷	0.862(20) ⁸⁷	1.492(20) ⁸⁷	<u>1-Methyl-3-isopropyl-benzene, "m-Cymene", C₁₀H₁₄</u> --KMnO ₄ oxid. g. iso-phthalic ac. (87). Cold fum. HNO ₃ g. 6-nitro-1-methyl-3-isopropyl-benzene wh. w. dil. HNO ₃ g. a nitro-n-toluic ac., M.P.=214°. (88). Gradual addn. to a cold mixture of 1 pt. fum. HNO ₃ +4 pts. conc. H ₂ SO ₄ + htg. until violent reaction is over, abt. 1 hr. at 100°, g. yellow-white plates fm. pet. ether, M.P.=72-3°, odor of musk on warming. (89).
96	175-6 ⁶⁹			<u>1,2,3-Trimethyl-benzene, "Hemellitene", C₉H₁₂</u> . -- Long treatment w. HNO ₃ -H ₂ SO ₄ g. 4,5,6-trinitro-1,2,3-trimethyl benzene, prisms fm. EtOH, M.P.=209°. (89).
98	175-6 ⁴⁴²	0.970(20/4) ⁴⁴⁴	1.512(20) ⁴⁴⁴	<u>p-Cresyl-methyl ether, C₈H₁₀O</u> . *
100	175-6 ⁴⁶⁸	0.874(22/0) ⁴⁶⁸	1.501 ⁴⁶⁸	<u>1-Methyl-2-isopropyl-benzene, "o-Cymene", C₁₀H₁₄</u>
102	^{22, 58} 176-7	0.857(20/4) ⁸³	1.493(14) ⁸³	<u>1-Methyl-4-isopropyl-benzene, "p-Cymene", C₁₀H₁₄</u> For oxid. see Beilstein 1910, Vol. V, pg. 422. HNO ₃ -H ₂ SO ₄ g. a trinitro cymene, M.P.=118°. (90).
104	^{91, 92} 176- 7.5	0.863(16) ^{91, 92}		<u>1-Methyl-3-n-propyl-benzene, C₁₀H₁₄</u> . -- Br+I at rm. temp. g. 4,6-dibrom-

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
106	will be	found at the top of	Page	<p>307</p> <p>1-methyl-3-propyl-benzene wh. w. HNO₃-H₂SO₄ g. 4,6- dibrom-2,5-dinitro-1- methyl-3-propyl-benzene, needles fm. pet. ether, M.P.=140-1°.(92).</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
106	^{82,94} 176-7	0.963(16/4) ⁹⁵		<u>Hydrindene</u> , C_9H_{10} . - Tinged yellow by conc. H_2SO_4 . (96). Sulphonated by cold conc. H_2SO_4 . (442). M.P. sulfonamide; 91-2°. (442).
108	^{95,97} 176-7	0.914(20/4) ⁹⁷	1.549(20) ⁹⁷	<u>1-Phenyl-propene(1)</u> , C_9H_{10} .
110	⁴⁴⁴ 176-7	0.977(15/15) ⁴⁴⁴	1.506(20) ⁴⁴⁴	<u>m-Cresyl methyl ether</u> , $C_8H_{10}O$. - Oxid. w. $KMnO_4$ g. m-methoxy benzoic ac. (443).
112	^{93,98,99} ¹⁰² 180-1	0.875(20/4) ¹⁰²	1.494(20) ¹⁰²	<u>n-Butyl-benzene</u> , $C_{10}H_{14}$.
114	¹⁰³ abt. 180 w. dec.			<u>1,4-Diethenyl-benzene</u> , $C_{10}H_{10}$. -- HBr g. 1',4'-dibrom-1,4-diethyl benzene. (103).
116	⁴⁴² 180-1	0.958 ⁴⁴²		<u>o-Cresyl ethyl ether</u> , $C_9H_{12}O$.
118	^{82,112} 181-2	1.000(15/15) ¹¹²	1.577(19) ¹¹²	<u>Benzo-cyclopentadiene</u> , "Indene", C_9H_8 . -- Aq. $KMnO_4$ g. 1,2-dioxyhydrindene, then homophthalic ac. + phthalic ac. (113). Diln. w. EtOH+shak. w. Ph-CHO+KOH+ steam distn. of impurities g. 1-[α -oxybenzyl]-3-benzal-indene, m.p.:135°. (114). - Conc. H_2SO_4 g. brown resin. (442). Adds br. (442). - Forms picrate, M.P.:98°, golden yellow exp. when dry. (453). Cpd. w. 1,3,5-trinitrobenzene (1 mol= 1 mol), yellow needles, M.P.:101-2 (115). - Cpd. w. picryl chloride (1mol:1mol), yellow needles, M.P.=39°. (115). α -Nitrosite fm. pet ether powder, M.P.=107-9° w. dec., wh. w. boil. abs. EtOH g. β -nitrosite, needles fm. C_6H_6 , M.P.=136-7°. (116). - Conc. H_2SO_4 g. para indene. (117)

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
120	181-2 ¹⁰⁵	0.902(15/4) ¹⁰⁵	1.528(15) ¹⁰⁵	<u>1-Phenyl-2-methyl-propene-(1), C₁₀H₁₂.</u> -CrO ₃ oxid. to benzoic ac. (106).- HNO ₂ g. nitrosite, M.P.=112°.(107).
122	181-2 ¹⁰⁸	0.860(20/4) ¹⁰⁸		<u>1,3-Diethyl-benzene, C₁₀H₁₄.</u> Oxid. w. dil. HNO ₃ g. m-ethyl benzoic ac.+iso-phthalic ac.(108). 2,4,5,6-Tetrabrom-1,3-diethyl-benzene, prisms fm. EtOH, M.P.=74°.(108).
124	181-2 ¹²⁸			<u>1-Phenyl-propine-(1), C₉H₈.</u> Slowly combines w. HgCl ₂ in H ₂ O g. 2 C ₉ H ₈ +3 HgO+-3 HgCl ₂ wh. w. HCl g. EtPhCo.(129).
126	182-3 ¹⁰⁹	0.868(14/4) ³⁷	1.498(14) ³⁷	<u>1,4-Diethyl-benzene, C₁₀H₁₄</u> - Htg. w. dil. HNO ₃ g. p-ethyl-benzoic ac.+tere-phthalic acid.(109,110).- 120 g. Br dropped into 25 g. hy. at 155° g. 1,1,4,4-tetrabrom-1,4-diethyl-benzene, M.P.=157°.(111).
128	182-8 ¹¹⁸			<u>1,3-Dimethyl-5-ethyl-benzene, C₁₀H₁₄.</u> - Oxid. g. 3,5-dimethyl-benzoic ac.(118). <i>of # 150</i>
130	^{119,120} 183-4	0.868(15) ¹¹⁹		<u>1-Methyl-4-propyl-benzene, C₁₀H₁₄.</u> - Bromination g. 2,5-dibrom-1-methyl-4-propyl-benzene wh. w. HNO ₃ -H ₂ SO ₄ g. 2,5-dibrom-3,6-dinitro-1-methyl-4-propyl-benzene, needles, M.P.=156-7°.(121).
132	⁴⁴² 183-4	0.942(0) ⁴⁴²	1.472(20) ⁴⁴⁴	<u>Pinol, C₁₀H₁₆O.</u> - Odor like that of eucalyptol.(442).- Unsat. (442).- Dissolved in 2 vols. glac. AcOH+ treated w. Br. g. stable dibromide wh. xtal. fm. ether-alc. w. M.P.=94°(442)

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
134	184-5 ¹²²	0.907(16/0) ^{133,138}	1.528(n _D) ¹³²	<u>1-Methyl-4-[methoethenyl]-benzene, C₁₀H₁₂.</u> - KMnO ₄ in cold alk. soln. g. p-toluic ac. (123). Iodohydrin+HgO g. p-tolyl-acetone. (122). - Nitroschloride, xtals. fm. MeOH, M.P.=100-2°. (124).
136	184-5 ³⁷	0.877(16/4) ³⁷	1.503(16) ³⁷	<u>1,5-Dimethyl-2-ethylbenzene, C₁₀H₁₄.</u> - Dil. HNO ₃ g. 2,4-dimethylbenzoic ac. (125). - HNO ₃ -H ₂ SO ₄ g. 3,4,6-trinitro-1-5-dimethyl-2-ethyl-benzene needles, M.P.=127°. (125, 126).
138	184-5 ¹²⁷	0.866(18/4) ¹²⁷		<u>1,2-Diethyl-benzene, C₁₀H₁₄.</u> - 3,4,5,6-Tetra-brom-1,2-diethyl-benzene, prisms fm. EtOH, M.P.=64.5°. (127).
140	184-5 ⁴⁷⁴ 62.5 (13mm.) ⁴⁷³	0.879(20/4) ⁴⁷¹	1.538(20) ⁴⁷¹	<u>[Methyl-ethyl-methylene]-cyclopentadiene, ω-Methyl-ω-ethyl-fulvene, C₉H₁₂.</u> - Orange colored liquid. (474). - W. HAc-H ₂ SO ₄ g. red color and yellow ppt. (473).
142	Abt. 184 ¹³⁰ 86 (20mm.) ³⁷ 68 (11mm.)	0.895(15/4) ³⁷	1.538(15) ³⁷	<u>1-Ethyl-4-ethenyl-benzene C₁₀H₁₂.</u> - Na+EtOH g. p-diethyl benzene. (37)
144	185-6 ^{131,133}	0.904(22/0) ¹³³	1.53(n _D) ¹³³	<u>1-Methyl-3-metho-ethenyl-benzene, C₁₀H₁₂.</u> - I+HgO g. iodohydrin wh. w. AgNO ₃ or Hg ⁰ g. m-tolyl acetone. (122).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
146	147, 442 185-6			Ethyl benzyl ether, C ₉ H ₁₂ O. -Treatment w. P ₂ O ₅ g. ethylene + anthracene. (Test 912). (442). <i>diphenyl-methane (Ann. 298 255)</i>
148	³⁷ Abt. 185 69 (10 mm.)	0.907(18/4) ³⁷	1.524(18) ³⁷	1,4-Dimethyl-2-ethenyl-benzene, C ₁₀ H ₁₂ . -Na+EtOH g. 1,4-dimethyl-2-ethylbenzene. (37).
150	¹³⁴ 185-6	0.861 ¹³⁴		1,3-Dimethyl-5-ethylbenzene, C ₁₀ H ₁₄ . - Dil. HNO ₃ nitric ac. (135). - HNO ₃ -H ₂ SO ₄ g. 2,4,6-trinitro-1,3-dimethyl-5-ethylbenzene, needles fm. EtOH, M.P.=238°. (135). <i>cf #128</i>
152	^{37, 69} 185-6	0.882(17/4) ³⁷	1.503(17) ³⁷	1,4-Dimethyl-2-ethylbenzene, C ₁₀ H ₁₄ . - Dil. HNO ₃ g. 2,5-dimethylbenzoic acid. (132). - Br.+AlBr ₃ g. tetrabrom-p-xylene+3,5,6-tribrom-1,4-dimethyl-2-ethylbenzene, needles fm. EtOH, M.P.=89°. (69). - 3,5,6-Trinitro-1,4-dimethyl-2-nitro-1,4-dimethyl-2-ethylbenzene, prisms fm. EtOH, M.P.=129°. (69).
154	¹³² 187-9, 100 80-1 (20mm.)	0.909(22/4) ¹³²	1.529(22) ¹³²	1-Phenyl-propadiene-(1,2), C ₉ H ₈ . - Tetrabromide, xtals. fm. alc., M.P.=75°. (442).
156	⁴⁸² around 50° (23mm.)	0.869(16/4) ⁴⁸²	1.509(15) ⁴⁸²	1,1-Dimethyl-4-methylene-cyclohexadiene-(2,4), C ₉ H ₁₂ . - After 3 distns., $d_{15}^{15}=0.843$, $n_D^{15}=1.509$.
170	¹⁴⁴ 186-9	0.867(16/4) ¹⁴⁴	1.497(16) ¹⁴⁴	2,4-Dimethyl-3-phenyl-butane, (482).
158	¹⁴³ 185.5- 6.5	0.858(18/4) ¹⁴³	1.488(18) ¹⁴³	1-Phenyl-2,2-dimethylpropane, C ₁₁ H ₁₆ .
160	¹³⁹ 186-7	0.920(20/0) ¹³⁹	1.505(n _D) ¹³⁹	1-Methyl-2-phenyl-cyclopropane, C ₁₀ H ₁₂ .
176	¹⁴⁰ 189-91	0.939(0) ¹⁴⁰		

DIVISION B, SECTION 1

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
162	186-8 ¹⁴⁰			<p><u>1-Methyl-3-ter-butyl-benzene, C₁₁H₁₆.</u> - CrO₃ mixt. slowly oxid. to isophthalic ac. (140). Oxid. by hot. dil. HNO₃ g. m-ter-butyl-benzoic ac. (141). - Cold HNO₃ (d=1.5) g. esodinitro-3-ter-butyl-toluene, needles, M.P.=92° (142). - Addn. of 3 pts. hy. w. cooling to mixture of 5 pts. HNO₃(d:1.5)+10 pts. fum. H₂SO₄ (15%SO₃) htg. 8-9 hrs. on H₂O bath, pouring into H₂O+re-nitration g. yellow-white needles fm. EtOH of 2,4,6-trinitro-1-methyl-3-ter-butyl-benzene (artificial musk), M.P.=96-7° (441).</p>
164	187-9 ¹³⁶ ₁₀₀ 80-1 (20mm.)	0.909 (22/4) ¹³⁷	1.529 (22) ¹³⁷	<p><u>2-Phenyl-butene-(2), C₁₀H₁₂.</u></p>
166	187-8 ¹²⁵			<p><u>1-2-Dimethyl-4-ethyl-benzene, C₁₀H₁₄.</u> - Oxid. w. dil HNO₃ g. 3,4-dimethyl-benzoic ac. (143). Br g. a dibrom cpd., needles fm. EtOH, M.P.=201° xs. Br g. tribrom cpd. (149). - 3,5,6-trinitro-1,2-dimethyl-4-ethyl-benzene, needles fm. EtOH, M.P.=121° (125).</p>
168	187-8 ¹⁴⁴	0.876 (15/4) ¹⁴⁴	1.499 (16) ¹⁴⁴	<p><u>3-Phenyl-pentane, C₁₁H₁₆.</u></p>
170	188-9 ¹⁴⁴	0.867 (16/4) ¹⁴⁴	1.497 (16) ¹⁴⁴	<p><u>2-Methyl-3-phenyl-butane, C₁₁H₁₆.</u></p>
172	188-9 ¹⁴⁵			<p><u>2-Methyl-3-phenyl-butene-(2), C₁₁H₁₄.</u></p>
174	73, 188-9 ¹⁴⁶	0.912 (16/4) ¹⁴⁶	1.541 (16) ¹⁴⁶	<p><u>1-Phenyl-butene-(1), C₁₀H₁₂</u></p>
176	189-91 ¹⁵⁴	0.939 (0) ¹⁵⁴		<p><u>1-Phenyl-butene-(3), C₁₀H₁₀</u></p>

DIVISION B, SECTION 1

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
178	189-91	0.939(0)		1-Phenyl-butane (3), C₁₀H₁₀
180	^{151, 152} 189-90	¹⁵¹ 0.861(23)	¹⁵³ 1.494(17)	<u>1-Methyl-4-ter-butyl-benzene, C₁₁H₁₆</u> . - Oxid. w CrO ₃ g. p-ter-butyl-benzoic ac. beside other cpds. (151). Maleic anhydride+AlCl ₃ g. two β-[Methyl-ter-butyl-benzyl]-acrylic acs. (Beilstein 1910, Syst. No. 1296). (153) -Htg. 9 hrs. on H ₂ O bath w. 5 times its wgt. of mixture of 1 pt. HNO ₃ (d=1.52)+2 pts. fum. H ₂ SO ₄ g. iso-dinitro-p-ter-butyl-toluene, xtals fm. dil. EtOH, odor of musk, M.P.=94-5°. (151).
182	¹³⁹ 189.5- 92.5	¹³⁹ 0.901(20/0)	¹³⁹ 1.524(n _D)	<u>1-Phenyl-2-methyl-propene- (1), C₁₀H₁₂</u> . - Nitrosite, M.P.=122°. (139).
184	^{157, 158} ¹⁵⁹ 189-90	¹⁵⁷ 0.866(22/4)	¹⁵⁷ 1.492(23)	<u>2-Methyl-2-phenyl-butane, C₁₁H₁₆</u> *
186	¹⁵⁰ 190-2	¹⁵⁰ 0.909(18)	¹⁵⁰ 1.54(n _D)	<u>1-Ethyl-4-ethinyl-benzene, C₁₀H₁₀</u> . - Anise odor. (150) -Dibromide, B.P.=168-72°, d ₁₈ =1.598. (150). Gives test 906. (150). - Bright yellow Cu salt+alk. K ₃ Fe(CN) ₆ g. p-diethyl-diphenyl-diacetylene. (150)
188	¹⁵⁶ 190-2			<u>1-Ethyl-3-isopropyl-benzene, C₁₁H₁₆</u> .
190	⁴⁴ 191-3	⁴⁴ 0.859(21/4)	⁴⁴ 1.488(21)	<u>2-Phenyl-pentane, C₁₁H₁₆</u> .
192	¹⁴⁴ 191.5- 2.5	¹⁴⁴ 0.899(14/4)	¹⁴⁴ 1.518(14)	<u>2-Methyl-3-phenyl-butene- (3), C₁₁H₁₄</u> .
194	⁴⁴² 192-3	⁴⁴² 0.965(0/0)	⁴⁴⁴ 1.513(20)	<u>m-Cresyl ethyl ether, C₉H₁₂O</u> *
196	⁴⁴² 193-4	⁴⁴² 0.885(18)		<u>2-Methyl-4-phenyl-butane, C₁₁H₁₆</u> . - Test 905-2 g. benzoic ac. (442). - Br in

DIVISION B, SECTION 1

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
198	194-5 ¹⁶²			sunlight g. Br deriv., M.P.=128-9°.(442). <u>1,5-Dimethyl-2-isopropylbenzene, C₁₁H₁₆.</u> - 3,4,6-Tribrom-1,5-dimethyl-2-isopropylbenzene, needles M.P.=261°.(162). - 3,4,6-Trinitro-1,5-dimethyl-2-isopropylbenzene, needles, M.P.= 182°.(162).
200	73,164 195-7	0.906(13) ^{73,164}	1.548(n _D) ^{73,164}	<u>1-Methyl-4[propene-(4¹)-yl]-benzene, C₁₀H₁₂.</u> - Dibromide, B.P.,=140-3°.(73). EtO ₂ NO+AcCl, cold, g. nitroso chloride, flakes, M.P.=135°.(75,164).
202	195-6 ¹⁶³			<u>1-Methyl-3-[butene-3³]-yl]-benzene, C₁₁H₁₄.</u>
204	195- ¹¹⁸ 210			<u>Dimethyl-isopropylbenzene C₁₁H₁₆.</u>
206	195-7 ¹⁶⁵ ?	0.896(0/4) ¹⁶⁶		<u>1,2,3,5-Tetramethyl-benzene "Isodurene", C₁₀H₁₄.</u> - For oxid. see Beilstein, Vol.V, pg. 430,431. - Br-aq.(167) or Br in presence of I (168) g. 4,6-dibrom-1,2,3,5-tetramethyl-benzene, needles, M.P.=198°.(166, 167). HNO ₃ -H ₂ SO ₄ g. 4,6-dinitro-1,2,3,5-tetramethyl-benzene, prisms fm. EtOH, M.P.=156-7°.(166,168).
208	195-6 ⁸⁷	0.861(16/4) ⁸⁷	1.493(16) ⁸⁷	<u>1-Ethyl-4-isopropylbenzene, C₁₁H₁₆.</u> - Br+ AlBr ₃ g. 2,3,5,6-tetra-brom-1-ethyl-4-iso-propylbenzene, xtals. fm. dil. EtOH, M.P.=246°.(87).
210	abt.196 ¹³⁰ 92.0- 2.3	0.888(16/4) ¹³⁶	1.509(n _D) ¹³⁶	<u>1-Phenyl-pentene-(2), C₁₁H₁₄.</u>

(18.5 mm.)

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
212	abt. 196 ³⁷ 79-80 (12mm.)	0.902(22/4) ³⁷	1.521(22) ³⁷	<u>1,5-Dimethyl-2-ethenyl-benzene, C₁₀H₁₂.</u> - Na+ EtOH g. 1,5-dimethyl-2-ethyl-benzene.(37).
214	144,169 ¹⁴⁴ 197-8	0.917(14/4) ¹⁴⁴	1.527(15) ¹⁴⁴	<u>3-Phenyl-pentene-(2), C₁₁H₁₄.</u> - Na+EtOH g. 3-phenyl-pentane.(144). - I + yellow HgO g. iodohydrin wh. w. AgNO ₃ g. 2-phenyl-pentanone(3).(170). - Nitrosochloride by alc. HCl or AcCl+EtONO at -10°, M.P.=117°. (144).
216	197-8 ⁹³	0.863(15/4) ⁹³	1.488(15) ⁹³	<u>2-Methyl-4-phenyl-pentane, C₁₂H₁₈.</u>
218	197-8 ¹⁷¹	0.862(18/4) ¹⁷¹	1.493(18) ¹⁷¹	<u>1-Methyl-3-n-butyl-benzene C₁₁H₁₆.</u>
220	198-9 ¹⁶⁰	0.863(16/4) ¹⁶⁰	1.487(16) ¹⁶⁰	<u>2-Methyl-4-phenyl-butane, C₁₁H₁₆.</u> - CrO ₃ mixture g. benzoic ac. slowly.(161).
222	198-9 ¹⁷¹	0.861(14/4) ¹⁷¹	1.491(14) ¹⁷¹	<u>1-Methyl-4-n-butyl-benzene, C₁₁H₁₆.</u>
224	77 ⁴⁷⁹ (12mm.)	0.877(20/4) ⁴⁷⁹	1.515(20) ⁴⁷⁹	<u>1,1,3,6-Tetramethyl-4-methylene-cyclo-hexadiene-(2,4), C₁₁H₁₆.</u> - Rearrangement g. penta-methyl-benzene.(479).
226	89-90 ⁴⁷⁹ (15mm.)	0.881(20/4) ⁴⁷⁹	1.517(20) ⁴⁷⁹	<u>1,1,4,6-Tetramethyl-4-methylene-cyclo-hexadiene-(2,4), C₁₁H₁₆.</u> - Rearrangement g. penta-methyl-benzene.(479).
228	100-3 ⁴⁷⁹ (18mm.)	0.880(20/4) ⁴⁷⁹	1.513(20) ⁴⁷⁹	<u>1,1,3,6-Tetramethyl-4-ethylidene-cyclopentadiene-(2,4), C₁₁H₁₆.</u>
230	83-5 ⁴⁷⁹ (13mm.)	0.858(20/4) ⁴⁷⁹	1.504(20) ⁴⁷⁹	<u>1,1-Dimethyl-4-propylidene-cyclohexadiene-(2,5), C₁₁H₁₆.</u> -- Rearranges to assym-propyl-o-xylene (Ber. 23,2349), B.P.=201-3

DIVISION B, SECTION 1

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
232	⁴⁷⁹ 85-6 (15mm.)	0.879(20/ ⁴⁷⁹ 4)	1.516(20) ⁴⁷⁹	$d_4^{20}=0.864$, $n_D^{20}=1.4954$. (479). <u>1,1,3-Trimethyl-4-ethylidene-cyclohexadiene-(2,4)-C₁₁H₁₆.</u> - Rearranges to sym.-ethyl-pseudocumene whose dibromide m. at 55°. (479).
234	⁴⁸⁵ 67-7.5 (11mm.)	0.869(20) ⁴⁸⁵	1.507(20) ⁴⁸⁵	<u>Tetramethyl-cycloheptatriene, C₁₁H₁₆.</u> - Odor of camphor. (485).
236	¹⁷² 198-200	0.879(20) ¹⁷²		<u>1-Methyl-3,5-diethylbenzene, C₁₁H₁₈.</u> - Oxid. w. HNO ₃ g. uvitic ac. (172). 2,4,6-Tribrom-1-methyl-3,5-diethylbenzene, fine needles, M.P.=206°. (172). - Htg. to boiling w. fum. HNO ₃ +conc. H ₂ SO ₄ g. 2,4,6-trinitro-1-methyl-3,5-diethylbenzene, yellow xtals. fm. EtOH, M.P.=86-7°. (172).
238	¹⁷³ 198-202	0.914(0) ¹⁷³		<u>2-Phenyl-pentene-(1), C₁₁H₁₄.</u> - Gives a liquid dibromide. (173).
240	¹⁷⁴ 199-200	0.873(21/ ¹⁷⁴ 4)	1.499(21) ¹⁷⁴	<u>1,2-Dimethyl-4-isopropylbenzene, C₁₁H₁₆.</u>
242	¹⁷⁵ 199-200	0.898(21/ ¹⁷⁵ 4)	1.520(27) ¹⁷⁵	<u>2-Phenyl pentene-(2), C₁₁H₁₄.</u> - Fruit-like odor. (175). -Na+EtOH g. 2-phenyl-pentane. (175). HgO+I in EtOH g. an iodo-hydrin wh. w. AgNO ₃ in aq. ether g. (Et)(Ph)CH.CO.Me. (173).
244	¹¹⁸ 200-3			<u>1,2-Diethyl-5-methylbenzene, C₁₁H₁₆.</u> - Oxid. g. MeC ₆ H ₃ (CO ₂ H) ₂ . (118).
246	abt. 200 ²¹⁹ 95 (24mm.)			<u>2-Methyl-3-phenyl-butadiene-(1,3), C₁₁H₁₆.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
248	200-1	0.870(18/4)	1.497(18)	<u>1-Methyl-2-n-butyl-benzene, C₁₁H₁₆.</u>
250	200-1 ¹⁸¹	0.884(16/4) ¹⁸¹	1.506(16) ¹⁸¹	<u>1-Phenyl-pentene-(2), C₁₁H₁₄.</u> - G. an oily dibromide. (181). O ₃ splitting g. PhCH ₂ CHO. (182).
252	200.5- ¹⁷⁸ 2.5			<u>1,3-Dimethyl-5-ter-butyl-benzene, C₁₂H₁₈.</u> - Oxid. w. HNO ₃ g. 3,5-dimethylbenzoic ac. (178). - CrO ₃ oxid. g. trimesic ac. (178). - Br+I w. cooling g. xtals. fm. EtOH of 2-brom-1,3-dimethyl-5-ter-butyl-benzene, M.P.=45°. (178). - Fum. HNO ₃ in HAc, cold, g. 2-nitro-1,3-dimethyl-5-ter-butyl-benzene, needles fm. EtOH, M.P. 85°. (178). - Addn. of 50 gms. of 2-nitro-1,3-dimethyl-5-ter-butyl-benzene to mixture of 80 g. 85% HNO ₃ +200 g. conc. H ₂ SO ₄ +htg. to 50-60° g. 2,4-dinitro-1,3-dimethyl-5-ter-butyl-benzene, yellow needles fm. EtOH, M.P.=68°. (180). H ₂ SO ₄ + fum. HNO ₃ on H ₂ O bath g. 2,4,6-trinitro-1,3-dimethyl-5-ter-butyl-benzene, needles fm. EtOH, M.P.=110°, strong odor of musk. (178).
254	201-3 ¹⁸⁴ (u.c.)	0.923(21) ¹⁸⁴		<u>1-Phenyl-butene-(1), C₁₀H₁₀.</u> - I in CHCl ₃ at 100° g. 1,2-diiodo-1-phenyl-1-butene. (185).
256	201-2 ¹⁸⁶ (u.c.)	0.860(22) ¹⁸⁶		<u>n-Amyl-benzene, C₁₁H₁₆.</u>
258	202-5 ¹⁸⁷	0.867(19) ¹⁸⁷		<u>1-Ethyl-4-n-propyl-benzene, C₁₁H₁₆.</u> - HNO ₃ (d=1.07) oxid. to p-n-propyl-benzoic ac. (187).
260	203-4 ¹⁸⁸	0.880(15/4) ³⁷	1.520(15) ³⁷	<u>1-Ethenyl-4-isopropyl-benzene, C₁₁H₁₄.</u>

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
262	203-7 ¹⁸⁹	0.858(25/4) ¹⁸⁹	1.483(25) ¹⁸⁹	<u>1-Phenyl-2-methyl-pentane,</u> <u>C₁₂H₁₈.</u>
264	203-4 ¹⁷⁶	0.889(20/4) ¹⁷⁶	1.507(20) ¹⁷⁶	<u>1-Phenyl-pentene-(4),</u> <u>C₁₁H₁₄.</u>
266	Abt. 203 84-6 ¹⁷⁷ (13mm.)			<u>2-Methyl-4-phenyl-butene-</u> <u>(3), C₁₁H₁₄.</u> -- KMnO ₄ in Me ₂ CO g. benzoic ac. + Me ₂ CHCOOH. (177).
268	203.5- ¹⁹⁰ 4.5	0.860(19/4) ¹⁹⁰	1.490(19) ¹⁹⁰	<u>2-Methyl-1-phenyl-butane,</u> <u>C₁₁H₁₆.</u>
270	204-5 ¹⁹¹			<u>1,3-Diisopropyl-benzene,</u> <u>C₁₂H₁₈.</u> - Oxid. w. boiling dil. HNO ₃ g. isophthalic ac. (191). - Trinitro-1,3- diisopropyl benzene, M.P.= 110-1°. (191).
272	204-6 ¹⁰⁵ Slight dec.	0.904(19/4) ¹⁰⁵	1.518(19) ¹⁰⁵	<u>1-[1²-Etho-butene-(11)-yl]-benz-</u> <u>C₁₂H₁₆.</u> - KMnO ₄ g. benzoic ac. (105). - Adds Br. (105). Nitroschloride, M.P.=99°. (105).
274	204-5 ¹⁹²			<u>1,2,3,4-Tetramethyl-benz-</u> <u>ene, "Prehnitene", C₁₀H₁₄.</u> -M.P.=-4°. (192). Dil. HNO ₃ g. 2,3,4-trimethyl-benzoic ac. (192). - Cold HNO ₃ - H ₂ SO ₄ g. 5,6-dinitro-1,2, 3,4-tetramethyl-benzene, M.P.=178°. (192). - Picrate, yellow needles, M.P.=92-5°. (454).
276	204-6 ²⁰⁰	0.877(15/4) ²⁰⁰	1.497(17) ²⁰⁰	<u>3-Methyl-3-phenyl-pentane,</u> <u>C₁₂H₁₈.</u>
278	205-6 ²⁰⁰	0.880(10/4) ²⁰⁰	1.496(17) ²⁰⁰	<u>2-Methyl-2-phenyl-pentane,</u> <u>C₁₂H₁₈.</u>
280	205-6 ⁴⁴⁴	1.086(15) ⁴⁴⁴		<u>Pyrocatechol dimethyl-</u> <u>ether, "Veratrol", C₈H₁₀O₂</u>

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
282	205-6 ¹⁹⁶	0.968(27/4) ¹⁹⁷	1.559(27) ¹⁹⁷	M.P.=22.5°.(444). - Htd. w. HI g. pyrocatechol+MeI. (442). <u>1-Methyl-indene, C₁₀H₁₀.</u> - Resinified by H ₂ SO ₄ +conc. HCl.(196). - Picrate, M.P.=76-8°.(198). - 1 cc. hy. added to 4 cc. HAc+ 7 cc. 90% EtOH + 1.25 g. NaNO ₂ added, by cooling + shak., g. blue, then green liquid wh., when turbid, dil. w. H ₂ O g. nitromethyl indene, yellow xtals. w. HAc of xtaln., wh. when dried in vac. over soda-lime+H ₂ SO ₄ , M.P.=107-8°.(199).
284	205-10 ¹¹⁸			<u>Methyl-amyl-benzene, C₁₂H₁₈</u>
286	205-6 ¹⁸³	0.891(18/4) ¹⁸³	1.513(18) ¹⁸³	<u>3-Methyl-1-phenyl-butene-(2), C₁₁H₁₄.</u> - O ₃ splitting g. phenyl-acetaldehyde + Me ₂ CO.(183). Br g. dibromide, xtals. fm.AcOH, M.P.=66°.(183). Nitroschloride, xtals. fm.H ₂ Cl ₂ or C ₆ H ₆ , M.P.=146-7°.(183)
288	206-7 ²⁰¹	0.907(18/4) ²⁰¹	1.530(18) ²⁰¹	<u>1,3,5-Trimethyl-2-ethinyl-benzene, C₁₁H₁₂.</u> -- 80% H ₂ SO ₄ polymerizing g.xtals fm. EtOH + ligroin, M.P.=62-4°, B.P. ₁₈ =178-80° w. slight dec.(202).
290	206-8 ²⁰³	0.903(13) ²⁰³		<u>1,5-Dimethyl-2-[propene-(2¹)yl]-benzene, C₁₁H₁₄.</u>
292	206-10 ²⁰⁴			<u>1,3-Dimethyl-5-n-propyl-benzene, C₁₁H₁₆.</u> - Boiling w. HNO ₃ (d=1.1) g. 3,5-dimethyl-benzoic ac.(204).
294	206-7 ²⁰⁵			<u>1,4-Dimethyl-2-propyl-benzene, C₁₁H₁₆.</u> - 3,5,6-Tribrom-1,4-dimethyl-2-propyl-benzene, M.P.=49°.(205). 3,5,6-Trinitro-1,4-

DIVISION B; SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
296	^{193,194} 206-7	0.966(20) ¹⁹³	1.540(20) ¹⁹³	dimethyl-2-propyl-benzene, M.P.=85°.(205). <u>1,2,3,4-Tetrahydronaphthalene, C₁₀H₁₂.</u> - Oxid. w. HNO ₃ g. phthalic ac.(195). Conc. H ₂ SO ₄ colors wine yellow.(195). Conc. HNO ₃ g. picric ac.(442).
298	^{37,206} 206-8	0.889(14/4) ³⁷	1.508(14) ³⁷	<u>1,2,4-Trimethyl-5-ethylbenzene, C₁₁H₁₆.</u> - Xs. Br in presence of I g. needles fm. EtOH, M.P.=218°.(206).
300	¹⁰⁵ 206-7	0.891(15/4) ¹⁰⁵	1.513(15) ¹⁰⁵	<u>2-Methyl-3-phenyl-pentene-(2), C₁₂H₁₆.</u>
302	⁷³ 206-8	0.903(13) ⁷³	1.534(15) ⁷³	<u>1,3-Dimethyl-4[propene-(4²)-yl]-benzene, C₁₁H₁₄.</u> - Dibromide, B.P. ₉ =151-3°, d_{15} =1.545.(73).
304	⁹³ 206-7	0.895(15/4) ⁹³	1.516(15) ⁹³	<u>4-Methyl-2-phenyl-pentene-(2), C₁₂H₁₆.</u> - Na+EtOH g. 2-methyl-4-phenyl-pentane.(93).
306	¹³⁸ 206.5- 9.5	0.893(23/4) ¹³⁸	1.527(23) ¹³⁸	<u>1-Methyl-4-[ethoethenyl]-benzene, C₁₁H₁₄.</u> Adds Br readily.(138).
308	¹³³ 207-8	0.890(15/4) ¹³³	1.525(20) ¹³³	<u>3-Methyl-1-phenyl-butene-(1), C₁₁H₁₄.</u> - Na+EtOH g. isocamyl-benzene.(133). I+ HgO in aq.-ether g. iodo-hydrin wh. w. HgO g. iso-propyl-phenyl-acetaldehyde(173).
310	^{37,201} 207-9			<u>1,3,5-Trimethyl-2-ethylbenzene, C₁₁H₁₆.</u> - Soln. in cold fum. HNO ₃ g. 4,6-dinitro-1,3,5-trimethyl-2-ethyl-benzene, needles fm. EtOH, M.P.=123°.(445).
312	²⁰⁷ 208-9	0.890(18) ²⁰⁷		<u>1-Methyl-3-[butene-(3²)-yl]-benzene, C₁₁H₁₄.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
314	²⁰⁸ 208-9	²⁰⁸ 0.869(15/4)	²⁰⁸ 1.492(n _D ¹⁵)	<u>2-Phenyl-hexane, C₁₂H₁₈.</u>
316	²⁰⁵ 208-9			<u>1,5-Dimethyl-2-n-propylbenzene, C₁₁H₁₆.</u> - Oxid. g. <u>2,4-dimethyl-benzoic ac.</u> (²⁰⁵). <u>3,4,6-Tribrom-1,5-dimethyl-2-n-propylbenzene, M.P.=39°.</u> (²⁰⁵). <u>3,4,6-Trinitro-1,5-dimethyl-2-n-propylbenzene, M.P.=110°.</u> (²⁰⁵).
318	Polymer. on boiling. Abt. ²⁰⁸ ⁴⁴⁵ 86 (11mm.) ⁴⁴⁶ 90-2 (16mm.)	⁴⁴⁷ 0.9309(16/4)	⁴⁴⁷ 1.613(16)	<u>1-Phenyl-butadiene-(1,3), C₁₀H₁₀.</u> - Leaflets in ice, M.P.=+4.5°.(⁴⁹⁶) - Htd. at 150-5°(⁴⁴⁹) or w.pyridine(⁴⁴⁸) g. "bis-phenyl-butadiene". Reduced by Na or Na-Hg in alc. to 1-phenyl-butene(2).(⁴⁵⁰). G. two tetrabromides, M.P.=151°+76°.(⁴⁵¹).
320	²⁰⁵ 209-10			<u>1,2-Dimethyl-4-n-propylbenzene, C₁₁H₁₆.</u> - Oxid. g. <u>3,4-dimethyl-benzoic ac.</u> (²⁰⁵). - <u>3,5,6-Tribrom-1,2-dimethyl-4-propylbenzene, M.P.=48°.</u> (²⁰⁵).
322	¹⁹¹ 209-10			<u>1,2-Diisopropylbenzene, C₁₂H₁₈.</u> - Oxid. g. <u>phthalic ac.</u> (¹⁹¹).
324	²⁰⁹ 209-13			<u>1-Ethyl-4-ter-butylbenzene, C₁₂H₁₈.</u>
326	¹⁰⁵ 210-2	¹⁰⁵ 0.890(16/4)	¹⁰⁵ 1.507(16)	<u>2-Methyl-3-phenyl-hexene-(2), C₁₃H₁₈.</u> - Not reduced by Na+EtOH.(¹⁰⁵).
328	¹³⁸ 210-2	¹³⁸ 0.884(27/4)		<u>2-Methyl-3-phenyl-butene-(1), C₁₁H₁₄.</u>
330	²¹⁰ 210-5 ²¹¹ 82 (9mm.)	²¹¹ 0.892(15/4)	²¹¹ 1.514(15)	<u>1-Phenyl-pentene-(1), C₁₁H₁₄.</u> - Forms a solid dibromide.(^{210,211}).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
332	210-1 ⁴⁴²	0.950(0) ⁴⁴²		<u>n-Butyl phenyl ether</u> , [*] <u>C₁₀H₁₄O.</u>
334	210-1 ²²³	0.893(20/4) ²²³	1.516(20) ²²³	<u>2-Methyl-4-phenyl-pentene-(2)</u> , <u>C₁₂H₁₆.</u> - Sol. in conc. H ₂ SO ₄ .(223). Adds 2 atoms of Br.(223). - Nitroso- chloride, M.P.=140°.(223).
336	210-2 ⁷³	0.889(20) ⁷³	1.439(20) ⁷³	<u>1-Methyl-4-[butene-(4¹)-</u> <u>yl]-benzene, C₁₁H₁₄.</u> - Di- bromide, B.P.=162-4°.(73). Nitroschloride, m. ¹⁶ p.: 148°.(73).
338	211-2 ²¹⁵	0.994(15/4) ²¹⁵	1.574(15) ²¹⁵	<u>1,4-Dihydro-naphthalene</u> , [*] <u>C₁₀H₁₀.</u> - Adds Br in CHCl ₃ , w. cooling by ice in NaCl mixture, g. 2,3- dibrom-1,2,3,4-tetrahydro- naphthalene.(215). - Frozen at +15.5°.(442). Cf. Div. A, Sect. 2, #46: mp: 24.5-48°.
340	211-3 ²²⁴	0.955(19/0) ²²⁴	1.550(n_D) ²²⁴	<u>1-Cyclopropyl-1-phenyl-</u> <u>ethene, C₁₁H₁₂.</u> - G. w. a drop of conc. H ₂ SO ₄ a golden ppt. wh. dissolves in alc. w. color of neutral litmus.(224).
342	212-4 ²⁰⁸	0.914(17/4) ³⁷	1.538(17) ³⁷	<u>1,2,4-Trimethyl-5-ethenyl-</u> <u>benzene, C₁₁H₁₄.</u>
344	abt. 212 ⁴⁷³ 74.5- 8.5 (19mm.)			<u>[Dimethyl-methylene]-</u> <u>cyclo-pentadiene, "ω,ω-</u> <u>Diethyl-fulvene", C₁₀H₁₄.</u> - Orange-yellow oil.(473)
346	60-5 ⁴⁷⁹ (15mm.)	0.866(20/4) ⁴⁷⁹	1.514(20) ⁴⁷⁹	<u>1,1,2-Trimethyl-4-methyl-</u> <u>ene-cyclohexadiene-(2,4),</u> <u>C₁₀H₁₄.</u> - Rearrangement g. isodurene.(479).
348	212-3 ²²⁵	0.864(9) ²²⁵		<u>1-Methyl-4-isoamyl benzene</u> , <u>C₁₂H₁₈.</u> - CrO ₃ oxid. to terephthalic ac.(225).
350	abt. 213 ²⁷³ 84 (10mm.)			<u>2-Phenyl-hexene-(3),</u> <u>C₁₂H₁₆.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
352	²⁴¹ 213-5	0.958(17) ²⁴¹	1.532 ²⁴¹	<u>Cyclopentyl-benzene,</u> <u>C₁₁H₁₄.</u>
354	⁴⁴² 213-6			<u>n-Butyl benzyl ether,</u> <u>C₁₁H₁₆O.</u>
356	⁴⁴² 214-5	1.080(0/4) ⁴⁴²		<u>Resorcinol dimethyl ether,</u> <u>C₈H₁₀O₂.</u> - Vol. kw. st. (442) *
358	²²⁶ 214-6	0.871(0) ²²⁷		<u>1-n-Propyl-4-isopropyl-</u> <u>benzene, C₁₂H₁₈.</u> - Boil- ing dil. HNO ₃ oxid. to n- propyl benzoic ac. + tere- phthalic ac. (226). - 10 pts. hy. + 20 pts. Br + 1 pt. I w. ice-cooling g. 2,5(?)-dibrom-1-propyl-4- isopropyl-benzene, liquid, wh. w. HNO ₃ (d=1.51) g. 2,5(?)-dibrom- <u>iso</u> -dinitro- 1-propyl-4-isopropyl- benzene, fine needles fm. pet. ether, M.P.= 124-5°. (226).
360	¹⁷⁴ 214-5	0.867(21/4) ¹⁷⁴	1.497(21) ¹⁷⁴	<u>1-Methyl-2-ethyl-4-iso-</u> <u>propyl-benzene, C₁₂H₁₈.</u> - Odor of cymene. (174).
362	²²⁹ 214-5	0.857(16) ¹⁸⁶		<u>2-Methyl-5-phenyl-pentane,</u> <u>C₁₂H₁₈.</u>
364	²²⁸ 214.5- 5.5	0.86(19/4) ²²⁸	1.488(19) ²²⁸	<u>2,2-Dimethyl-1-phenyl-</u> <u>butane, C₁₂H₁₈.</u>
366	⁴⁴² 215-20	0.920(21/4) ⁴⁴²		<u>Isoamyl phenyl ether,</u> <u>C₁₁H₁₆O.</u> *
368	⁴⁴² 216-7	0.953(0) ⁴⁴²		<u>Methylthymyl ether,</u> <u>C₁₁H₁₆O.</u>
370	^{73,203} 216-8	0.907(18) ^{73,203}	1.544(n_D) ^{73,203}	<u>1-Ethyl-4-[propene-(4¹)-</u> <u>yl]-benzene, C₁₁H₁₄.</u> - Dibromide, B.P.=162-5°, d ₁₅ =1.574. (73).
372	²³⁰ 216-7	0.863(20/4) ³⁷	1.495(17) ³⁷	<u>1,3,5-Triethyl-benzene,</u> <u>C₁₂H₁₈.</u> - CrO ₃ mixt. oxid.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				to trimesic ac. (231). - Br.+I g. 2,4,6-tribrom-1,3,5-triethyl-benzene, xtals fm. EtOH, M.P.=105-6°. (231). Conc. H ₂ SO ₄ +fum. HNO ₃ g. bright yellow needles, fm. dil. EtOH of 2,4,6-trinitro-1,3,5-triethyl-benzene, M.P.=108-9° explode w. r. htg. (231).
374	217-8 ²³²	0.874(15/4) ²³²	1.494(17) ²³²	<u>2,4-Dimethyl-2-phenyl-pentane, C₁₃H₂₀.</u>
376	217-8 ³⁷	0.882(17/4) ³⁷	1.498 ³⁷	<u>1,2,4-Triethyl-benzene, C₁₂H₁₈.</u> - Br+AlBr ₃ +I g. 1,2,4-triethyl-3,5,6-tribrom-benzene, needles fm. EtOH, M.P.=88-9°. (37).
378	abt. 217 ^{213,223} 108 (16mm.)	0.890(16/4) ^{213,223}	1.506(16) ^{213,223}	<u>1-Phenyl-hexene(2), C₁₂H₁₆.</u>
380	218-9 ²¹²	0.889(25) ²¹²		<u>1-Methyl-4-[butene-(4¹)-yl]-benzene, C₁₁H₁₄.</u>
382	480,481 72-4 (9mm.)	0.900(20/4) ^{480,481}	1.503(20) ⁴⁸¹	<u>α-Tetrameride of propadiene-(1,2), C₁₂H₁₈.</u>
384	480,481 101 (10mm.)	0.935(20/4) ^{480,481}	1.526(20) ^{480,481}	<u>β-Tetrameride of propadiene-(1,2), C₁₂H₁₈.</u> - Normal type of allene polymer.
386	218.5- ⁹³ 20.5 w. dec.	0.928(20/4) ⁹³	1.524(20) ⁹³	<u>2-Methyl-4-phenyl-pentadiene-(2,3), C₁₂H₁₄.</u> - Odor of lemons. (93). - Na+EtOH g. 2-methyl-4-phenyl-pentene-(2). (93). Br g. tetrabromide. (93).
388	219-20 ¹⁰²	0.861(20/4) ¹⁰²	1.490(20) ¹⁰²	<u>Hexyl-benzene, C₁₂H₁₈.</u>
390	219-23 ²³³	0.926(22) ²³³		<u>1,4-Dimethyl-2-[propene-(2¹)-yl]-benzene, C₁₁H₁₄.</u>
392	220-3 ⁷³	0.926(22) ⁷³	1.544(n _D) ⁷³	<u>1,4-Dimethyl-2-[propene-(2¹)-yl]-benzene, C₁₁H₁₄.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
394	220-1 ²³⁴	0.864(15/4) ²³⁴	1.490(n _D) ²³⁴	Dibromide, B.P. ₁₇ =163-6°, d ₁₆ =1.459.(73). <u>3-Methyl-1-phenyl-pentane, C₁₂H₁₈.</u> - D-form=odor of cymene; [α] _D ^{14.5} =+17.2°. (234).
396	220-2 ²³²	0.866(25/4) ²³²	1.492(25) ²³²	<u>3-Ethyl-3-phenyl-pentane, C₁₃H₂₀.</u>
398	130-2 ⁴⁸³ (20mm.)	0.928(20/4) ⁴⁸³	1.517 ⁴⁸³	<u>1-Methyl-6-cyclohexylidene-cyclohexene-(1), C₁₃H₂₀.</u>
400	107-8 ⁴⁸⁴ (13mm.)	0.880(15/4) ⁴⁸⁴	1.503(15) ⁴⁸⁴	<u>1-Methyl-2-n-propyl-4-methoxyphenyl-cyclohexadiene-(2,6), C₁₃H₂₀.</u> - [α] _D ²² +86.20°. (484). - Warming w. 3% soln. of HCl in HAc g. 1-methyl-2-propyl-4-isopropyl-benzene. (484). Br reacts instantaneously. (484).
402	^{238,239} 220-1	0.876(25/4) ²³⁹	1.501(25) ²³⁹	<u>1,3,5-Trimethyl-2-n-propylbenzene, C₁₂H₁₈.</u> - Odor like mesitylene. (238). - Oxid. w. dil. HNO ₃ g. 2,4,6-trimethyl-benzoic ac. (238). - Soln. in cold fum. HNO ₃ g. fine needles fm. EtOH, M.P.=93-4° (besides a cpd. of M.P.=135°) (238).
404	220-1 ⁷³	0.896(18) ⁷³	1.528(20) ⁷³	<u>1,4-Dimethyl-2-[butene-(2)-yl]-benzene, C₁₂H₁₆.</u> - Dibromide, M.P.=75°. (73).
406	220.5- ²³⁶ 1.5			<u>1,4-Di-n-propyl-benzene, C₁₂H₁₈.</u> - Boiling dil. HNO ₃ oxid. g. p-propyl-benzoic ac. (237). - Dropping hy. w. cooling into xs. Br g. <i>iso</i> -dibrom-1,4-di-n-propyl-benzene, xtals. fm. EtOH, M.P.=48° (237), wh. by soln. w. warming in HNO ₃ (d=1.52) g. <i>iso</i> -dibrom- <i>iso</i> -dinitro-1,4-dipropyl benzene, M.P.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
408	¹³⁸ 221-4			= 145°.(226). - Soln. in fum. HNO ₃ g. <u>eso-dinitro-1,4-dipropyl-benzene</u> , xtals. fm. EtOH, M.P.=65°.(237). <u>1-Methyl-4-[propoethenyl]-C₁₂H₁₆.</u>
410	²⁴⁰ 221.5- 3.5	0.880(21/4) ²⁴⁰	1.506(21) ²⁴⁰	<u>1,2,4-Trimethyl-5-isopropyl-benzene, C₁₂H₁₈.</u>
412	¹⁷⁵ 22-3	0.870(15/4) ¹⁷⁵		<u>2-Methyl-5-phenyl-hexane, C₁₃H₂₀.</u> - Odor like cymene (175).
414	abt.222 ⁷⁴ 128-30 (50 mm.)	0.898(10) ⁷⁴	1.527(n_D) ⁷⁴	<u>1-Methyl-4-isopropyl-2-ethinyl-benzene, C₁₂H₁₄.</u> - Dibromide, B.P. ₂₄ =150-5°.(74). *G. Ag salt.(74).
416	²⁴¹ 223-5	0.965(20) ²⁴¹	1.536(n_D) ²⁴¹	<u>1-Phenyl-cyclopentene-(2) or -(3), C₁₁H₁₂.</u> - Immediately decolorizes Aq. Br+KMnO ₄ .(241).
418	²⁴² 223-5			<u>n-Butyl-o-cresyl ether, C₁₁H₁₆O.</u>
420	²³⁹ 225.5- 4.5	0.899(21/4) ²³⁹	1.523(21) ²³⁹	<u>1,3,5-Trimethyl-2-[propene-(2¹)-yl]-benzene, C₁₂H₁₆.</u> - Odor of turpentine.(239). Unattacked by Na+EtOH.(239). - Nitrosochloride, xtals., M.P.=146.5°.(130).
422	^{73,203} 224-6	0.915(18) ^{73,203}	1.556(n_D) ^{73,203}	<u>1,2-Dimethyl-4-[propene-(4¹)-yl]-benzene, C₁₁H₁₄.</u> - Dibromide, B.P. ₁₆ =165-8°, d_{13} =1.591 ₉ (73).
424	²⁴² 224-6			<u>1,5-Diethyl-2-isopropyl-benzene, C₁₃H₂₀.</u> - Conc. H ₂ SO ₄ + fum. HNO ₃ g. <u>2,4,6-trinitro-1,3-diethyl-benzene.</u> (242).
426	abt.224 ¹⁰⁴ 128-30 (50 mm.)	0.888(17) ¹⁰⁴		<u>1-Methyl-4-isopropyl-2-ethinyl-benzene, C₁₂H₁₄.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydro carbon
428	225-6 ²⁴³	0.869(15/4) ²⁴³	1.496(15) ²⁴³	<u>1-Methyl-2-n-propyl-4-isopropyl-benzene, C₁₃H₂₀.</u> - Br+AlBr ₃ g. pentabromtoluene.
430	225-35 ¹¹⁸			<u>Di-ter-butyl-benzene(?)</u> , C ₁₄ H ₂₂ .
432	225-35 ⁷³	0.931(22) ⁷³	1.543(n _D) ⁷³	<u>1-Isopropyl-4-[propene-(4¹)-yl]-benzene, C₁₂H₁₆.</u> - Dibromide, B.P. ₂₀ =169-72°, d ₁₅ =1.512.(73).
434	226-8 ⁷³	0.890(19) ⁷³	1.523(n _D) ⁷³	<u>1-Methyl-4-isopropyl-3-[propene-(3¹)-yl]-benzene, C₁₃H₁₈.</u> - Dibromide, B.P. ₁₉ =167-70°, d ₁₅ =1.432.(73).
436	226-8 ⁷³	0.897(18) ⁷³	1.535(20) ⁷³	<u>1,3-Dimethyl-4-[butene-(4¹)-yl]-benzene, C₁₂H₁₆.</u> - Dibromide, B.P. ₁₅ =167-9°.(73).
438	226-9 ⁴⁴²	0.933(0) ⁴⁴²		<u>Ethyl thymyl ether, C₁₂H₁₈O.</u> - At 360°-400° splits to thymol+ethylenene.(442).
440	226-8 ²⁰³	0.890(18) ²⁰³		<u>1-Methyl-4-isopropyl-3-[propene-(3¹)-yl]-benzene C₁₃H₁₈.</u>
442	226-7 ²¹¹	0.901(18/4) ²¹¹	1.51(18) ²¹¹	<u>3-Methyl-1-phenyl-pentene-(2), C₁₂H₁₆.</u> - Nitroschloride, M.P.=151.(211).
444	226-8 ²⁰³	0.894(19/4) ²⁰³		<u>1,5-Dimethyl-2-[butene-(2¹)-yl]-benzene, C₁₂H₁₆.</u> - Nitroschloride, needles, M.P.=135°.(203).
446	226.5-7.5 ²³⁹	0.890(19/4) ²³⁹	1.516(19) ²³⁹	<u>1,3,5-Trimethyl-2-[2²-methopropene-(2¹)-yl]-benzene, C₁₃H₁₈.</u> -AcCl+hy.+EtONO g. nitroschloride, needles fm. hot EtOH, M.P.=136°.(239).
448	abt.227 106-8 ^{3c} (19.5mm)			<u>3-Benzylidene pentane, C₁₂H₁₆.</u>

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
450	²⁴⁴ 227-8			<u>4-Phenyl-heptene-(3),</u> <u>C₁₃H₁₈.</u> - Nitrosochloride, M.P.=112° w. dec. (244).
452	Abt.228 ³⁷ 96-7 (12mm.)	<i>0.892(1/4)</i> ³⁷	<i>1.514(16)</i> ³⁷	<u>1,4-Diethyl-2-ethenyl-</u> <u>benzene, C₁₂H₁₆.</u> - Na+EtOH g. 1,2,4-triethyl-benzene. (37).
454	²³⁹ 228.5- 30.5	0.878(18/4) ²³⁹	1.500(18) ²³⁹	<u>1,3,5-Trimethyl-2-iso-</u> <u>butyl-benzene, C₁₃H₂₀.</u>
456	²⁴⁵ 229-30	0.89(15) ²⁴⁵		<u>4-Isopropyl-1-[propene-</u> <u>(1¹)-yl]-benzene, C₁₂H₁₆.</u>
458	Abt.229 ¹⁵⁵ 93.5-4 (10mm.)			<u>1-Methyl-4-[4¹methoprop-</u> <u>ene(4¹)-yl]-benzene,</u> <u>C₁₁H₁₄.</u>
460	Abt.229 ¹⁷⁶ 94-5 (10mm.)	0.844(20/4) ¹⁷⁶	1.503(20) ¹⁷⁶	<u>1-Phenyl-hexene-(5),</u> <u>C₁₂H₁₆.</u>
462	²⁵⁰ 229-30	0.916(17) ²⁵⁰		<u>1-Methyl-4-isopropyl-2-</u> <u>isobutyl-benzene, C₁₄H₂₂.</u>
464	⁷³ 230-3	0.907(20) ⁷³	1.541(20) ⁷³	<u>1-Ethyl-4-[butene-(4¹)-</u> <u>yl]-benzene, C₁₂H₁₆.</u> - Dibromide, B.P.c=146-9°. (73).
466	²⁴⁶ 230-2	0.937 ²⁴⁶	1.521(16) ²⁴⁶	<u>1-Methyl-3-phenyl-cyclo-</u> <u>pentane, C₁₂H₁₆.</u> - De- colorizes aq. Br+alk. KMnO ₄ at once.(246). - KMnO ₄ at 100° or CrO ₃ g. benzoic ac. (246).
468	²²⁵ 232-3	0.895(9) ²²⁵		<u>Dimethyl-isoamyl-benzene,</u> <u>C₁₃H₂₀.</u>
470	^{251, 494} 232-3	0.857(20/4) ²⁵⁷	1.487(20) ²⁵⁷	<u>n-Heptyl-benzene, C₁₃H₂₀.</u>
472	⁴⁴² 233-4	0.989(28) ⁴⁴²	1.554(34) ⁴⁴⁴	<u>p-Anethole*, C₁₀H₁₂O.</u> - See Div. A, Sect. 2, No. 40.-M.P.=22.5°.(444).

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
474	⁴⁴² 233-4	1.108(15) ⁴⁴²	1.543(11) ⁴⁴⁴	Safrole* <u>C₁₀H₁₀O₂</u> . - Strong sassafras odor. (442). - M.P. after solidification by cold, 11°. (442). - Quickly reduces a 1% neutral KMnO ₄ soln. upon shak. (442). - Is violently attacked and completely carbonized in Test 907 w. conc. H ₂ SO ₄ . (442).
476	²⁵⁵ 233.5- 4.5	1.009(4/4) ²⁵⁵		<u>1,2-Benzo-cycloheptadiene-(1,3), C₁₁H₁₂</u> . - Odor like naphthalene. (255). - Dec. by htg. - Adds Br. (255). - Reacts w. KMnO ₄ . (255).
478	^{175, 256} 234-6	0.899(15/4) ^{175, 256}		<u>1,2,4-Trimethyl-5-[5¹-methoxypropene-(5¹)-yl]-benzene, C₁₃H₁₈</u> .
480	²⁵³ 234-6			<u>1,3,5-Triisopropyl-benzene, C₁₅H₂₄</u> . - HNO ₃ at 190-200° oxid. g. trimesic ac. (253).
482	²⁵⁴ 234-5	0.889(15) ²⁵⁴		<u>1-Isopropyl-4-[4²-methoxypropene-(4¹)-yl]-benzene, C₁₃H₁₈</u> . - Aq. Br g. liquid dibromide. (254).
484	²⁵⁰ 234-5	0.892(17) ²⁵⁰		<u>1-Methyl-4-isopropyl-2-n-butyl-benzene, C₁₄H₂₂</u> .
486	⁴⁴⁴ 235-6			<u>Resorcinol diethyl ether, C₁₀H₁₄O₂</u> . - M.P.=12.4°. (444).
488	Abt. 235 ¹⁷⁵ 121 (20mm.)	0.881(15/4) ¹⁷⁵		<u>5-Methyl-2-phenyl-hexene-(2), C₁₃H₁₈</u> . - Na+EtOH g. 2-methyl-5-phenyl-hexane. (175).
490	Abt. 235 ²¹⁷ 88 (7 mm.)			<u>2-Methyl-4-phenyl-butene-(1)-ene-(3), C₁₁H₁₆</u> .
492	²⁵² 235-7			<u>2,4-Dimethyl-1-phenyl-pentadiene-(1,3), C₁₃H₁₆</u> .

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
494	237-40 ²⁵⁸			<p><u>ter-Butyl-hydrindene, C₁₂H₁₈.</u> - 10 pts. hy.+40 pts. fum. HNO₃+80 pts. fum. H₂SO₄ (15%SO₃), w. cooling g. odorless xtals. fm. EtOH, of dinitro-ter-butyl-hydrindene, M.P.=121°, wh. w. 4 pts. fum. HNO₃+8pts. fum. H₂SO₄ (30-40%SO₃) at 50-5° g. xtals. fm. EtOH of trinitro-ter-butyl-hydrindene, M.P.=140°, odor of musk. (258).</p>
496	238-40 ⁴⁸⁶	0.905(15)		<p><u>"8-Butenyl-camphenylidene" C₁₃H₂₀.</u></p>
498	247, 248 238-9	0.944(20/0) ²⁴⁸		<p><u>Phenyl-cyclohexane, C₁₂H₁₈.</u> - Hot alk. KMnO₄ g. benzoic ac. (248). - HNO₃ (d=1.075) in sealed tube at 100-110° g. 1-nitro-1-phenyl-cyclohexane, needles, M.P.=54.5-6°, besides other products. (249). - Addn. of HNO₃ (d=1.52) to hy. w. ice cooling till dissolved (140 cc. HNO₃ per 30 g. hy) g. prismatic xtals. fm. EtOH, M.P.=57.5-8.5°. (249)</p>
500	238-9 ⁷³	0.911(15) ⁷³	1.546(20) ⁷³	<p><u>1,2-Dimethyl-4-[butene-(4¹)-yl]-benzene, C₁₂H₁₈.</u> - Dibromide, B.P._c=155-7°. (73).</p>
502	239-40 ²³⁹	0.890(20/4) ²³⁹	1.511(20) ²³⁹	<p><u>1,3,5-Trimethyl-2-[2³-methobutene-(2¹)-yl]-benzene, C₁₄H₂₀.</u> - Ester-like odor. (239). - Nitroschloride, needles, M.P.=185°. (239).</p>
504	240-50 ²⁵⁹	0.920(20) ²⁵⁹		<p><u>Hexahydrofluorene, C₁₂H₁₈.</u> - From French gas coal. (259). - Violet fluorescence in benzene. (259). - Polymerizes on boiling. (259). - 3 pts. fum. HNO₃. + 3 pts. conc. H₂SO₄. +</p>

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
506	²¹³ 214 240-60 ²¹³ 116 (16mm.)	0.938(13/4) ²¹³	1.611(13) ²¹³	5 pts. glac. AcOH g. di-nitro-tetrahydrofluorenone, light yellow amorphous powder fm. EtOH + H ₂ O, dec. without melting at 95-100°. (259). <u>1-Phenyl-pentadiene-(1,3), C₁₁H₁₂.</u> - Xtalizes at -4°. (213). - Polymerizes on standing to a thick oil. (213). - Na+EtOH g. 1-phenyl-pentene(2). (213). - Adds 4 atoms of Br. (214).
508	^{260, 261} 240-3	1.001(19) ²⁶⁰		<u>1-Methyl-naphthalene, C₁₁H₁₀.</u> - M.P.=22°. (260). - Picrate, yellow, M.P.=141-2°. (457). FP -22
510	¹⁴² 240-5			<u>1-Methyl-x,x-butyl-benzene, C₁₅H₂₄.</u> - HNO ₃ -H ₂ SO ₄ g. <i>eso</i> -trinitro-di- <i>ter</i> -butyl-toluene, xtals. fm. EtOH, M.P.=152-3°. (142).
512	²⁴⁴ 241-3	0.854(14) ²⁴⁴		<u>4-Benzyl-heptane, C₁₄H₂₂.</u> <i>1 phenyl 2 propyl pentane</i>
514	⁷³ 241-4	0.935(14) ⁷³	1.527(20) ⁷³	<u>1-Methyl-4-isopropyl-3-[butene-(3¹)]-yl-benzene, C₁₄H₂₀.</u> - Dibromide, B.P. ₇ =150-2°. (73).
516	¹¹⁸ 241-7			<u>Tri-<i>ter</i>-butyl-benzene, C₁₈H₃₀.</u>
518	²⁶² 241.5- 3.5	0.875(23/4) ²⁶²	1.498(23) ²⁶²	<u>1,3,5-Trimethyl-2-iso-amyl-benzene, C₁₄H₂₂.</u>
520	⁴⁴² 242-3 (th.i.)			<u>2-Methyl-naphthalene, C₁₁H₁₀.</u> - Picrate, M.P.=116-7°. (456). *
522	⁷³ 242-3	0.893(14) ⁷³	1.533(20) ⁷³	<u>1-Isopropyl-4-[butene-(4¹)]-yl]-benzene, C₁₃H₁₈.</u> - Dibromide, B.P. ₁₀ =152-7°. (73).

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
524	242-3 ²⁴⁵	0.888(15) ²⁴⁵		<u>1-Isopropyl-4-[butene-(4¹)-yl]-benzene, C₁₃H₁₈.</u> - Aq. Br.g. a solid dibromide. (245)
526	243-8 ²⁶³			<u>1-Methyl-3,5-di-n-propylbenzene, C₁₃H₂₀.</u> - Oxid. w. dil. HNO ₃ g. uvitic ac. (263).
528	243-4 ⁴⁴²	1.117(15/4) ⁴⁴²	1.563(15) ⁴⁴⁴	<u>cis-iso-Safrole, C₁₀H₁₀O₂.</u>
530	244-5 ⁴⁴²			<u>Dimethyl orcynyl ether, C₉H₁₂O₂.</u> - Almost insol. in aq. (442).
532	244-5 ²⁵⁰	0.89(17) ²⁵⁰		<u>1-Methyl-4-isopropyl-2-isoamylbenzene, C₁₅H₂₄.</u>
534	abt. 245 ²³⁵ 133(20 mm.)	0.994(14/4) ²³⁵	1.569(14) ²³⁵	<u>1-Phenyl-cyclohexene-(1), C₁₂H₁₄.</u>
536	245-55 ²⁶⁵			<u>6-Methyl-1-phenyl-heptane, C₁₄H₂₂.</u> <i>Isooctyl benzene</i>
538	abt. 245 ⁴³⁰ 117 (18 mm.)			<u>3-Benzyl-hexane, C₁₃H₂₀.</u>
540	246-8 ²⁴⁴	0.902(19) ²⁴⁴		<u>1-[1²-Propopentene-(1¹)-yl]-benzene, or 4-Benzylheptene-(3), C₁₄H₂₀.</u> - Nitrosochloride, M.P. = 115°. (244).
542	247-9 ²⁶⁶			<u>Phenyl-cyclohexadiene-(x,x), C₁₂H₁₂.</u> - Adds 2 atoms of Br directly. (266).
544	Abt. 247 ²⁶⁴ 245 (716mm.)			<u>Phenyl-cyclohexene-(x), C₁₂H₁₄.</u> - Adds 2 atoms of Br. (264).
546	116 ³²⁴ (20mm.)			<u>1-Methyl-2-phenyl-cyclopentene-(2), C₁₂H₁₄.</u>

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
548	²⁶⁷ 248-52	0.958(22/4) ²⁶⁷	1.540(22) ²⁶⁷	<u>1-Methyl-3-phenyl-cyclohexene-(4 or 5), C₁₃H₁₆.</u> - Immediately decolorizes dil. KMnO ₄ +HCCl ₃ soln. of Br.(267).
550	²⁵⁴ 248-50			<u>4-Methyl-1-phenyl-pentadiene-(1,3), C₁₂H₁₄.</u>
552	⁴⁴⁴ 248-9	1.123(15/4) ⁴⁴⁴	1.574(15) ⁴⁴⁴	<u>trans-iso-Safrole, C₁₀H₁₀O₂*</u>
554	⁴⁴⁴ 249-50	1.055(15) ⁴⁴⁴		<u>Eugenol methyl ether, C₁₁H₁₄O₂*</u>
556	^{37,288} 249-50	0.888(16/4) ³⁷	1.504(16) ³⁷	<u>1,2,4,5-Tetraethyl-benzene, C₁₄H₂₂.</u> - M.P.=+13°.(288). - Oxid. g. pyromelletic ac.(37). -Br g. 3,6-dibrom-1,2,4,5-tetraethyl-benzene, M.P.=113°(37); B.P.=325-30°.(289).
558	²⁴⁹ 249.5-50.5	0.943(18/4) ²⁴⁹	1.525(18) ²⁴⁹	<u>1-Methyl-3-phenyl-cyclohexane, C₁₃H₁₈.</u> - L form shows $[\alpha]_D = -1.06^\circ$.(249).
560	Abt.250 ²²⁰ 113-5 (15mm.) ²¹⁸ 102-3 (9mm.)	0.930(13/4) ^{220,221}	1.583(13) ^{220,221}	<u>3-Methyl-5-phenyl-pentene-(2)-ine-(4), or 3-Methylene-1-phenyl-pentene-(1), C₁₂H₁₈.</u> - Conc. H ₂ SO ₄ g. 1-phenyl-3-methylpentene (1)-ol(3). (218).
562	²⁷⁹ 250-3	0.949(20) ²⁷⁹	1.529(20) ²⁷⁹	<u>1-Cyclohexene-(1)-yl-cyclohexene(1), C₁₂H₁₈.</u>
564	²⁹² 250-1	1.008(0) ²⁹²		<u>2-Ethyl-naphthalene, C₁₂H₁₈.</u> - Freezes in a cooling mixture of -19°.(291). - Dil. HNO ₃ oxid. to β -naphthoic ac.(293). -Picrate, yellow needles, M.P.=71°.(452).
566	Abt.251 ²³⁴ 100-103 (9mm.)	0.891(15/4) ²³⁴	1.528(15) ²³⁴	<u>3-Methyl-1-phenyl-pentene-(1), C₁₂H₁₈.</u> - D form shows $[\alpha]_D^{15} = +50.3^\circ$.(234). -Na+EtOH g. optically active 3-methyl-1-phenyl-pentane.(234).

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
568	⁴⁸⁷ 103-6 (17mm.)	0.862(22/ ⁴⁸⁷ 4)	1.522(22) ⁴⁸⁷	<u>1,3-Dimethyl-5-[2-methylene-propylidene]-cyclohexene-(3), C₁₂H₁₈.</u>
570	²⁹⁴ 251-2	1.008(25/ ²⁹⁴ 4)	1.607(25) ²⁹⁴	<u>1-Methoethenyl-naphthalene, C₁₃H₁₂.</u>
572	abt.253 ²⁷⁸ 138 (20mm.)	0.970(14/ ²⁷⁸ 4)	1.545(14) ²⁷⁸	<u>Benzylidene cyclohexane, C₁₂H₁₆.</u>
574	²⁸⁸ 253-4	0.887(20/ ²⁹⁵ 4)	1.508(20) ²⁹⁵	<u>1,2,3,4-Tetraethyl-benzene, C₁₄H₂₂.</u> - KMnO ₄ oxid. g. prehnitic ac.(²⁹⁶). - 5,6-Dinitro-1,2,3,4-tetraethyl-benzene, weak, lemon yellow, transparent prisms fm. EtOH, M.P.= 115°.(²⁹⁶).
576	²⁶⁸ 253-4			<u>3,4,5,11-Tetrahydroacenaphthene, C₁₂H₁₂.</u> - Cpd. w. picryl chloride, M.P.=82-3°.(²⁶⁹). - Picrate, M.P.=152-3°.(²⁶⁹). - Br in HCCl ₃ g. dibromacenaphthene - tetrahydride(3,4,5,11), xtals. fm. C ₆ H ₆ , M.P.= 138°.(^{266,270,271}).
578	⁴⁴⁴ 254-5	1.021(20/ ⁴⁴⁴ 4)	1.530(10) ⁴⁴⁴	<u>Eugenol ethyl ether, C₁₂H₁₆O₂.</u>
580	abt.256 ²⁹⁹ 118-9 (14 mm.)	0.895(14/ ²⁹⁹ 4)		<u>1,3,5-Trimethyl-2-[butene-(2¹)-yl]-benzene, C₁₃H₁₈.</u> - Odor of mesitylene.(²⁹⁹). - Nitroschloride, xtals., M.P.= 122-2.5°.(²⁹⁹).
582	abt. 256 ^{232,233} 113-5 (9mm.)	0.940(20) ^{232,233}	1.527(20) ^{232,233}	<u>1,1,6-Trimethyl-1,4,9,10-tetrahydronaphthalene, "Irene", C₁₃H₁₈.</u> - CrO ₃ + HAc oxid. to trioxdehydroirene, cf. Beilstein, 1910, Vol. 5, pg. 506.

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
584	²⁹⁰ 257-9.5 w. slight dec.	1.012(12/0) ²⁹⁰		<u>1-Ethyl-naphthalene, C₁₂H₁₂-Picrate, lemon-yellow, M.P.=98°.(290). -Much xs. Br g. x,x,x-tribrom-1-ethyl-naphthalene, fine white needles fm. ether, M.P.=127°.(290).</u>
586	²⁸⁷ 155-8 (15mm.)			<u>1-Ethynyl-naphthalene, C₁₂H₈. - 1+xs. HgO in aq. ether g. α-naphthylacet-aldehyde.(287).</u>
588	³¹⁷ 130.5 (105mm)			<u>1-[1⁴-Phenyl-ethylidene]-cyclopentadiene-(2,4), -"ω-Methyl-ω-phenyl-fulvene," C₁₃H₁₂. -Red oil w. odor of azobenzene.(317)</u>
590	²⁴⁹ 257-8	0.937(18/4) ²⁴⁹	1.524(18) ²⁴⁹	<u>1-Methyl-3-cyclohexylbenzene, C₁₃H₁₈. - Htg. w. dil. HNO₃ (d=1.080) g. isophthalic ac.(249).</u>
592	⁴⁵⁹ 258-60 ³¹⁸ 143 (20mm.)	0.960(23) ⁴⁵⁹	1.555(20) ³¹⁸	<u>1-Methyl-3-phenyl-cyclohexene-(2 or 3), C₁₃H₁₆. - Nitrosochloride, M.P.=124-7° w. dec.(459).</u>
594	²⁹⁸ 260-2			<u>1-Methyl-4-[4²-methocyclohexene-(4¹)-yl]-benzene, C₁₄H₁₈. - Adds Br.(298). - Dil. HNO₃ g. terephthalic ac.(298).</u>
596	²⁷⁸ 260-1	0.970(14/4) ²⁷⁸	1.541(14) ²⁷⁸	<u>1-Phenyl-1-cyclohexylidene-ethane, C₁₄H₁₈.</u>
598	²⁴⁹ 260-1	0.937(18/4) ²⁴⁹	1.523(18) ²⁴⁹	<u>1-Methyl-4-cyclohexylbenzene, C₁₃H₁₈. - Htg. w. dil. HNO₃ (d.=1.080) g. terephthalic ac.(249)g</u>
600	abt.261 ¹⁸³ 116 (16mm.)	0.927(18/4) ¹⁸³	1.521(18) ¹⁸³	<u>3-Methyl-1-phenyl-hexene-(2), C₁₃H₁₈.</u>
602	⁴⁶⁰ 261-4	1.010(20/4) ⁴⁶¹		<u>2-Methyl-diphenyl, C₁₃H₁₂. -KmnO₄ g. o-phenyl-benzoic ac.(460).</u>

DIVISION B, SECTION I

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
604				<u>Octahydrophenanthrene, C₁₄H₁₈.</u> - M.P. abt. -4°. (302).
606	³⁰³ 261-3 u.c.	0.849(15) ³⁰³		<u>n-Octyl-benzene, C₁₄H₂₂.</u> - Freezes at -7°. (304). - KMnO ₄ oxid. g. benzoic ac. (304). - Treatment w. fum. HNO ₃ in the cold, filtration of m-nitro cpd. +htg. of filtrate g. xtals. of p-nitro-octyl-benzene by rextaln. fm. EtOH + sublimation fm. m-nitro cpd. (m-cpd. comes over first, then on strong htg. the p-cpd.) yellow needles, M.P.=204°. (304).
608	¹⁷⁶ 115-7 (8 mm.)	0.879(20/4) ¹⁷⁶	1.500(n _D) ¹⁷⁶	<u>1-Phenyl-octene-(7?), C₁₄H₂₀</u>
610	²⁷³ 104 (8 mm.)			<u>3-Phenyl-octene-(4), C₁₄H₂₀</u>
612	⁴⁶⁷ 111-3 (11 mm.)			<u>2-Methyl-3-isopropyl-butene-(3), C₁₄H₂₀.</u> ^{4-phenyl}
614	⁴⁶⁷ 121-3 (10 mm.)			<u>2-n-Propyl-1-phenyl-pentene-(1), C₁₄H₂₀.</u>
616	³¹⁵ 115-6 (10 mm.)			<u>1-[14,14-Dimetho-1³-methyl-enopentene-(1⁴)-yl]-benzene, C₁₄H₁₆.</u>
618	⁴⁷⁰ 262-3	1.006(15) ⁴⁷⁰		<u>1,6-Dimethyl-naphthalene, C₁₂H₁₂.</u> - Picrate, orange-red needles, M.P.=111-2° (114°). (470).
620	⁴⁶⁹ 147-9 (15mm.)			<u>1,7-Dimethyl-naphthalene, C₁₂H₁₂.</u> - Picrate, M.P.=125°. (469).
622	³⁰⁵ 262.5- 4.5	^{306, 307} 1.018(16/4)	³⁰⁶ 1.616(16)	<u>1,4-Dimethyl-naphthalene, C₁₂H₁₂.</u> - Xs. Br g.x, x, x-tribrom-1,4-dimethyl-naphthalene, needles fm.

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
624	³⁹³ 143-4 (25mm.) ²⁸⁷ 135-8 (15m.m.)	³⁹³ 1.057		<u>CHCl₃, m.p.: 228°(305)-Picrate, orange needles, m.p.: 141°(462).</u> <u>1-Ethynyl-naphthalene,</u> <u>C₁₂H₈.</u> - G. test 906.(393) -H ₂ SO ₄ g. methyl- α -naphthyl- -ketone.(393). -I+xs. HgO in aq. ether g. α -naphthylacetaldehyde.(287).
626	⁴⁴² 263-7	⁴⁴² 1.015(27)		<u>4-Methyl-diphenyl, C₁₈H₁₂.</u> -Freezes at (-2°)-(-3°). (442).
628	⁴⁴⁴ 264-5	⁴⁴⁴ 1.055(20/4)		<u>iso-Eugenol methyl ether,</u> <u>C₁₁H₁₄O₂.</u> *
630	³⁰³ 264-6	³⁰³ 0.977(17)	³⁰³ 1.549(17)	<u>Phenylcyclohexylethane,</u> <u>C₁₄H₂₀.</u>
632	³⁰⁹ 265-7			<u>1-[Propene-(1²)-yl]-</u> <u>naphthalene, C₁₃H₁₂.</u> - Boiling alc. KOH g. 1- [propene-(1 ¹)-yl]-naphthal- ene.(309).
634	²⁹³ 265-6	²⁹³ 0.990(0)		<u>2-Isopropyl-naphthalene,</u> <u>C₁₃H₁₄.</u> - Dil. HNO ₃ at 170° g. β -naphthoic ac. (293). -Picrate, lemon yellow needles. M.P.= 89°.(293).
636	³¹⁰ 265-6	³¹⁰ 0.863(16/4)	³¹⁰ 1.492(15)	<u>1-Isopropyl-4-[4³-metho-</u> <u>pentyl]-benzene, C₁₅H₂₄.</u> - D-form shows $[\alpha]_D^{15} = +15.91^\circ$ (310).
638	³⁹⁵ 124-6 (20 mm.) 110-2 (395) (10 mm.)	³⁹⁵ 0.873(17/4)	³⁹⁵ 1.498(25)	<u>2,6-Dimethyl-4-phenyl-</u> <u>heptene-(3), C₁₅H₂₂.</u> - Adds Br easily.(395).
640	³⁴² 139-40.5 (9.5mm.)	³⁴² 0.880(16/4)	³⁴² 1.518(16)	<u>1-Isopropyl-4-[4³-metho-</u> <u>pentene-(4⁴)-yl]-benzene,</u> <u>C₁₅H₂₂.</u> - D-form shows $[\alpha]_D^{16} = +41.89^\circ$.(342).
642	⁴⁸¹ 100 (8 mm.)	⁴⁸¹ 0.858(20/4)	⁴⁸¹ 1.487(20)	<u>Trimeride of 2-Methyl-</u> <u>butadiene-(2,3), C₁₅H₂₄.</u> - Oxid. w. benzyl peroxide g. C ₁₅ H ₂₄ O ₂ , M.P.=49°, B.P.=137°.(481). -KMnO ₄ g. xtaln. cpd., M.P.= 120-2°+ketone, C ₁₂ H ₁₈ O,

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
644	⁴⁸¹ 108-10 (17 mm.)			B.P. ₁₇ =112-3°, whose semi-carbazone, M.P.=288°.(481). <u>Trimeride of Pentadiene-(2,3), C₁₅H₂₄.</u>
646	^{480,481} 131-2 (10.5 mm.)	^{480,481} 0.950(20/4)	^{480,481} 1.528(20)	<u>Pentameride of propadiene-(1,2), C₁₅H₂₀.</u>
648	³⁸⁸ 152 (9.5 mm.)	³⁸⁸ 0.895(17/4)	³⁸⁸ 1.528(17)	<u>3,7-Dimethyl-1-phenyl-octadiene-(1,x), C₁₆H₂₂.</u> - Br both adds and substitutes in the cold.(388). - L-form shows [α] _D ¹⁷ =65.11°.(388).
650	³¹¹ Abt.265	³¹¹ 0.887(0)		<u>Diamyl-benzene, C₁₆H₂₆.</u>
652	⁴⁶³ 145-6 (9.5 mm.)	⁴⁶³ 0.884(12/4)	⁴⁶³ 1.503(n _D)	<u>3,7-Dimethyl-1-phenyl-octene-(5,6, or 7), C₁₆H₂₄.</u> - L-form shows [α] _D =-7.26°.
654	²¹¹ Abt.267 130 (20 mm.)	²¹¹ 0.959(19/4)	²¹¹ 1.537(n _D)	<u>3-Methyl-1-phenyl-pentadiene-(1,3), C₁₂H₁₄.</u> - Turpentine odor.(211). - Na+EtOH g. 3-methyl-1-phenyl-pentene-(2).(211).
656	⁴⁴² 267.5- 8.5			<u>o-Tolyl phenyl ether,</u> * <u>C₁₂H₁₂O.</u> - M.P.=21.5-2.0°.(442). - Boiling w.KMnO ₄ soln. g. phenyl ether of salicylic acid. (442).
658	²⁴³ 268-9	²⁴³ 0.978(15/4)	²⁴³ 1.568(15)	<u>2-Methyl-5-isopropyl-diphenyl, C₁₆H₁₈.</u>
660	^{308,312} 268-70	^{175,303} 0.988(25/4)	^{308,314} 1.576(20)	<u>1,1-Diphenyl-ethane, C₁₄H₁₄</u> - Solidifies in freezing mixture and melts at ordinary temp.(313). - CrO ₃ oxid. g. benzophenone.(313)
662	⁴⁴² 269-70	⁴⁴² 1.096(14/4)	⁴⁴⁴ 1.623(14)	<u>Methyl-α-naphthyl ether, C₁₁H₁₀O.</u> - G. red xtaln. cpd. w. picric ac. - Split by conc. HCl at 150°.(442).

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No.	Boiling Point (c°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
664	²³⁵ Abt. 269 148 (20mm.)	²³⁵ 0.973(12/4)	²³⁵ 1.551(12)	<u>1-Benzyl-cyclohexene-(1),</u> <u>or Benzylidene-cyclo-</u> <u>hexane, or probably both,</u> <u>C₁₃H₁₆.</u>
666	⁷⁴ Abt. 270 124-6 (14-6 mm.)	⁷⁴ 0.900(21)		<u>2,4,6-Triethyl-1-ethinyl-</u> <u>benzene, C₁₄H₁₈.</u> - G. yellow, amorphous, Cu salt. (74).
668	¹⁰⁴ Abt. 270 110-20 (10mm.)	¹⁰⁴ 0.912(17)		<u>1-Ethinyl-4-isopropyl-</u> <u>benzene, C₁₁H₁₂.</u>
670	³¹⁶ 270-1			<u>2,3'-Dimethyl-diphenyl,</u> <u>C₁₄H₁₄.</u>
672	²³⁹ 270-2	²³⁹ 0.884(17/4)	²³⁹ 1.514(17)	<u>1,3,5-Trimethyl-2-[heptene-</u> <u>-(2¹)-yl]-benzene, C₁₆H₂₄.</u> -Faint odor like mesityl- ene. (239). - G. a di- bromide. (239). - Nitroso- chloride, xtals., M.P. = 160° w. dec. (239).
674	³¹⁸ Abt. 270	³¹⁸ 0.948(17)	³¹⁸ 1.528(17)	<u>1-Ethyl-3-phenyl-cyclo-</u> <u>pentane, C₁₃H₁₈.</u>
676	³¹⁸ 270-1	³¹⁸ 0.959(20)	³¹⁸ 1.547(n _D)	<u>1-Methyl-3-benzyl-cyclo-</u> <u>hexene-(2 or 3), C₁₄H₁₈.</u>
678	¹⁵⁰ Abt. 271 110-20 (10mm.)	¹⁵⁰ 0.918(17)		<u>1-Ethinyl-4-isopropyl-</u> <u>benzene, C₁₁H₁₂.</u>
680	³²¹ 271-2			<u>2-Methyl-1,1,1,2-tetra-</u> <u>phenyl-propane, C₂₃H₂₆.</u>
682	²³⁹ 271.5- 2.5	²³⁹ 0.875(17/4)	²³⁹ 1.497(17)	<u>1,3,5-Trimethyl-2-n-heptyl-</u> <u>benzene, C₁₆H₂₆.</u>
684	³¹⁹ 272-7	³¹⁹ 1.051(0)		<u>3-Methyl-diphenyl, C₁₃H₁₂.</u> <u>KMnO₄ g. m. phenyl-</u> <u>benzoic ac. (320).</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
684	³¹⁹ 272-7	1.031(0) ³¹⁹		<u>3-Methyl-diphenyl, C₁₃H₁₂.</u> -KMnO ₄ g. m. phenyl- benzoic ac. (320).
686	²¹³ Abt. 272 128 (16 mm.)	0.925(12/4) ²¹³	1.603(12) ²¹³	<u>1-Phenyl-hexadiene-(1,3),</u> <u>C₁₂H₁₄.</u> - Na+EtOH g. 1- phenyl-hexene-(2). (213).
688	³²² 272-80			<u>2,4'-Dimethyl-diphenyl,</u> <u>C₁₄H₁₄.</u> - CrO ₃ +HAc g. 1st 2-methyl-diphenyl-carbonic acid(4') and then tere- phthalic ac. (322). -Br g. 2'- or 3'-brom-2,4-dimethyl- diphenyl, fm. EtOH, M.P.= 93-5° conc. (322). -Br in CS ₂ g. x,x-dibrom-2,4'- dimethyl-diphenyl, fm. EtOH, M.P.=156° corr. (322)
690	³²⁵ 274-5			<u>1-Propyl-naphthalene,</u> <u>C₁₃H₁₄.</u> - Picrate, M.P.= 141-2°. (325).
692	Abt. 274 ²¹⁸ 120-2 (12mm.)			<u>2,3-Dimethyl-5-phenyl-</u> <u>pentene-(2)-ine(4), C₁₃H₁₄.</u>
694	³²⁷ 274-5	0.879(11/4) ³²⁷	1.496(n _D) ³²⁷	<u>1-[1³,1⁷-Dimethooctyl]-</u> <u>benzene, C₁₆H₂₂.</u> - L-form shows† [α] _D ^{10.5} : -1.82°. (327). - Stable to KMnO ₄ + Br. (327). -Fum. H ₂ SO ₄ (6%SO ₃) g. a sulfonic ac. (327).
696	⁴⁴³ 274.5- 5.5			<u>m-Tolyl phenyl ether,</u> <u>C₁₂H₁₂O.</u>
698	³⁰⁹ 275-8 w.p. polym.			<u>1-[Propene-(1⁴)-yl]-</u> <u>naphthalene, C₁₃H₁₂.</u> - I+HgO in aq. ether + removal of HI g. C ₁₀ H ₇ . CHMe.CHO. (309). -Picrate, red xtals., M.P.=110°. (464).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
700	³³⁶ 125 (8 mm.)	1.014(9/4) ³³⁶	1.614(9) ³³⁶	<u>1-Methoethenyl-naphthalene, C₁₃H₁₂.</u> - I+xs. HgO in aq. ether g. iodohydrin wh. by removal of HI g. α-naphthyl-acetone.(309). - Picrate, M.P.=91°.(336).
702	³²⁶ 275-6	0.997(18) ³²⁶		<u>1-Methyl-3-benzyl-benzene, C₁₄H₁₄.</u> - Htg. w. much HNO ₃ (d.:1.4) at 90° pptg. w. H ₂ O, washing ppt. w. cold ether, + xtaln. fm. EtOH g. a di-nitro cpd., needles fm. HAc, M.P.=141°.(326).
704	^{465a, 331} 276-7	^{465a, 335} 1.038(14/4)	^{465a, 335} 1.610(14)	<u>1,1-Diphenyl-ethene, C₁₄H₁₂.</u> -Na+EtOH g. 1,1-diphenyl-ethane.(335). - I + yellow HgO+Aq. EtOH g. iodohydrin wh. by removal of HI g. desoxybenzoin.(332). CrO ₃ mixture g. benzophenone.(333).
706	²³⁴ 142-3 (21mm.)			<u>1,2-Diphenyl-ethene, "Iso-stilbene", C₁₄H₁₂.</u> - Odor, especially dil. "bluten ahnlichen".(234). -Htg. at 170-8° g. stilbene.(234). -KMnO ₄ soln. in EtOH etg. MgSO ₄ g. benzoin.(234). - Br in ether g. principally 237° melting dibromdibenzyl.(235). -Xs. Br in CS ₂ in dark w. cooling g. principally 110-110,5° melting cpd.(236).
708	³²³ 277-8	0.896(20/4) ³²⁹	1.513(n _D ³²⁹)	<u>Pentaethyl-benzene, C₁₆H₂₆.</u> - Br in HAc g. 6-brom-1, 2,3,4,5-pentaethyl-benzene, needles fm. EtOH, M.P.= 47.5°, B.P. abt. 315° (323). -Dec. by fum. H ₂ SO ₄ to tetraethyl+hexaethyl benzene.(442).
710	³³⁴ 277-9			<u>2-n-Propyl-naphthalene, C₁₃H₁₄.</u> - Picrate, M.P.= 90-2°.(325).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
712	⁴⁴³ 277.5- 8.5			<u>p-Tolyl phenyl ether,</u> <u>C₁₃H₁₂O.</u>
714	^{330,338} 278.5- 80	^{335,338} 0.992(14/4)	^{335,333} 1.566(14)	<u>1,1-Diphenyl-propane,</u> <u>C₁₅H_{16.}</u>
716	³³⁸ 279-81	³³⁸ 1.004(24/0)	³³⁸ 1.587	<u>1,1-Diphenyl-propene-(2),</u> <u>C₁₅H_{14.}</u>
718	Abt.279 277-8 (²⁵²) (724mm.)			<u>2,6-Dimethyl-4-benzylidene-</u> <u>heptadiene(2,5), C₁₆H_{20.}</u>
720	³⁴⁰ 279-80			<u>ter(?) - Butyl-naphthalene,</u> <u>C₁₄H_{16.}</u> - HNO ₃ -H ₂ SO ₄ g. trinitro cpd., brown-red xtals. fm. EtOH, sinter at 50°, liquify at 79-80°. (340). - Picrate, M.P.=96°. (340).
722	⁴⁴² 280-1 ³⁴⁶ 112-3 (6mm.)			<u>2-Isobutyl-naphthalene,</u> <u>C₁₄H_{16.}</u> - Faint odor.(346). Picrate, yellow needles, M.P.=96°.(442).
724	³⁴⁶ 136-8 (11mm.)			<u>1-Isobutyl-naphthalene,</u> <u>C₁₄H_{16.}</u>
726	³⁴¹ 280-5	³⁴¹ 0.993(15)	³⁴¹ 1.537(15)	<u>Octahydrophenanthrene,</u> <u>C₁₄H_{18.}</u> - Liquid at -10°. (341). - Easily oxid. without g. phenanthra- quinone. (341).
728	³⁴³ 281-3			<u>1-Methyl-4-n-octyl-benz-</u> <u>ene, C₁₅H_{24.}</u> - M.P.=11-2°. (343). - Oxid. w. KMnO ₄ g. terephthalic ac.(343).
730	⁴⁴² 281-2	⁴⁴² 1.057(0/4)	⁴⁴⁴ 1.602(20)	<u>Ethyl-α-naphthyl ether,</u> <u>C₁₂H₁₂O.</u> - M.P.= 5.5°. (442).
732	³³⁴ 281-3			<u>1-n-Butyl-naphthalene,</u> <u>C₁₄H_{16.}</u> - Picrate, m.p.: 104-6°(³²⁵).
734	³⁴⁴ 281-2			<u>2,2-Diphenyl-propane, C₁₅H₁₆</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
736	⁴⁸⁷ 109-11 (3 mm.)			<u>1,3-Dimethyl-2-isopropyl-1-phenyl-butene-(1)</u> , <u>C₁₅H₂₂</u>
738	³⁴⁵ 282-3 (282 at 737)	1.012(20/ ³⁴⁵ 4)	1.560(20) ³⁴⁵	<u>Octahydrophenanthrene</u> , <u>C₁₄H₁₈</u> . - M.P. = (-12°) - (-11°). (345). - Sol. in abt. in 15 pts. MeOH + abt. 10 pts. EtOH. (345). - CrO ₃ in AcOH oxid. to phenanthraquinone. (345). - Warm conc. H ₂ SO ₄ g. 1st wine red, then brown + black color. (345). - Conc. H ₂ SO ₄ + K ₂ Cr ₂ O ₇ g. green-black soln. (345).
740	³³⁴ 283-5			<u>2-n-Butyl-naphthalene</u> , <u>C₁₄H₁₈</u> . - Picrate, M.P. = 71-4°. (325).
742	³⁴⁷ 283-4	1.043(0) ³⁴⁷		<u>3-Ethyl-diphenyl</u> , <u>C₁₄H₁₄</u> . - CrO ₃ oxid. g. m-phenylbenzoic acid. (347).
744	³⁴⁸ 283-8	0.939(20/0) ³⁴⁸	1.518(20) ³⁴⁸	<u>1-Methyl-4-isopropyl-3-phenyl-cyclo-hexane</u> , " <u>Phenyl-p-menthane</u> ", <u>C₁₆H₂₄</u> .
746	³⁵⁰ 284-9 9	1.025 ³⁵⁰		<u>x,x-Dimethyl-diphenyl</u> , <u>C₁₄H₁₄</u> .
748	³⁴⁹ 284.5- 7.5	0.984(15) ³⁴⁹		<u>2-Methyl-1,2-diphenylpropane</u> , <u>C₁₆H₁₈</u> . - Fluorescent. (349).
750	³⁷ 285-6	0.986(17/4) ³⁷	1.455 ³³⁸	<u>1,2-Diphenylpropane</u> , <u>C₁₅H₁₆</u> . - HNO ₃ (d.=1.075) g. 3-nitro-1,2-diphenylpropane (besides other products), prisms, M.P. = 153-5°. (330).
752	²⁷² Abt. 285 128 (6 mm.)			<u>1-Methyl-2-phenyl-cyclohexene-(2)?</u> , <u>C₁₃H₁₆</u> . - Absorbs Br energetically g. violet color. (272).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
754	⁴¹³ 167-8 (10mm.)			<u>1-Phenyl-2-[2³-methocyclohexene-(2¹ or 2⁶)]-yl-benzene, C₁₉H₂₀.</u>
756	³⁸³ 142 (20mm.)	0.971(12/ ³⁸³ 4)	1.549(12) ³⁸³	<u>1-p-Tolyl-cyclohexene-(1), C₁₃H₁₆.</u>
758	³⁸⁴ 147 (23mm.)	0.972(14/ ³⁸⁴ 4)	1.555(14) ³⁸⁴	<u>1-Methyl-4-phenyl-cyclohexene-(3), C₁₃H₁₆.</u>
760	²¹³ Abt. 285 136 (16mm.) ²¹³ 143 (22mm.)	0.925(20/ ²¹³ 4)	1.587(20) ²¹³	<u>5-Methyl-1-phenyl-hexadiene-(1,3), C₁₃H₁₆.</u> - Odor faintly of cinnamon. (213).
762	³⁷⁹ 126-8 (8mm.)			<u>1-Ethyl-3-phenyl-cyclohexadiene-(1,3), C₁₄H₁₆.</u>
764	³⁸⁵ 170 (42mm.)	0.981(18) ³⁸⁵	1.453(18) ³⁸⁵	<u>1-Methyl-2-benzyl-cyclohexene-(2)?, C₁₄H₁₈.</u> - Odor of lemons. (385). - Absorbs Br w. blue coloration. (385).
766	³⁸⁵ 158-60 (12mm.)	0.961(20) ³⁸⁵	1.541(20) ³⁸⁵	<u>1-Methyl-2-o-tolyl-cyclohexene-(2)?, C₁₄H₁₈.</u> - Yellow liquid. (385). - Absorbs Br g. violet color. (385).
768	³⁸⁴ 160 (30mm.)	0.957(16/ ³⁸⁴ 4)	1.542(16) ³⁸⁴	<u>1-Methyl-4-benzyl-cyclohexene-(3), or 1-Methyl-4-benzylidene-cyclohexane, or mixture of both, C₁₄H₁₈.</u>
770	³⁵¹ 285-6	0.994(18) ³⁰²		<u>Phenyl-p-tolyl-methane, C₁₄H₁₄.</u> - Freezes at abt. -30°. (352). -HNO ₃ -H ₂ SO ₄ g. prisms fm. HCCl ₃ or C ₆ H ₆ , M.P.=160-1°. (353).
772	³⁵⁴ 285-6	1.021(0) ³⁵⁵		<u>1-Phenyl-1-p-tolyl-ethene, C₁₅H₁₄.</u> - I+HgO in aq. alc. g., lst, iodo-hydrin wh. by subtraction of HI g phenyl-p-xylyl-ketone. (355).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
774	^{355, 356} 285-7	1.009(18/0) ³⁵⁶	1.587(n ₁₈) ³⁵⁶	<u>1,2-Diphenyl-butane, C₁₆H₁₈</u>
776	³⁵⁶ 285-6	0.978(16/0) ³⁵⁶	1.56(n ₁₆) ³⁵⁶	<u>1,1-Diphenyl-2-methyl-propane, C₁₆H₁₈</u> .
778	³⁵⁷ Abt. 288 286-7 (713mm.) ³⁵⁸ 286 (716mm.)			<u>3,3'-Dimethyl-diphenyl, C₁₄H₁₄</u> . - By long cooling at -16° freezes to xtaline mass wh. M.P.=5-7° (359). - CrO ₃ mixt. g. isophthalic ac. (359). - Conc. HNO ₃ +Conc. H ₂ SO ₄ at abt. 75° g. 4,4'-dinitro cpd., faintly yellow needles fm. EtOH, M.P.=228 (359).
780	³⁵⁶ 286-8	0.975(16/0) ³⁵⁶	1.554(n ₁₆) ³⁵⁶	<u>1,1-Diphenyl-butane, C₁₆H₁₈</u> .
782	^{356, 335} 286-7 w. slight dec.	^{356, 335} 1.030(18/4)	^{356, 335} 1.592(18)	<u>1,1-Diphenyl-butene-(1), C₁₆H₁₆</u> . - Na+EtOH reduces only w. difficulty. (335).
784	³⁶⁰ 286.5- 91.5	0.959(20/0) ³⁶⁰	1.527(20) ³⁶⁰	"Phenyl-dihdropinene", <u>C₁₆H₂₂</u> . - Sat'd. character. (360).
786	⁴¹⁹ 157-8 (13-14 mm.)	0.980(15/4) ⁴¹⁹	1.554(n _j ¹²) ⁴¹⁹	<u>C₁₆H₂₀ from phenyl fenchol</u> -D form shows [α] _D ¹⁵ =+0.60. (419). - Does not add HBr in AcOH. (419).
788	⁴¹⁹ 139-41 (16mm.)			<u>C₁₆H₂₀ from phenyl-fenchol</u> - M.P.=16-7°. (419). - D-form shows [α] _D ¹⁸ = +22.60°. (419). - Slowly adds HBr in AcOH soln. g. cpd. M.P.=115-6°. (419).
790	²⁷⁵ Abt. 287 138-41 (10mm.)	0.974(18/11) ²⁷⁵		<u>1,7,7-Trimethyl-2-phenyl-bicyclo-[1,2,2]-heptene-(2)(?)</u> , "β-Phenyl-Camphene", <u>C₁₆H₂₀</u> .
792	³⁶¹ 287-8			<u>2-[2²-Methopropene-(2¹)-yl]-naphthalene, C₁₄H₁₄</u> .

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
794	⁴¹¹ 128 (14mm.)	⁴¹¹ 0.99	⁴¹¹ 1.546(20)	<u>3-Methyl-1,2,3,4,12,13-hexahydrofluorene, C₁₄H₁₈.</u> - Decolorizes neither Br nor KMnO ₄ . (411).
796	²⁹³ 288-92	²⁹³ 0.973(0)		<u>2-Isoamyl-naphthalene, C₁₅H₁₈.</u> - Dil. HNO ₃ at 170° g. β-naphthoic ac. (293). - Picrate, lemon yellow, M.P.=110°. (293).
798	³⁴⁵ 290-1	³⁴⁵ 1.045(20/4)	³⁴⁵ 1.570(20)	<u>Hexahydrophenanthrene, C₁₄H₁₈.</u> - M.P.=(-8°)-(-7°). (345). - Sol. in abt. 15 pts. MeOH + abt. 10 pts. EtOH. (345). - Sol. in warm conc. H ₂ SO ₄ w. brown-black color, in presence of K ₂ Cr ₂ O ₇ w. green-black color. (345). - "G. no picrate." (345). - CrO ₃ in AcOH oxid. (345).
800	¹⁰⁴ Abt. 291 110 (10mm.)	¹⁰⁴ 0.909(18)		<u>1-Ethyl-4-ethinyl-benzene, C₁₀H₁₀.</u> - Odor strongly like anise. (104).
802	³³⁷ 291-3	³³⁶ 0.98		<u>1-Phenyl-1-[1⁴-methoxyphenyl]-ethane, C₁₅H₁₆.</u> - Oxid. g. p-benzyl-benzoic ac. (336).
804	³⁵⁶ 292-3	³⁵⁶ 1.024(16/0)	³⁵⁶ 1.596(n ₁₆)	<u>1,1-Diphenyl-2-methylpropene-(1), C₁₆H₁₆.</u>
806	³⁶² 293-5			<u>1-Phenyl-2-[2⁴-ethoxyphenyl]-ethane, C₁₆H₁₈.</u> - Blue fluorescence. (362).
808	³⁶⁵ 293.5- 4.5			<u>1,4-Dimethyl-2-benzylbenzene, C₁₅H₁₈.</u>
810	³⁶³ 294-5	³⁶³ 0.985(19)		<u>1-Ethyl-4-benzyl-benzene, C₁₅H₁₆.</u> - Oxid. g. p-benzoyl-benzoic ac. (363).
812	³⁶⁴ 294-5	³⁶⁴ 0.974(20/4)		<u>1,1-Di-p-tolyl-ethane, C₁₆H₁₈.</u> - CrO ₃ mixture g. di-p-tolyl-ketone+ p-tolyl-benzoic ac. (364).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
814	³⁶⁵ 295-6			<u>1,5-Dimethyl-2-benzyl-benzene, C₁₅H₁₆.</u> - Oxid. g. benzophenone-dicarboxic ac- (2,4). (365).
816	⁴⁴² 295-8 (th.i.)	1.036(16) ⁴⁴²		<u>Benzyl ether, C₁₄H₁₄O.</u> - Htd. above 315° g. benzaldehyde, toluene, + resinous matter. (442).
818	³⁶⁶ 295-6			<u>1,2-Di-m-tolyl-ethane, C₁₈H₂₂.</u>
820	³³⁵ Abt. 296			<u>1,2-Diphenyl-butene-(1), C₁₆H₁₆.</u> - KMnO ₄ in H ₂ SO ₄ g. benzoic ac. + EtPhCO. (335). Cf. Div. A, Sect. 2, No. 188.
822	296-7	0.964(21/0) ³⁶⁸	1.551(21) ³⁶⁸	<u>2-Methyl-4,4-diphenyl-butane, C₁₇H₂₀.</u> - Slightly fluorescent. (368).
824	³⁶⁷ 296.5- 7.5	0.969(15/4) ³⁶⁷	1.557(15) ³⁶⁷	<u>1-Methyl-4-isopropyl-2-benzyl-benzene, C₁₇H₂₀.</u>
826	³⁶⁹ 298- 302			<u>1,4-Dimethyl-6-ethyl-naphthalene, C₁₄H₁₆.</u>
828	³⁶⁸ 298-9	0.979(21/0) ³⁶⁸	1.581(21) ³⁶⁸	<u>1,1-Diphenyl-pentene-(1), C₁₇H₁₈.</u> - "Yellow". (368).
830	³⁷⁸ 184-5 (10mm.)			<u>1,5-Diphenyl-pentene-(2), C₁₇H₁₈.</u>
832	³⁷⁸ 186 (11mm.)			<u>1,5-Diphenyl-pentene-(1), C₁₇H₁₈.</u>
834	³⁷⁸ 187-9 (10mm.)			<u>1,5-Diphenyl-pentane, C₁₇H₂₀.</u>
836	^{370, 372} 298-9	1.007(20/0) ³⁷⁰	1.576(20) ³⁷⁰	<u>1,3-Diphenyl-propane, C₁₅H₁₆.</u> - Dropping fum. HNO ₃ into AcOH soln. of hy. g. x,x-dinitro cpd., needles fm. HCl ₃ , M.P.= 139°. (371). - Dropping hy. into fum. HNO ₃ g. x,x,x,x-

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
838	⁴¹⁷ 178-9 (15 mm.)			tetranitro cpd., needles fm. $HCCl_3$, m.p.: $162-4^\circ$ (371). <u>1,3-Diphenyl-propene, $C_{15}H_{14}$.</u> - Hyacinth odor. (417). - $KMnO_4$ g. chiefly benzoic ac., besides other products. (417). - Br g. liquid dibromide. (417).
840	³⁷⁴ Abt. 300			<u>1,3-Diphenyl-butane(?)</u> , $C_{16}H_{18}$.
842	³⁹⁶ 301-2	0.972 (21/ ³⁹⁶ 4)		<u>2,5-Diphenyl-hexene-(2)?</u> , $C_{18}H_{20}$. - Not reduced by $Na+EtOH$. (396). - Decolorizes hot $KMnO_4$. - Slowly adds 2 atoms of Br. (396, 397).
844	⁴⁹⁴ 206-8 (20mm.)			<u>1,6-Diphenyl-hexane, $C_{18}H_{22}$</u> - To identify, slowly drop hy. into 10 pts. HNO_3 ($d=1.52$) at -15° at rate of 20 g./hr., let stand 0.5 hr. in ice, 2 hrs. at rm. temp., finally a few min. on H_2O bath, + pour into cold H_2O ; g. tetranitro-diphenyl-hexane, needles, M.P.= 90° , wh. w. CrO_3 g. 2,4-dinitro-benzoic ac., + wh. w. alc. $(NH_4)_2S$ g. a little mono-amino-trinitro cpd. + p,p'-diamino-o,o'-dinitro cpd., M.P.= 124° , + o,o'-diamino-pp' dinitro cpd., reddish yellow leaves, M.P.= $150-1^\circ$ + wh. w. $Sn+HCl$ g. tetramine, needles, M.P.= 138° , whose tetrahydrochloride, m.p.: 275° , needles, M.P.= $213-5^\circ$, whose tetrabenzoyl deriv., M.P.= 151° , whose tetraacetyl deriv., obtained by boiling 10 min. w. xs. Ac_2O , M.P.= 270° , whose p,p'-diacetyl deriv., obtained by htg. w. $AcOH$ ctg. few drops H_2O for 1 hr., M.P.= 167° .
846	³⁹⁰ 302-3	1.080 (20/ ³⁴⁵ 4)	³⁴⁵ 1.582 (20)	<u>2,7,9,10-Tetrahydrophenanthrene, $C_{14}H_{14}$.</u> - Br in

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
848	³⁹³ 302-3			<p>HCCL₃ g. 2,7-dibrom-phenanthrene+ small amts. of 9-bromphenanthrene. (391) -Picrate, orange-red needles, M.P.=105-6°. (391)</p> <p><u>1-Iso-amyl-naphthalene, C₁₅H₁₈.</u> - Picrate, M.P.=85-90°. (393).</p>
850	⁴⁰¹ 165-6 (13mm.)			<u>9-Ethyl-fluorene, C₁₅H₁₄.</u>
852	³⁹⁹ 303-4			<p><u>1,4-Diphenyl-2,3-dibenzyl-butene-(2), C₃₀H₂₂.</u> - Faint violet fluorescence. (399). -Absorbs Br w. formation of cpd., M.P.=188°. (399). -Fum. HNO₃ at -4° g, white needles w. green sheen fm. C₆H₆+EtOH of tetra-nitro cpd., M.P.=156° wh. dec. by exposure to sunlight or on long standing. (399).</p>
854	³⁴⁵ 303-4	³⁴⁵ 1.085 (20/4)	³⁴⁵ 1.582 (20)	<p><u>x,x,x,x-Tetrahydro-phenanthrene, C₁₄H₁₄.</u> - M.P.=(-3)-(-4°). (345). "Gives no picrate". (345).</p>
856	³⁵⁶ 303-4	³⁵⁶ 1.018 (16/0)	³⁵⁶ 1.593 (n ₁₆)	<u>2-Methyl-1,3-diphenyl-propene, C₁₆H₁₆.</u>
858	³⁸⁸ 139-40 (10mm.)	³⁸⁸ 0.946 (16/4)	³⁸⁸ 1.580	<p><u>1-Methyl-4[methoethenyl]-3-phenyl-cyclohexane, C₁₆H₂₂.</u> - D-form shows [α]_D=+17°. (388).</p>
860	⁴⁹⁵ 149-50 (12mm.)	⁴⁹⁵ 0.938 (14/4)	⁴⁹⁵ 1.528 (14)	<u>1-Methyl-3-[3⁴-isopropo-phenyl]-cyclohexene-(4 or 5), C₁₆H₂₂.</u>
862	³⁹² 157-8 (8mm.)			<u>1,3-Diphenyl-cyclobutane, C₁₆H₁₆.</u>
864	³⁶³ 304-5	³⁶³ 0.974 (21/0)	³⁶³ 1.553 (21)	<u>1-Phenyl-2-benzyl-butane, C₁₇H₂₀.</u> - Fluorescent. (363).
866	³⁵⁰ 304-10	³⁵⁰ 0.999 (0)		<u>x,x-Diethyl-diphenyl, C₁₆H₁₈.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
868	305-7 ⁴⁰⁰	1.043(15) ⁴⁰⁰	1.580(15) ⁴⁰⁰	Hexahydrophenanthrene, <u>C₁₄H₁₆</u> . - M.P.=-3°. -Pale yellow.(400). - Easily oxidized without g. phenanthraquinone.(400). - Br in AcOH g. 2 xtaln. Br cpds., one M.P.=150°, insol. in ether, the other M.P.= 142° sol. in ether.(400). - Picrate, M.P.= 106°.(400).
870	Abt. 306 ³⁶⁸ 165 (30mm.)			<u>2,3-Dimethyl-1-phenyl-butadiene-(1,3), C₁₂H₁₄</u> .
872	306-7 ³⁶⁸	1.016(21/0) ³⁶⁸	1.589(21) ³⁶⁸	<u>1-Phenyl-2-benzyl-butene-(1), C₁₇H₁₈</u> .
874	307-8 ⁴⁰²	0.969(15) ⁴⁰²		<u>1-Methyl-4-isopropyl-2-(or 3)-benzyl-benzene, C₁₇H₂₀</u> . - Oxid. g. benzoyl-terephthalic ac.(402).
876	152-4 ⁴¹⁹ (14-5 mm.)		1.547(19) ⁴¹⁹	<u>1,3,3-Trimethyl-2-benzylidene-bicyclo-[1,2,2]-heptane, C₁₇H₂₂</u> . - D-form shows [α] _D ¹⁸ =71.89° (in alc. soln.),(449). - O ₃ g. benzaldehyde +fenchone. In AcOH or ether soln. adds HBr or HCl.(419).
878	163-6 ⁴¹⁹ (13-4 mm.)			<u>C₁₇H₂₂ from benzyl-fenchol - Isomeric with No. (419).</u>
880	166-9 ⁴²¹ (10mm.)			<u>C₁₇H₂₂ from benzyl-dihydrocarveol.</u>
882	170-1 ⁴¹⁰ (20mm.) 160-1 ⁴¹⁰ (10mm.)			<u>1,7,7-Trimethyl-3-benzyl-5-benzyl-bicyclo-[1,2,2]-heptene (?), C₁₇H₂₂</u> . - D-form shows [α] _D ¹⁸ =+8°20'. (410).
884	153-5 ⁴¹⁶ (18mm.)	0.867(26/4) ⁴¹⁶	1.499(27) ⁴¹⁶	<u>2,8-Dimethyl-5-phenyl-nonene-(4), C₁₇H₂₆</u> .

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
886	²⁰² 308-12	1.015(18/4) ²⁰²		<u>1,3,4-Trimethyl-6-benzyl-benzene, C₁₆H₁₈.</u>
888	²⁰² 309-10	1.007(18/4) ²⁰²		<u>1-Isopropyl-4-benzyl-benzene, C₁₆H₁₈.</u> - Faint odor, (202).
890	⁴⁰³ 310-2	1.016(15) ⁴⁰³		<u>1,3-Diphenyl-butene-(2)</u> (403) or (1) (404), "Liquid Distyrene, (403,404). <u>C₁₆H₁₆.</u> - Freshly prepared has blue fluorescence wh. almost entirely disappears by long standing. (403). - K ₂ Cr ₂ O ₇ mixture g. benzoic ac. (403). - Br in CS ₂ g. dibromide, needles, M.P. = 102°. (403). - HI+P g. 1,3-diphenyl-butane, yellow oil, B.P. = 295°. (404).
892				<u>1-Phenyl-2-[2²-etheno-phenyl]-ethene, C₁₆H₁₄.</u> - Yellow-brown oil. (380). - Picrate, orange-red, M.P. = 95-100°. (380).
894	Abt. 310 ²⁷⁴ 154-6 (15mm.)			<u>1,2,6-Trimethyl-naphthalene</u> C ₁₃ H ₁₄ . - Odor of orange blossoms. (274). - Picrate, orange-yellow, M.P. = 122-3°. (274).
896	⁴⁰⁶ 311-2	0.987(15) ⁴⁰⁶		<u>1-Phenyl-1-[1²,1⁴-dimetho-phenyl]-ethane, C₁₆H₁₈.</u> - Faint fluorescence. (406).
898	⁴⁰⁷ 312-4	0.970(20/0) ⁴⁰⁷		<u>1,2-Ditolyl-propane,</u> <u>C₁₇H₂₀.</u>
900	⁴⁰⁵ 313-4			<u>1,1-Diphenyl-hexene-(1),</u> <u>C₁₈H₂₀.</u>
902	³⁷³ 178 (16mm.)	0.973(25/4) ³⁷³	1.575(18) ³⁷³	<u>4-Methyl-1,1-diphenyl-pentene-(1), C₁₈H₂₀.</u>
904	²²² 190 (11mm.)	0.992(20) ²²²	1.588(20) ²²²	<u>1,4-Diphenyl-hexene-(1),</u> <u>C₁₈H₂₀.</u> - Oxid. w. KMnO ₄ g. benzoic ac. + phenyl succinic ac. (222).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
906	⁴³⁶ 185 (12mm.)	0.963(15) ⁴³⁶	1.544 ⁴³⁶	<u>2,5-Diphenyl-hexane, C₁₈H₂₂.</u>
908	⁴⁰⁵ 164 (10mm.)			<u>1,1-Diphenyl-hexane, C₁₈H₂₂.</u>
910	⁴⁰⁰ 206-8 (20mm.)			<u>1,6-Diphenyl-hexane, C₁₈H₂₂.</u>
912	⁴⁴² 324-5			<u>1-Phenyl-naphthalene, C₁₆H₁₂</u> - Feeble blue fluorescence. (⁴⁴²). - Oxid. in alk. soln. to o-benzoyl benzoic ac. (⁴⁴²).
914	²⁷⁶ Abt. 326 165 (15mm.)			<u>4-Methyl-1-isopropyl-2-benzyl-bicyclo-[0,2,3]-, C₁₇H₂₂.</u> - By dehydration of benzyl-tanacetyl alcohol. (²⁷⁶).
916	²⁷⁷ Abt. 327 162-4 (10mm.)			<u>1-Methyl-4-[methoethenyl]-2-benzyl-cyclohexene-(2 or 3), C₁₇H₂₂.</u> - By dehydration of benzyl-pulegol. (²⁷⁷).
918	³²⁷ Abt. 327 195-200 (40mm.)			<u>1,1,2-Trimethyl-2-phenyl-cyclopentane-(4)?, C₁₄H₁₈.</u>
920	³²⁹ 153-5 (10mm.)			<u>1-Methyl-4-isopropyl-1,2,3,4,9,10-hexahydrofluorene, C₁₇H₂₄.</u>
922	³³⁵ 333.5- 4.5			<u>1,1-Diphenyl-heptane, C₁₉H₂₄.</u> - M.P.=14°. (³³⁵).
924	³⁷⁸ 207-8 (12mm.)			<u>1,7-Diphenyl-heptane, C₁₉H₂₄.</u>
926	³⁷⁸ 201-3 (10mm.)			<u>1,7-Diphenyl-heptene-(2 or 3 or mixture), C₁₉H₂₂</u>
928	³⁷⁷ 191-5 (20mm.)			<u>3,5-Diphenyl-heptadiene-(2,4), C₁₉H₂₀.</u> - KMnO ₄ g. benzoic ac. + EtPhCO whose semicarbazone, needles from alc., M.P.=176°. (³⁷⁷).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
930	²³⁰ Abt. 335 106-7 (10mm.) 112-5 ²³¹ (14mm.)	^{230, 231} 0.934(20)	^{230, 231} 1.524(n _D)	<u>1,1,6-Trimethyl-1,2,3,9-</u> <u>(or 1,2,9,10)-tetrahydro-</u> <u>naphthalene, "Ionene",</u> <u>C₁₃H₁₈.</u> - Oxid. w. dil. alk. KMnO ₄ at ord. temp. g. ionegenontricarbonic ac., C ₁₃ H ₁₈ O ₇ , (230), cf. Beilstein 1910 Syst. No. 1370.
932	¹⁰⁴ Abt. 341 168-75 (20mm.)	¹⁰⁴ 0.873(17)		<u>1,3,5-Trimethyl-2-ethinyl</u> <u>-benzene, C₁₁H₁₂.</u>
934	⁴¹⁵ Abt. 350 344-8 (714mm.)			<u>Dehydrofichtelit, C₁₃H₂₀.</u> -Blue-fluorescing oil. (415). -By htg. fichtelit w. I. (415).
936	⁴⁴² 350-1			<u>Cuminyll ether, C₂₀H₂₆O.</u> Dist. w. partial dec. to cymene+cuminic aldehyde. (442).
938	³⁷⁶ Abt. 354 170-5 (12mm.)			<u>1-Ethyl-3-phenyl-cyclo-</u> <u>pentadiene-(2,4)(?),</u> <u>C₁₃H₁₄.</u>
940	²¹³ Abt. 313 146-7 (15mm.)	²¹³ 0.951(20/4)		<u>6-Methyl-1-phenyl-hepta-</u> <u>diene-(1,3), C₁₄H₁₈.</u>
942	⁴⁰⁶ 316-7			<u>1-Phenyl-1-[1^x,1^x-di-</u> <u>methophenyl]-ethane,</u> <u>C₁₆H₁₈.</u>
944	⁴⁰⁶ 316-7			<u>1-Phenyl-1-[1²,1⁵-di-</u> <u>methophenyl]-ethane,</u> <u>C₁₆H₁₈.</u>
946	^{465b} 322-3			<u>2,3-Dimethyl-1-phenyl-</u> <u>1,2,3,4-tetrahydro-</u> <u>naphthalene, "Methronol",</u> <u>C₁₃H₂₀.</u> - Br in CS ₂ immediately liberates HBr. (465b). - Oxid. w. CrO ₃ mixture g. o-benz- oyl-benzoic ac., then CO ₂ , AcOH, benzoic ac., + anthraquinone. (465b).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
948	⁴⁰⁸ 280 (50mm.)			<u>Tetrahydroretene, C₁₂H₂₂.</u>
950	164-5 (11mm.)			<u>2,2-Dimethyl-3,4-diphenyl-butene-(3), C₁₈H₂₀.</u>
952	⁴¹² 323-5	0.966(20/4) ⁴¹²		<u>1,1-Di-[1²,1⁴-dimetho-phenyl]-ethane, C₁₈H₂₂.</u> - Blue fluorescence.(412).
954	⁴⁰⁶ 323-4			<u>1-Phenyl-1-[1^x1^x1^x-trimethophenyl]-ethane, "α-Phenyl-α-pseudocumyl-ethane, C₁₇H₂₀.</u>
956	^{480,481} 170 (10mm.)	^{480,481} 0.972(20/4)	^{480,481} 1.539(20)	<u>Hexameride of propadiene-(1,2), C₁₈H₂₄.</u> - Nearly odorless. - Oxid. g. (CO ₂ H) ₂ +CH ₂ (CO ₂ H) ₂ .(481).
958	³⁶⁸ 323-4	0.981(19/0) ³⁶⁸	1.559(19) ³⁶⁸	<u>1,5-Diphenyl-pentane, C₁₇H₂₀.</u> - Fluorescent.(368).
960	²⁵⁷ 202-6 (20mm.)			<u>Phenyl-O-xylyl-propane, C₁₇H₂₀.</u>
962	205-6 (40mm.)			<u>2-Methyl-1,4-diphenyl-butene-(1), C₁₇H₁₈.</u>
964	³³⁹ 178-81 (12mm.)	0.980(25/4) ³³⁹	1.570(25) ³³⁹	<u>1-[1²-Methopentadiene-(1¹,1³)-yl]-naphthalene, C₁₆H₁₆.</u>
966	⁴⁴³ 325-6			<u>1-Phenyl-x,x-dihydro-naphthalene, "Atronol", C₁₆H₁₄.</u> - CrO ₃ mixt. g. o-benzoyl-benzoic ac.(413)
968	³³¹ 183-5 (13mm.)			<u>1-Benzyl-indene, C₁₆H₁₄.</u> - Condenses in presence of KOH in MeOH w. PhCHO g. 1-benzyl-3-benzal-indene; w. anisaldehyde g. 1-benzal-3 anisal-indene.(331). G. yellow color w. H ₂ SO ₄ .(331).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
970	⁴²⁹ 175-8 (10mm.)	0.973(⁴²⁹ 9/4)	1.591(⁴²⁹ 9)	<u>5-Methyl-2-[β-naphthyl]-hexene-(1 or 2), $C_{17}H_{20}$.</u> - Picrate, orange yellow needles, M.P.=46-7°.(429).
972	⁴³¹ 235-45 (16mm.)			<u>Diindene, $C_{18}H_{16}$.</u> - Stable to oxid. agents.(431).
974	⁴⁴⁰ 217-9 (30mm.)			<u>1,4-Diphenyl-hexene-(1), $C_{18}H_{20}$.</u>
976	^{420, 432} 180 (10mm.)			<u>Cyclohexyl-phenyl-cyclohexylidene-methane, $C_{19}H_{26}$.</u> - Fum. HNO_3 at 0° g. mononitro cpd., M.P.=130°.(432). - Adds Br readily.(432).
978	^{394, 420} 210-2 (20mm.)	^{394, 420} 0.989(13)		<u>Dicyclohexyl-phenyl-methane $C_{19}H_{26}$.</u> - Fum. HNO_3 at 10° g. mononitro cpd., light yellow needles, M.P.=113°.(394, 420).
980	⁴²³ 224-6 (20mm)	1.069(⁴²³ 22/4)		<u>1-p-Tolyl-1-α-naphthyl-ethene, $C_{19}H_{16}$.</u>
982	²⁵⁷ 208-10 (8mm.) ³⁷⁸ 215(378) (12mm.)			<u>1,8-Diphenyl-octane, $C_{20}H_{26}$</u>
984	⁴³⁴ 192-3 (10mm.)	0.954(⁴³⁴ 20/4)		<u>2,7-Diphenyl-octane, $C_{20}H_{26}$</u>
986	⁴³⁹ 152 (22mm.)			<u>1-Ethylidene-3,5-diphenyl-cyclohexene-(2), $C_{20}H_{20}$.</u> - $KMnO_4$ in Me_2CO g. β -phenyl γ -benzoyl-butyric ac.(439).
988	⁴³⁴ 186-8 (8mm.)	0.946(⁴³⁴ 20/4)		<u>2,5-Dimethyl-1,6-diphenyl-hexane, $C_{20}H_{26}$.</u>
990	⁴²⁴ 204-6 (10-12mm)	1.018(15)		<u>1,2-Diphenyl-4,5-endo-</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
992	will be	found at the	top of page	<p>355.</p> <p><u>ethylene-bicyclo-[0,2,2]-hexane, C₂₀H₂₀.</u> - Yellow-green oil w. blue fluorescence and unpleasant odor. (424). Br does not add (427) but, without a diluent, substitutes, liberating HBr. (424). Unattacked by KMnO₄. (427).</p>

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
992	⁴²⁴ 217-20 (17mm.)	1.033(20/4) ⁴²⁵	1.602(20) ⁴²⁵	<u>Bis-[phenyl-butadiene]-</u> <u>"Dimer of 1-phenyl-buta-</u> <u>diene-(1,3)," C₂₀H₂₀.</u> - KMnO ₄ in Me ₂ CO g. benzoic ac.+4-phenyl-cyclo-butane- tricarboxylic acid(1,2,3). (466).
994	⁴²³ 235 (15mm.)			<u>9-Phenyl-hexahydroanthra-</u> <u>cene, C₂₀H₂₀.</u> - Green- yellow oil whose solns. in ether+C ₆ H ₆ fluoresce blue. (423).
996	³⁷⁸ 231-3 (12mm.)			<u>1,9-Diphenyl-nonene-(4),</u> <u>C₂₁H₂₆.</u>
998	³⁷⁸ 235 (12mm.)			<u>1,9-Diphenyl-nonane, C₂₁H₂₂.</u>
1000	⁴²² 365-6			<u>1,1,2-Triphenyl-propane,</u> <u>C₂₁H₂₀.</u>
1002	⁴²⁸ 255-8 (20mm.)			<u>9-Benzyl-1,2,3,4,13,14-</u> <u>hexahydroanthracene, C₂₁H₂₂.</u> - Sol. in alc., C ₆ H ₆ , ether, w. blue fluorescence (428).-Picrate, yellow needles, M.P.=120°.
1004	⁴³³ 253-4 (21mm.)			<u>2-Methyl-1,1,1-triphenyl-</u> <u>propane, C₂₂H₂₂.</u> - Fluoresces blue.(433). - Nitration g. 2-Methyl-1,1, 1-tris-[4-nitro-phenyl]- propane, xtals. fm. AcOH, M.P.=262-3° wh. g. the fuchsine reaction of Gomberg and Cone.(433).
1006	⁴⁴² 396-400			<u>1,1,2-Triphenyl ethane,</u> <u>C₂₀H₁₈.</u> - Shows a violet fluorescence.(442).
1008	⁴¹⁴ 236.5-7 (15mm.)	0.862(11/4) ⁴¹⁴		<u>1-Methyl-3-hexadecyl-benz-</u> <u>ene, C₂₃H₄₀.</u> -M.P.=11-2°(414).

DIVISION B, SECTION I

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
1010	⁴¹⁴ 238.5-9 (15mm.)	0.868(9/4) ⁴¹⁴		<u>1-Methyl-2-hexadecyl-benzene, C₂₃H₄₀.</u> - M.P.=8-9°. (414).
1012	⁴³⁷ 285 (50mm.)			<u>1,2,4-Triphenyl-cyclopentane, C₂₆H₂₂.</u> - By long standing small amounts of needle-like xtals. separate (437).
1014	⁴³⁵ 254 (17mm.)	0.868(20/4) ⁴³⁵		<u>1,5-Dimethyl-2-[hexadecene-(2¹)-yl]-benzene, C₂₄H₄₀.</u>
1016	¹⁰² 240 (20mm.)			<u>1,12-Diphenyl-dodecane, C₂₄H₃₄.</u>
1018	⁴³³ 260-2 (28mm.)			<u>1-Methyl-2,3,5-triphenyl-cyclopentane, C₂₄H₂₄.</u> - See Div. A, Sect. 2, No. to wh. this slowly goes over on standing. (433). - "Straw-yellow liquid." (433).
1020	⁴³³ 246-8 (25mm.)			<u>1,3-Dimethyl-2,4,5-triphenyl-cyclopentane, C₂₅H₂₆.</u> - See Div. A, Sect. 2, No.
1022	¹⁰² 262-5 (8 mm.)			<u>1,14-Diphenyl-tetradecane, C₂₆H₃₈.</u>

DIVISION B

.....
Liquid Hydrocarbons

Section 2

.....
Aliphatic Ethers

DIVISION B SECTION 2

No.	Boiling Point (C°)	Specific Gravity (20-4)	M.p. of the 3,5-dinitrobenzoate Test 928	Ether ¹
2	70.3	0.763 (0)		<u>Methyl Butyl Ether, C₅H₁₂O.</u>
4	70.74			<u>Methyl Isocrotyl Ether, C₅H₁₀O.</u> -- Dec. by 2-3 hrs. htg. at 140° w. 1% H ₂ SO ₄ giving MeOH + isobutyl aldehyde.
6	78-80	0.751		<u>Ethyl Isobutyl Ether, C₆H₁₄O.</u>
8	92	0.752 (20)		<u>Ethyl Butyl Ether, C₆H₁₄O.</u>
10	92-4			<u>Ethyl Isocrotyl Ether, C₆H₁₂O.</u> -- Unsat. -- Dec. by htg. w. 1% H ₂ SO ₄ giving isobutyric ald. and C ₂ H ₅ OH.
12	111-4			<u>Ethyl Valeryl Ether, C₇H₁₄O.</u> -- Htd. w. 1% H ₂ SO ₄ at 130-140° g. methylethyl acetaldehyde + C ₂ H ₅ OH.
14	112	0.764 (18)		<u>Ethyl Isoamyl Ether,* C₇H₁₆O.</u>
14	112	0.764 (18)		<u>Ethyl Isoamyl Ether,* C₇H₁₆O.</u>
16	116-8			<u>Hexenyl Ether, C₁₂H₂₂O.</u> -- Oil w. very pungent odor; i. aq.
18	117.1	0.777 (0)		<u>Propyl Butyl Ether, C₇H₁₆O.</u>
20	120			<u>Allyl Isoamyl Ether, C₈H₁₆O.</u>
22	120-1	0.756 (21)	75.6 ^{2,3}	<u>Sec. Butyl Ether, C₈H₁₈O.</u>
24	122-2.5	0.762 (15)	87-8 ⁴	<u>Isobutyl Ether, C₈H₁₈O.</u> -- <u>2-Naphthyl-amine addn.</u> product w. the dinitrobenzoate, m.p. = 105.5-6.5°. (4).
26	123.5	0.799 (0)		<u>Glycol Diethyl Ether, C₆H₁₄O₂.</u>
28	134.7			<u>Ethyl Hexyl Ether, C₈H₁₈O.</u>

DIVISION B SECTION 2

No.	Boiling Point (C°)	Specific Gravity (20-4)	M.p. of the 3,5-dinitrobenzoate Test 928	Ether ¹
30	141	0.769 (20)	64 ^{1,2}	Butyl Ether*, $C_8H_{18}O$. -- <u><i>L</i>-Naphthyl-amine addn. product w. the dinitrobenzoate, m.p. = 92.5-3°.</u> (4).
32	140-1	0.835 (25-25)		Trimethylene Glycol Diethyl Ether, $C_7H_{16}O_2$. -- Fruity odor - i. aq.
34	143-5	0.890 (0)		<u>Crotonyl Ether, $C_8H_{14}O$.</u>
36	145	0.831 (15)		<u>Octylene Oxide, $C_8H_{16}O$.</u>
38	149.8	0.795 (0)		<u>Methyl Heptyl Ether, $C_8H_{18}O$.</u>
40	166	0.795 (0)		<u>Ethyl Heptyl Ether, $C_9H_{20}O$.</u>
42	170-80			<u>Diamylene Oxide, $C_{10}H_{20}O$.</u> -- Reduces ammon. $AgNO_3$ sol.
44	173	0.801 (0)		<u>Methyl Octyl Ether, $C_9H_{20}O$.</u>
46	173 C.		62 ^{2,4}	Isoamyl Ether*, $C_{10}H_{22}O$. -- n_D^{20} : 1.408 - <u><i>L</i>-Naphthyl-amine addn. product w. the dinitrobenzoate, m.p. = 104-5°.</u> (4).
48	180			<u>"Diallyl" Ether, $C_{12}H_{22}O$.</u>
50	189.2	0.801 (0)		<u>Ethyl Octyl Ether, $C_{10}H_{22}O$.</u>
52	209		60-1 ⁴	<u>Hexyl Ether, $C_{12}H_{26}O$.</u> -- <u><i>L</i>-Naphthyl-amine addn. product w. the dinitrobenzoate, m.p. = 103-4°.</u> (4).
54	261	0.815 (0)	47-8.5 ⁴	<u>Heptyl Ether, $C_{14}H_{30}O$.</u> -- <u><i>L</i>-Naphthyl-amine addn. product w. the dinitrobenzoate, m.p. = 57-8.5°.</u> (4).

DIVISION B SECTION 2

No.	Boiling ¹ Point (C°)	Specific ¹ Gravity (20-4)	M.p. of the 3,5-dinitro- benzoate Test 928	Ether ¹
56	291-7	0.820 (0)	62 ²	<p>Octyl Ether, C₁₆H₃₄O. -- α-Naphthyl-amine addn. product w. the dinitro- benzoate, m.p. = 48-9.5°. (4).</p>

DIVISION B

Liquid Hydrocarbons

Section 3

Acyclic acetylenes and polyolefines, terpenes, and cyclic unsaturated hydrocarbons of all sorts.

Note! See Test 929 - 3.

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
1	18-19 ¹			<u>Butadiene-(1,2)</u> , C ₄ H ₆ -- Htg. w. Na in ether g. Na cpd. of ethyl-acetylene.(2) - Alcoholic KOH at 170° g. dimethyl-acetylene. (2).
3	28-29 ^{3,4}			<u>Butine (2)</u> , C ₄ H ₆ -- HClO g. 3,3-dichlorbutanone-(2) (5).
5	28-29 ⁶	0.685 (0°) ⁶		<u>2-Methyl-butene-(3)</u> , C ₅ H ₈ -- CrO ₃ in tube g. Me ₂ CO, AcOH, Me ₂ CHCO ₂ H.(7). - Shak. w. H ₂ SO ₄ (1.65) g. Me ₂ CH.CO.Me.(6,7). - Htg. w. CdCl ₂ or ZnCl ₂ at 150° g. same ketone.(8). - Ammon. CuCl g. yellow ppt. (9). - AgNO ₃ (ammon.) g. white ppt., (9), Test 906. - Na cpd. + CO ₂ g. isopr acetylene carbonic acid. (10,11). - Quinoline HBr in tube at 165-70° or sat'd. HBr at rm. temp. g. 3-brom-2-methyl butene (3).(12).
6	31-2 ⁷¹⁶	0.944 (13) ⁷¹⁶		<u>Furane</u> ,* C ₄ H ₄ O, -- Peculiar odor (716) - Insol. in aq.; easily sol. in alc. or ether(716) - Colors a pine splinter moistened w. conc. HCl emerald green. (716) - Conc. HCl attacks vigorously, g. brown resinous body. (716) - Maleic anhydride g. addn. cpd., M.P.: 125°. (717)
7	32-33 ¹³		1.416 (20°) ¹³	<u>2-Methyl-butene-(1)-ene (3)</u> , C ₅ H ₈ .
9	14,15,16,17,18) 33.5-34.5	16,17,18 0.682 (20/4)	1.422 (20°) ^{16,18}	<u>2-Methyl-butadiene-(1,3)</u> ,* "Isoprene", C ₅ H ₈ -- Htg. w. dil. alc. HCl g. Me ₂ C=C=CH ₂ .(19). Dil. CrO ₃ oxid. to CO ₂ , formic ac., AcOH.(20). - HNO ₃ oxid. g. C ₂ H ₂ O ₄ .(20). - Addn. of HCl or HBr g. chiefly CH ₂ =CH-C (Hlg.)-Me ₂ . (21,22,23). - HClO g. C ₅ H ₁₀ O ₂ Cl ₂ , M.P. = 82°.(14). Does not g. Test 906.(20). - M.P. abt. -120° (indistinct). (24). - Anylene free isoprene scarcely affected by long standing w. AlCl ₃ . - 2, 4-dinitro benzene diazonium chloride g.

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
11	37-38 ^{32,35}			<p>cpd. M.P.=98° w. explosion, orange-yellow xtals. (25).- Maleic anhydride g. 5-methyl-cyclohexene-(4)-dicarbonic ac. (1,2) anhydride, M.P.=63-40° (713).- B.b.no.= above 410, Test 925.</p> <p>109625</p> <p><u>Methyl-cyclobutene-(1), C₅H₈</u> -- This together with methylene-cyclobutane constitutes Gustavson's "vinyl-trimethylene" of J. prakt. Chem. 54, 97; 56, 93 and Zelinski's "Spiro-cyclane" of J. Russ. Phys. Chem. Soc. 44, 1870 (cf. Ber. 46, 160) and Fecht's "spiro cyclane" of C.A. 2, 104. (32, 33, 34)</p>
13	27, 29, 30 37.5-38.5	0.674 (15) ^{27, 29}	1.377 (15) ²⁷	<p><u>3,3-Dimethyl-butene-(1), C₆H₁₀</u> -- M.P.= (-81.2°). (27).- G. Test 906. (28).- Cu salt exists in 2 forms, red xtals + yellow powder (or plate-like xtals), (28), yellow form at 80° gives red, (28), both melt at abt. 140° (28); xtalln. frm. C₆H₈ of yellow form g. red; frm. CHCl₃ of red form g. yellow (28);- htg. dry yellow form in tube at 150° g. Cu mirror + white sublimate of di-t-butyl diacetylene, M.P.= 130-1°, (28), of Div. A, Sect. 2, No. 560.- Oxid. w. alk. K₃Fe(CN)₆ g. above by M.P. 130-1° + Me₃C.CH₂. CO₂H, B.P.= 176-80° (28).- 12 g. COCl₂ in Et₂O added in 1 portion to 40 g. Na acetylide (formed by Na in boiling ether soln.) at C° + dec. w. H₂O + extn. w. Et₂O g. tri [t-butyl acetylenyl] carbinol, (Me₃C.C≡C)₃ COH, M.P. = 102.5°, sublimes in vacuo, characteristic eosin red w. HOAc, and color similar to that of C₃COH w. H₂SO₄, (29) cf. Div. A, Sect. 3 No. 1018 - Di [3,3-dimethyl butinyl]-Hg, M.P.= 91-2°, Test 926. (31).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
15	12.42 39.5-40.5	0.678 ⁴²	1.409 (20) ⁴²	<p>-Ag salt difficulty sol. in alc. + C₆H₆, slowly g. a silver mirror from C₆H₆. (28). - Na cpd. + acetone g. cpd. vol. w. steam, M.P. 102.5° wh. g. characteristic color w. AcOH. (29).</p> <p><u>2-Methyl-butadiene-(2,3), C₅H₈</u> -- Does not g. test 906. (41). - G. an ozonide at -70° wh. w. H₂O g. CO₂. (43) - Htg. w. Na g. Na salt of isopropyl acetylene. (11). - CrO₃ oxid. g. AcOH + acetone. (44).</p>
17	36.37.38. 39.40 39.5-40.5	0.694 (17) ⁴⁰	1.388 (17) ⁴⁰	<p><u>Pentene-(1)*, C₅H₈</u> -- M.P. = (-95°), (36). - G. test 906 (38). - Cu salt + I₂ g. liquid triiodide (38). - Na salt (by NaNH₂ in C₆H₆, then removal of NH₃ in vacuo.) + CO₂ g. n-hexine acid, B.P. = 120°; d (20) = 0.982, n (20) = 1.465. (40) - Dipentynyl Hg, M.P. = 118.5° -- 119°, * Test 926.</p>
19	46.47 41-42	0.805 (18.6/4)	1.445 (18.6)	<p><u>Cyclopentadiene*, C₅H₆</u> -- Polymerizes at ord. temp. giving dicyclopentadiene, (46) B.P. = 24.70°, M.P. = 32° (47), cf. Div. A, Sect. 2, No. 78. - G. an Ag mirror w. ammon. Ag soln. (46). - G. addn. product w. quinone. (48). - Pseudonitrosite, C₁₀H₁₂O₆N₄, by nitrous gases + ether soln., yellow xtaln. flakes M.P. = 38°, quickly dec. (50). - Nitrosochloride, C₁₀H₁₂O₂N₂Cl₂, by Am ONO + alc. KOH, cold, (50; 51), needles frm. EtOH, AcOH, or acetone, begin to darken at 100° - (51) 105° (50) and dec. w. slight explosion at abt. 142° (50) - 144° (51). - MeMgI g. C₅H₅MgI wh. w. 4-MeOC₆H₄CHO g. p-methoxy phenylfulvene, red, m.p. = 70°; w. Bz Ph g. diphenyl fulvanol, CH₂ = CH-CH=CH-CH-C₆H₄OH, M.P. = 123-4°, spontaneously dehydrated w. formation of diphenyl-fulvene, red prisms, M.P. = 82°. (52) -</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
21	32, 35, 45 41.5-42.5	0.743 (15/4) ⁴⁵	1.424 (15) ⁴⁵	<p>Maleic an. g. cis-endomethylene-3,6 - Δ^4-tetrahydro-phthalic an., M.P. = 164-5° (frm. ligroin); ac., M.P. = 177-9°, wh. latter w. Pd + H g. hexahydro ac., M.P. = 160-1° (718) <i>page 626</i></p> <p><u>Methylene-cyclobutane, C₅H₈</u> -- Odor somewhat allyl-like (45). - Reacts vigorously w. Br. (45) - Instantly decolorizes KMnO₄. (45) - N₂O₃ g. nitrosite, leaflets frm. AcOEt, M.P. abt. 140° w. gradual dec. cf. Methyl cyclobutene (1). (45)</p>
23	18, 45 ^a , 53 42-43	18, 45 ^a , 54 0.685 (20/4)	18, 45 ^a , 54 1.431 (20°)	<p><u>Pentadiene-(1,3)</u>, C₅H₈ -- Br. without a solvent g. 1,2,3,4 - tetra-bromopentane, M.P. = 116°. (58, 57, 45^a). - KMnO₄ g. formic ac. + AcOH (55, 56). - Does not g. Test 906. (53). - P-nitro-benzene-diazonium chloride g. [p-nitro-benzene-azo-piperylene] M.P. = 137° (25). - O, p-dinitro-benzene-diazonium sulfate g. o, p-dinitrobenzene-azo-piperylene, M.P. = 121° w. dec., beginning to darken at 105° (25). - Maleic anhydride g. 6-Methyl-cyclohexene-(4)-dicarbonic ac.-(1,2) anhydride, M.P. = 62° (713). - B.b.no. = 470, Test 925. <i>page 625</i></p>
24		0.674 (15/4) ⁵⁹	1.377 (15) ⁵⁹	<p><u>Dimethyl-butylene, C₆H₈</u> -- M.P. = (-81.2°). (59).</p>
25	44-45 ^{60, 61}	0.689 (20°) ⁶⁰	1.415 (20°) ⁶⁰	<p><u>Pentadiene-(1,2)</u>, C₅H₈ -- Ppts. HgCl₂ frm. its solns. (60). - Br. g. tetrabromide, B.P.₃ = 120°, d₂₀²⁵ = 2.284, n_D⁽²²⁾ = 1.592 (60). - HBr in AcOH at 0° g. n-PrCBr₂Me, B.P.₁₈ = 62-3°, d₁₆ = 1.645, n_D⁽¹⁶⁾ = 1.503 (61). - Odor like garlic (60). - H₂SO (d=1.84) at -10° g. C₃H₇.CO.Me. (714).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
27	62:63:64,515,516) 45-6	0.775 (14/4)	1.421 (14)	<u>Cyclopentene</u> , C_5H_8 -- O_3 g. oz ⁿ ide (in hexane) wh. w. boiling H_2O g. glutaric di-aldehyde + glutaric aldehyde-acid. (64,515).
29	49-51 ⁶⁵	0.702 (20/0) ⁶⁵		<u>Pentadiene-(2,3)</u> , C_5H_8 -- Na + CO_2 g. $CH_3.CH_2.CH_2.C\equiv C.CO_2H$ (see pentine (1)). (65). - Alc. KOH g. pentine-(2). (65). - Does not g. Test 906. (65).
31	55-56 ^{39,41}	0.713 (17/4)	1.405 (17 ⁰)	<u>Pentine-(2)</u> , C_5H_8 -- M.P. (-101 ⁰) (69) - CrO_3 ox. g. AcOH + Et CO_2H . (41,66). - Dil. H_2SO_4 g. PrCOMe. (41). - HClO g. MeCOCCl ₂ Et + n-Pr-CCl ₂ -CHO. (68). - Br g. MeCBr = CBr-Et. (69). - Does not g. Test 906. (41,67).
33	59.0-60.0 ⁷⁰	0.688 (20/4) ⁷¹	1.401 ⁷¹ (20 ⁰)	<u>Hexadiene (1,5)</u> , C_6H_{10} -- CrO_3 oxid. g. CO_2 + HAc. (72). - Dil. HNO_3 oxid. g. succinic ac. • (73). - 2.57 g. $KMnO_4$ + 1 g. diallyl in neutral soln. g. hexane tetrol-(1,2,5,6). (74). - Br. g. tetrabromide wh. after rextaln. frm. pet. ether M.P. = 64-50. (75).
35	61.5-62.5 ³⁷	0.724 (00) ³⁷		<u>4-Methyl-pentine-(1)</u> , C_6H_{10} -- G. Test 906. (37). - Ppt. w. alc. $AgNO_3$, brilliant plates, sol. in hot alc. (37).
37	63-65	0.770 (20/4)	1.423 (20 ⁰)	<u>1,2-Dimethylene-cyclobutane</u> , C_6H_8 -- Readily polymerized. - Obtained by polymerization of allene.
39	64-66 ⁷⁸			<u>Hexadiene-(1,4)</u> , C_6H_{10} --
41	64.5-5.5 ⁴²⁶	0.849 (20) ⁴²⁶	1.476 (20) ⁴²⁶	<u>a 1,1,3,4-Tetramethyl-cycloheptadiene</u> , $C_{11}H_{18}$ -- This b.p. is undoubtedly in error - R.L.W.

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
43	67-69 ¹³		1.433(20°) ¹³	<u>3-Methyl-pentene-(3)-ine-(1).</u> C ₆ H ₈ --
45	69-70 ¹⁸	0.725 (20/4) ¹⁸	1.438 (20°) ¹⁸	<u>2,3-Dimethyl-butadiene-(1,3).</u> * C ₆ H ₁₀ -- F.P. = (-65°) (24) - NO ₂ (in cold ether) g. nitrosate, needles frm. CS ₂ , M.P. = 72-3° (79). - KMnO ₄ g. formic acid, AcOH, pinacoline. (80). - P-nitro-benzene diazonium chloride (aq. soln.) g. cpd. M.P. = 177° (25). - O, p-dinitro-benzene diazonium sulfate g. cpd. exploding at 126° (25). - HBr in glac. AcOH g. Me ₂ CBrCMe = CH ₂ , B.P. ₁₀₀ = 84-6°, d ₂₀ = 1.2201; wh. w. HBr g. 30% CMe ₂ BrCMe ₂ Br, M.P. = 173° frm. ether + 70% CMe ₂ BrCHMeCH ₂ Br, B.P. _{16.5} = 88-9, d ₂₀ = 1.6065. (81). - Htg. w. H ₂ O + some H ₂ SO ₄ at 100° g. pinacoline. (79). - HBr at low temp. g. Me ₂ CH=CMe ₂ CH ₂ Br, B.P. ₂₀ = 51-4°, 15 B.P. ₇₆₀ = 144-8 (dec.), d ₁₅ = 1.254; wh. by shak. w. aq. 15 Na ₂ CO ₃ → Me ₂ C(OH)CMeCH ₂ , d ₁₅ = 0.8527, (82), whose monobromide (by HBr) in ice cold benzine + ONH ₂ in ether after standing several days → di-isohexenyl-aniline, ON(C ₆ H ₁₁) ₂ , present in roset. form from MeOH, M.P. = 58-9° (82). - Maleic anhydride g. 4,5-dimethyl-cyclohexene-(4)-dicarbonic ac. -(1,2) anhydride, M.P. = 78-9° (713). - B.b.no. = 385, * Test 925. <i>may 625</i>
47	^{87,88} 69-71	0.766 (18/4) ⁸⁷	1.422 (18°) ⁸⁷	<u>1-Methyl-cyclopentene-(2).</u> C ₆ H ₁₀ -- Oxid. w. KMnO ₄ g. α-methyl glutaric ac. (88) - [α] _D : + 59.07° for d-form. (87).
49	70-1 ⁷¹⁴	0.706 (22) ⁷¹⁴	1.423 (22) ⁷¹⁴	<u>4-Methyl-pentadiene - (1,2).</u> C ₆ H ₁₀ -- NaNH ₂ g. 4-methyl-pentine - (1). (714).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
51	70-71 ⁸³	0.858 (18.2°) ⁸³		<u>Hexene-(5)-ene-(1)</u> , C ₆ H ₁₀ -- Ammon. Cu ₂ Cl ₂ soln. g. siskin colored ppt., C ₆ H ₇ Cu + (H ₂ O) ₃ (83) G. Test 906. (83).
53	70-71 ⁸⁶	0.731 (0/0) ⁸⁶		<u>3-Methyl-pentadiene-(1,2)</u> , C ₆ H ₁₀ .
55	70.5-71.5 ⁹¹	0.752 (20/0) ⁹¹	1.425 ⁹¹ (20°)	<u>2-Cyclopropyl-propene</u> , C ₆ H ₁₀ -- KMnO ₄ g. acetyl trimethylene, B.P. 75.7 = 112-2.5°, d ₂₀ ²⁰ = 0.8993; n _D (20°) = 1.4244 (wh. g. semicarbazone, M.P. = 110-2°, C ₆ H ₁₁ ON ₃ , xtalizes w. H ₂ O of xtaln.) + a hydroxy acid, CO ₂ HC(Me)(OH) - CH $\begin{cases} \text{CH}_2 \\ \text{CH}_2 \end{cases}$, colorless needles, M.P. = 75.5° (forms a hydrate 2C ₆ H ₁₀ O ₃ .H ₂ O M.P. = 54-5°). (91). - Alexejew, J. Russ. Phys. Chem. Soc. 37, 419 Chem. Zentr. 1905 II, 403 described this as Me ₂ C=C $\begin{cases} \text{CH}_2 \\ \text{CH}_2 \end{cases}$
57	40.84 71-72	0.721 (17°) ⁴⁰	1.402 ⁴⁰ (19°)	<u>Hexene-(1)</u> , C ₆ H ₁₀ -- G. Test 906 (85) - NaNH ₂ in boiling ether g. Na salt wh. w. CO ₂ g. n-heptene acid, B.P. 78°, d ₂₀ = 1.00, n _D (20) = 1.458. (84, 11) = Dihexinyl Hg, M.P. = 90-1.5* (Test 926).
59	71-72.5 ⁹⁰	0.722 (0°) ⁹⁰		<u>2-Methyl-pentene-(3)</u> , C ₆ H ₁₀ .
61	89.98 71.5-72.5	0.715 (16.1) ⁹⁰		<u>2-Methyl-pentadiene-(2,3)</u> , C ₆ H ₁₀
63	72-73 ⁹²	0.776 (20/0) ⁹²		<u>1-Methyl-cyclopentene-(1)</u> , C ₆ H ₁₀ .
65	72-74 ⁸⁶			<u>3-Methylene-pentene-(1)</u> , C ₆ H ₁₀ .
67	72-74 ¹⁰³	0.714 (12°) ¹⁰³		<u>Hexadiene-(1,3)</u> , C ₆ H ₁₀ -- (Impure) tetrabromide → Et - diacetylene B.P. abt. 80°, (58).
69	72-74 ⁹³			<u>2-Ethyl-butadiene-(1,3)</u> , C ₆ H ₁₀

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
71	74-75 ^{24,94}	0.723 (16/4) ²⁴	1.446 (13°) ²⁴	2-Methyl-pentadiene (2,4), C₆H₁₀.
73	75.5-76.5 ^{24,95,94}	0.722 (20/4) ^{24,95}	1.447 (20) ^{95,240}	<u>4-Methyl-pentadiene-(1,3),</u> [*] C ₆ H ₁₀
75	76-77 ^{96,97}	0.718 (16°) ⁹⁶		<u>2-Methyl-pentadiene-(1,3),</u> C ₆ H ₁₀ .-- Maleic anhydride g. 3,5-dimethyl-cyclohexene-(4)- dicarbonic ac.-(1,2) anhydride, M.P.= 56-7°. (713).
77	76-79 ⁹⁸	0.747 (16.3/4) ⁹⁸	1.454 (16.5°) ⁹⁸	<u>3-Methyl-pentadiene-(1,3),</u> C ₆ H ₁₀ .
79	77-78	0.738 (20°)	1.450 (20°)	<u>Metho-ethenyl-cyclopropane (?)</u> C ₆ H ₁₀ .
81	77-78.5 ¹⁰⁰	0.749 (13.5°) ¹⁰⁰	1.488 (13.5°) ¹⁰⁰	<u>Hexatriene-(1,3,5),</u> C ₆ H ₈ .-- Conc. H ₂ SO ₄ polymerizes at once to solid, absorbs O ₂ .
83	78-79 ¹⁰²	0.720 (17°) ¹⁰²	1.430 (17°) ¹⁰²	<u>Hexadiene-(1,2),</u> C ₆ H ₁₀ -- Tetrabromide, B.P. ₃ = 130°, d ₁₅ = 2.187, n _D (15) = 1.585. (714).-- NaNH ₂ g. hexine -(1). (714).
85	78-79 ¹¹¹	0.758 (20/0) ¹¹¹	1.419 (20°) ¹¹¹	<u>1,1-Dimethyl-cyclopentene-(2),</u> C ₇ H ₁₂ -- Oxid. w. HNO ₃ g. α, α-dimethyl glutaric ac. (111).-- Colors green by warming w. conc. H ₂ SO ₄ . (111).
87	78-81 ¹¹⁰	0.78 (19°) ¹¹⁰	1.436 (19°) ¹¹⁰	<u>Methylene-cyclopentane,</u> C ₆ H ₁₀ -- Oxid. w. KMnO ₄ at 0° g. 1-methylol-cyclopentanol-(1) + cyclopentanone-(110) - nitroso-chloride dec. at 80-81°, w. NaO Me g. oxime of 1-methylalicyclopentene-(1). (110).
89	78-83 ⁷⁸	0.825 (0°) ⁷⁸		<u>Hexadiene-(1,4),</u> C ₆ H ₈ -- Remains liquid at -60°. (78).-- Polymerizes easily. (78).-G. Test 906 (78).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
91	108, 107, 105, 104) 80-81	108, 107, 104) 0.840 (20/4)	108, 107, 104) 1.474 (20°)	<u>Cyclohexadiene-(1,3), C₆H₈</u> -- G. a dark red color w. conc. H ₂ SO ₄ . (104). - Br. g. tetrabromide, prisms, M.P.=87-9° (104). - Odor of leeks. (105). - HNO ₃ oxid. g. C ₂ H ₂ O ₄ + succinic ac. (106). - W. NHMe ₂ in cold conc. C ₆ H ₈ soln. the dibromide g. quant. Δ ² -tetramethyldiamine-cyclohexene, B.P. ₁₀ =90.5 -2.5°; B.P. ₇₂₅ =219.5-23.5°; d ₄ ⁰ = 0.920; chloroplatinate blackens at 240°, decomp. 259-60°, methiodide, M.P.=236° (dec.). (108). - The pure diene, treated w. Br in CHCl ₃ to exact decolorization g. the dibromide, M.P.=108°, wh. on further bromination g. the tetrabromide M.P.=87-8° (109). Maleic an. g. addn. cpd., M.P.=147°. (718).
93	58, 18 80.5-82	54, 18 0.718 (21/4)	54, 18 1.446 (21°)	<u>Hexadiene-(2,4)*, C₆H₁₀</u> -- Br. g. dibromide, B.P. ₁₃ = 94° (58), tetrabromide, M.P.=183° (58). - Tetrabromide w. xs. alc. KOH g. dimethyl-diacetylene, M.P.=65° B.P. = 129-130° (58), cf. Div. A, Sect. 1, No. 109. - Maleic anhydride g. addn. product, M.P. = 95-6° (713).
95	113, 114 81-82.5	113, 114 0.718 (20/0)		<u>2,2-Dimethyl-pentadiene-(3,4), C₇H₁₂</u> -- Oxid. w. KMnO ₄ g. HCO ₂ H + Me ₃ C.CO ₂ H. (114). - Na at 150° g. salt (yellow powder) of 2,2-dimepentine-(4) wh. w. CO ₂ g. Me ₃ C-CH ₂ .C=C.CO ₂ H, B.P. ₂₀ = 129.5-30°, M.P. = 48-9.5°, frm. ligroin. (112).
97	100 82.5-83.5	123 0.718 (20/4)	120 1.407 (20.1°)	2,2-Dimethyl-pentene (2), C₇H₁₂
99	517 82-3	517 0.718 (20/4)	517 1.407 (20)	<u>2,2-Dimethyl-pentene-(3), C₇H₁₂</u> .
101	115, 116, 117, 118) 83-84	115, 118, 119) 0.814 (15.6/4)	119 1.449 (15.1°)	<u>Cyclohexene*, "Hexanaphthylene", C₆H₁₀</u> -- Htg. w. HNO ₃ in sealed tube at 100° g. little adipic ac. + ac. of M.P.=137-9° (116). - I + yellow HgO + H ₂ O + ether soln. g. 2-iodo-cyclo-hexanol(1) (120). = Nitrosite, flakes,

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
103	84-85 ⁴⁰	0.740 (21 ⁰) ⁴⁰	1.414 (21 ⁰) ⁴⁰	M.P. = abt. 150° w. dec. (121).- Nitrosate by dropping 1 cc. conc. HNO ₃ onto a well cooled mixture of 1 gm. cyclohexene, 1.5 g. isoamyl nitrite, + 2 g. AcOH, needles, M.P. = 150° w. sudden decomp. (121).- Nitrosochloride, white xtals frm. ether, M.P. = 152-153° w. dec., stable at rm. temp. (121,122).- Dibromide htd. 9 hrs. at 110-50° in sealed tube w. 6 mols. NHMe ₂ in 18% C ₆ H ₆ soln. g. 75% of Δ^2 -dimethyl-amino-cyclohexene, B.P. ₈₀ = 89-91.5°, B.P. ₇₂₅ = 160.5-2.5°; chloroplatinate prisms, M.P. = 185°; methiodide, needles, M.P. = 173-4° (108).- B.b.no. \neq 192 ₂ *.
105	86-87 ⁷⁸	0.805 (20/4) ¹²⁶		<u>Hexine-(2), C₆H₁₀</u> -- M.P. = (-92°) (85). - CrO ₃ oxid. g. CO ₂ , AcOH, PrCO ₂ H (124). - H ₂ SO ₄ g. hexanone-(2) + hexanone-(3). (125). - Htg. w. Na g. hexine(1) (11).
107	86-7 ⁵²⁰	0.715 (22 ⁴) ⁵²⁰	1.425 (22) ⁵²⁰	<u>Hexadine-(1,5), C₆H₆</u> -- M.P. = (-6°) (78). - Stable in absence of air. (78). - After a day it no longer distills completely from a waterbath, and explodes by htg. to 110-120° violently. (127).- G. Test 906. (78,127,128). Cu epd. w. xs. I g. Cl ₂ =Cl.CH ₂ .CH ₂ .Cl=Cl ₂ (127). - Forms a di-Mg deriv. wh. w. CO ₂ g. octadiindioic ac. (127).
107	86-7 ⁵²⁰	0.715 (22 ⁴) ⁵²⁰	1.425 (22) ⁵²⁰	<u>2,4-Dimethyl-pentadiene-(2,3), C₇H₁₂</u> -- Htg. to 150-175° partly rearranges to 2,4-dimethyl-pentadiene-(1,3), partly polym. to 1,1,2,2-tetramethyl-3,4-di-isopropylidene-cyclobutane. (520). - H + Pt. black g. 2,4-dimethyl-pentane. (520).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
109	86-8 ⁴⁴³	0.846 (16 ⁴⁴³ / ₄)	1.495 (16 ⁴⁴³)	<p><u>1,1,2,2-Tetramethyl-3,4-di-isopropenyl-cyclobutane</u>, $C_{14}H_{24}$ - $KMnO_4$ g. 1,1,2,2,-tetramethyl-3-isopropenyl-cyclobutanone, B.P.₂₀=104-5⁰, semicarbazone, M.P.=229-30⁰. (443). - CrO_3 g. $(Me_2CCO_2H)_2$. (443). - This B.P. is undoubtedly in error. = R.L.W.</p>
111	129,130 92-93	0.737 (17 ⁰) ¹³⁰	1.408 (17 ⁰) ¹³⁰	<p><u>2-Methyl-hexine-(5)</u>, C_7H_{12} -- The K cpd. w. propionyl chloride in ether g. isoamyl propionyl acetylene. (130).</p>
113	131,132 92-93	0.729 (18.5 ¹³² / ₄)	1.424 (17.3 ⁰) ¹³²	<p><u>2-Methyl-hexadiene-(1,5)</u>, C_7H_{12} - Adds 4 atoms of Br. (131). - Absorbs O_2 frm. the air. (131). Nitrososchloride, $C_7H_{12}ONCl$, needles frm. dil. MeOH, M.P.=75-76⁰. (131).</p>
115	133,134 92.5-93.5	0.734 (23 ¹³⁴ / ₄)	1.439 (23 ⁰) ¹³⁴	<p><u>2,4-Dimethyl-pentadiene-(1,3)</u>, C_7H_{12} -- 80% H_2SO_4 g. dimer, $C_{14}H_{24}$. (133). - $EtO_2CN=NCO_2$-Et g. N,N¹-dicarb-ethoxy-3-dimethyl-5-methyl-tetrahydro-pyridazine, B.P._{0.5}=136⁰. (135). - <i>Br adds readily, but dibromide</i> readily splits out HBr g. resinous products. (135). - Maleic anhydride g. addn. product, M.P. = 49⁰. (713).</p>
117	93-94 ¹³⁶	0.773 (19 ¹³⁶ / ₄)	1.430 (19 ⁰) ¹³⁶	<p><u>3-Methyl-1-methylene-cyclopentane</u>, C_7H_{12} -- -D-form shows $[\alpha]_D^{25} = +57.67^0$ - CrO_3 (136) or $KMnO_4$ (138) oxid. g. 1-methyl-cyclopentanone-(3).</p>
119	139,140 96-98	0.736 (23.9 ¹⁴⁰ / ₄)	1.437 (23.9 ⁰) ¹⁴⁰	<p><u>3-Ethyl-pentadiene-(1,2)</u>, C_7H_{12} - Does not g. Test 906. (139). - HBr in 45% AcOH g. Et_2CBrCH_2. - $CH_2Br+Et_2C=CH=CH_2Br$. (139). - Na g. salt of 3-ethyl-pentine (1). (140).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
121	96-7 ⁷¹⁴	0.723 (19) ⁷¹⁴	1.428 (19) ⁷¹⁴	<u>5-Methyl-hexadiene-(1,2)</u> , C ₇ H ₁₂ --NaNH ₂ g. 5-methyl-hex- ine -(1). (714).
123	97-99 ⁵⁴	0.719 (24.5/4) ⁵⁴	1.427 (24.5) ⁵⁴	<u>2-Methyl-hexadiene-(2,4)</u> , C ₇ H ₁₂ Garlic odor. (54).
125	99.5-100.5 ^{40*}	0.750 (19 ⁰) ⁴⁰	1.418 (19 ⁰) ⁴⁰	<u>Heptene-(1)</u> *, C ₇ H ₁₂ --Ether suspension of K salt + CH ₃ COBr + 4 hrs. standing in freezing mixture, overnight at 200, + 2 hrs. shak. g. n-pentyl- acetyl-acetylene, B.P. ₁₂ =84-5 ⁰ , D ₄ ²⁰ = 0.879; semicarbazone frm. EtOH + aq., M.P. =89-90 ⁰ . (142). - EtMgBr at rm. temp. 1 hr., warmed till no more ethane evolution + gaseous HCHO + nitrogen for 4 hrs. in cooling mixture g. octine (2) ol (1), B.P. ₁₂ =99-104 ⁰ . (141). H ₂ SO ₄ (1 vol. monohydrate + 1 vol. H ₂ O) g. heptanone (2); (143) 2,4-dinitro-phenyl- hydrazone, M.P.=88 ⁰ .* Test 926. = Na salt + CO ₂ g. n-pentyl propionic acid - G. Test 906 (9,145) - Diheptyl Hg. M.P.= 610.* (31), Test 926.- Na salt +CO ₂ g. Na salt of pentyl propionic acid. (144). - B.D. 12. Test 926.
127	101-103 ¹⁵¹	0.741 (25/4) ¹⁵¹	1.452 (25 ⁰) ¹⁵¹	<u>3-Methyl-hexadiene-(1,3)</u> , C ₇ H ₁₂
129	101.5-105.5 ¹⁵⁰	0.795 (20 ⁰) ¹⁵⁰	1.441 (20 ⁰) ¹⁵⁰	<u>1,2-Dimethyl-cyclopentene</u> , C ₇ H ₁₂ Probably mixture of Δ1, Δ5, + methylene-cyclopentane.
131	146, 152, 153 102-103	0.800 (20/4)	1.442 (18 ⁰)	<u>1-Methyl-cyclohexene-(3)</u> , C ₇ H ₁₂ Oxid. w. KMnO ₄ g. inactive ³ - methyl adipic acid, (148, 152)- D-forms shows=[α] _D ²⁰ =+ 110 ⁰ . (152).
133	146, 147, 148 102-103	115, 146, 148 0.802 (20/4)	115, 146, 148 1.451 (18 ⁰)	<u>Methylene-cyclohexane</u> , C ₇ H ₁₂ -- KMnO ₄ oxid. g. 1-methylol- cyclohexanol-(1) + adipic acid (147) + cyclohexanone. (148). - AcOH + H ₂ SO ₄ g. acetate of 1-methyl-cyclohexanol-(1). (148).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
135	¹⁵⁴ 103-104	0.792 (20/0) ¹⁵⁴	1.445 ¹⁵⁴ (13.50)	Nitrosochloride, $C_7H_{12}ONCl$ by $KNO_2 + HCl$, plates frm. C_6H_6 , M.P., slow htg., 118° ; rapid htg., 145° w. dec.; w. NaOMe or by warming w. NaAc + AcOH g. oxime of 1-Methylol cyclohexene (1). (148;149).
137	^{146, 156^a, 156^b} 104-105	0.801 (20/4) ¹⁴⁶	1.445 ¹⁴⁶ (17°)	<u>1,2-Dimethyl-cyclopentene-(1)</u> , C_7H_{12} -- HNO_3 oxid. g. $C_2H_2O_4$, succinic ac., + cpd. $C_7H_{12}O_4N_2$, M.P. = 202° . (154). - Nitrosochloride $C_7H_{12}ONCl$ by HCl + $NaNO_2$, M.P. = $73-75^\circ$. (155).
139	¹⁰² 105-106	0.731 (18°) ¹⁰²	1.432 ¹⁰² (18°)	<u>Heptadiene-(1,2)</u> , C_7H_{12} -- Tetrabromide, B.P. ₃ = 140° , $d_{20} = 2.068$, $n_D^{20} = 1.572$. (714). $NaNH_2$ g. heptene-(1). (714).
141	¹⁶⁷ 105.5-106.5	0.827 (20/4) ¹⁶⁷	1.468 ¹⁶⁷ (20°)	<u>1-Methyl-cyclohexadiene-(2,4) ?</u> , C_7H_{10} -- Frm. 3,4-dibrom 1-methyl-cyclohexane. (167).
143	^{40, 157 *} 106-107	0.765 (190)	1.423 (19°)	<u>Heptene-(3)</u> *, C_7H_{12} -- H_2SB_4 g. butyrene. (157). - $HClO$ g. dichlorheptanone. (158). - Does not g. Test 906. - $HgCl_2$ g. white ppt. wh. w. dil. HCl g. butyrene. (157) - B.b.no. = 233, Test 925.
145	^{18 *} 106-107	0.731 (20/4) ¹⁸	1.453 ¹⁸ (20°)	<u>Heptadiene-(2,4)</u> *, C_7H_{12} -- G. no ppt. w. aq. or alc. $HgCl_2$. (54). - Mixture of tetrabromides g. very little propyl-diacetylene + more methyl-ethyl-diacetylene w. xs. alc. KOH. B.P. = 143° . (58). B.b.no. = 325, Test 925.
147	⁹⁸ 107-108	0.763 (15/4) ⁹⁸	1.461 ⁹⁸ (15°)	<u>3-Methyl-hexadiene-(2,4)</u> , C_7H_{12} .
149	⁹⁹ 107-109	0.774 (20°) ⁹⁹	1.445 ⁹⁹ (20°)	<u>2-Cyclopropyl-butene-(2)</u> , C_7H_{12} .
151	¹⁵⁹ 107-110	0.798 (20°) ¹⁵⁹	1.443 ¹⁵⁹ (20°)	<u>1-Ethyl cyclopentene-(1)</u> , C_7H_{12}

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
153	107-8 *	0.737 (25/4vac) *	1.443 (25) *	<u>2,2-Dimethyl-hexadiene-(3,4).</u> * C_8H_{14} -- B.b.no.= 290, Test 925. *
155	^{160,161,162} 108.5-109.5	^{160,161} 0.781 (20/4)		<u>1,1,2-Trimethyl-cyclopentene(2).</u> "Isolaurolene", C_8H_{14} -- Odor like turpentine + camphor. (163).
157	^{164,147,146} 110-111	^{164,146} 0.810 (20/4)	¹⁴⁶ 1.450 (17.50)	<u>1-Methyl-cyclohexene-(1).</u> * C_7H_{12} -- G. a yellow ppt. w. $HgSO_4$. (165). An alc. soln. + conc. H_2SO_4 , alc. assumes a blue color, separated layer a yellow color soon going to orange, and by shaking the mixture turns green. (156a).- W. HNO_3 (11.45) it g. a blue-green coloration. (156a). HNO_2 + AcOH in ligroin g. nitrosite, $C_7H_{12}O_3N_2$, yellow needles, M.P. = 102°. (165). - Amyl nitrite + HNO_3 g. nitro- sate, $C_7H_{12}O_4N_2$, needles frm. MeOH, M.P. = 106-7°. (166). - Frm. HCl + $NaNO_2$ at 00 g. nitroso chloride, colorless plates frm. ligroin, M.P. = 97.50, dec. at 115°. (147). - Latter w. NaOMe g. oxime of 1-methyl cyclohexene-(1)-one-(6) + $H_2C \begin{cases} \text{CH}_2-C \rightleftharpoons NOH \\ \text{CH}_2-CH_2-C(O, CH_3).CH_3 \end{cases}$ (148). B.b. no. = 159, * Test No. 925.
159	110-1 *	0.728 (25/4vac) *	1.421 (25) *	<u>4-Methyl-heptadiene-(1,5).</u> * C_8H_{14} B.b.no. = 245, Test 925. *
161	¹⁶⁷ 110-111	¹⁶⁷ 0.829 (20/4)	¹⁶⁷ 1.471 (20°)	<u>1-Methyl-cyclohexadiene-(1,5)(?)</u> C_7H_{10} -- Fr. 1,2-dibrom-1- methyl-cyclohexane+quinoline. (167).
163	¹⁷⁰ 110-111	¹⁷¹ 0.816 (14/4)	n_D given	<u>1,1-Dimethyl-cyclohexadiene-(2,4)</u> C_8H_{12} -- Air g. yellow gelatinous mass. (170). - Cold oxid. w. $KMnO_4$ g. α, α -dimethyl succinic ac. (170). - Nitroso- chloride in poor yields by cold AmONO in AcOH + conc. HCl, needles frm. MeOH, melts grad- ually between 118.5-126° w. dec. (170).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
165	¹⁶⁸ 111-112	0.813 (16.3/4)	n_D given	<u>1,1-Dimethyl-cyclohexadiene-(2,5)</u> , C_8H_{12} -- Cold oxid. w. $KMnO_4$ g. dimethyl malonic acid. -Fum. HNO_3 + conc. H_2SO_4 g. 3,4,5 + 3,4,6-trinitro-1,2-dimethyl benzene. (168).
167	¹⁴⁵ * 111-113	0.763 (0°) ¹⁴⁵		<u>Heptene-(2)</u> * C_7H_{12} . B. b. no. 122, Test 925.
169	¹⁷² 113-114	0.764 (20/4) ¹⁷²	1.516 ¹⁷² (19.8)	<u>1,3,5-Heptatriene</u> , C_7H_{10} .
171	¹⁷³ 113-114	0.742 (19/4) ¹⁷³	1.432 ¹⁷³ (12°)	<u>Octadiene-(2,6)</u> , C_8H_{14} .
173	¹⁷⁴ 113-114	0.751 (20°) ¹⁷⁴	1.431 ¹⁷⁴ (20°)	<u>2,5-Dimethyl-hexadiene-(1,5)</u> , C_8H_{14} -- Ozone in presence of H_2O g. formaldehyde + acetyl acetone. (175). - HBr g. <u>2,5-dibromadiisocrotyl oxide</u> (anhydride of 2,5-dimethyl-hexandiol-(2,5)). (176).
175	¹⁵⁹ 113-117	0.802 (20°) ¹⁵⁹	1.448 ¹⁵⁹ (20°)	<u>Ethylidene-cyclopentane</u> , C_7H_{12} -- G. a nitrosochloride wh. by boiling w. $NaOAc$ + $AcOH$ g. oxime of 1-acetyl-cyclopentene-(1). (159).
177	⁹⁸ 114-115	0.764 (16.5/4) ⁹⁸	1.455 ⁹⁸ (16.5)	<u>2,4-Dimethyl-hexadiene-(2,4)</u> , C_8H_{14}
179	¹⁷⁷ 114-115			<u>Heptatriene-(1,3,6)</u> , C_7H_{10} .
181	^{178, 179} 114.5-115.5	0.823 (20/4) ¹⁸⁰	1.453 ¹⁸⁰ (20°)	<u>Cycloheptene</u> , C_7H_{12} .
183	¹⁸ 116-117	0.733 (20/4) ¹⁸	1.451 ¹⁸ (20°)	<u>2-Methyl-heptadiene-(3,5)</u> , C_8H_{14} -- G. no ppt. w. aq. or alc. $HgCl_2$. (54).
185	116-118	0.741 (22°)		<u>2-Methyl-heptadiene-(4,6)</u> , C_8H_{14}
187	¹⁸² 116-118	0.741 (20/4) ¹⁸²	1.450 ¹⁸² (20°)	<u>2,5-Dimethyl-hexadiene-(1,3)</u> , C_8H_{14} -- Does not solidify at -80°. (182) - W. $KMnO_4$ g. little Me_2CO , small amt. of an xtl. erythritol, HCO_2H , $AcOH$, + Me_2CHCO_2H . (182).
189	^{179, 183} 117-118	0.888 (18.3/4) ¹⁸⁴		"Tropilidene" <u>Cycloheptatriene-(1,3,5)</u> , C_7H_{10} -- CrO_3 mixture g. benzoic ac. + benzaldehyde. (183).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
191	¹⁸⁶ 117-119			<u>Octadiene-(2,6), C₈H₁₄</u> -- Adds 3.5 atoms Br. (187).
193	²⁰⁶ 77-82 (80 mfn.)			<u>2-Methyl-heptatriene-(4,5,6) ?</u> C ₈ H ₁₂ -- HgCl ₂ in aq. - alc. soln. g. white ppt. (206). - Htg. in tube at 110-120° w. K g. K cpd. of methyl heptenine. (206).
195	¹⁸⁵ 117-120.5			<u>2-Methyl-hexene-(3)-ene-(5),</u> C ₇ H ₁₀ -- G. Test 906. (185)
197	⁹⁵ 119-123	0.764 (20/4) ⁹⁵	1.451 (20) ⁹⁵	<u>2,5-Dimethyl-hexadiene-(2,3)(?)</u> C ₈ H ₁₄ -- M.P. lies between -23° and -80°. (95).
199	¹⁸⁸ 119.5-121.5	0.803 (20/4) ¹⁸⁸	1.445 (16.20) ¹⁸⁸	<u>1,1-Dimethyl-cyclohexene-(3),</u> C ₈ H ₁₄ .
201	¹⁹¹ 121-122	0.795 (20/4) ¹⁹²	1.442 (20) ¹⁹²	<u>1,2,3-Trimethyl-cyclopentene-(1),</u> C ₈ H ₁₄ -- Cold aq. KMnO ₄ g. 3-methyl heptandione (2,6) (193) - D-form shows $\alpha_D^{15} = +23.60$. (193)
203	¹⁷⁹ 121.5-122.5	0.868 (17.60) ¹⁷⁹	n_D given	<u>Cycloheptadiene-(1,3), C₇H₁₀.</u>
205	¹⁸⁹ 122-123	0.760 (14/0) ¹⁸⁹		<u>3-Ethyl-hexadiene-(2,5), C₈H₁₄</u> -- CrO ₃ oxid. g. CO ₂ , AcOH, + Et CO ₂ H. (189).
207	^{195,196} 122-123	^{195,196} 0.792 (20°)	^{195,196} 1.445 (20°)	<u>4-Methyl-1-methylene-cyclohexane</u> C ₈ H ₁₄ -- Oxid. w. 1% KMnO ₄ at 0° g. an ac., 4-methyl-1-methylol-cyclohexanol-(1), + 1-methyl-cyclohexanone-(4). (195,196). - G. a nitroso-chloride wh. by loss of HCl g. the oxime of Δ^1 -tetrahydro-p-toluyaldehyde. (195).
209	¹⁹⁴ 122-125	0.808 (22°) ¹⁹⁴	1.452 (22°) ¹⁹⁴	<u>2-Methyl-1-methylene-cyclohexane</u> C ₈ H ₁₄ -- Oxid. w. KMnO ₄ g. 2-methyl-1-methylol-cyclohexanol-(1) + 1-methyl-cyclohexanone-(2) (194). - G. a solid nitroso-chloride wh. by loss of HCl g. the oxime of tetrahydro-o-toluyaldehyde. (194).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
211	123-124 ¹⁹⁰	0.790 (15°) ¹⁹⁰	1.486 ("n") ¹⁹⁰	<u>2,5-Dimethyl-hexadiene-(1,5) iner(3)</u> , C_8H_{10} -- Easily resinifies in the air, especially w. ht. (190). - H + Pt. g. 2,5-dimethyl hexane. (190).
213	123-4 ⁷¹³	0.879 (13/4) ⁷¹³		<u>2-Methylene-bicyclo-[1,2,2] - heptane</u> , "Nor-camphene", C_8H_{12} -- 1 cc. hy. + 1.75 cc. $C_5H_{11}ONO$ + slow addn. of (cold) 1 cc. fum. HCl g., in abt. 19 min., nitroschloride, xtals fm. AcOEt, M.P. = 125°. (713).
215	123-4 ¹⁹⁸	0.794 (20) ¹⁹⁸	1.447 (18) ¹⁹⁸	<u>1-Methyl-3-methylene-cyclohexane</u> , C_8H_{14} -- L-form shows $[\alpha]_D^{20} = -290$ (without solvent). (198). - $KMnO_4$ oxid. g. 3-methyl-1-methylol-cyclohexanol- (1) + 1-methyl-cyclohexanone- (3). (198). - G. in poor yield a nitroschloride wh. w. alc. KOH g. the oxime of tetrahydro- <i>m</i> -tolualdehyde. (198).
217	123.5-4.5 ²⁰⁵	0.806 (20/4) ²⁰⁵	1.445 (20) ²⁰⁵	<u>1,2-Dimethyl-cyclohexene-(3 and 4)</u> , C_8H_{14} .
219	124-5 ²⁰¹	0.801 (18/4) ²⁰¹	1.443 (18) ²⁰¹	<u>1,3-Dimethyl-cyclohexene-(4)</u> , C_8H_{14} -- $HNO_3-H_2SO_4$ g. 2,4,6-trinitro-1,3-dimethyl-benzene. (201). - 1 vol. H_2SO_4 + 4 vol. (201) EtOH g. first red color to violet, then violet, finally blue. (201).
221	126-7 ²⁰²	0.797 (21/4) ²⁰²	1.444 (202)	<u>1,3-Dimethyl-cyclohexene-(5)</u> , C_8H_{14} -- G. a nitroschloride w. difficulty. (202). - $KMnO_4$ oxid. does not g. keto ac. (202).
223	126-9 ²⁰⁶			<u>2-Methyl-heptene-(4)-ine-(6)</u> , C_8H_{12} -- G. Test 906. (206).
225	127-8 ¹⁹⁹	0.801 (20/4) ²⁰³	1.446 (20) ^{199,203}	<u>1,4-Dimethyl-cyclohexene-(1)</u> , C_8H_{14} -- 1% $KMnO_4$ g. 1,2-dihydroxy-1,4-dimethyl-cyclohexane, M.P. - 77°, wh. is also formed by addn. of Br. in AcOH soln., treatment of dibromide w. AcOAg, + sapon. w. KOH. (199).-

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
227	127-8 ²⁰⁸	0.821 (18/4) ²⁰⁸	1.469 (19) ²⁰⁸	Nitrosochloride (by EtNO ₂ , HCl, +AcOH), M.P. = 83-4° (199). - Nitrolpiperidide, M.P. = 169-70° (199). <u>1,3-Dimethyl-cyclohexadiene(3,5)</u> , C ₈ H ₁₂ -- In glac. AcOH at 0° adds 2 HCl g. C ₈ H ₁₄ Cl ₂ , B.P. = 93-7° (208).
229	127-8 ^{213*}	0.762 (17) ²¹³	1.426 (17) ²¹³	<u>Octine-(1)</u> ,* C ₈ H ₁₄ -- H ₂ SO ₄ g. octanone (2). (214) - G. test 906. - B.b.no. = 142,* test 925 - Hg salt, test 926, M.P. = 79-80°*
231	127-8 ²¹⁵			<u>2-Methyl-3-ethyl-hexadiene-(1,5)</u> , C ₉ H ₁₆ -- Odor of peppermint and lemon. (215) - Adds 4 atoms of Br. (215)
233	127-30 ²²⁵			<u>3-Ethyl-hexadiene-(2,4) (?)</u> , C ₈ H ₁₄ -- Adds only 2 atoms of Br. (225).
235	127.5-8.5 ¹⁹⁹	0.802 (22/4) ²⁰⁰	1.447 (22) ²⁰⁰	<u>1,3-Dimethyl-cyclohexene-(3)</u> , C ₈ H ₁₄ -- D-form shows [α] _D = +95° (200). - Nitrosochloride, M.P. = 118-90° (199). - Nitrolpiperidide, M.P. = 130-1° (199).
237	127.5-8.5 ²⁰⁴	0.801 (20/4) ²⁰⁴	1.446 (22) ²⁰⁴	<u>1,4-Dimethyl-cyclohexene-(3)</u> , C ₈ H ₁₄ .
239	129-30 ²¹⁷	0.764 (20) ²¹⁷	1.458 (20) ²¹⁷	<u>3-Cyclopropyl-pentene-(2) (?)</u> , C ₈ H ₁₄ .
241	129.5-30.5 ²⁰³	0.823 (20/4) ²⁰³	1.468 (20) ²⁰³	<u>1,3-Dimethyl-cyclohexadiene-(1,5)</u> , C ₈ H ₁₂ -- D-form shows [α] _D ²⁰ = +27.38°. (203).
243	129.5-31.5 ²¹⁶	0.767 (13/4) ²¹⁶	1.461 (13) ²¹⁶	<u>2-Methyl-heptadiene-(1,3)</u> , C ₈ H ₁₄ -- Easily resinified. (216).
245	130-2 ²⁴¹	0.842 (20/0)	1.460	<u>Ethynyl-cyclohexane</u> , C ₈ H ₁₂ .
247	131-2 ²¹³	0.755 (21) ²¹³	1.430 (21) ²¹³	<u>Octine-(3)</u> , C ₈ H ₁₄ .
249	131-2 ²²⁶	0.763 (20/4) ²²⁶	1.463 (20) ²²⁶	<u>4-Methyl-heptadiene-(3,5)</u> , C ₈ H ₁₄

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
251	131-2 ²²⁷	0.755 (25/4) ²²⁷	1.462 (25) ²²⁷	<u>4-Methyl-heptadiene-(2,4), C₈H₁₄</u>
253	131.5-3.5 ²²⁸			<u>1-Isopropyl-cyclopentene-(1), C₈H₁₄</u> -- 8.4 cc in 20 cc AcOH + 7.6 cc. C ₅ H ₁₁ NO ₂ treated w. 17 cc conc. HNO ₃ in 17 cc. AcOH during 1.5 hrs., then addn. of Me ₂ CO till oil goes into soln. g. nitrosochloride, needles w. blue lustre, M.P. = 88° (228). - Nitrolpiperidide, C ₈ H ₁₄ NONC ₅ H ₁₀ , fine needles, M.P. = 93.5° (228). - The nitrosochloride treated w. AcONa + AcOH and then dil. H ₂ SO ₄ g. 2-isopropyl-cyclopentene(2)-one, whose semicarbazone, M.P. = 203-4° (228).
255	132-3 ²³⁰	0.768 (20/0) ²³⁰	1.442 ²³⁰	<u>1,1-Dimethyl-2-[2-methopropene-(2¹)yl]-cyclopropane, C₉H₁₆</u>
257	132-5 ²²⁹	0.767 (15/4) ²²⁹	1.465 (15) ²²⁹	<u>3-Methyl-heptadiene-(2,4), C₈H₁₄</u>
259	132.5-3.5 ⁵¹⁸	0.822 (20/4) ⁵¹⁸	1.468 (20) ⁵¹⁸	<u>1,4-Dimethyl-cyclohexadiene-(1,5), C₈H₁₂</u>
261	133-4 [*]	0.743 (25/4 vac) [*]	1.454 (25) [*]	<u>Octadiene-(2,4), C₈H₁₄</u> -- B. b. no.: 279, Test 925. [*]
263	134-5 ²⁰⁹			<u>1,2-Dimethyl-cyclohexadiene-(x,x), "Cantharene", C₈H₁₂</u> -- Oxid. w. dil. HNO ₃ g. o-toluic ac. + phthalic ac. (209). - Conc. H ₂ SO ₄ or conc. H ₂ SO ₄ + alc. g. orange color (210). - Ac ₂ O + H ₂ SO ₄ g. red-brown color. (210). - Ac ₂ O + Br. g. blue-green color. (210).
265	82-3.5 ²⁴⁶ (96 min.)	0.794 (11/4) ²⁴⁶	1.533 (15) ²⁴⁶	<u>3-Methyl-hentatriene-(2,4,6), C₈H₁₂</u> -- Absorbs O very quickly. (246). - Reduces HgCl ₂ to Hg ₂ Cl ₂ . (246).
267	134-5 ²¹⁸	0.773 (18) ²¹⁹		<u>2,5-Dimethyl-hexadiene-(2,4), C₈H₁₄</u> -- M.P. = +11° (221) - Very unstable. (220). - Absorbs O quickly frm. air. (220). Polym. by stg. (220). - G. liquid tetrabromide. (220). - Cf. No. 281.

DIVISION B SECTION 3

No.	Boiling Point (°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
269	222, 223, 224 134-5	0.804 (15/4) ⁴⁹³	1.445 (20) ⁴⁹³	<p><u>1,1,2,3-Tetramethyl-cyclopentene</u> (2). "Campholene", $C_{10}H_{16}$ -- $KMnO_4$ oxid. g. oxalic ac. + β, β-dimethyl levulinic ac. (222). Shak. w. conc. H_2SO_4 g. dicampholene, B.P. = 266-70° (partial dec.), B.P.₃₀ = 165-80°, d_4^{20} = 0.8993. (224). - Nitrosochloride by $NaNO_2$ + cold HCl, indigo-blue mass of odor of camphor, M.P., rapid hgt. = 25° (224).</p>
271	232, 233 134-5	0.826 (20) ²³²	1.458 (20) ²³²	<p><u>1-Ethyl-cyclohexene-(1)</u>, C_8H_{14} -- G. a nitrosochloride wh. w. NaOAc + AcOH + dec. of reaction products w. H_2SO_4 g. a 1-ethyl-cyclohexene (1)-one-(6). (232)</p>
273		0.809 (20/0) ²³⁵	1.451 (20) ²³⁵	<p><u>1-[1-Ethoxypropylidene]-cyclobutane</u>, C_9H_{16} -- Menthene-like odor. (235).</p>
275	237, 238 134-5	0.862 (20/4) ^{237, 238}	1.481 (20) ^{237, 238}	<p><u>1,4-Dimethylene-s-spiroheptane</u>, "Trimeride of allene", C_9H_{12}.</p>
277	234 134.5-5.5	0.861 (26/4) ²³⁴	1.458 (26) ²³⁴	<p><u>Apoisofenchene</u>, C_9H_{16} -- $KMnO_4$ g. trans-apofencho-camphoric ac., M.P. = 143.5-4.5°. (234).</p>
279	135-6 ²⁰⁷	0.837 (20/4) ²⁰⁷	1.486 ²⁰⁷	<p><u>1,3-Dimethyl-cyclohexadiene - (1,3)</u>, C_8H_{12} -- Odor of pinene. (207). - G. orange color w. H_2SO_4 and claret-red w. H_2SO_4-Ac_2O. (207).</p>
281	135-6 ²³¹	0.765 (18/4) ²³¹	1.480 (20) ²³¹	<p><u>2,5-Dimethyl-hexadiene-(2,4)</u>, "Diisocrotyl", C_8H_{14} -- M.P. = 14.5°. (231). - Stable if pure. - Br. in CCl_4 g. dibromide wh. is readily hydrolyzed in damp air, M.P. = 45° when htd. slowly, M.P. = 72° when htd. suddenly, after hgt. at 60° takes 2 hrs. of cooling to xtalize, even in presence of seeds, after hgt. at 100° and sudden cooling xtalizes immediately; $Me_2CBrCH=CHCBrMe_2$, solid, M.P. = 72° + $Me_2CBr.CBr.CH=CMe_2$, liquid, equilibrium mixture = M.P. = 45°; adds no more Br. (231). - Cf. no. 267.</p>

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
283	135-6 ²³⁶	0.817 (21) ²³⁶	1.453 (15) ²³⁶	<u>Octadecane (1.7)</u> , C ₁₈ H ₃₈ -- Absorbs O on stg. (236).
285	135-8 ²¹¹	0.830 (20/4) ²¹¹	1.480 (19) ²¹¹	<u>1,4-Dimethyl-cyclohexadiene - (1.3)</u> , C ₈ H ₁₂ -- Polym. quickly in moist state. (211).-KMnO ₄ at 0° g. acetone + acetyl-acetone. (211).-Complete nitration g. trinitro-p-xylene. (211). Dissolves in conc. H ₂ SO ₄ g. orange-red color, in alc. H ₂ SO ₄ g. yellow color, in Ac ₂ O + H ₂ SO ₄ g. first a reddish, then a yellow color. (211).
287	199,203 135.5-6.5	0.823 (20/4) ^{199,203}	1.459 (20) ¹⁹⁹	<u>1,2-Dimethyl-cyclohexene-(1)</u> , C ₈ H ₁₄ -- Nitrosochloride at first forms a deep blue oil, then colorless xtals, M.P.=58-60°, wh. do not yield an oxime, but split off NOCl. (199). Glycol, M.P.=38-9° (199).- Dibromide, M.P.=154-60. (199).
289	135.5-6.5 ²⁰²	0.792 (15/4) ²⁰²	1.446 (15) ²⁰²	<u>1,3-Dimethyl-5-methylene-cyclohexane</u> , C ₉ H ₁₆ -- Does not g. nitrosochloride. (202).- KMnO ₄ oxid. g. 1,3-dimethyl-5-cyclohexanone. (202).
291	135.5-6.5 ²⁰⁷	0.852 (20/4) ²⁰⁷	1.490 ²⁰⁷	<u>1,2-Dimethyl-cyclohexadiene (2.6)</u> , C ₈ H ₁₂ -- Odor of turpentine. (207).- H ₂ SO ₄ g. orange-red color. (207).- Ac ₂ O + H ₂ SO ₄ g. carmine changing to brown. (207).- HNO ₃ (dil.) oxid. to o-toluic ac; M.P.=103°. (207).
293	135.5-7 ^{218*}	0.787 (17) ²¹⁸	1.437 (17) ²¹⁸	<u>Octine-(2)*</u> , C ₈ H ₁₄ -- H ₂ SO ₄ g. octanone-(2) + octanone-(3). (242).- Na at 110° g. octine - (1). (242).- Does not g. Test 906. (242).- G. oily tetra-bromide. (243).- B.b.no.:160,* Test 925.

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
295	Abt. 136 133-4 ⁽²¹²⁾ (720 mm)			<u>1,4-Dimethyl-cyclohexadiene - (1,4) (?)</u> , C ₈ H ₁₂ --Odor of turpentine. (212). - G. xtaln. addn. product w. HBr. (212).
297	136-7 ²³⁹	0.817 (20) ²³⁹	1.458 (20) ²³⁹	<u>Isopropylidene-cyclopentane</u> , C ₈ H ₁₄ --Oxid. w. 1% cold KMnO ₄ g. cyclopentanone, besides acs. + a xtaln. glycol of M.P. = 61-30. (239).
299	^{202,244} 136-7	^{202,244} 0.824 (18/4)	^{202,244} 1.464 (18)	<u>Ethylidene-cyclohexane</u> , C ₈ H ₁₄ -- Nitrosochloride, M.P. = 1320, (245), wh. htd. 10 min. at 60-50 w. NaOAc + glac. AcOH g. 1-acetyl-1-acetoxy-cyclohexane-oxime, M.P. = 1030, (245), and wh. w. MeOH + MeONa g. 1-acetyl-1-methoxy-cyclohexaneoxime, (244).
301	²⁴⁷ 137.5-8.5	0.824 (20) ²⁴⁷	1.458 (20) ²⁴⁷	<u>1-Methyl-cycloheptene-(1)</u> , " <u>Δ¹-Methyl-suberene</u> ", C ₈ H ₁₄ --EtNO ₂ + conc. cold HNO ₃ + AcOH g. white nitrosate, C ₈ H ₁₄ O ₄ N ₂ ; M.P. = 97-80 (dec.). (247).
303	²⁴⁹ 137.5-9	0.891 (20/4) ²⁵⁰	1.484 (20) ²⁵⁰	<u>Bicyclo-[O, X, X]octene</u> , C ₈ H ₁₂ -- Immediately decolorizes Br + KMnO ₄ . (249). - Conc. H ₂ SO ₄ g. slight yellow color after 1-2 min. (249).
305	²⁵⁵ 138-9	0.791 (22) ²⁵⁵	1.438 (22) ²⁵⁵	<u>1-Methyl-3-isopropyl-cyclo-pentene-(2)</u> , " <u>Pulegene</u> ", C ₉ H ₁₆ -- KMnO ₄ oxid. g. AcOH + 2,6-dimethyl-heptanone-(5)-ac-(1). (256). - Nitrosochloride, frm. EtONO + conc. HCl in AcOH at -15 to -200, M.P. = 74-50, wh. w. NaOMe in MeOH g. pulegenon-oxime. (255).
307	^{253,258} 138-9	0.803 (20) ²⁵⁸	1.446 (22) ²⁶⁰	<u>1,1,3-Trimethyl-cyclohexene-(3)</u> , " <u>Δ-Cyclogeraniol-ene</u> ", C ₉ H ₁₆ -- Conc. H ₂ SO ₄ g. red color. (260). - Aq. KMnO ₄ g., besides other products, iso-geranic ac. (259). - Nitrosate, by conc. HNO ₃ + cold AcOH + C ₅ H ₁₁ ONO, xtals, M.P. = 102-40, wh. w. alc. KOH g. oxime of 1,1,3-trimethyl-cyclohexene-(2)-one-(4). (258). - C ₅ H ₁₁ ONO +

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
309	138-40 ²⁴⁷	0.824 (20) ²⁴⁷	1.461 (20) ²⁴⁷	<p>conc. HCl in cold AcOH g. nitrosochloride, blue xtals, M.P. = 100-120° (frm. aq. + MeOH), wh. w. boiling alc. KOH g. oxime of 1,1,3-trimethyl cyclohexene-(2)-one-(4). (258).</p> <p><u>Methylene-cycloheptane.</u> <u>"Methylene-suberane", C₈H₁₄</u> -- KMnO₄ oxid. at 0° g. suberone, 1-methylol-cycloheptanol-(1) + 1-cycloheptane-carbonic ac.-(1). (247).</p>
311	138-40 ²⁵¹			<p><u>Octadiene-(3,5), C₈H₁₄</u> --O₃ in cold CCl₄ g. yellowish-red, thick, oily ozonide, wh. on dec. w. boil. H₂O (1/2 hr.) g. H₂O₂, EtCHO, + (CHO)₂, wh. on further treatment w. boil. HNO₃ g. (CO₂H)₂ + EtCO₂H. (251). - Hy. gradually turns yellow on stg. (251).</p>
313	138-40 ²⁶¹			<p><u>1,1,2-Trimethyl-3-methylene-cyclopentane, C₉H₁₆</u> -- Turpentine odor. (261) - KMnO₄ oxid. g. 1,1,2-trimethyl-cyclopentanone-(3). (261).</p>
315	138-41 ²⁵⁴	0.851 (20/0) ²⁵⁴	1.483 ²⁵⁴	<p><u>Ethenylidene-cyclohexane, C₈H₁₂</u> -- Warming w. Na to 100° g. partly cyclohexyl-acetylene, partly a C₈H₁₄ (?). (254).</p>
317	139-40 ⁷⁰⁵	0.843 (20/4) ⁷⁰⁵	1.449 (20) ⁷⁰⁵	<p><u>2,2,4-Trimethyl-bicyclo-[1.2.2]heptene -(5), "Qvist's 'Isufenchylene', Semmler's 'Iso-allofenchene', 'Nemetkin's 'Fenchylene', 'Isufenchene (new) 'δ-Fenchene', (705) C₁₀H₁₆</u> -- $n_D^{20} = 57.28$ (711, 712) Active form eagerly absorbs Br. w. evolution of some HBr + oil formation. (712). - EtONO + HCl g. nitrosochloride, M.P. = 131° wh. oxid. to fencho camphoric ac. (fm. 1-form of δ-fenchene). (712). - Cf. Nos. 365, 401, 425.</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
319	139-41 ²⁴⁸	0.801 (10) ²⁴⁸	1.448 (10) ²⁴⁸	<u>3,3-Dimethyl-methylene-cyclohexane</u> , C_9H_{16} --
321	139.5-40.5 ²⁶³	0.874 (15/4) ²⁶⁴	1.454 (20) ²⁶³	<p><u>2,3-Dimethyl-bicyclo-[1,2,2]-heptene</u> -(2), "Santene", (265), C_9H_{14} -- Occurs in Austrian sandalwood oil, various pine-needle oils, etc. (262). - Odor somewhat resembles that of menthene. $[\alpha]_D = -0.50$ (1=100 mm.) (greatest reported rotation). (266). - O_3 in C_6H_6 soln. in presence of H_2O + dec. of reaction products w. H_2O vapor g. 1,3-diethylon-cyclopentane. (265). - 33% HCl + ether soln. of hy. mixed w. $NaNO_2$ in H_2O g. nitrosite, green-blue xtals frm. EtOH + pet. ether, M.P. = 124-5° w. dec. (265, 267). - 7.5 cc conc. HCl dropped into mixture of 5 g. hy., 5 g. AcOH, + 6 g. EtONO in freezing mixture, g. α-santene-nitroschloride, M.P. = 109-10° (dec.), wh. by polym. in air g. β-nitroschloride, white, stable xtals. g. at 90° the blue α modification. (265). - Htg. w. H_2SO_4-AcOH g. the acetate, $d_4^{20} = 0.988$; $n_D^{20} = 1.460$, wh. sap'd. w. EtOK g. isocamphenilol wh. latter w. CrO_3 g. isocamphenilone or santenone whose semi-carbazone, M.P. = 225-6° + wh. w. $KMnO_4$ g. santenic ac., M.P. = 170-1° whose anhydride, M.P. = 115-6° and whose anilide, M.P. = 204-5° (267).</p>
323	Abt. 140 (501) 42.2-2.4 (17 mm.)	0.923 (20/4) ⁴⁹⁸	1.539 (20) ⁴⁹⁸	<p><u>Cyclooctatetraene</u>, C_8H_8 -- M.P. = abt. -27°. (498) - Yellow liquid. (501) - Cf. Div. B, Sect. 1, No. 20.</p>
325	140-1 ²⁶⁸	0.810 (15/4) ²⁶⁸	1.449 (15) ²⁶⁸	<p><u>1,1,4-Trimethyl-cyclohexane</u>-(3), C_9H_{16} -- Conc. H_2SO_4 g. blood-red soln. (268). - Nitroschloride, M.P. = 118-22° dpdg. on rate of htg. (268). - O_3 g. 1,1-dimethyl-3-aceto-cyclopentene-(3), whose semi-carbazone, M.P. = 156-8°. (268).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
327	140-1 ²⁷⁰	0.868 (20/4) ²⁷⁰	1.469 ²⁷⁰ (20)	<p><u>2,2-Dimethyl-bicyclo-[1,2,2]-heptene-(3), "Camphenilene",</u> $C_{10}H_{14}$ --Hydrochloride, plates, M.P. = 60-1°.(270). - Nitrosite, blue-green prisms frm. ligroin, M.P. = 122°.(270). - O_3 g. ketone-aldehyde, B.P. = 123-5°, d_4^{20} = 1.033, n_D^{20} = 1.469, whose disemicarbazone, M.P. = 205-6°.(270).</p>
329	141-3 ²⁷²	0.801 (20/0) ²⁷²	1.446 ²⁷²	<p><u>1-Methyl-2-methoxyethyl-cyclopentane, C_9H_{16}.</u></p>
331	580, 581 142-3	0.795 (21) ⁵⁸⁰	1.440 ⁵⁸⁰ (21)	<p><u>1-Methyl-3-isopropyl-cyclopentene-(5), "Apofenchene",</u> $C_{11}H_{18}$ --$[\alpha]_D$: +66.21°.(580, 581). Oxid. by cold neutral $KMnO_4$ g. 3-isopropyl-hexanone-(5) acid-(1). (581). - $EtONO + HCl$ in $AcOH$ g. mixture of oily + solid nitrosochlorides of wh. 1, M.P. = 115°.(580).</p>
333	142-3 ²⁵⁹	0.757 (20) ²⁵⁹	1.437 ²⁵⁹	<p><u>2,6-Dimethyl-heptadiene-(1,5), "Geraniolene", $C_{10}H_{16}$</u> -- Inversion w. H_2SO_4 g. mixture of α-+(3-cyclogeraniolene.(274) HBr g. 2,6-dibrom-2,6-dimethyl-heptane. (260). - O_3 g. diazonide $C_9H_{16}O_6$ wh. by htg. w. aq. g. levulinic aldehyde. (275). - $KMnO_4$ g. acetone + levulinic ac. (276).</p>
335	142-4 ⁶⁴⁹	0.773 (22) ⁶⁴⁹	1.424 ⁶⁴⁹	<p><u>Dihydropulegene, C_9H_{18}.</u></p>
337	202, 271 142.5-3.5	0.797 (21) ^{202, 271}	1.445 ^{202, 271} (21)	<p><u>1,3,5-Trimethyl-cyclohexene-(1), "Tetrahydro-mesitylene", $C_{10}H_{18}$</u> -- Nitrosochloride, M.P. = 134°.(271). - Nitropiperidide, M.P. = 122-3°.(271) - Oxid. g. 1,3,5-trimethyl-1,2-dihydroxy-cyclohexane, M.P. = 104°.(271).</p>
339	273 142.5-144.5			<p><u>1-Methyl-2-methoxyethyl-cyclopentene-(4), C_9H_{14}</u> -- H_2SO_4 g. to soln. of hy. in Ac_2O blood-red color turning violet on htg. (273).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
341	143-4 ²⁴⁹	0.887 (0/4) ²⁴⁹		<u>Cyclooctadiene-(1,4) ?,"3-Cyclooctadiene", C₈H₁₂ -- Energetically reduces KMnO₄. (249).</u>
343	143-4 ²⁷⁶	0.772 (16/4) ²⁷⁶	1.445 (22) ²⁷⁶	<u>2,6-Dimethyl-heptadiene-(X,X), C₉H₁₆ --KMnO₄ g. acetone.(276)</u>
345	143-5	0.870 (20/0)	1.491	<u>1-Ethenyl-cyclohexene-(1), C₈H₁₂ -- Shows no optical exaltation. - Unaffected by htg. w. Na.</u>
347	31 ³⁰⁸ (7 mm.)	0.792 (22) ³⁰⁸	1.461 (22) ³⁰⁸	<u>2,6-Dimethyl-heptadiene-(4,6), C₉H₁₆ --Very fleeting unpleasant odor. (308).- Adds 2 atoms of Br quickly, further Br very slowly w. HBr evolution. (308).- O₃ g. HCHO + methyl-glyoxal. (308).</u>
349	143-5 ²⁷⁷	0.765 (10/4) ²⁷⁷	1.462 (10) ²⁷⁷	<u>2,6-Dimethyl-heptadiene-(1,3), C₉H₁₆.</u>
351	143.5-4.5 ²⁷⁹	0.808 (20/0) ²⁷⁹	1.446 ²⁷⁹	<u>1,1-Diethyl-cyclopentene-(2), C₉H₁₆ --G. a green color w. H₂SO₄ + alc. (279).- Hot KMnO₄ g. an ac., C₇H₁₄ (CO₂H)₂, M.P.= 85°, wh. w. Ac₂O g. an anhydride. M.P.= 10-10°, B.P.=190° wh. latter w. PhNH₂ g. C₇H₁₄ (CO₂H)-CONHPh, M.P.= 142° that on warming g. dianilide, M.P.=163°. (279).</u>
353	144-5 ²⁸²	0.805 (20/4) ²⁸²	1.448 (20) ²⁸²	<u>1,2,4-Trimethyl-cyclohexene-(4), C₉H₁₆.</u>
355	144-6 ²⁷⁸	0.775 (14/4) ²⁷⁸	1.463 (14) ²⁷⁸	<u>2,5-Dimethyl-heptadiene-(3,5), C₉H₁₆.</u>
357	Abt. 144-6 ²⁸⁰	0.801 (20) ²⁸⁰	1.448 (20) ²⁸⁰	<u>1-Methyl-3-isopropyl-cyclopentene-(1 or 2, or both), C₉H₁₆</u>
359	144-6 ²⁸⁸	0.805 (20/4) ²⁸⁸	1.448 (20) ²⁸⁸	<u>1,2,3 (?) -Trimethyl-cyclohexene-(4), C₉H₁₆.</u>
361	145-6 ²⁸¹	0.855 (20/4) ²⁸¹	1.474 (20) ²⁸¹	<u>Cyclooctene, C₈H₁₄ --Decolorizes KMnO₄ + Br. solns. immediately. (281).- G. no color w. H₂SO₄ - Odor of parsley.(281).Polymerizes rather readily to white powder. -HBr + glac. AcOH g. bromo-cyclooctane, oil w. sweet odor</u>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
363	39.5 ²⁵² (15.5 mm)	0.889 (0/4) ²⁵²		of peppermint, B.P. ₁₀ =90.5-1.5; d ₄ ²⁰ =1.290; n _D ²⁰ =1.511. (281). <u>Cyclooctadiene-(1,5), C₈H₁₂</u> -- Polym. on stg. at rm. temp. in 2 or 3 days to xtaln. dimer, plates frm. gasoline or ether, sinter at 1060, M.P.=114°.(252) O ₃ in CCl ₄ g. diozonide wh.w. boiling H ₂ O g. succindialdehyde + succinic ac. (252) - Immediately decolorizes KMnO ₄ .(252) Does not reduce Ag soln.,(252) cf. Test 906. - Alc. soln. + Conc.H ₂ SO ₄ g. orange-yellow color. (252).
365	145-7 ⁷⁰⁵	0.855 (17/4) ^{705,710}	1.461 (17) ^{705,710}	2,2,5-Trimethyl-bicyclo-[1,2,2]-heptene-(5). "1-Fenchene", C₁₀H₁₆ -- Cf. Nos. 317,401, 425.
367	146-7 ²⁸²	0.814 (20/4) ²⁸²	1.449 (20) ²⁸²	<u>1^c,2^c,4-Trimethyl-cyclohexene-(4), C₉H₁₆.</u>
369	146-9 ²⁸⁷			<u>2,3-Dimethyl heptadiene-(2,5 or 3,5) or 2-Methyl-3-methylene-heptene-(5), C₉H₁₄</u> -- Isomerisation product resulting by action of H ₂ SO ₄ g. nitrosate, M.P.=122.50. (287).
371	147-8 ⁵⁰² 133-5 ⁵⁰⁰ (715 mm)	0.903 (20/4) ⁴⁹⁸	1.528 (20) ⁴⁹⁸	<u>Cyclooctatriene, C₈H₁₀</u> -- Cf. Div. B, Sect. 1, No. 30.
373	226,302 148-9	0.751 (20/4) ^{226,302}	1.455 (15) ^{226,302}	<u>2-Methyl-octadiene-(4,6), C₉H₁₆</u> G. a bis-hydro-bromide w. HBr in AcOH + a tetrabromide w. Br. (302)2
375	148-9 ⁴⁹⁵	0.814 (25/4) ⁴⁹⁵		<u>1,2-Diethyl-cyclopentene, C₉H₁₆.</u>
377	148-9 ²⁸⁵	0.817 (16/4) ²⁸⁵	1.453 (16) ²⁸⁵	<u>1-Methyl-4-ethyl-cyclohexene-(3), C₉H₁₆.</u>

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
379	148-9 ²⁹⁶	0.820 (13) ²⁹⁶	1.454 (13) ²⁹⁶	<p><u>1-Cyclohexyl-propene-(2)</u>, C₉H₁₆ -- Dibromide, B.P.₁₆=143-40, d₀=1.537. (296). - HgO + 1 g. syrupy mixture of 2 iodohydrins wh. when dried and treated in Et₂O w. anhyd. KOH g. cyclohexyl-propene-oxide, B.P.=182-50, d=1.003, isomerizing under influence of dil. H₂SO₄ to C₆H₁₁(CH₂)₂CHO whose semi-carbazone, M.P.=1370. (297). - Mixture of iodohydrins treated at 80-100° for several hrs. w. Me₂NH + condensation products washed w. C₆H₆, soln. + washings evapd. w. aq. HCl + subsequently rendered alk. w. NaOH g. the yellow amine, C₆H₁₁.CH₂.CH(OH).CH₂.NMe₂, B.P.₁₉=133-40, d₀=0.934 whose methiodide, M.P.=1750; and whose benzoate hydrochloride, M.P.=1820. (297). - B.b.no: 124*, Test 925.</p>
381	148-9 ⁶⁶⁹	0.821 (20/4) ⁶⁶⁹	1.447 (20) ⁶⁶⁹	<p><u>(3)-Thujene</u>, C₁₀H₁₆ -- D-form shows [α]_D: +109.090. (670) - Only very difficultly oxid. by KMnO₄. (670).</p>
383	148-51 ²⁶⁸	0.822 (20/4) ²⁶⁸	1.456 ²⁶⁸	<p><u>1,1,2-Trimethyl-cyclohexene-(2)</u>, C₉H₁₆ -- Nitrosochloride, M.P.=133-40°. (268).</p>
385	226, 295 149-50	0.771 (20/4) ²²⁶	1.466 (20) ²²⁶	<p><u>4-Methyl-octadiene-(3,5)</u>, C₉H₁₆</p>
387	84-6 ²⁸⁹ (102 mm)	0.791 (4/15) ²⁸⁹	1.461 (15) ²⁸⁹	<p><u>3-Ethyl-heptadiene-(1,3)</u>, C₉H₁₆</p>
389	149-50 ³⁰³	0.798 (20/4) ³⁰³	1.468 (20) ³⁰³	<p><u>1,1-Dimethyl-2-methylene-3-isopropylidene-cyclobutane</u>, C₁₀H₁₆ -- Kerosene odor. (303). - G. xtaln. cpd. w. NaNO₂, M.P.=100° dec. - O₃ g. 1-methyl-ene-2,2-dimethyl-cyclobutanone-(4), B.P.₅₀=59-60°, d₄²⁰=0.8684, n_D²⁰=1.447 wh. latter exposed to air develops xtals. M.P.129°, exploding at higher temp. and wh. also g. semicarbazone, M.P.=160-900, dec. (303). - O₃ also g. 1,1-dimethyl-3-isopropylidene-cyclobutanone of B.P._{11.5}=58-65°.</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
391	149-50 ²⁷³			unstable in air. (303). <u>1-Methyl-3-methoxyethyl-cyclopentene (1 or 5 or mixture of both), C₉H₁₄.</u>
393	149-51 ²⁷²	0.810 (20/0) ²⁷²	1.452 ²⁷²	<u>1-Methyl-2-isopropylidene-cyclopentane, C₉H₁₆.</u>
395	149-51 ^{283,200}	0.809 ^{200,283} (25/4)	1.454 ²⁸⁴ (19)	<u>1-Methyl-3-ethyl-cyclohexene (2 or 3 or both), C₉H₁₆</u> -- Nitrosochloride softens at 118° M.P. = 124-6° (283). - D-form shows $[\alpha]_D = +56.80$. (200,284)
397	150-1 ²⁸⁶	0.828 (20/4) ²⁸⁶	1.459 (20) ²⁸⁶	<u>1,2,3-Trimethyl-cyclohexene, C₉H₁₆</u> -- Nitrosochloride is a blue oil. (286).
399	151-2 ²¹³	0.760 (20) ²¹³	1.423 (20) ²¹³	<u>Nonene-(1), C₉H₁₆</u> -- G. Test 906. (40) - Hg salt, Test 926, M.P. = 69-70°.
401	151-3 ^{705,710}	0.860 ^{705,710} (20/4)	1.466 ^{705,710} (20)	<u>2,2-Dimethyl-5-methylene-bicyclo-[1,2,2]-heptane, "Wallach's 'D-d-and L-l-Fenchene' (optical antipodes)", "Semmler's 'Isosfenchene' (old)" "β-Fenchene." (705) C₁₀H₁₆</u> -- $[\alpha]_D^{17} = +150.46$ (l=10 cm). (708) Much more quickly attacked by KMnO ₄ than α-fenchene. (709). - hydroxy-β-fenchenic ac., needles, M.P. = 124-5° (710) wh. w. PbO ₂ + H ₂ SO ₄ g. dl-β-fenchocamphorone, cf. below, wh. latter w. KMnO ₄ g. "apofenchocamphoric ac., (710), i.e. 1,1-dimethyl-cyclopentan-dioic-(2,4) ac., M.P. = 144-5° (705). - O ₃ g. β-fenchocamphorone, M.P. = 64-6°, B.P. = 196-70, whose semicarbazone, M.P. = 193-5° (705,710). - Cf. Nos. B17, 365,425.

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
403	151-2 ^{669, 527}	0.830 (20/4) ⁶⁶⁹	1.452 (20) ⁶⁶⁹	<p><u>4-Methyl-1-isopropyl-bicyclo-[0.1.3]-hexene-(3). "α-Thuiene", C₁₀H₁₈</u> --Occurs in resin oil of <i>Boswellia serrata</i>. (527). - KMnO₄ oxid. g. either α-thujaketoacid, M.P.=75-6°, semicarbazone, M.P.=182-3°; or d-α-tanacetogen-dicarbonic ac., M.P.=140-3°; or a d-ac., C₈H₁₄O₄, M.P.=116-7°. (527, 594).</p>
405	152-3 ²⁹⁰	0.812 (19) ²⁹⁰	1.457 (19) ²⁹⁰	<p><u>1-Methyl-4-ethylidene-cyclohexane, C₉H₁₆</u> --Nitrosochloride, xtals frm. ether - MeOH, blue, M.P.=108-10°, (290), wh. is a mixture of isomers, less sol., M.P.=117-8°, whose nitrolpiperidide, M.P.=130-1°; + more sol., M.P.=113-4°, whose nitrolpiperidide, M.P.=119-20° w. partial formation of higher melting isomer + of oxime. (291). Both forms of nitrosochloride g. same oxime (of 1-methyl-4-ethylone-cyclohexane-(3)), M.P.=116-7°, g. 1-methyl-4-acetoxy-4-acetoxime, M.P.=111-2° as intermediate. (291).</p>
407	152-3 ²⁹⁴	0.813 ²⁹⁴	1.458 ²⁹⁴	<p><u>1-Methyl-3-ethylidene-cyclohexane, C₉H₁₆</u> --L-form shows: [α]_D = -500. (293). - Nitrosochloride, prisms, M.P.=114°. - Nitrolpiperidide, needles, M.P.=101-20°. (293). - Mixing w. ice + 1% KMnO₄ g. 1-methyl-3-α-hydroxy-ethyl-cyclohexanol-(3), silky needles, M.P.=68°. (293). - Nitrosochloride w. AcONa + glac. AcOH w. gentle htg. and then 10 min. boil. g. d-3-acetyl-1-methyl-cyclohexene-(2)-oxime, prisms, M.P.=79° whose benzoyl deriv. needles, M.P.=85-60°. (293).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
409	²⁹² 153-4	0.815 (22) ²⁹²	1.451 ²⁹²	<u>1-Methyl-4-ethyl-cyclohexene-(3)</u> , $C_{10}H_{16}$ -- Nitrosochloride exists in two forms, (a) difficultly sol., large, transparent prisms, M.P.= 103-4°, (b) easily sol., M.P.= 98-9°, both wh. g. same nitrolpiperidide, M.P.= 134° + oxime, M.P.= 59-60°. (292) - Dil. $KMnO_4$ g. 1-methyl-4-ethyl-4,5-dihydroxy-cyclohexane. (292).
411	²⁵⁷ 61-6 (37 mm)	0.828 (20/4) ²⁵⁷	1.481 ²⁵⁷ (20)	<u>1,3-Dimethyl-5-methylene-cyclohexene-(3)</u> , $C_{10}H_{14}$ -- For its transformation to mesitylene see Auwers, Peters, Ber. <u>43</u> , 3076.
413	⁵⁰⁷ Around 50 (23 mm) 38-40 (15 mm)	0.869 (16/4) ⁵⁰⁷	1.509 ⁵⁰⁷ (15)	<u>1,1-Dimethyl-4-methylene-cyclohexadiene-(2,5)</u> , $C_{10}H_{12}$ -- After 3 distns. in H, $d_4^{15} = 0.843$, $n_D^{15} = 1.509$. (507). - A few drops of conc. HCl in AcOH g. pseudocumene.
415	153-4 *	0.761 (25/4 vac) *	1.438 (25) *	<u>4,5-Dimethyl-octadiene-(2,6)</u> , $C_{10}H_{18}$ -- B.b.no: 193, * Test 925.
417	³⁰⁵ 153-5	0.777 (20/4) ³⁰⁵	1.445 ³⁰⁵ (20)	<u>3,6-Dimethyl-octadiene-(2,6)</u> , $C_{10}H_{18}$ -- $KMnO_4$ g. CO_2 , succinic ac., AcOH. (305)
419	154-6	0.755 (15)	1.435 (15)	<u>2,6-Dimethyl-octadiene-(2,7)</u> , "Menthonylene", $C_{10}H_{18}$.
421	⁶⁴⁸ 154-6	0.868 ⁶⁴⁸	1.471 ⁶⁴⁸ (20)	<u>Isopinene</u> , $C_{10}H_{16}$ -- $[\alpha]_D = 2.30^0$. (648) - G. hydrochloride, M.P.= 36°. (648)
423	^{306, 307} 154.5-5.5	0.838 (19) ³⁰⁶	1.458 ³⁰⁶ (19)	<u>1-Propyl-cyclohexene-(1)</u> , $C_{10}H_{16}$ -- Nitrosochloride, M.P.= 104°, wh. w. MeONa + MeOH + ht. g. oxime of a ketone $C_9H_{14}O$. (306).
425	⁷⁰⁵ 155-6	0.867 (20/4) ⁷⁰⁵	1.471 ⁷⁰⁵ (20)	<u>7,7-Dimethyl-2-methylene-bicyclo-[1,2,2]-heptane</u> , "Wallach's 'D-I-and I-d-Fenchene' (optical antipodes)", (705) "Isopinene", (706) "I-Fenchene", (705) $C_{10}H_{16}$ -- Odor similar to that of camphene. (527). - Its occurrence in nature has been

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
				<p>neither definitely shown nor disproven. (527). Optical activities of $[\alpha]_D^{20} = -32.320$, (707), + 43.930 (706) have been described. - The $[\alpha] = 43.930$ g. hydrochloride, M.P. = 35-7°. (706). - Active forms w. O_3 g. d l-α-fenchocamphorone whose semicarbazone, M.P. = 220° (706,707) + a monobasic ac., M.P. = 105° (706). - The l-form g. a d-dibromide, M.P. = 87-8°. (527). - AcOH + H_2SO_4 g. the acetate of isofenchyl alc. whose free alc. M. = 61.5-2° and g. phenyl urethane, M.P. = 106-7°. (527). - Cf. Nos. 317, 365, 401. For a discussion of the structure and reactions of the fenchenes cf. Komppa, Roschier, Ann. 470, 129 (1929).</p>
427	155-6 *	0.772 (25/4 vac) *	1.433 (25) *	<p>Nonene-(4) *, $C_{18}H_{34}$ --B.b.no. = 106, Test 925. *</p>
429	634, 635, 636 155-6	0.860 (20/4) ⁶³⁷	1.466 ⁶³⁵ (20)	<p><u>2,6,6-Trimethyl-bicyclo-[1.1.3]heptene-(2) *</u>, "<u>α-Pinene</u>", "<u>Pinene</u>", as "<u>Australene</u>", $C_{10}H_{16}$ - l-Pinene formerly known as "<u>Terebenthene</u>", d-Pinene as "<u>Australene</u>". Widely distributed in essential oils frm. leaves, rinds, and woods. - Occurs in turpentine, hazel-nut, myrrh, cajeput, peppermint, corianda, etc. oils. (527, 594). - D-form shows $[\alpha]_D = +46.73^\circ$. (635). - L-form shows $[\alpha]_D = -43.40$. (638). - Long htg. at 250-700 g. polym. products + dipentene. (590). - 10-15 hrs. htg. w. Et_2SO_4 at 120° in a sealed tube oxid. it to p-cymene w. formation of Et_2O, SO_2, H_2O. (639). - Mixt. of hy. + alc. - aq. - HCl in air begins to separate terpin hydrate after 1 hr. (640). - 50% (by vol) H_2SO_4 at 80° g. mixture of terpenes. (603). - For active forms, shake 100 g. hy. w. soln. of 233 g. $KMnO_4$ in 3 l. H_2O w. ice cooling, remove unoxid. hy.,</p>

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				<p>extract neutral oxid. product w. ether; acid. w. H₂SO₄ g. pinonic ac., (641), (inactive form, M.P.= 103-5°), active form, M.P.= 69.6-70.5°) (527) whose semicarbazone, M.P.= 204° (641).- EtONO, AcOH, + HCl g. nitrosochloride, inactive form, M.P.= 103-15° dec., (527, 642), -wh. w. NaOMe g., besides nitrospinene, C₁₁H₁₉O₁N, xtals. frm. EtOH, M.P.= 101-2° (643), and whose nitrolpiperidide, M.P.= 118-9°; and whose nitrol-benzylamide, M.P.= 122-3° (527).- 10 g. d-pinene nitrosochloride, + 10 g. piperidine, + 30 cc. alc. htd. + pptd. w. H₂O g. d-nitrospinene, prisms frm. Et₂O, M.P.= 131-2°, + d-pinene-nitrolpiperidide, M.P.= 118-9° (644).- For strongly active hy., mix equal vols. hy., EtONO, absolute alc., treat w. HCl in alc. below -5°, filter off inactive nitrosochloride, dil. w. 1-2 vols. alc., cool below -10°; needles of active nitrosochloride separate, M.P.= 81-1.5° α ± 322° (alc. or CHCl₃), wh. g. nitrolpiperidide, M.P.= 84°, wh. g. nitrolbenzylamide, M.P.= 144-5° (645).- Htg. pinene w. 1/10 pt. by wgt. picric ac. to boiling, whereby reaction proceeds to an end, continually boil without further addn. of ht., g. straw yellow plates frm. boiling EtOH, M.P.= 133° (646).- Gaseous HCl or HBr + cold hy. g. d-pinene hydrochloride, [α]_D = +33.19°, M.P.= 127°, B.P.= 207.5°; d-pinene hydrobromide, [α]_D = +31.31°, M.P.= 80°, boils w. dec. (647).- To distinguish frm. β-pinene, cf. latter, No. 495, B.b.no.= 236-254* (Test 925).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
431	155-6 ²⁵³			<u>1,1-Dimethyl-3-ethyl-cyclohexene (2 or 3 or both), C₁₀H₁₈</u>
433	155-6 ⁶⁷⁴	0.854 (15) ⁶⁷⁴	1.462 ₂ (20) ⁶⁷⁴	<p>C₁₀H₁₆; Terpene frm. Tsuga heterophylla. (674) - Carrot-like odor. $[\alpha]_D = +4^\circ 50$ (674). Nitrosochloride dec. at abt. 85° (674). - Nitrolpiperidide, M.P. = 194-5° (674). - Nitrolamine, M.P. = 105-8° (674)</p>
(Nos. 435-451 on next page).				

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
435	155-7 ²⁰⁷	0.815 (20/4) ²⁰⁷	1.489 ²⁰⁷	<u>1-Methyl-2-methoxyethyl-cyclopentene-(1)</u> , C_9H_{14} .
437	155-7 ^{298,304}	0.829 (20) ²⁹⁸	1.461 (20) ²⁹⁸	<u>1-Isopropyl-cyclohexene-(1)</u> , C_9H_{16} --Nitrosochloride, white prisms frm. C_6H_6 , M.P.=129-30° wh. by htg. w. alc, piperidine soln. or AcONa in AcOH g. oxime of a ketone, $C_9H_{14}O$. (298).
439	155.5-6.5	0.750 (25/4 vac)	1.431 (25)	<u>4-n-Propyl-heptadiene-(1,5)</u> , $C_{10}H_{18}$ --B.b.no.= 181, Test 925.
441	155.5-7.5 ²¹⁶	0.782 (13/4) ²¹⁶	1.463 (13) ²¹⁶	<u>3-Methyl-octadiene-(2,4)</u> , C_9H_{16} -- Ctg. some 3-methylene-octene-(4). (216)
443	85-7 ³⁰¹ (36 mm)			<u>Triene frm. 5-methyl-1,5-octadien-4-ol + KHSO₄</u> , C_9H_{14} -- Boiled at 760 mm. g. polymer. (301).- Unstable in presence of ac., polym. (301).- Adds Br rapidly. (301).- Polym.on htg. w. Na. (301).- Absorbs O rapidly. (301).- NH_3 -Ag ₂ O is not reduced, but $HgCl_2$ in EtOH is quickly reduced to $HgCl$. (301).
445	Abt. 156 153-4(497) (717 mm) 46 (497) (11 mm)	0.881 (20/4)	1.547 (20)	<u>1-Isopropylidene-cyclopentadiene-(2,4) "α,α-Dimethylfulvene"</u> , C_8H_{10} --Cf. Div. B, Sect. 1, No. 36.
447	157-8 ³⁰⁶	0.821 (19) ³⁰⁶	1.463 (19) ³⁰⁶	<u>n-Propylidene-cyclohexane</u> , C_9H_{16} Nitrosochloride, prisms frm. C_6H_6 , M.P.= 119°, wh. w. NaOMe in MeOH g. oxime of ethyl [1-methoxy-cyclohexyl] ketone. (306).
449	157-8 ³⁰⁹			<u>1,1,2-Trimethyl-5-ethenyl-cyclopentene-(2)</u> , $C_{10}H_{16}$ -- Odor like camphene. (309).
451	40,299 157-8	0.844 (18) ^{40,299}	1.460 (18) ^{40,299}	<u>1-Cyclohexyl-propene-(2)</u> , C_9H_{14} -- G. leaflets w. $AgNO_3$. (300) -- (G. Test 906). (40).- $NaNH_2 + CO_2$ g. cyclohexyl-butinoic ac., (213) M.P.= 74-5°, (296), whose methyl ester, B.P. ₁₅ =1350, $d_{16}^4=0.998$, $n_D^{12}=1.484$. (296).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
453	157-8 ³¹¹	0.81 (20) ³¹¹	1.47 ³¹¹	<u>1-Methyl-2-ethylidene-cyclohexane, C₉H₁₆.</u>
455	158-9 ³¹⁰	0.766 (21/0) ³¹⁰		<u>4-n-Propyl-heptadiene-(1,4), C₁₀H₁₈ --G, unstable tetra-bromide. (310). -- CrO₃ g. AcOH, MeCH₂CO₂H, + Me(CH₂)₂CO₂H. (310).</u>
457	158-9 ⁶⁹³	0.863 (20) ⁶⁹³	1.466 (20) ⁶⁹³	<u>2,7,7-Trimethyl-bicyclo-[1,1,3]heptene-(3), "Dihydroverbenene", C₁₀H₁₆ -- [α]_D = 36.52° (693). -- Adds Br without any sharp end-pt. and resulting bromide does not solidify. (693). -- 5 g. g. 0.5 g. nitrosochloride, M.P. = 103-4° wh. w. NaOEt g. nitrospinene, M.P. = 131.2° (693). -- 5 g. in 10 g. cold CS₂ sat'd. w. dry HCl g. 1.7 g. pinene hydrochloride, M.P. = 130° (693). -- 3 g. shak. 24 hrs. w. 100 cc. 10% H₂SO₄ g. 1.4 g. cis-terpinol hydrate, M.P. = 117° (693).</u>
459	159-60 ⁵¹³	0.882 (20) ⁵¹³		<u>4,7,7-Trimethyl-bicyclo-[1,1,3]heptadiene-(1,3), C₁₀H₁₄ -- Cf. Div. B, Sect. 1, No. 48.</u>
461	159-63 ³¹⁸	0.846 (20/4) ³¹⁸		<u>1- [Propene(1²)-ylidene] - cyclohexane, C₉H₁₄ --G. a tetrabromide. (318).</u>
463	160-1	0.774 (20/4)	1.443 (20)	<u>1-Methyl-1- [1⁵-methopentene-(1⁴) yl] -cyclopropane, C₁₀H₁₈ -- Oxid. g. Me₂CO; 1-methyl-1- [1⁵-methopenton-(1³)-yl] -cyclopropane, M.P. = 14.5-15°, B.P. = 223°, d₄²⁰ = 0.941, n_D²⁰ = 1.449, whose semicarbazone M.P. = 149-51°; + 3- [3¹-methocyclopropyl] -propanoic ac. - (1), B.P. = 219-22°, d₄¹⁵ = 0.992, n_D¹⁵ = 1.444 whose anilide, M.P. = 126-7°.</u>

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
465	160-1	0.833 (20/0)	1.466 (20)	<u>1,3-Dimethyl-3-ethenyl-cyclohexene-(6)</u> , C ₁₀ H ₁₆ --By hot polym. of isoprene.
467	160-1 ²¹³	0.768 (21) ²¹³	1.434 (21) ²¹³	<u>Nonene-(2)</u> , C ₉ H ₁₆ .
469	160-1 ²⁹⁸	0.836 (20) ²⁹⁸	1.472 (20) ²⁹⁸	<u>Isopropylidene-cyclohexane</u> , C ₉ H ₁₆ --Nitroschloride, deep blue, camphor odor, volatile w. steam, oil wh. gradually solidifies to colorless xtals. M.P.= 83° (298). - KMnO ₄ oxid. g. glycol of M.P.=82° + cyclohexanone. (298).
471	160-1 ³¹²	0.833 (20/0) ³¹²	1.447 (20) ³¹²	<u>1,3-Dimethyl-3-ethenyl-cyclohexene-(6)</u> , C ₁₀ H ₁₆ --Dihydrobromide, M.P.= 34-5° (312).
473	160-2 ³⁷⁴	0.835 (20) ³⁷⁴	1.467 (20) ³⁷⁴	<u>1-Methyl-2-isopropylidene-cyclohexane</u> , "o-Menthene-(2(8))". C ₁₀ H ₁₈ --1% KMnO ₄ oxid. in cold g. 1-methyl-cyclohexanol-(2) + other products. (374).
475	160-3 ³¹³	0.843 (22) ³¹³	1.476 (22) ³¹³	<u>1-Methyl-4-ethylidene-cyclohexene-(1)</u> or <u>1-Methyl-4-ethenyl-cyclohexene-(1)</u> , or both, C ₉ H ₁₄ .
477		0.833 (20/4) ²⁵⁷	1.481 (18) ²⁵⁷	<u>1,3-Dimethyl-5-ethylidene-cyclohexene-(3)</u> , C ₁₀ H ₁₆ .
479	160.5-1.5	0.829 (17/4)	1.484 (18)	<u>1-(Propene(1¹)-yl)-cyclohexene-(1)</u> , C ₉ H ₁₄ .
481	160.5-1.5 ³¹⁵	0.839 (18) ³¹⁵	1.493 (18) ³¹⁵	<u>1-Methyl-4-ethyl-cyclohexadiene-(1,3)</u> , C ₉ H ₁₄ .
483	160.5-7.5 ⁵⁸⁷	0.801 (20/20) ⁵⁸⁷	1.458 (20) ⁵⁸⁷	<u>Heptarene</u> , C ₁₀ H ₁₈ . --By passing vapors of spinacene at 45 mm. over htd. Pt. (dull redness). (587) - Tetrabromide, M.P.= 136°. (587).
485	161-2 ³⁰⁴			<u>1-Methoethenyl-cyclohexene-(1)</u> , C ₉ H ₁₄ .

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
487	333:335 161-71	333:334 0.768 (25)- 0.782 (15)	333:334 1.446 - 1.453 (15)	<u>2,6-Dimethyl-octadiene-(1 or 2, 6 or 7), "Linaloolene", "Dihydromyrcene", C₁₀H₁₈</u> -- Several preps. of doubtful structures and varying constants have been described - Tetrabromide, M.P.= 88-8.5° (334).- Tetrabromide, M.P.= 92° (335).- Nitrosate, M.P.= 95° dec. (by AcOH, C ₅ H ₁₁ ONO + fum. HNO ₃ , then xtaln. frm. Me ₂ CO + aq.) (333).- AcOH + H ₂ SO ₄ g. cyclodihydromyrcene + dihydrolinalylacetate. (334).
489	162-3 ³¹⁴	0.768 (20/0) ³¹⁴	1.442 ³¹⁴	<u>2,6-Dimethyl-octadiene from citral hydrazone, C₁₀H₁₈</u> -- Sabatier reduction g. 2,6-dimethyl-octane. (314).
491	162-7 ²⁴⁶	0.802 (19/4) ²⁴⁶		<u>4-Methyl-octatriene-(3,5,7), C₁₀H₁₄</u> -- Polymerizes by distn. at ord. press. (246).
493	163-4 ³⁴⁹	0.826 (0) ³⁴⁹	1.497 ³⁴⁹	<u>Octadiene-(3,5), C₈H₁₀</u> --4 days htg. w. alc. HgCl ₂ in tube at 100° g. small amt. of octadiene-(3,5). (349).
495	163-4	0.866 (22)	1.472 (22)	<u>5,6-Dimethyl-2-methylene-bicyclo-[1,1,3]-heptane, "α-Pinene", "Nopinene", "Pseudopinene, C₁₀H₁₆</u> --Occurs in most turpentine oils, in various other oils as citronella, coriander, etc. (527;594).- L-form especially abundant in oils frm. Douglas fir. (527).- L-form shows: [α] _D = -22° 21' (l=10 cm.) (615) - Oxid. w. cold 1% KMnO ₄ g. (β-pinene glycol M.P.= 76-8° (615;527) + l-nopinic ac; M.P.= 126-70 (527;615).- Htg. several hrs. w. AcOH-H ₂ SO ₄ at 60° g. terpinene. Shak. 300 g. hy. w. soln. of 700 g. KMnO ₄ + 150 g. NaOH in 9 l. H ₂ O, without cooling, distn. w. steam, concn. of filtered residue, under CO ₂ , to 3 l. g. xtals. of Na salt of nopinic ac. on cooling. (616;617).

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon																					
				<p>-Nopinic ac. + PbO₂ or KMnO₄ in H₂SO₄ soln. g. nopinone, whose semicarbazone, M.P. = 188°. (527). - To distinguish β-pinene frm. α-pinene, add hy. to a cold alc. Hg(OAc)₂; after stg. 2-3 days β-pinene soln. remains clear, while α-pinene ppts. Hg₂(OAc)₂ and is oxid. to sabinol + oxyhydrocarbone. (527). - Sol. in 100 vols. EtOH: (619):</p> <table style="margin-left: auto; margin-right: auto; border-collapse: collapse;"> <thead> <tr> <th style="border-bottom: 1px solid black;">%EtOH</th> <th style="border-bottom: 1px solid black;">Vols. -pinene</th> <th style="border-bottom: 1px solid black;">Vols. Pinene</th> </tr> </thead> <tbody> <tr> <td>95</td> <td>35.8</td> <td>30.4</td> </tr> <tr> <td>90</td> <td>13.0</td> <td>9.75</td> </tr> <tr> <td>85</td> <td>8.25</td> <td>5.1</td> </tr> <tr> <td>80</td> <td>6.0</td> <td>2.3</td> </tr> <tr> <td>70</td> <td>1.6</td> <td>0.2</td> </tr> <tr> <td>60</td> <td>0.5</td> <td>trace</td> </tr> </tbody> </table> <p>B.b.no. 256, Test 925.</p>	%EtOH	Vols. -pinene	Vols. Pinene	95	35.8	30.4	90	13.0	9.75	85	8.25	5.1	80	6.0	2.3	70	1.6	0.2	60	0.5	trace
%EtOH	Vols. -pinene	Vols. Pinene																							
95	35.8	30.4																							
90	13.0	9.75																							
85	8.25	5.1																							
80	6.0	2.3																							
70	1.6	0.2																							
60	0.5	trace																							
497	163-4 ⁶⁵⁰	0.848 (15/15) ⁶⁵⁰	1.468 (20) ⁶⁵¹	<p>1-Isopropyl-4-methylene-bicyclo-[0.1.3]-hexane,* <u>"Sabinene", C₁₀H₁₆</u> -- Occurs in oils of sassafras, cubeb, cardamom frm. Ceylon, etc. (527, 594) [α]_D = +63° 50' (1=100 mm) (650) - Shak. w. cold dil. H₂SO₄ quickly g. d-terpineol-(4) and then p-menthandiol-(1,4) of M.P. = 137° (terpinene terpin). (652). - Boil. w. dil. H₂SO₄ (1:7) g. ordinary terpinene. (651). - Alk. KMnO₄ g. very difficultly sol. Na salt of sabinene ac. whose free ac. M.P. = 57° and wh. by oxid. g. sabinaketone whose semicarbazone, M.P. = 141-2°. (527). - KMnO₄ g. sabinene-glycol, M.P. = 54°. (527). - B.b.no: 250, * Test 925.</p>																					
499	163-6 ³²⁴			<p><u>1,3-Diethyl-cyclohexene-(4), C₁₀H₁₈</u> -- G. oily dibromide. (324).</p>																					

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
501	^{321,322} 163.5-4.5	0.785 (20/4) ³²²	1.448 (20) ³²²	<u>2,7-Dimethyl-octadiene-(2,6), C₁₀H₁₈</u> --Tetrabromide, M.P.= 124-70. (323). - KMnO ₄ g. AcOH + succinic ac. (322).
503	164-5 ²⁴⁸	0.832 (11) ²⁴⁸	1.464 (11) ²⁴⁸	<u>2,3,3-Trimethyl-1-methylene-cyclohexane, C₁₀H₁₈</u> .
505	164-6 ⁶⁶⁷	0.810 (21) ⁶⁶⁷	1.447 ⁶⁶⁷	<u>1,2-Dimethyl-3-isopropyl-cyclopentene-(5), "Thuiamenthene", (668) C₁₀H₁₈</u> -- Nitroschloride, M.P.= 108°; easily splits off HCl by htg. soln. g. isothujone oxime, M.P.= 119°. (667).
507	164-7 ²²⁹	0.783 (15/4) ²²⁹	1.467 (15) ²²⁹	<u>2,6-Dimethyl-octadiene-(4,6), C₁₀H₁₈</u> .
509	164.5-5.5 ²²⁷	0.775 (25/4) ²²⁷	1.461 (25) ²²⁷	<u>2,5-Dimethyl-octadiene-(3,5), C₁₀H₁₈</u> .
511	^{282,331} 165-6	0.817 (20/4) ^{282,331}	1.457 (20) ^{282,331}	<u>1^c, 2^t, 4,5-Tetramethyl-cyclohexene-(4), C₁₀H₁₈</u> .
513	165-8			<u>1-Methyl-2-isopropyl-cyclohexene-(1 or 2), "o-Menthene-(1 or 2)", C₁₀H₁₈</u> --Odor like peppermint.
515	^{328,336} 165-8	0.788 (20) ^{328,336}	1.455 ^{328,336}	<u>2,6-Dimethyl-octadiene-(2,7), C₁₀H₁₈</u> .
517	165-70			<u>Cyclohexyl-propine; mixture; C₉H₁₄</u> --Na + CO ₂ g. cyclohexylbutinoic ac., M.P.= 74-5°. (296).
519	³²⁵ 166-7	0.792 (12) ³²⁵	1.452 (15) ³²⁵	<u>2,6-Dimethyl-octadiene-(2,6), C₁₀H₁₈</u> --Weak mint-like odor. (325). - G. red color w. conc. H ₂ SO ₄ . (325). - KMnO ₄ g. sirupy glycol wh. oxid. to Me ₂ CO, AcOH, + a little levulinic ac. (326). - O ₃ g. ozonide wh. w. H ₂ O g. Me ₂ CO, levulinic ac., + AcOH. (327). - Br. g. tetrabromide. (326,328). - AcOH + H ₂ SO ₄ g. cyclodihydromyrcene. (328).
521	166 ³³² -7	0.845 (21) ³³²	1.491 ³³²	<u>1-Methyl-3-isopropyl-cyclopentadiene-(2,5), C₉H₁₄</u> -- Unstable in the air. (332).

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
523	166-8 ³²⁴			<u>1,3-Diethyl-cyclohexadiene</u> (probably 3,5), $C_{10}H_{16}$.
525	166-70 ³³⁰	0.849 (20/4) ³³⁰	1.469 (20) ³³⁰	<u>1-Methyl-cyclooctene</u> -(1), $C_{10}H_{16}$ — O ₃ g. a ketone whose semi-carbazone M.P. = 182-3° (probably nonanone-(8)-ol-(1)). (330).
527	167-8	0.822 (20/20)	1.457 (20)	<u>1-Methyl-3-isopropyl-cyclohexene</u> -(5), $C_{10}H_{18}$ --Decolorizes $KMnO_4$ + Br.
529	167-8 ³⁴¹	0.812 (20/4) ³⁴¹	1.452 (20) ³⁴¹	<u>1-Methyl-4-isopropyl-cyclohexene</u> -(3), * $C_{10}H_{18}$ --Odor like cymene. (343). - D-form shows $[\alpha]_D = +116.74^\circ$ (highest reported value). (341). - EtONO + fum. HCl in AcOH w. cooling g. nitrosochloride, M.P. = 127-8°; (342) ; wh. w. Na + EtOH g. menthylamine; (342); + wh. w. NaOMe g. menthenone oxime. (345). - Htg. w. anhyd. $CuSO_4$ in sealed tube at 250° g. -cymene. (344). - Htg. w. HNO_3 (d=1.075) in a sealed tube at 100° g. nitromenthene. (346). - Nitrosate, by HNO_3 + EtONO in cold AcOH, cubes, M.P. = 97.5-8°, sol. in 80 pts. ether + 9 pts. CCl_4 . (347). - B.b.no. = 159* (Test 925).
531	55-6 ³⁶⁸ (12 mm)	0.824 (20) ³⁶⁸	1.461 (20) ³⁶⁸	<u>1-Methyl-4-isopropyl-cyclohexene</u> -(2), $C_{10}H_{18}$.
533	167-70 ³³⁷			<u>3,6-Dimethyl-octadiene</u> -(3,5), $C_{10}H_{18}$ --Dil. H_2SO_4 g. oxide $C_{10}H_{20}O$ (anhydride of 3,6-dimethyl-octandiol-(3,6)). (337).
535	168-70 ³³⁸	0.750 (20) ³³⁸		<u>Decadiene</u> -(1,3), $C_{10}H_{18}$.
537	168-71 ²⁸³	0.830 (15) ²⁸³	1.456 ²⁸³	<u>1-Methyl-3-n-propyl-cyclohexene</u> -(2 or 3), $C_{10}H_{18}$ -- Nitrosochloride, M.P. = 128-31°. (283).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
539	168-9 ³⁴⁰			<u>1-Methyl-3-isopropyl-cyclohexene-(2 or 3), "m-Menthene-(2 or 3)"</u> , C ₁₀ H ₁₈ -- C ₅ H ₁₁ ONO + conc. HCl in MeOH g. nitrosochloride, colorless leaves very difficultly sol. in alc., M.P. = 130-2°. (340).
541	168.5-9.5 ²⁸²	0.828 (20/4) ²⁸²	1.461 (20) ²⁸²	<u>1^c, 2^c, 4,5-Tetramethyl-cyclohexene</u> , C ₁₀ H ₁₈ .
543	168.5-9.5	0.822 (21/16)	1.457	<u>3-Dihydro-limonene</u> , C ₁₀ H ₁₈ .
545	169-70 ³⁴⁸			<u>cis-1-Methyl-2-methoxyethyl-cyclohexene-(3)</u> , C ₁₀ H ₁₆ .
547	169-70 ³⁵⁰	0.820 (16/4) ³⁵⁰	1.456	³⁵⁰ <u>1-Methyl-3-isopropyl-cyclohexene-(4 or 5), "m-Menthene-(4 or 5)"</u> , C ₁₀ H ₁₈ .
549	169-70 ³⁵⁴	0.807 (20) ³⁵⁴	1.498	³⁵⁴ <u>3,6-Dimethyl-octadiene-(2,6)-ine-(4)</u> , C ₁₀ H ₁₄ -- Resinifies in the air. (354). - H + Pt black g. 3,6-dimethyl-octane. (354).
551	169-70 ³⁵⁵			<u>trans-1-Methyl-2-methoxyethyl-cyclohexene-(3)</u> , "trans- <u>Δ²⁰-¹⁰7</u> " <u>Menthadiene</u> , C ₁₀ H ₁₆ .
553	169-70 ³⁵⁶			<u>Decadiene-(1,9)</u> , C ₁₀ H ₁₈ .
555	169-71 ³⁶²	0.852 (20/20) ³⁶²	1.473 (20) ³⁶²	<u>1-Methyl-3-isopropyl-cyclohexadiene-(4,6)</u> , C ₁₀ H ₁₆ -- Reduces alk. KMnO ₄ . (362). - Adds only 2 atoms of Br. (362). - H ₂ SO ₄ g. red color. (362). - H ₂ SO ₄ + alc. g. reddish-brown color. (362). - Ac ₂ O + H ₂ SO ₄ g. violet color. (362). - On standing the hy. undergoes change to d ₂₀ ²⁰ = 0.861, n _D ²⁰ = 1.474. (362).
557	65 ³¹² (22 mm)	0.811 (20/4) ³¹²	1.479 (20) ³¹²	<u>1,2-Dimethyl-3,4-diethylidene-cyclobutane</u> , C ₁₀ H ₁₆ .
559	60-5 (15 mm)	0.866 (20/4)	1.514 (20)	<u>1,1,2-Trimethyl-4-methylene-cyclohexadiene-(2,5)</u> , C ₁₀ H ₁₄ -- Rearranges to isodurene.

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
561	³⁵⁸ 169-72	0.822 (15) ³⁵⁹	1.460 (15) ³⁵⁹	<u>1,1,2,3-Tetramethyl-cyclohexene-(3), "Cyclodihydromyrcene", C₁₀H₁₈.</u>
563	Abt. 170 ⁵⁴⁶ 165.5-7 (707 mm)	0.855 (30/30) ⁵⁴⁶	1.474 (30) ⁵⁴⁶	<u>3,7,7-Trimethyl-bicyclo-[0.1.4]heptene-(2), "Δ^4-Carene", "Pinolene", C₁₀H₁₆ -- $[\alpha]_D^{30} = +62.2^\circ$. (546). - Occurs in the essential oil of Andropogon javarancusa, (546), etc. KMnO₄ in Me₂CO at 0° g. d-1,1-dimethyl-2-γ-ketobutyl-cyclopropane-3-carboxylic ac., whose semicarbazone, M.P.=182-3° (546). - HCl in AcOH g. dipentene + silvestrene dihydrochlorides, M.P.= 48-50° and 72° resp. (527). Nitrosochloride, M.P.= 101-2° (527). - Nitrosocarene, M.P.= 89-90° (527). -HCl g. silvestrene dihydrochloride, M.P.= 72° (527). - KMnO₄ oxid. g., beside other products, cis + trans-caron-acid, dec. 174-5° + M.P. = 213° resp., + cis-homocaron acid, C₈H₁₂O₄, M.P.= 137° (527).</u>
565	Abt. 170 ³⁵¹ w. partial depolym. 95 (351) (55 mm)	0.977 (33/4) ³⁵¹	1.505 (35) ³⁵¹	<u>Dicyclopentadiene*, C₁₀H₁₂ -- Two forms, M.P.=32.5° + M.P.= 19.5°; (352); cf. Div. A. Sect. 2, Nos. 38, 78.</u>
567	170-1 ³⁵⁵			<u>1-Methyl-2-methoethenyl-cyclohexene-(4), "$\Delta^{4:8(9)}$-α-Menthadiene", C₁₀H₁₆.</u>
569	170-1 ³⁵⁵			<u>1-Methyl-2-methoethenyl-cyclohexene-(5), "$\Delta^{5:8(9)}$-α-Menthadiene", C₁₀H₁₆ -- Odor of lemons and eucalyptus. (355).</u>
571	170-3 ³⁵⁷	0.814 (19) ³⁵⁷	1.459 (19) ³⁵⁷	<u>1-Methyl-3-n-propylidene-cyclohexane, C₁₀H₁₈ -- L-form: shows $[\alpha]_D = -34^\circ 28'$ (l=1 dm.); nitrosochloride g. mixture of oximes by PhNMe₂. (357).</u>

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
573	170.5-1.5 ³⁶¹	0.810 (20) ³⁶⁰		<u>1-Methyl-4-methoxyethyl-cyclohexane, "p-Menthene-(8(9))", C₁₀H₁₈.</u>
575	170.5-2.5 ²¹⁶	0.787 (13/4) ²¹⁶	1.460 (13) ²¹⁶	<u>4-Methyl-nonadiene-(3,5), C₁₀H₁₈-Unpleasant odor. (46).</u>
577	171-2 ⁶²¹ ₆₂₂ 57 (11 mm)	0.852 (20) ⁶²²	1.479 (20) ⁶²²	<p><u>4-Isopropyl-1-methylene-cyclohexene-(2)*, "p-Menthadiene-(2,1(7))", "β-Phellandrene", C₁₀H₁₆ --In turpentine oil frm. Pinus contorta, Japanese pepper oil, oil of citronella, etc. (527, 594). - [α]_D = +18° 54' (622) or up to + 65° (527). - The hy. is unstable and oxid. quickly in the air. (527). - Pleasant, geranium odor, burning taste. (622). - Long boil. or htg. in sealed tube for 20 hrs. at 140-50° g. diphellandrene. (621). - Htg. w. alc. H₂SO₄ + dil. NaNO₂, sepn. of isomers by Me₂CO + fractional pptn. w. H₂O g. α-nitrosite, M.P. = 102° (less sol.) + β-nitrosite, M.P. = 97-80. (622). - Several hrs. action of aq. NH₃ on nitrosite g., beside nitro β-phellandrene, an ac., C₁₀H₁₇O₄N₃, needles frm. ligroin, M.P. = 75-60 whose Cu salt [Cu(C₁₀H₁₆O₄N₃)₂] dried in vacuum, sky blue microscopic plates, M.P. = 108° dec. (621). - L-form g. nitrite, M.P. = 203-40, [α]_D = 159.9°, (623), D-form g. nitrite, M.P. 2010, [α]_D = 159.3° (623). - EtONO in alc. soln. + alc. HCl g. 2 nitrosochlorides, M.P. = 101-2°, + M.P. = 100° (527). - Oxid. w. 1% KMnO₄ g. sirupy glycol (B.P.₁₀ = 150°) wh. by htg. w. dil. H₂SO₄ g. dihydrocuminalc. + tetrahydrocuminaldehyde whose semicarbazone, M.P. = 204-5°. (527). - 50 g. mixed w. 150 cc. 96% EtOH + sat'd. w. HCl, in 24 hrs. g. red-brown oil frm. wh. in hot EtOH xtalize shining laminas of α-terpinene-dihydrochloride, M.P. = 51-2°. (624). - B. b. no: 155-198,* Test 925.</u></p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
579	171-2 ⁵⁴²	0.896 (12/4) ⁵⁴²	1.484 (12) ⁵⁴²	<u>Endocamphene, C₁₀H₁₆.</u>
581	171-2 ⁶⁰⁹ (slight dec. (610))	0.798 (20/4) ⁶¹¹	1.471 (20) ⁶¹¹	<p><u>2-Methyl-6-methylene-octadiene-(2,7) "Myrcene", C₁₀H₁₆</u> -- Occurs in oil of bay, etc. (527, 594). -NiH g. 2,6-dimethyl octane. (614). -Na + EtOH g. 2,6-dimethyl-octadiene-(2,6), (609), whose tetrabromide, M.P. = 88°. (527). - Htg. in sealed tube at 250-60° g. α-camphorene, tetrahydrochloride, frm. cold ether, rextald. frm. absolute EtOH, M.P. = 129-30°. (527). - Stg. (527) or htg. 4 hrs. at 360° g. dimyrcene, B.P. 13 = 160-200°, whose nitrosite M.P. = 163° (dec.) (613). -H₂SO₄ + AcOH g. myrcenol. (612). - To distinguish frm. ocimene cf. latter, No. 641. - Maleic anhydride g. isohexenyl-4-cis-Δ⁴-tetrahydrophthalic ac. anhydride, M.P. = 34-5°, whose ac., M.P. = 122-3°, wh. latter g. w. aq. HBr at 100° in sealed tube (12 hrs.) dimethyl-1,1-octahydronaphthalenedi-carbonic ac-6,7, M.P. = 206-7°, whose anhydride, M.P. = 215-70. (713).</p>
583	105-7 ²⁸⁹ (90-5 mm)	0.798 (4/15) ²⁸⁹	1.466 (15) ²⁸⁹	<u>4-Ethyl-octadiene-(2,4), C₁₀H₁₈.</u>
585	82-3 ³¹⁶ (17 mm)	0.895 (23/4) ³¹⁶	1.486 (23) ³¹⁶	<u>1-Cyclopentyl-cyclopentene-(1)? C₁₀H₁₆.</u>
587	171-2 ³⁶³	0.927 (17/4) ³⁶³	1.515 (17) ³⁶³	<u>Bicyclo- [0,3,4]-nonadiene-(2,4) C₉H₁₂.</u>
589	171-3 ³⁶⁹	0.836 (20/4) ³⁶⁹		<u>1-Methyl-3-[propene(3²)-yl]-cyclohexene, C₁₀H₁₆.</u>
591	172-3 ³⁷⁰	0.854 (15/4) ³⁷⁰	1.485 ³⁷⁰	<u>2-Methyl-3-[cyclohexene-(3¹)-yl]-propene-(2), C₁₀H₁₆.</u>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
593	373;374 172-4	0.831 (21) ^{373;374}	1.465 (21) ^{373;374}	<p><u>1-Methyl-4-isopropylidene-cyclohexane</u>, "<u>Dihydro-terpinolene</u>", "<u>p-Menthene (4_g)</u>", <u>C₁₀H₁₈</u> --1% KMnO₄ g. 1-methyl-cyclohexanone-(4) + Me₂CO. (373;374).- Nitrosochloride by C₅H₁₁ONO + HCl or by hy. + HCl + HNO₃, deep blue oil wh. solidifies on long stg. g. white xtals., M.P.= 101-3° (373;374).</p>
595	Abt.172 168-9(545) (705 mm)	0.859 (30/30) ⁵⁴⁵	1.469 (n ₃₀) ⁵⁴⁵	<p><u>3,7,7-Trimethyl-bicyclo-[0,1,4]heptene-(3)</u>, "<u>Δ³-Carene</u>", "<u>Isodiprene</u>", <u>C₁₀H₁₆</u> --Occurs in Indian turpentine oil frm. Pinus longifolia, German turpentine oil, etc.- D-form shows [α]_D+7.69° (545).- Ac₂O soln. + a drop of H₂SO₄ g. transient green color - 5 g. mixed w. 2 cc AcOH + 4 g. C₅H₁₁ONO + 3.5 g. HNO₃ (d=1.4) in the cold g. d-carene nitrosate, glistening prisms frm. CHCl₃-pet. ether, dec. 147.5° (547).= 50 g. hy. in ice cold NaOH (50 g. in 500 cc) w. KMnO₄ (116 g. in 1.5 l.) after steam dist'n. to remove unchanged hy. + extn. w. Et₂O g. d-carene glycol, long needles, M.P.= 69-70°, B.P.₄₂=166-7° [α]_D in CHCl₃ = 16.05° (545).</p>
597	172-4 ³⁷⁷	0.809 (20) ³⁷⁷	1.451 (20) ³⁷⁷	<p><u>1-Ethylidene-3-isopropyl-cyclopentane</u>, <u>C₁₀H₁₈</u>.</p>
599	172-5 ⁶⁷¹	0.840 (20) ⁶⁷¹	1.479 (17) ⁶⁷²	<p><u>1,2-Dimethyl-3-isopropyl-cyclopentadiene ?</u>, "<u>Isothuiene</u>", "<u>Tanacetene</u>", <u>C₁₀H₁₆</u>.</p>
601	172.5-3 ³⁷¹	0.837 (20/4) ³⁷¹	1.470 (20) ³⁷¹	<p><u>1-Methyl-4-methoxyethyl-cyclohexene-(2) (?)</u>, "<u>Isolimonene</u>", <u>C₁₀H₁₆</u> --L-form shows [α]_D= -140.58° (371).- D-form shows [α]_D²⁰= +121.93° (372).-Boil 1-form w. K g. 1-hy., B.P.= 180-2° (371).</p>

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
603	173-4 ³⁶⁵	0.823 (17/4) ³⁶⁷	1.460 ³⁶⁷ 1.458 (30) ³⁶⁶	<p><u>1-Methyl-4-isopropyl-cyclohexene-(1)</u>, "<u>2,3,4,5-Tetrahydro-p-cymene</u>", "<u>p-Menthene-β1</u>", "<u>Carvomenthene</u>", "<u>Dihydrophellandrene</u>", "<u>Dihydrolimonene</u>", C₁₀H₁₈ -- Last two names applied in literature to strongly d-forms. (364). - KMnO₄ g. AcOH, 3-isopropylglutaric ac-(1), + small amts. of 2-isopropyl-4-acetyl-n-valeric ac.-(1). (365). - D-form has shown [α]_D as high as +40°. (365).</p>
605	173-4 ³⁷⁶	0.811 (20) ³⁷⁶	1.457 ³⁷⁶	<p><u>1-Methyl-4-p-propylidene-cyclohexane</u>, C₁₀H₁₈ -- Nitrosochloride, M.P. = 138-40°. (376). Nitrolpiperidide, M.P. = 148-90. (376) - Nitrosochloride g. oxime, M.P. = 105-60°. (376).</p>
607	173-4 ⁶⁸⁴	0.838 (22) ⁶⁸⁴	1.475 ⁶⁸⁴ (22)	<p><u>4-Isopropyl-1-methylene-cyclohexene-(3)</u>, "<u>p-Menthadiene-(3,1 (7))</u>", "<u>β-Terpinene</u>", C₁₀H₁₆ -- Reacts only slowly w. HNO₂, then g. α-terpinene nitrosite. (685). - Shak. w. O in presence of H₂O + sunlight g. dihydro-cuminaldehyde wh. oxid. to cuminaldehyde. (685).</p>
609	173-5 ⁶⁰⁸	0.853 (18) ⁶⁰⁸	1.482 ⁶⁰⁸	<p><u>1-Methylene-4-isopropylidene-cyclohexane ?</u>, "<u>p-Menthadiene-(1 (7), 4 (8))</u>", "<u>Crithmene</u>", "<u>Moslene</u>", C₁₀H₁₆. -- In essential oil of Mosla japonica, Maxim, (608), etc. Nitrosochloride, M.P. = 111°. (608). - Nitrolpiperidide, M.P. = 142-30°. (608). - Nitrolanilide, M.P. = 126-80°. (608). - Dihydrochloride, M.P. = 52°. (608). - Nitrosate, M.P. = 124°. (608). - 10 g. of the nitrosochloride htd. w. 2 g. Na + 40 cc. alc. g. (C₁₀H₁₃)₂N₂O, M.P. = 52-30°, sometimes C₁₀H₁₃N=NC₁₀H₁₃, orange-red, M.P. = 85-70°. (608). - Nitrolbenzylamine, M.P. = 106-70° (527). - KMnO₄ oxid. g. γ-terpinene-erythrite, M.P. = 235°. (527).</p>

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
611	173-5 ³⁷⁹	0.825 (20) ³⁷⁹	1.467 (20) ³⁷⁹	<u>1-Methyl-3-isopropylidene-cyclohexane</u> , $C_{10}H_{18}$ --Oxid. w. 1% $KMnO_4$ in the cold g. 1-methyl-cyclohexanone-(3). (379).
613	173-6 ³⁷⁶			<u>1-Methyl-4-propyl-cyclohexane</u> -(3), $C_{10}H_{18}$ --Nitrosochloride, M.P. = 134-5° (376). --Nitrosate, M.P. = 119° (376). --Nitrolpiperidide, M.P. = 150-2° (376).
615	625, 626 175-6	0.841 (20/4) ⁶²⁷	1.473 (20) ⁶²⁷	<u>1-Methyl-4-isopropyl-cyclohexadiene</u> -(1,5), " <u>p-Menthadiene</u> -(1,5)", " <u>α-Phellandrene</u> ", $C_{10}H_{16}$ --Occurs in many essential oils, among them gingergrass, elemi, and Australian eucalyptus. (527, 594). --l-form shows $[\alpha]_D^{20} = -112.76^\circ$ (527, 627). --D-form shows $[\alpha]_D^{20} = +44^\circ 40'$ ($l = 1$ dm.). (625). --Rotation altered by sunlight (629) + hy. distn. at ord. press. (628). --Unstable to acids. (527). -- $KMnO_4$ g. α -oxy- β -isopropylglutaric ac. (630). --Na + EtOH g. p-menthene-(1) of same sign of rotation. (631). --Br in AcOH g. dibromide wh. w. alc. KOH g. p-cymene (630). --Alc. H_2SO_4 g. terpinene w. part. charring. (603). --100 g. hy. in 400 g. ligroin treated w. dil. H_2SO_4 + aq. $NaNO_2$ below 4° g. $\alpha + \beta$ -nitrosites, separable by Me_2CO + fractional pptn. w. H_2O ; α -ppts. 1st, M.P. rapid htg. 113-4° (622), slow htg. 105-6°; (632, 633) β -, M.P. = 105° (622). -- α -nitrosite is less sol. in CS_2 than β + can be thus septd. frm. latter, $[\alpha]_D^{20} = -160^\circ$ (in $CHCl_3$), shows mutarotation. (633). --25 cc. hy. in 150 cc light pet. poured into 53 cc 44% $NaNO_2$ in freezing mixture, treated w. 25 cc glac. AcOH, + xtaln. α -nitrosite removed frm. sticky yellow mass by washing w. EtOH + rextaln. frm. Me_2CO -EtOH, g.

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
617	675, 683 175-6	0.834 (20/4) ⁶⁷⁵	1.478 (20) ⁶⁷⁵	<p>less than 5 g., M.P.=121-2°. (627) (differs frm. M.P. reported on otherwise prepared α-cpd., cf. above).-Maleic an. g. addn. cpd., M.P.=126-7. (718).</p> <p><u>1-Methyl-4-isopropyl-cyclohexadiene-(1,3)</u>, "<u>p-Menthadiene-(1,3)</u>", "<u>α-Terpinene</u>", "<u>Carvenene</u>", C₁₀H₁₆ -- Optically inactive. (528).- Occurs in various essential oils, cardamon, coriander, etc. (527, 594).- Odor like lemons (527) (like limonene). (675).- Alk. KMnO₄ g. only α, α^1-dioxy-α-methyl-α^1-isopropyl-adipic ac., no p-menthane tetrol-(1,2,4,5) (difference frm. γ-terpinene). (675, 676, 677, 678).- Chromyl chloride in CS₂ g. cymene, wh. w. chromyl chloride + H₂O g. α-p-tolyl-propionaldehyde + methyl p-tolyl-ketone. (679).- Repeated reduction w. Na + C₅H₁₁OH g. p-menthene-(2). (680).- 3 cc. hy. + 1.5 cc AcOH + 4.5 cc H₂O + cold conc. aq. soln. of 1.5 g. NaNO₂; nitrosite (best obtained by seeding), M.P.=155°; nitrol-piperidide, M.P.=153-4°, nitrolbenzylamine, M.P.=137° (527).- Nitrosite in AcOH w. HNO₃ (d-1.4) g. C₁₀H₁₅O₆N₃, light yellow xtals frm. EtOH, M.P.= abt. 73°, wh. in Et₂O w. PhNH₂ in Et₂O g. C₁₆H₂₁O₄N₃, yellow needles, M.P.=145°. (681) Nitrosite in MeOH w. NaOMe g. C₂₀H₃₁O₄N₃, needles frm. Me₂CO, M.P.=163-4°, dec. at abt. 175° whose benzyl deriv. M.P.= 127°. (682).</p>
619	175-6 ³⁸⁰			<p><u>1-Methyl-3-methoxyethyl-cyclohexene-(5)</u>, C₁₀H₁₆ -- D-form shows $[\alpha]_D^{25} = 25.3^\circ$; L-form shows $[\alpha]_D^{25} = -29.6^\circ$ in EtOAc. (380).</p>

DIVISION B SECTION 3

No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
621	175-7 ³⁵⁷	0.814 (19) ³⁵⁷	1.452 (19) ³⁵⁷	<u>1-Methyl-4-n-propylidene-cyclohexane, C₁₀H₁₈</u> --KMnO ₄ g. much 1-methyl-cyclohexanone-(4). (357).
623	175-7 ³⁸⁰			<u>1-Methyl-3-methoxyethyl-cyclohexene-(4), C₁₀H₁₆.</u>
625	175-8 ²⁶⁹ 269 71-3 (16 mm)	0.857 (20/4) ²⁶⁹	1.514 (20) ²⁶⁹	<u>1,1-Dimethyl-4-ethylidene-cyclohexadiene-(2,5), C₁₀H₁₄</u> -- HCl + AcOH g. 1,2,4-dimethyl-ethylbenzene, d ₄ ²⁰ = 0.874, n _D ²⁰ = 1.503. (269). - A Br deriv. cannot be obtained because of the rearrangement. (269). - Htg. at 150-60° for 1 hr. does not change the hy. (269).
627	175.5-6.5 ⁵⁹¹	0.840 (21/4) ⁵⁹¹	1.474 (21) ⁵⁹¹	<u>dl-1-Methyl-4-methoxyethyl-cyclohexene-(1)* "dl-Limonene", "Dipentene", C₁₀H₁₆</u> --Occurs in Finnish and Russian turpentine oils, citronella, lemon-grass, cubeb, coriander oils, etc. - Dimer of isoprene. (594). - Odor of lemon. (594). - Chromyl chloride in CS ₂ , then H ₂ O g. Me-p-tolyl-ketone + o-p-tolyl-propionaldehyde. (601). - Br g. oily products + tetrabromide, M.P. = 125°.- Shak. w. conc. H ₂ SO ₄ g. p-cymene + part. charring. (602). - Htg. w. alc. H ₂ SO ₄ g. part. charring. (603). - C ₅ H ₁₁ ONO + HNO ₃ in AcOH w. cooling g. nitrosate, M.P. = 84° (dec.), wh. w. warm alc. KOH g. inactive carboxime, (604), M.P. = 93° (527). Nitrosochloride, M.P. = 78°, by further htg. 103-40; wh. w. alc. KOH g. dl carboxime; + wh. w. alc. benzylamine g. α-nitrolbenzylamine, M.P. = 110° (527). B.b. not Test 925 - Cf. No. 629.
629	589, 590, 591, 592) 176-7	589, 591 0.841 (20/4)	591, 593 1.474 (21)	<u>1-Methyl-4-methoxyethyl-cyclohexene-(1)* "p-Menthadiene-(1,8(a))", "Limonene", "Carvone", "Hesperidene", "Citral", (the dl-form is "Dipentene", see No. 627), C₁₀H₁₆</u> -- Odor like

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				<p> lemon. (527). - The d-form occurs in gingergrass, lemon-grass, lemon, bergamot, myrrh, etc. oils. (594). - The l-form occurs in many essential oils, among them cascarilla, Russian and American peppermint oils. (594). - D-form shows $[\alpha]_D^{20} = +126^{\circ}$ (589, 592) 8.41 - L-form shows $[\alpha]_D^{20} = -122^{\circ}6'$ (589, 591) No reaction w. Na + EtOH (595) - Addn. of Br in slightly moist solvent g. solid tetrabromide, M.P. = 104-5°; aq. free solvents g. liquid products. (590) - Slow addn. of ice cold ether soln. of Br to soln. of hy. in amyl alc. + ether (1:2) g. tetrabromide. (596) - H₂SO₄ in AcOH g. almost complete polym. (597) - To well cooled mixture of 5 cc hy., 7 cc C₅H₁₁ONO or 11 cc EtONO + 12 cc AcOH, add, in small portions, mixture of 6 cc HCl (d=1.155) + 6 cc AcOH and finally 5 cc EtOH; purify by wash. w. alc., dissolve in 3 pts. by wgt. cold HCCl₃ (β cpd. left behind), filter, extract w. 2-3 pts. by wgt. cold dry ether and let filtrate evap., g. α-nitrosochloride, M.P. = 103-4°, (598) wh. by htg. w. EtONa g. an oil + an xtaln. oxime, M.P. = 72°. (599) - β-nitrosochloride, M.P. = 105-6°, indistinct. (598) - BzO₂H g. limonene-1,2-oxide, B.P. 14-5° = 82-40, D₄²⁰ = 0.929, n_D²⁰ = 1.470, wh. by shak. w. 5 pts. 1% H₂SO₄ for 6 hrs. g. p-menthene-(8(9))-diol-(1,2), M.P. = 72.5-3°, wh. by catalytic reduction g. menthane-diol-(1,2), M.P. = 89-9.5° (600) - B.b.no. = 242, Test 925. </p>

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
631	176-7 ⁶⁶⁰	0.848 (20) ⁶⁶¹	1.478 (18) ⁶⁶²	<p><u>1-Methyl-3-methoxyethyl-cyclohexene-(1), "m-Menthadiene-(1,8(9))", "Silvestrene", "Carvestrene", C₁₀H₁₆</u> -- In turpentine oils etc. (594). - Odor of pine-wood. (659) - D-form shows $[\alpha]_D^{20} = +66.320$ (in CHCl₃ p=14.3) (662) - L-form shows $[\alpha]_D^{20} = -68.20$. (666) - Br. in cold AcOH g. oily products + silvestrene tetrabromide, M.P. = 135-6° (603) - C₅H₁₁ONO + fum. HCl, cold, g. nitrosochloride, xtals. frm. MeOH, M.P. = 106-7° (663) - Ac₂O soln. + 1 drop H₂SO₄ g. deep blue. (603) - Boil w. alc. H₂SO₄ g. much resinification + polym., but no rearrangement to other terpenes. (603) - Decolorizes KMnO₄ immediately. (664) - Oxid. by cold CrO₃. (664) - Not reduced by Na + EtOH. (665)</p>
633	176-7 ²⁴⁰	0.850 (20/20) ²⁴⁰	1.491 (n_D^{20}) ²⁰	<p><u>1-Methyl-3-methoxyethyl-cyclohexene-(6), "Isocarvestrene", C₁₀H₁₆</u> -- Adds only 2 atoms of Br - Ac₂O soln. + conc. H₂SO₄ g. violet, quickly diluting, color. (240)</p>
635	68 (9 mm)			<p><u>1,3-Diethyl-cyclohexadiene-(1,3)</u> C₁₀H₁₆.</p>
637	74 ³⁸² (12 mm)	0.809 (20/4) ³⁸²		<p><u>2,7-Dimethyl-octadiene-(3,5),</u> C₁₀H₁₄.</p>
639	95 ⁶²⁰ (29 mm)			<p>"Alloocimene", C₁₀H₁₆ -- For d + n of various alloocimene fractions cf. Enklaar, J. prakt. chem. [2] 84, 41; Rec. trav. chim. 36, 235. - Na + EtOH g. 2,6-Dimethyl-octadiene-(2,6). (700).</p>

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
641	176-8 ⁷⁰² dec.	0.799 (21/4) ⁷⁰¹	1.486 (18) ⁷⁰⁰	<u>2,6-Dimethyl-octatriene-(2,5,7)</u> , (620) " <u>Ocimene</u> ", $C_{10}H_{16}$ -- Occurs in Java basilicum oil, etc. (527, 594) - Slow htg. g. <u>alloocimene</u> , (700) - Na + EtOH g. <u>2,6-dimethyl-octadiene-(2,6)</u> , (700) whose tetrabromide, M.P. = 88°. (527). - AcOH + H_2SO_4 g. <u>ocimenol</u> whose phenylurethane M.P. = 72° (700) - $KMnO_4$ oxid. g. an ac. whose Pb salt xtalizes in diamonds (difference frm. <u>myrcene</u> wh. g. an ac. whose Pb salt xtalizes in needles), (527)
643	177-8 ³²⁹			<u>1-Methyl-2-methoxyethyl-cyclohexene-(1)</u> , $C_{10}H_{16}$ -- Odor like pine wood and <u>lemon</u> , (329) Adds only 2 atoms of Br directly. (329). - Soln. in Ac_2O + 1 drop conc. H_2SO_4 (329) g. quickly diluting rose coloration. - Na + boil. alc. g. <u>o-menthene-(2(8))</u> , (329)
645	177-8 ³⁸⁴	0.884 (15) ³⁸⁴		<u>8-Methyl-camphene</u> , $C_{11}H_{18}$ -- $[\alpha]_D = +40$, (384).
647	177-9			<u>1,1,2-Trimethyl-5-methoxyethyl-cyclopentene-(2)</u> , $C_{11}H_{18}$ -- Odor of <u>lemon</u> .
649	178-9 ³⁸⁶		1.478 (20) ³⁸⁶	$C_{10}H_{16}$ - By <u>menthanendiol-(2,5)</u> + $KHSO_4$ at 190°.
651	178-80³⁸⁵	0.868 (12)³⁸⁵	1.481³⁸⁵	<u>1-Methylene-4-isopropylidene-cyclohexene</u>?, "<u>Citronene</u>", $C_{10}H_{16}$.
653	178-9* ⁵⁴⁸	0.756 (25/4 vac)* ⁵⁴⁸	1.434 (25)* ⁵⁴⁸	<u>4-Propene-(4²)-yl-octene-(2)</u> ,* $C_{11}H_{20}$ -- B. b. no. = 170*, Test 925.
655	179-80.5 ⁵⁴⁹	0.845 (20) ⁵⁴⁹	1.491 ⁵⁴⁹	<u>1-Methyl-4-isopropyl-cyclohexadiene-(1,3)</u> " <u>Carvenene</u> ", $C_{10}H_{16}$.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
657	179-81 ³⁰³	0.842 (20/4) ³⁰³	1.501 (20) ³⁰³	<p><u>1,2-Diisopropylidene-cyclobutane, C₁₀H₁₆</u> -- Aromatic odor. (303) - Obtained by polym. of dimethyl allene. (303) - O₃ g. succinic anhydride, AcMe, acetone-peroxide, + isopropylidene-2-cyclobutanone whose B.P.₁₁ = 57°, B.P.₇₆₀ = 171°, D₄²⁰ = 0.933, n_D²⁰ = 1.486, quinone odor, semicarbazone M.P. = 241° (dec.) (303) - Xtaln. cpd. w. NaNO₂, M.P. = 141°. (303)</p>
659	179-82 ⁶⁸⁶	0.857 (20/4) ⁶⁸⁶	1.478 (17) ⁶⁸⁶	<p><u>1-Methyl-4-isopropyl-cyclohexadiene-(1,4)</u>, "<u>p-Menthadiene-(1,4)</u>", "<u>γ-Terpinene</u>", C₁₀H₁₆ -- Not yet obtained pure. (527, 594) - An integral constituent of ordinary terpinene. (687) - (Constants given are for an impure α-terpinene.) - KMnO₄ g. p-menthanetretol-(1,2,4,5) M.P. = 237-8°; difference frm. α-terpinene. (527, 677)</p>
661	180-1 ³⁸⁷			<p><u>1-Methyl-3-methoxyethyl-cyclohexene-(3)</u>, C₁₀H₁₆ -- D-form shows [α]_D = 17.5°. (387) - L-form shows [α]_D = -12.9°. (387)</p>
663	180-2 ²⁰⁰	0.819 (26/4) ²⁰⁰	1.456 (26) ²⁰⁰	<p>C₁₁H₂₀ -- By 1,5-dimethyl-2-methoxyethyl-cyclohexanol-(1) + oxalic ac. - [α]_D = + 88.53°. (200)</p>
665	181-2 ³⁹²	0.827 (16) ³⁹²		<p><u>1-Methylene-2-isopropyl-5-methyl-cyclohexane, C₁₁H₂₀</u> -- [α]_D¹⁸ = 87.25° (in alc.) (392)</p>
667	181-3 ²⁴⁸	0.825 (11) ²⁴⁸	1.463 (12) ²⁴⁸	<p><u>2,2,3,3-Tetramethyl-methylene-cyclohexane, C₁₁H₂₀</u>.</p>
669	67-7.5 ⁵¹⁰ (11 mm)	0.869 (20) ⁵¹⁰	1.507 (20) ⁵¹⁰	<p><u>1,1,3,4-Tetramethyl-cycloheptatriene-(2,4,6)</u>, or an isomer of the same carbon skeleton, C₁₁H₁₆.</p>

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No.	Boiling Point (°C)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
671	³⁸⁸ 181.5-2.5	0.791 (0/0) ³⁸⁹		<u>Decene-(1)</u> , $C_{10}H_{18}$ --M.P. = -36°. (388) - Hg salt (Test 926) M.P. 82-30* (390).
673	²¹⁶ 181.5-3.5	0.793 (14/4) ²¹⁶	1.457 ²¹⁶ (14)	<u>2,4-Dimethyl-nonadiene-(3,5)</u> , $C_{11}H_{20}$.
675	³²⁵ 181.5-3.5	0.801 (10) ³²⁵	1.458 ³²⁵ (10)	<u>2,6-Dimethyl-nonadiene-(2,6)</u> , $C_{11}H_{20}$ -- Odor like mint. (325)
677	³⁹³ 182-3	0.863 (20) ³⁹³	1.497 ³⁹³ (20)	$C_{10}H_{14}$ from 1,4,8-tribrom-p-menthane + NaOEt. (393)
679	³²⁵ 182-3	0.810 (11) ³²⁵	1.461 ³²⁵ (11)	<u>2,6,7-Trimethyl-octadiene-(2,6)</u> , $C_{11}H_{20}$.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
681	182-3 ³⁹¹			<p>1-Methyl-3-Methoethenyl-cyclohexene-(2), C₁₀H₁₆. - D-form shows (α)_D = +64°. (391). - Pungent lemon odor. - Adds 2 atoms of Br (340). - Soln. in Ac₂O w. 1 drop H₂SO₄ g. intense methylene-blue color. (391, 340).</p>
683	182.5-4.5 ²⁴⁸	0.816 (10) ²⁴⁸	1.462(10) ²⁴⁸	<p>3,3-Dimethyl-2-ethyl-methylene-cyclohexane, C₁₁H₂₀.</p>
685	183-4 ³⁹⁷			<p>2-Methyl-decadiene-(1,3), C₁₁H₂₀.</p>
687	183-5 ⁶⁸⁸	0.854 (20) ⁶⁸⁹	1.484 ⁶⁸⁹	<p>1-Methyl-4-isopropylidene-cyclohexene-(1), "p-Menthadiene-(1,4(8))", "Terpinolene", C₁₀H₁₆. - Occurs in oils of elemi, orange, + probably coriander. (527, 594). - Changes by boil. at ordinary press. (688). - Htg. w. a. c. g. terpinene. (690). - 7 g. hy. shak. w. 33 g. KMnO₄, 14 g. KOH, 400 g. ice, + 400 cc. aq.; unattacked hy. distd. off, MnO₂ filtered off + soln. evapd. to dryness on H₂O bath under CO₂, residue washed w. alc., taken up in H₂O + extracted w. AcOEt several times; xtals. of p-menthantetrol-(1,2,4,8)+1H₂O, wh. begins to sinter at 90° + m. above 100° w. gas evolution; m.p. of cpd. slowly dried at 145°, 149-50°. (527, 676).</p>
689	184-5 ²⁹¹	0.858 (19) ²⁹¹	1.492 (19) ²⁹¹	<p>1-Methyl-4-methoethenyl-cyclohexene-(3), C₁₀H₁₆. - D-form shows (α)_D = +100°. - 1% KMnO₄ at 0° g. i-β-methyl-adipic ac. (291).</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
691	184-5 ³⁹⁵	0.857 (25-25) ³⁹⁵	1.484 ³⁶⁰	1-Methyl-4-methoethenyl-cyclohexene-(3), "p-Mentha-diene-(3,8(9))", C ₁₀ H ₁₆ . - D-form shows (∞)D ²⁰ : +98.2° (in C ₆ H ₆). (395). - Odor like d-limonene or lemon. (395).
693	184-5 ³⁹⁸	0.809 (22) ³⁹⁸	1.450 (22) ³⁹⁸	1-Methyl-3-(3 ² -methopropyl)-cyclohexene-(4 or 5), C ₁₁ H ₂₀ . -- 1 vol. H ₂ SO ₄ + 2 vol. EtOH g. first a yellow color, then yellow-red, + finally a violet-red color. (398).
695	184-6 ³⁹⁴	0.845 (10-4) ³⁹⁴	1.473 (10) ³⁹⁴	1,3-Dimethyl-1-(propene-(1 ²)-yl)-cyclohexene-(3)(?), C ₁₁ H ₁₈ .
697	184-5 ⁴⁹⁹ 62.5 (13 mm.)	0.879 (20-4) ⁴⁹⁸	1.538 (20) ⁴⁹⁸	1-sec.-Butylidene-cyclopentadiene-(2,4), "ω-Methyl-ω-ethyl-fulvene", C ₉ H ₁₂ . - Orange colored liquid. (499). Cf. Div. b, Sect. 1, No. 140.
699	70 ³⁵³ (17 mm.)	0.845 (20) ⁴⁰	1.462 (20) ⁴⁰	1-Cyclohexyl-butane-(3), C ₁₀ H ₁₆ . -- G. Test 906. (40). - Cyclohexylpentanoic ac., M.P. = 37.5-9°. (40).
701	184.5-8.5 ⁵⁸⁸	0.854 (20-4) ⁵⁸⁸	1.471 (20) ⁵⁸⁸	Homoverbanene, C ₁₁ H ₁₆ .
703	185-7 ³⁹⁹	0.824 (20-0) ³⁹⁹		C ₁₁ H ₂₀ from 1,3-dimethyl-4-isopropyl-3-chloro-cyclohexane.
705	185-9 ²²⁷	0.778 (25-4) ²²⁷	1.462 (25) ²²⁷	2,6-Dimethyl-nonadiene-(4,6), C ₁₁ H ₂₀ .
707	186-7 ³⁴⁰			1-Methyl-3-methoethenyl-cyclohexene-(3), C ₁₀ H ₁₆ . -- Adds 2 atoms of Br. (340). Ac ₂ O + conc. H ₂ SO ₄ g. a red-violet color. - Odor like lemon. (340).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
709	187-8 ⁴⁰⁰			Undecadiene-(1,10), C ₁₁ H ₂₀ . KMnO ₄ oxid. g. azelaic ac. in alk. soln. (356,400). - NO ₂ in pet. ether g. green addn. product. (400).
711	187.5-8.5 ²¹⁶	0.786 (14-4) ²¹⁶	1.460 (14) ²¹⁶	5-Methyl-decadiene-(4,6), C ₁₁ H ₂₀ .
713	188-9 ⁷⁰⁰	0.899 (22-4) ⁷⁰¹	1.545 (21) ⁷⁰⁰	2,6-Dimethyl-octatriene-(2,4,6), C ₁₀ H ₁₆ .
715	188-90 ⁴⁰²	0.831 (16) ⁴⁰²	1.467 (16) ⁴⁰²	1,1,2-Trimethyl-3-(3 ^a -methopropene-3 ^a -yl)-cyclopentene-(2), C ₁₂ H ₂₀ .
717	189-90 ^{411,462}	0.918 (18) ^{411,462}	1.495 (n _D ¹⁸) ^{411,462}	1(or 3)-Cyclopentyl-cyclopentene-(1), C ₁₀ H ₁₆ . -- Br + AlBr ₃ g. C ₁₀ H ₄ Br ₆ , m.p. = 308-9°. (411,462). - Br in CS ₂ g. dibromide, m.p. = C ₅ H ₂ C ₅ H ₇ Br ₂ , m.p. = 160°, wh. w. K ₂ CO ₃ g. dihydroxycyclopentyl-cyclopentane, m.p. = 87-8° (411), wh. latter is also found by hy. + I + HgO. (462).
719	190-1 ³⁷⁰	0.844 (15-4) ³⁷⁰		1-Methyl-3-(3 ^a -methopropene-3 ^a -yl)-cyclohexene-(3), C ₁₁ H ₁₈ . -- D-form shows (L): + 63.9°. (370). - Adds theor. amt. of Br. (370). - Alk. KMnO ₄ g. - methyl-adipic ac. (370).
721	190-1 ⁴⁴⁶	0.825 (20-4) ⁴⁴⁶	1.483 (20) ⁴⁴⁶	1,2-Dimethyl-3,4-diisopropylidene-cyclobutane, C ₁₂ H ₂₀ . -- Oxid. g. 1,2-dimethyl-3-isopropylidene-cyclobutane, m.p. = 83-6°, quinone odor, whose semicarbazone, m.p. = 200-1°. (446).
723	190-2 ⁴⁰³	0.914 (17) ⁴⁰³	1.499 (17) ⁴⁰³	Bicyclo-(0,4,4)-decene-(1 or 2 or both?), "L-naphthanene", C ₁₀ H ₁₆ . -- Br in HCCl ₃ g. a dibromonaphthalenedecahydride of m.p. = 143°. (403).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
725	190-2 ⁴⁰³	0.901 (13) ⁴⁰³	1.491 ⁴⁰³	Bicyclo-(0,4,4)-decene-(3), " <u>β-Naphthanene</u> ", C ₁₀ H ₁₆ . At 0° in CHCl ₃ w. Br g. two 2,3-dibromnaphthalenedecahydrides, m.p. = 41° + 85° (403). - Combines directly w. org. acs. to esters of decanhydro-β-naphthol of m.p. = 75° (403). - This hy. is probably a mixture of the two forms, cis: d ₄ ²⁰ : 0.917-0.913, n _D ²⁰ : 1.499-1.494; trans, d ₄ ¹⁹ : 0.894, n _D ²⁰ : 1.484, m.p. = -24° (404).
727	192-3 ³⁷⁰	0.853 (15-4) ³⁷⁰	1.480 ³⁷⁰	1-Methyl-3-(3 ² -methopropene-(3 ¹)-yl)-cyclohexene-(2), C ₁₁ H ₁₈ . -- D-form shows (α _p) = 54.8° (370).
729	192-5 ²⁸³			1-Methyl-3-isobutyl-cyclohexene, C ₁₁ H ₂₀ .
731	193-4 ⁴⁰⁵	0.846 (12-4) ⁴⁰⁵	1.463 (12) ⁴⁰⁵	C ₁₁ H ₂₀ from 1-isoamyl-cyclohexanol-(1).
733	194-5 ⁴¹²			Bicyclo-(0,4,4)-decadiene-(2,4), " <u>Hexahydronaphthalene</u> ", C ₁₀ H ₁₄ .
735	79 ³⁵³ (17 mm.)	0.855 (20) ³⁵³	1.47 (20) ³⁵³	1-Cyclohexyl-butine-(2), C ₁₀ H ₁₆ .
737	195-6* slight dec.	0.832 (25-4 vac.) [*]	1.460(25) [*]	4-Cyclohexyl-pentene-(2)*, C ₁₁ H ₂₀ . -- B.b. no. = 112, Test 925.
739	195-6 ⁴¹³	0.825 (23-4) ⁴¹³	1.465 (23) ⁴¹³	1,1-Dimethyl-3-(3 ² -methopropene-(3 ¹)-yl)-cyclohexene-(4), C ₁₂ H ₂₀ . -- Adds 4 atoms of Br. (413).
741	195.5- ⁴⁰⁷ (dec.) 7.5	0.814 (11-4) ⁴⁰⁷	1.487 (11) ⁴⁰⁷	2,6-Dimethyl-nonatriene-(2,6,8), C ₁₁ H ₁₈ . -- 80% H ₂ SO ₄ g. cyclic isomeric hy. (407).
743	92-3 ²⁷⁶ (63 mm+)	0.800 (13-4) ²⁷⁶	1.459 (15) ²⁷⁶	5-Ethyl-nonatriene-(1,4,8) or 5-Ethylidene-nonadiene-(1,8), C ₁₁ H ₁₈ .

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
745	196.5- ⁴¹⁹ 8.0	0.908 (20) ⁴¹⁹	1.494 ⁴¹⁹	1-Cyclopentyl-pentene-(1), <u>C₁₀H₁₈</u> . -- Nitrosochloride, m.p. = 113-4°. (419).
747	197-9 ⁴⁰⁸	0.910 (20) ⁴⁰⁸		Octahydronaphthalene (?), <u>C₁₀H₁₆</u> . -- Hydrobromide, p.p. ₂₀ = 95-100°. (408). - Dibromide, xtals. fm. C ₆ H ₆ , m.p. = 169°. (408).
749	198-200 ⁴²³	0.867 (25-4) ⁴²⁰		Undecine-(1), "Rutylidene", <u>C₁₁H₂₀</u> . -- M.p. = -33°. (420). - G: Test 906. (421, 422).
751	199-201 ⁴²⁵			Undecine-(2), C ₁₁ H ₂₀ . -- Does not g. test 906. (424). - 94% H ₂ SO ₄ g. methyl-nonyl- ketone + ethyl-octyl-ketone. (424). - KMnO ₄ g. pelargonic ac. + AcOH. Br g. dibromide† (424).
753	106-9 ²⁵⁷ (46 mm.)	0.847 (22-4) ²⁵⁷	1.495 (23) ²⁵⁷	1,3-Dimethyl-5-isopropyl- idene-cyclohexene-(3), <u>C₁₁H₁₈</u> .
755	abt. 200 ³⁹²	0.830 (16) ³⁹²		1-Ethylidene-2-isopropyl-5- methyl-cyclohexane, C ₁₂ H ₂₂ . -- D-form shows (L) _D ⁺ 34.79°. (392).
757	201-2 ⁴³²	0.855 (18) ⁴³²	1.480 (18) ⁴³²	1-Ethyl-4-methoxyethyl- cyclohexene-(1), C ₁₁ H ₁₈ .
759	201-3 ²⁴⁸	0.813 (10) ²⁴⁸	1.462 (11) ²⁴⁸	3,3-Dimethyl-2-n-buty- lidene-cyclohexane, C ₁₂ H ₂₂ .
761	201.5- ³²⁵ 2.5	0.803 (10) ³²⁵	1.459 (11) ³²⁵	2,6-Dimethyl-decadiene- (2,6), C ₁₂ H ₂₂ .
763	84-4.5 ³⁵³ (16 mm.)	0.846 (20) ³⁵³	1.463 (20) ³⁵³	1-Cyclohexyl-pentene-(4), <u>C₁₁H₁₈</u> . -- Cyclohexyl- hexinoic ac., m.p. = abt. 30°. (213). - G: Test 906. (40). - Odor like anise. (40).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
765	203-5 ⁴³³	0.917 (20) ⁴³³		8-Vinyl-camphenylidene, C ₁₂ H ₁₈ . -- D-form shows (L) _D ²⁰ : 76.39°. (433). - K ₂ Cr ₂ O ₇ g. camphenylidene-acetic ac. + some corresp. aldehyde whose semicarbazone m.p. = 235°. (433).
767	70-1 ⁴²⁷ (16 mm.)	0.905 (20-4) ⁴²⁷	1.464 (20) ⁴²⁷	1,3-Dimethyl-4-isopropylidene-cyclohexane-(2) or 1-Methyl-3-methylene-4-isopropylidene-cyclohexane, C ₁₁ H ₁₈ . -- D-form shows (L) _D ²⁰ = 114.30-9.92°. (427). - O ₃ g. mixt. of diozonide w. little monoozonide + some oxozide (perhaps) wh. w. H ₂ O g. Me ₂ CO, 4-methyl-5-acetylpentanoic ac.-(1), b.p. ₁₄ = 171°, whose semicarbazone, needles m.p. = 150° + an aldehyde b.p. ₂₀ = 130-40° whose semicarbazone, m.p. = 266°. (427).
769	74.5-8.5 ⁴²⁷ (19 mm.)			1-Diethylmethylene-cyclopentadiene-(2,4), "ω,ω-Diethyl-fulvene", C ₁₀ H ₁₄ . Orange-yellow oil. (427).
771	61-2 ⁴⁰¹ (9 mm.)	0.773 (20-4) ⁴⁰¹		2,6-Dimethyl-nonadiene-(1,7 + 2,7), C ₁₁ H ₂₀ . -- L-form shows (L) _D ²⁰ = -10.37°. (401). - D ₂ O ₃ splitting g. 2-methyl-5-acetylpentanoic ac.-(1) + L-methylglutaric ac. (401).
773	81-2 ²⁷⁶ (20 mm.)	0.816 (19-4) ²⁷⁶	1.474 (18) ²⁷⁶	2,6-Dimethyl-nonatriene-(1 or 2, 5, 8), or an isomer of the same carbon skeleton, C ₁₁ H ₁₈ . -- Oxid. + polym. easily in the air. (276).
775	152 ⁴²⁹ (67 mm.)			2-Methyl-dodecadiene-(1,11) C ₁₃ H ₂₄ . -- Faint odor of lemon: (429).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
777	77 ⁵⁰⁴ (12 mm.)	0.877 (20-4) ⁵⁰⁴	1.515 (20) ⁵⁰⁴	<u>1,1,3,6-Tetramethyl-4-methylene-cyclohexadiene-(2,5), C₁₁H₁₆.</u> -- Cf. Div. B, Sect. 1, No.224.
779	205-6 ⁴³⁴	0.860 (20) ⁴³⁴	1.481 ⁴³⁴	<u>1,3-Dimethyl-3-methoxyphenyl-cyclohexene-(6), C₁₁H₁₈.</u> -- By polym. of di-isopropenyl. (434).
781	95 ⁴⁰⁹ (20 mm.)	0.834 (12-4) ⁴⁰⁹	1.467(12) ⁴⁰⁹	<u>C₁₁H₂₀, from isobutyl-cyclohexyl-carbinol.</u> (409)
783	209-10 ²⁸⁵	0.821 (16-4) ²⁸⁵	1.485 (16) ²⁸⁵	<u>C₁₂H₂₂ from 1-Methyl-4-isoamyl-cyclohexene-(4).</u> (285).
785	209-11 ²⁸³	0.819 (20) ²⁸³	1.459 ²⁸³	<u>1-Methyl-3-isoamyl-cyclohexene-(2 or 3), C₁₂H₂₂.</u> -- Nitrosochloride, m.p. = 136°. (283).
787	213, 253 93-3.5 ⁴⁰ (17 mm.)	0.857 (20) ⁴⁰	1.471 (20) ⁴⁰	<u>1-Cyclohexyl-pentine-(3), C₁₁H₁₈.</u> -- NaNH ₂ in ether g. 1-cyclohexyl-pentine-(4). (213).
789	213-4 ²⁴⁸	0.811 (11) ²⁴⁸	1.461 (11) ²⁴⁸	<u>3,3-Dimethyl-2-isobutylidene-cyclohexane, C₁₂H₂₂.</u>
791	214-5 ³²⁵	0.804 (11) ³²⁵	1.460 (11) ³²⁵	<u>2,6,9-Trimethyl-decadiene-(2,6), C₁₃H₂₄.</u> -- Fruity odor. (325).
793	505, 506 72-4 (9 mm.)	0.896 (20-4) ^{505, 506}	1.533(20) ^{505, 506}	<u>C₁₂H₁₆, L-Tetrameride of propadiene-(1,2).</u>
795	504 83-5 (13 mm.)	0.858 (20-4) ⁵⁰⁴	1.504 (20) ⁵⁰⁴	<u>1,1,3-Trimethyl-4-ethylidene-cyclohexadiene-(2,5), C₁₁H₁₆.</u> --
799	415 75-6 (10 mm.)	0.875 (20) ⁴¹⁵	1.502 (20) ⁴¹⁵	<u>1,2-Dimethyl-4-methoxyphenyl-cyclohexadiene-(χ, χ), or an isomer, C₁₁H₁₆.</u> -- Odor like limonene. + (415). - D-form shows (α) _D ²⁰ = +103.49°. (414). - Rotation decreases by repeated distn. under reduced press. (416). - Very unstable. (416). - Colors in absence of air, becoming resinified. (416).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
801	214-7 ⁴³⁵	0.855 (20-4) ⁴³⁵		1-Methyl-4-isopropyl-3-(propene ³ -yl)-cyclohexene-(3), C ₁₃ H ₂₂ . -- (α) _D = 50.86° has been prepared. (435). - Adds 4 atoms of Br.(?). (435). Undecadiene, C ₁₁ H ₁₆ . -- (M) _D = 48.89°. (410). - G. Test 906. (410).
803	22.5-3 ⁴¹⁰ (12 mm.)	0.818 (21-4) ⁴¹⁰		
805	89-90 ⁵⁰⁴ (15 mm.)	0.881 (20-4) ⁵⁰⁴	1.517 (20) ⁵⁰⁴	1,1,2;6-Tetramethyl-4-methylene-cyclohexadiene-(2,5), C ₁₁ H ₁₆ . -- Rearranges to pentamethyl benzene. (504).
807	81-2 ⁴²⁸ (9 mm.)	0.781 (20-4) ⁴²⁸		2,6-Dimethyl-decadiene-(1,8 + 2,8), C ₁₂ H ₂₂ . -- L-form shows (α) _D = -6.64°. (428). - Not entirely miscible in all proportions w. alc.-O ₃ splitting g. 3-methyl-6-acetyl-hexanoic ac.-(1), 2-methyl-adipic ac. + acetone-superoxide. (428).
809	221-2 ⁴⁰² * dec.	0.780 (25-4) ⁴⁰² * vac.)	1.447 (25) ⁴⁰² *	4,5-Di-n-propyl-octadiene-(2,6), C ₁₄ H ₂₆ . *-- Turns golden-yellow on boil.* - B.b. no. = 146*, Test 925.
811	222-4 ⁴⁰²	0.869 (19) ⁴⁰²	1.469 (19) ⁴⁰²	1,1,2-Trimethyl-3-(3 ² -ethobutene-(3 ¹)-yl)-cyclopentene-(2), C ₁₄ H ₂₄ .
813	223-8 ⁴³⁶	0.911 (0) ⁴³⁶		C ₁₅ H ₂₈ , "Benylene", from triamylene-dibromide + alc. KOH. (436).
815	224.5-5.5 ⁴³⁸	0.815 (20-0) ⁴³⁸	1.473 ⁴³⁸	2,6-Dimethyl-undecatriene-(1 or 2, 5 or 6,8), "Pseudoionane", C ₁₃ H ₂₂ . -- Boil. w. AcOH + H ₂ SO ₄ g. L-ionane. (438).
817	122-3 ⁴⁴³ (29 mm.)	0.870 (17-4) ⁴⁴³	1.488 (17) ⁴⁴³	1,3,3,5,5-Pentamethyl-4-isopropyl-cyclohexene-(1), C ₁₄ H ₂₆ . -- By polym. of Me ₂ C=CH+C(:CH ₂)Me. (443).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
819	⁵¹² 103-6 (17 mm.)	0.862 (22-4) ⁵¹²	1.522 (22) ⁵¹²	<u>1,3-Dimethyl-5-(5²-methyl-eno-propylidene)-cyclohexene-(3), C₁₂H₁₈.</u>
821	⁴⁰⁶ 103-6 (17 mm.)	0.862 (22-4) ⁴⁰⁶	1.522 (22) ⁴⁰⁶	<u>1,3-Dimethyl-5-(propene-(5²)-ylidene)-cyclohexene-(6), C₁₁H₁₆.</u>
823	⁴⁴⁰ 226-8	0.844 (21) ⁴⁴⁰		<u>4-Cyclohexyl-heptene-(3), C₁₃H₂₄.</u> -- Nitrosochloride m.p. = 110° (dec.). (440).
825	³⁵⁰ 228-30	0.822 (22) ³⁵⁰	1.456 (22) ³⁵⁰	<u>1-Methyl-3-n-hexyl-cyclohexene-(4 or 5), C₁₃H₂₄.</u>
827	⁴³⁰ 95-7 (15 mm.)			<u>Dodecine-(1), C₁₂H₂₂.</u> -- G. Test 906. (430).
829	⁵⁰⁴ 100-3 (18 mm.)	0.880 (20-4) ⁵⁰⁴	1.513 (20) ⁵⁰⁴	<u>1,1,3,6-Tetramethyl-4-ethylidene-cyclopentadiene-(2,5), C₁₁H₁₆.</u>
831	⁴⁰ 101 (17 mm.)	0.846 (20) ⁴⁰	1.463 (20) ⁴⁰	<u>1-Cyclohexyl-hexene-(5), C₁₂H₂₀.</u> -- G. Test 906. (40). -- Cyclohexyl-heptinoic ac., m.p. = 40±1°. (40).
833	230-1* slight dec.	0.836 (25-4)* vac.)	1.463 (25)*	<u>4-Cyclohexyl-heptene-(2), C₁₃H₂₄.</u> -- Turns light yellow on boil.* -- B:B. No. = 82,* Test 925.
835	234-5			<u>Decahydroacenaphthene, C₁₂H₂₀.</u>
837	^{505,506} 101 (10 mm.)	0.935 (20-4) ^{505,506}	1.526 (20) ^{505,506}	<u>C₁₂H₁₆, β-Tetrameride of propadiene-(1,2).</u> -- Colorless; odor of petroleum. (505).
839	⁴⁴² 236-7.5	0.901 (20) ⁴⁵⁰	1.491 (20) ⁴⁵⁰	<u>Cyclohexyl-cyclohexene-(1), C₁₂H₂₀.</u> -- Nitrosochloride m.p. = 140°. (450). -- CrO ₃ in glac. AcOH g. ac., C ₆ H ₁₁ CO(CH ₂) ₄ CO ₂ H, xtals. fm. ligroin + Et ₂ O, m.p. = 58° whose semicarbazone, needles, m.p. = 172-3°, whose oxime, needles, m.p. = 105°. (450).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
841	124 ⁴⁰⁵ (20 mm.)	0.913 (14-4) ⁴⁰⁵	1.495 (14) ⁴⁰⁵	<u>C₁₂H₂₀</u> from 1-cyclohexyl-cyclohexanol-(†) + ZnCl ₂ at 160°. (405).
843	238-40 ⁵¹¹	0.905 (15) ⁵¹¹		8-Butenyl-camphenylidene, <u>C₁₄H₂₂</u> . -- Cf. Div. B, Sect. 1, No. 496.
845	100-1 ⁴¹⁶ (13.5 mm.)	0.886 (18-4) ⁴¹⁶	1.504 (18) ⁴¹⁶	1-Methyl-2-ethyl-3-methoxy-ethenyl-cyclohexadiene-(2,6)(?), <u>C₁₂H₁₈</u> . -- D-Form shows (α) _D ²⁰ : +86.19° (416). - Htg. w. 2% HCl in AcOH g. 1-methyl-2-ethyl-4-isopropyl-benzene. (416)
847	90-1 ⁴²⁸ (8 mm.)	0.787 (20-4) ⁴²⁸	1.449 (20) ⁴²⁸	2,6-Dimethyl-undecadiene-(1,8 + 2,8), <u>C₁₃H₂₄</u> .
849	239-40 ²⁸³	0.914 (18) ²⁸³	1.492 ²⁸³	1-Methyl-3-cyclohexyl-cyclohexene-(3), <u>C₁₃H₂₂</u> . -- Nitrosocchloride, softens at 138°, m.p. = 142-6°. (283).
851	239.5-40.5 ⁴⁵¹	0.793 (19-4) ⁴⁵¹	1.454 (19) ⁴⁵¹	2,11-Dimethyl-dodecadiene-(2,10), <u>C₁₄H₂₆</u> . -- Easily sol. in ether, difficultly in alc. (451). - Gradually oxid. by the air g. an aldehyde whose semicarbazone, m.p. = 203°. (451). KMnO ₄ oxid. g. Me ₂ CO, suberic ac., formic ac. + AcOH. (451).
853	abt.240 ⁴¹⁷	0.922 (20-4) ⁴¹⁷	1.509 (20) ⁴¹⁷	1,4-Dimethyl-hexahydro-naphthalene, <u>C₁₂H₁₈</u> . -- Boiling in 2% soln. of HCl in AcOH g. 1,2-dimethyl-4-isopropyl-benzene. (416). K ₃ Fe(CN) ₆ + 20% HCl g. white needles, easily sol. in alc. + H ₂ O. (414).
855	105 ⁴³¹ (15 mm.)	0.792 (15-4) ⁴³¹		Dodecine-(2), <u>C₁₂H₂₂</u> . -- Freezes in cooling mixt. + melts at (-9°). (431). - Na at 220° g. dodecine-(1). (431). - Does not g. Test 906. (431).

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
857	109-10 ³⁵³ (17 mm.)	0.853 (20) ⁴⁰	1.471 (20) ⁴⁰	<u>1-Cyclohexyl-hexine-(4),</u> <u>C₁₂H₂₀.</u>
859	240-50 ⁵⁴⁴	0.902 (16) ⁵⁴⁴	1.495 ⁵⁴⁴	Caparrapine, C ₁₅ H ₂₄ . -- By P ₂ O ₅ + caparrape oil. (⁵⁴⁴). (∞) _D : -2.21°. (⁵⁴⁴).
861	108-10 ⁵⁰⁶ (17 mm.)			<u>C₁₅H₂₄, Trimeride of</u> <u>Pentadiene-(2,3).</u>
863	100-1 ⁵⁰⁶ (8 mm.)	0.858 (20-4) ⁵⁰⁶	1.487 (20) ⁵⁰⁶	<u>C₁₅H₂₄, Trimeride of 2-</u> <u>Methyl-butadiene-(2,3),</u>
865	94-5 ⁴¹⁸ (8 mm.)	0.801 (20-4) ⁴¹⁸	1.473 (20) ⁴¹⁸	<u>2,6-Dimethyl-undecatriene-</u> <u>(1 or 2,8,10), or mixture</u> <u>of both, C₁₃H₂₂. --</u> Quickly resinifies in the air. (⁴¹⁸).
867	107-8 ⁵⁰⁹ (13 mm.)	0.880 (15-4) ⁵⁰⁹	1.503 (15) ⁵⁰⁹	<u>1-Methyl-2-n-propyl-4-</u> <u>methoxyethyl-cyclohexa-</u> <u>diene-(2,6), C₁₃H₂₀. --</u> Cf. Div. B, Sect. 1, No.
869	106-7 ⁵¹⁴ (10 mm.)	0.934 (20) ⁵¹⁴	1.542 ⁵¹⁴	<u>1,1,6-Trimethyl-1,2,3,9</u> <u>(or 1,2,9,10)-tetrahydro-</u> <u>naphthalene, "Ionene",</u> <u>C₁₃H₁₈. -- Cf. Div. B,</u> <u>Sect. 1, No.</u>
871	115.5 ⁵²¹ (12 mm.)	0.826 (21) ⁵²¹	1.454 (n) ⁵²¹	<u>Undecadiene-(1,10), C₁₁H₁₆</u> -- M.p. = (-3°)-(-2°). (⁵²¹). - G. Test 906 (yellow ppt. w. ammon. Cu ₂ Cl ₂). (⁵²¹).
873	247-8 ⁴⁴⁴			<u>1,4-Dimethyl-6-ethyl-</u> <u>naphthalene-octanhydride,</u> <u>C₁₄H₂₄. -- Odor of pepper-</u> <u>mint. (444).</u>
875	247.5- ⁴⁹⁴ 8.5 ⁴⁵² 102-3 ⁴⁵² (6 mm.)	0.896 (15) ⁴⁹⁴	1.493 (20) ⁴⁹⁴	<u>C₁₅H₂₄, -- Lower boil.</u> <u>sesquiterpene fm. C₁₅H₂₆O</u> <u>of eucalyptus oil by 90%</u> <u>formic ac. (⁴⁵²). - (∞)_D:</u> <u>-55°48' (l=10 cm.) (⁴⁹⁴).</u>

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
877	250-1 ⁴⁴⁶	0.860 (20-4) ⁴⁴⁶	1.477 (20) ⁴⁴⁶	1,2,4-Trimethyl-4-methoxyethyl-cyclonexene-(1), C ₁₂ H ₂₀ . -- KMnO ₄ g. 4-methyl-4-isopropenyl-2,7-octanedione, b.p. = 132-3°, semicarbazone, xtals., m.p. = 228°. (446).
879	250-3	0.949 (20)	1.529 (20)	1-(Cyclonexene-(1 ¹)-yl)-cyclonexene-(1), C ₁₂ H ₁₈ . -- Cf. Div. B, Sect. I, No. 562.
881	250-60 ⁵²²	0.916 (15) ⁵²²	1.507 (15) ⁵²²	"Amorphene", C ₁₅ H ₂₄ . -- In ethereal oil of <i>amorphna fruticosa</i> L. (522).
883	113.5-4.0 ⁵⁸⁵ (7 mm.)	0.935 (20) ⁵⁸⁵	1.505 ⁵⁸⁵	β -Gurjunene, C ₁₅ H ₂₄ . -- (L): +19°. (585). - Pt in AcOH ^d g. dihydro cpd., b.p. = 115-7°, d ₂₀ = 0.924, n _D = 1.495, (L) _D : -37°. (585). - Only difficultly attacked by KMnO ₄ in aq.-Me ₂ CO. (585). - O ₃ g. no definite dec. product (difference fm. cedrene). (585).
885	124 ⁵⁸³ (11 mm.)	0.895 (25-4) ⁵⁸³	1.495 (25) ⁵⁸³	Guaine, "Guajene" (not identical w. Div. A, Sect. 2, No. 422), C ₁₅ H ₂₄ . -- (L) _D ²⁵ : -66.11°. (583). - Dihydroguaine, b.p. ₁₅ = 124-5°, d ₄ : 0.956, n _D ²⁰ : 1.489. (583). - Htd. w. S g. guaisulene, for wn. cr. No. 925.
887	118-24 ⁴⁹⁰ (11 mm.)	0.918 (20-4) ⁴⁹⁰	1.520 (20) ⁴⁹⁰	Hexahydrochamazulene", -- By chamazulene + Na - C ₅ H ₁₁ OH. - Htd. w. S g. chamazulene. (490). Cf. 889.

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
889	119 ⁴⁹⁰ (11 mm.)			"Octahydrochamazulene", -- By chamazulene + Pt black in AcOEt. (490). - Htd. w. S g. chamazulene, b.p. ₁₂ = 161°, d ₄ ²⁰ : 0.988, whose picrate, m.p. = 115° (fm. EtOH or MeOH) + whose styphnate, m.p. = 95-6° (fm. EtOH or MeOH). (490).
891	123-30 ⁶⁹² (16 mm.)	0.936 (20) ⁶⁹²	1.511 ⁶⁹²	Tricyclic Vetivene, C ₁₅ H ₂₄ . -- Ctg. 1 double bond. (692). - In vetiver oil. (692). - (L) _D : 2°16'. (692).
893	125-7 ⁴⁶⁴ (18 mm.)	0.813 (19) ⁴⁶⁴	1.489 ⁴⁶⁴	4,7-Di-n-propyl-decadiene-(3,7)-ene-(5), C ₁₆ H ₂₆ . -- Yellowish liquid. (464). - H + Pt black g. 4,7-di-n-propyl-decane. (464).
895	251-2 ⁴⁵³	0.952 (15) ⁴⁵³	1.519 (n ₁₅) ⁴⁵³	C ₁₅ H ₂₄ , sesquiterpene fm. alcohol of copaiwa balsam oil. (453). - (L) _D : -61.7°. (453).
897	252-3 dec.	0.795 (25-4 vac.)	1.453 (25)	4,5-Dipropene(4 ¹ ,5 ¹)-yl-decane, C ₁₆ H ₃₀ . -- Turns deep golden yellow on boil. B.b. no. = 125, Test 925.
899	252-3 ⁶⁵⁴	0.913 (15) ⁶⁵⁴	1.492 (15) ⁶⁵⁴	1,2-Dimethyl-2-(2 ⁴ -methopentene(2 ³)-yl)-3,6-endo-methylene-bicyclo-(0,1,3)-heptane, "L-Santalene", C ₁₅ H ₂₄ . -- In Austrian sandalwood oil. (653). - (L) _D : -3°34' (l=10 cm.) (654). - Nitrosochloride, by NaNO ₂ + HCl, m.p. = 112-7°, (654); by NOCl in pet. ether m.p. = 122°. (653). - Nitropiperidide, m.p. = 108-9°. (527). - Htd. w. S g. eudalene. (527). - Cf. under selinene, No. 999.

DIVISION B SECTION 3

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
901	³⁸⁴ 254-5	0.900 (15) ³⁸⁴		<u>C₁₅H₂₄, "Sesquicamphene", "Amenyl-camphenylidene".</u> - <u>K₂Cr₂O₇ g. camphenylidene ac. + corresp. aldehyde.</u> (384).
903	⁵⁵³ 254-6	0.930 (20-4) ⁵⁵³	1.498 (20) ⁵⁵³	<u>Patschulene, C₁₅H₂₄.</u> -- <u>(L)_D²⁰: -38.08°.</u> (553). - <u>Htd. w. S g. neither cadalene nor eudalene.</u> (527).
905	⁴⁰⁵ 158 (35 mm.)	0.901 (10-4) ⁴⁰⁵	1.489 (10) ⁴⁰⁵	<u>C₁₃H₂₂ fm. 1-Methyl-4-cyclohexyl-cyclohexanol-(4) + ZnCl₂.</u> -- <u>Faint lemon-like odor.</u> (405).
907	⁵⁰⁸ 130-2 (20 mm.)	0.928 (20-4) ⁵⁰⁸	1.517 ⁵⁰⁸	<u>1-Methyl-6-cyclohexylidene-cyclohexene-(1),</u> <u>C₁₃H₂₀.</u>
909	⁴³⁹ 133 (20 mm.)	0.907 (14-4) ⁴³⁹	1.492 (14) ⁴³⁹	<u>Cyclohexyl-cyclohexylidene-methane, C₁₃H₂₂.</u>
911	⁵⁷² 112-4 (9 mm.)	0.919 (23) ⁵⁷²	1.505 (23) ⁵⁷²	<u>Dehydrobetulene, C₁₅H₂₂.</u> - <u>(L)_D: -68°.</u> (572).
913	⁵¹⁴ 113-5 (9 mm.)	0.940 (20) ⁵¹⁴	1.527 (20) ⁵¹⁴	<u>2,2,8-Trimethyl-bicyclo-(0,4,4)-decaatriene-(3,7,9)</u> <u>"Irene", C₁₃H₂₂.</u> -- <u>CF.</u> <u>Div. B, Sect. I, No. 582</u>
915	⁵⁸⁴ 114-6 (10 mm.)	0.918 (20) ⁵⁸⁴	1.501 ⁵⁸⁴	<u>α-Gurjunene, "Tricyclene-gurjunene", C₁₅H₂₄.</u> -- <u>Fm. East Indian copaiva balsam oil.</u> (584). - <u>(L)_D: -95°</u> (100 mm.). (584). - <u>Pt + H g. dihydro cpd., b.p.₁₂= 124-4.5, d₂₀: 0.920, n_D: 1.504, (L)_D= -52°.</u> (584). - <u>Easily oxid. by KMnO₄ in H₂O-Me₂CO.</u> (585).
917	⁵⁷⁴ 115-7 (10 mm.)	0.880 (20) ⁵⁷⁴	1.513 (14) ⁵⁷⁵	<u>Elemene, C₁₅H₂₄.</u> -- <u>Can be reduced to hexahydro cpd.</u> (574). - <u>Na + EtOH do not reduce it.</u> (574).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
919	⁵⁷⁴ 117-9 (10 mm.)	0.866 (20) ⁵⁷⁴	1.476 ⁵⁷⁴	Tetrahydroelemene, C ₁₅ H ₂₈ . -- (∞) _D : -20.40°. (574).
921	120-30 (14.5 mm.)	0.900 (20)	1.496	Sumbulene, C ₁₅ H ₂₄ . -- Occurs in musk root oil. - (∞) _D : 10°21'.
923	⁴⁴⁶ 130-2 (24 mm.)	0.849 (20-4) ⁴⁴⁶	1.478 (20) ⁴⁴⁶	1,1,3,3,4,4 Hexamethyl-2-(2 ² -methopropene(2 ¹)-yl)-cyclohexene-(3), C ₁₆ H ₂₈ . By polym. of diisocrotyl. (446).
925	⁴⁹⁰ 123-5 (11 mm.)	0.887 (20-4) ⁴⁹⁰	1.483 (20) ⁴⁹⁰	Octahydroguaiazulene, C ₁₅ H ₂₆ . -- Htd. w. S g. guaiazulene, (490), b.p. ₁₁ = 164°, d ₄ ¹⁸ : 0.976, picrate, black needles, m.p. = 122° styphnate, black needles (fm. MeOH), M.P. = 105-6°. (699).
927	⁴³⁰ 128 (15 mm.)			Tetradecine-(1), C ₁₄ H ₂₆ . -- G. Ag cpd. (430).
929	⁴⁵⁴ 258-9	1.012 (20-4) ⁴⁵⁴	1.506 (20) ⁴⁵⁴	Fluorene-decahydride, C ₁₃ H ₂₀ . -- Scarcely attacked by cold conc. H ₂ SO ₄ + conc. HNO ₃ . (454). - Nitrated by warm conc. HNO ₃ . (454). - Conc. H ₂ SO ₄ + K ₂ Cr ₂ O ₇ g. yellow brown, then dark violet coloration, wh. w. H ₂ O g. blue green color. (454).
931	⁵⁵² 258.5- 9.5	^{553, 561, 562} 0.903 (20-4)	^{536, 553, 561} 1.501-(20)	Caryophyllene*, C ₁₅ H ₂₄ . -- Natural caryophyllene is a mixt. of two isomers, α-Caryophyllene, "Humulene", (optically inactive)(554), and β-Caryophyllene, perhaps 1,3,6,6-Tetramethyl-2-(2 ² -methopropene(2 ¹)-yl)-bicyclo-(1,1,3)-heptene-(3), (527)(optically active). (554). -- Occurs

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				<p>in hop (556), betel (555), Ceylon cinnamon (557), copaiva balsam (527), clove (559), etc., oils. - Active α-form shows (L)_D²⁰: -8.95° (553). - Inactive α-Nitrosochloride, m.p. = 177° (rapid htg. 179°) (558), wh. w. piperidine g. nitrol piperidide, m.p. = 153° (563). - Active β-Nitrosochloride, m.p. = 159° g. w. benzylamine a nitrolamine, m.p. = 172-3° + w. EtONa a deriv. C₁₅H₂₅O₂N (or C₁₅H₂₃ON₂), m.p. = 163-4° (559). - Note -- The nitrosochlorides of α-isocaryophyllene + β-isocaryophyllene g. these same derivs.! (559). - The nitrosochlorides separate in the sunlight fm. a well cooled mixt. of 5cc. hy., 5cc. AcOEt, 5cc. EtOH, 5cc. EtONO, + 5cc. alc. HCl. (527). - α-Nitrosobromide, m.p. = 144-5° (dec.) (558). - β-Nitrosite, m.p. = 115° (blue) (527, 559, 560). - Careful addn. in the cold of 5cc. AcOH to a mixt. of 5cc. hy., 12cc. pet. ether, + 5cc. satd. soln. of NaNO₂ g. the blue nitrosite. (527). - This nitrosite g., w. benzyl amine, a base, m.p. = 172-3°; + w. 10% alc. KOH a cpd., m.p. = 162-3° C₁₅H₂₅NO₂ (or C₁₅H₂₃NO₂). (554, 559). - α-Nitrosate, m.p. = 162°, g. w. benzyl amine a nitrolamine base, m.p. = 126-8°, belonging to the Lseries. (559). - α-Nitrosate, m.p. = 130-5° (527). - Careful addn., w. thorough cooling, of a soln. of 5cc. conc. HNO₃ in 5cc. AcOH to mixt. of 5cc. hy., 5cc. AcOH, + 5cc. EtONO + addn. of alc. + 2 hrs. stg. g. nitrosate. (527). -</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				Addn. of mixt. of absolute ether + H ₂ SO ₄ .H ₂ O to hy. g. mixt. fm. wh. by distn., after making alk., β-caryophyllene alc., + then after acidification + distn. α-caryophyllene alc. is obtained. (527). - Active β-alc., M.P. = 94-6°, phenylurethane, m.p. = 136-7°. (527). - Inactive α-alc., m.p. = 117°, phenylurethane, m.p. = 180°. (527). - α-Hy. adds 4 atoms Br. (556). - Oxid. w. dil. KMnO ₄ at 0° g. cpd., C ₁₄ H ₂₂ O ₄ , m.p. = 120-120.5°. (558, 564). - G. neither cadalene nor eudalene when htd. w. S. (527).
933	125-6 (12 mm.)	0.912 (18-4)	1.507 (18)	1-Methyl-4-isopropylidene-bicyclo-(0,4,4)-decene-(5), C ₁₄ H ₂₂ . -- Htg. w. S g. 2-isopropyl-naphthalene, b.p. = 125°.
935	123-6 ⁵⁴⁰ (10.5 mm.)	0.922 (20-19) ⁵⁴⁰	1.506 ⁵⁴⁰	Calamene, C ₁₅ H ₂₄ . -- Fm. calamus oil. - (α) _D : +5°. (540). - Pt + H ₂ g. tetrahydro cpd., but Na + alc. do not. (540). - Htd. w. S g. cadalene, (527), cf. under "Bisabolene", No. 957.
937	260-70 ⁴⁶⁷ (dec.) 157 ⁴⁶⁷ (15 mm.)	0.864 (15) ⁴⁶⁷	1.518 (15) ⁴⁶⁷	C ₁₅ H ₂₄ , Light sesquiterpene fm. Citronella oil. (467). - (α) _D ¹⁵ : +1°28' (l=100 mm.). (467).
939	260-3 ⁵²³	0.915 (15-15) ⁵²³	1.509 (20) ⁵²³	Atracylene, C ₁₅ H ₂₄ . -- Adds Br. (523).
941	260-80 ⁴⁵⁵	0.939 (15-15) ⁴⁵⁵	1.522 ⁴⁵⁵	C ₂₀ H ₃₂ , diterpene fm. Sandarak rosin. - (α) _D : +55°. (455).
943	261-3 ⁵⁷⁰	0.922 (19-0) ⁵⁵⁸	1.474 (19) ⁵⁵⁸	Clovene, C ₁₅ H ₂₄ . -- α: +1.30° (l=10 cm.). (558).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
945	139-41 ⁴⁸⁷ (20 mm.)	0.931 (20-4) ⁴⁸⁷	1.517 ⁴⁸⁷	<u>1-Ethyl-6-cyclohexylidene-cyclohexene-(1), C₁₄H₂₂.</u>
947	141-3 ⁴⁸⁷ (20 mm.)	0.927 (20-4) ⁴⁸⁷	1.511 ⁴⁸⁷	<u>1-Ethyl-2-cyclohexyl-cyclohexene, C₁₄H₂₂.</u>
949	261-2.5 ⁴⁵⁶ 118-9 ⁴⁵⁶ (8 mm.)	0.910 (30-4) ⁴⁵⁶	1.496 (30) ⁴⁵⁶	C ₁₅ H ₂₄ , Sesquiterpene fm. <u>Balaoharz</u> balsam. - Adds Br in AcOH. (456).
951	124-8 ⁵³⁹ (12 mm.)	0.918 (20-4) ⁵³⁹	1.515 (20) ⁵³⁹	Isocadinene, C ₁₅ H ₂₄ . -- Fm. oil of cade. (539). - A hexahydrocadalene. (539) - G., w. S, cadalene, (539) cf. under "Bisabolene", No 957.
953	125-6 ⁵²⁶ (12 mm.)	0.916 ⁵²⁶	1.509 ⁵²⁶	<u>Hexahydrocadalene, C₁₅H₂₄.</u>
955	130-6 ⁵⁸⁶ (16 mm.)	0.943 (20) ⁵⁸⁶	1.513 (20) ⁵⁸⁶	Heerabolene, C ₁₅ H ₂₄ . -- (∞)D°: -14°.12'. (586).
957	261.5-2.5 ^{524, 525}	0.872 (21) ⁵²⁶	1.492 (21) ⁵²⁶	<u>1-Methyl-4-(4⁴, 4⁵-dimethohexadiene(4⁴, 4⁵))-cyclohexene-(1), "α-Bisabolene", together w. 1-Methyl-4-(4⁴ methylene-(4⁵)-methohexene-(4⁴)-yl)-cyclohexene-(1), "β-Bisabolene", together w. 1-Methyl-4-(4⁴, 4⁵-hexene-(4⁴)-ylidene)-cyclohexene-(1), "γ-Bisabolene", all three occur in both natural and synthetic "Bisabolene" "Limene", C₁₅H₂₄. -- Occurs in oil of bisabomyrrh, oil of limett, oil of opopanax, oil of bergamot, Siberian turpentine oil, oil of citronella, etc. (528). - HCl passed into ether soln. of hy. g. trihydrochloride, m.p. = 79-80°. (528). (All three g. the same trihydrochloride. (528). - Htg. w. S g. cadalene (1,6-dimethyl-4-isopropyl-naphthalene) b.p. = 291°, b.p.₁₂ = 145-65°.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				d: 0.979, $M_D: 67.78^\circ$; picrate, orange-yellow, m.p. = 115; styphnate, yellow, m.p. = 138°. (528 cf. 538). - Cadalene g. w. H_2CrO_4 , besides other products, 1,6-dimethyl-4-isopropyl- naphthaquinone whose oxime, m.p. = 178°, + 6-methyl-4- isopropyl-1-naphthoic ac. (528), m.p. = 161-2°, wh. latter distilled w. $Ca(OH)_2$ g. 6-methyl-4-isopropyl- naphthalene, b.p. ₁₃ = 139- 41°, d_4^{20} : 0.983, n_D : 1.588, orange yellow picrate, m.p. = 101-2°, yellow styphnate, m.p. = 163-4°. (529).
959	263-4 ⁶⁹¹ 137-40 ⁶⁹² (16 mm.)	0.932 (20) ^{691, 692}	1.519 ⁶⁹²	Bicyclic Vetivene, $C_{15}H_{24}$. -- Ctg. 2 double bonds. (692). - In vetiver oil. (691, 692). - L-form shows (∞) _D : -10°12'. (692). - D-form shows (∞) _D : +18°19'. (691). - Adds 4 atoms of Br g. blue coloration. (691).
961	137-9 ⁵⁸² (17 mm.)	0.918 (17) ⁵⁸²	1.513 (15) ⁵⁸²	Gonstylene, $C_{15}H_{24}$. -- (∞) _D : +40°. (582). - Adds Br in ether soln. (582).
963	134 ⁴³⁰ (15 mm.)	0.800 -15-4) ⁴³⁰		Tetradecine-(2), $C_{14}H_{26}$. -- G. no ppt. w. alc. $AgNO_3$. (430).
965	263-4 ⁵³⁰	0.936 (20-4) ⁵³⁶	1.502 (17) ⁵³⁶	Cedrene,* $C_{15}H_{24}$. -- In cedar oil. (565). - D-form shows (∞) _D : 85°32'. (530). - L-form shows (∞) _D : -60° (l=10 cm.). (565). - G. a nitrosochloride melting in- distinctly at 100-2°. (530). - CrO_3 in AcOH g. chiefly cedrone, $C_{15}H_{22}O$ (a ketone). (565). - O_3 in glac. AcOH g. a ketone, $C_{14}H_{20}O$ or $C_{14}H_{22}O$, liquid, b.p. ₁₃ = 120-30; d_{20} = 0.949, n_D = 1.489, semi- carbazone, m.p. = 218°;

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				<p>+ a netroaldehyde, C₁₅H₂₄O₂, b.p.₁₂ = 170-80°, d₂₀ = 1.039, n_D = 1.504; + an ac. C₁₅H₂₄O₃, b.p.₁₀ = 205-15° (567). - Emulsify 5 g. in a mixt. of 80 g. Me₂CO + 5 g. H₂O, add 5 g. pulverized KMnO₄ during abt. 1 hr., agitating continually + keeping at 30-5°; after complete reduction of KMnO₄ (effected finally, by addn. of few drops of alc.) drain off liquid, wash Mn oxides w. little Me₂CO, add 20cc. H₂O to combined washings, evap. Me₂CO on H₂O bath, shake aq. residue in separatory funnel w. 50 cc. pet. ether, let stand with out removing stopper; - in fm. few min. to 2 hrs. cedrene glycol, C₁₅H₂₆O₂, xtallizes fm. pet. ether as fine white needles, rextalld. fm. Me₂CO, m.p. = 167.5-8°; - the detn. can be carried out on 1-2 g. of hy. + takes abt. 3 hrs. (568). - G. neither cadalene nor eudalene when htd. w. S. (527). - Pt + H g. dihydrocedrene, b.p.₁₀₂₀ = 122-3°; d₂₀: 0.920, n_D: 1.493, (L)_D²⁰: +2°. B.b. no. = 121, Test 925.</p>
967	118-22 ⁶⁹⁴ (7 mm.)	0.912 (20) ⁶⁹⁴	1.506 ⁶⁹⁴	<p>Isozingiberene, C₁₅H₂₄. -- (L)_D: -51°36'. (694). - By zingiberene + 50% H₂SO₄ in glac. AcOH for 6 hrs. at 60-5° (694). - Dihydrochloride, m.p. = 169-70° ident. w. that fm. zingiberene. (694). - Dihydrobromide, m.p. = 175° (694). - Htd. w. S g. cadalene. (527).</p>
969	129-31 ⁵⁴¹ (12 mm.)	0.900 (13-4) ⁵⁴¹	1.495 (13) ⁵⁴¹	<p>Dihydrocadinene, C₁₅H₂₆.</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
971	126-7 ⁵⁷¹ (10 mm.)	0.921 (19-19) ⁵⁷¹	1.511 (16) ⁵⁷¹	Combanene, C ₁₅ H ₂₄ . -- By dehydration of combanol fm. cinnamon oil. - (∞) _D ¹⁸ -28°20'. (571).
973	129-32 ⁵⁷⁷ (12 mm.)	0.843 (19-4) ⁵⁷⁸	1.500 ⁵⁷⁷	2,6,10-Trimethyl-dodecetetraene-(χ, χ, χ, χ), "Farnesene", C ₁₅ H ₂₄ . -- (∞) _D : -3.6°: (577).
975	130 ⁶⁷³ (10 mm.)	0.929 (25) ⁶⁷³	1.498 (n _j) ⁶⁷³	Thunbergilene, C ₁₅ H ₂₄ . -- In Japanese Pinus thunbergii. (673). - Hydrobromide m.p. = 70°. (673). - Hydrochloride, m.p. = 60°. (673). - 1 of the very few sesquiterpenes in spirits of turpentine wh. give xtaln. derivs. (673).
977	125-6 ⁶⁵⁴ (7 mm.)	0.894 (20) ⁶⁵⁴	1.495 (20) ⁶⁵⁴	β-Santalene, C ₁₅ H ₂₄ . -- In Austrian sandalwood oil (653). - (∞) _D : -41°3' (l=10 cm.) - G. 2 isomeric nitrosochlorides, m.p. = 152° + 106. (653).
979	131-2 ^{505,506} (10.5 mm.)	0.950 (20-4) ^{505,506}	1.528 (20) ^{505,506}	C ₁₅ H ₂₀ , Pentameride of propadiene-(1,2).
981	140-50 ⁴⁸⁶			Dodecahydroanthracene, C ₁₄ H ₂₂ .
983	263-4 ⁴⁵⁸	0.910 (15-15) ⁴⁵⁸	1.503 (20) ⁴⁵⁸	C ₁₅ H ₂₄ , Sesquiterpene fm. ethereal oil of Pittosporum. - Odor of rose. (458).
985	263-5 ⁴⁵⁹	0.906 (20-4) ⁴⁶⁰	1.504 (20) ⁴⁶⁰	C ₁₅ H ₂₄ , Sesquiterpene fm. oil of Kadi.
987	265.5-6.5 ⁴⁵²	0.924 (15) ⁴⁵²	1.506 (20) ⁴⁵²	C ₁₅ H ₂₄ , Higher boiling sesquiterpene fm. C ₁₅ H ₂₆ O of eucalyptus oil + 90% formic ac. (452). - (∞) _D : +58°40' (l=10 cm.). (452).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
989	122-4 ⁵⁷⁶ (7 mm.)	0.920 (20) ⁵⁷⁶	1.509 ⁵⁷⁶	Eudesmene, C ₁₅ H ₂₄ . -- (L)D: +54°6'. (576). - Dihydrochloride, m.p. = 70°. (576). - Htd. w. S g. eudalene, cf. under selinene (576), No. 999.
991	124-6 ⁵⁷⁹ (7 mm.)	0.870 (20) ⁵⁷⁹	1.484 (20) ⁵⁷⁹	Ferulene, C ₁₅ H ₂₆ . -- Fm. gum ammoniac oil. (579). - Reduces to a tetrahydro cpd. wh. b.p. ₁₀ = 118-20°, d ₂₀ = 0.840, n _D ²⁰ : 1.458, (L) _D ²⁰ : 4.2°. (579).
993	120 ⁶⁰⁶ (3 mm.)	0.911 (23) ⁶⁰⁶	1.513 (23) ⁶⁰⁶	Machilene, C ₁₅ H ₂₄ . -- Fm. Machilus Kausai. (606). - (L) _D ²⁰ : +58.73°. (606).
995	143-4 ⁴⁶¹ (15 mm.)	0.920 (17-4) ⁴⁶¹	1.504 (17) ⁴⁶¹	C ₁₆ H ₂₆ , fm. a methyl-dimethanonadienyl-cyclohexanol.
997	abt. 527 268-70 ⁴⁸⁸ 138-40 (9 mm.)	0.849 (20) ⁴⁸⁸	1.533 ⁴⁸⁸	"Sesquicitronellene", C ₁₅ H ₂₄ . -- (L) _D : +0°36'. (527). - Principal constituent of the "light sesquiterpene fm. oil of Citronella", fm. oil fm. Java (488) + Ceylon (527). - Na + EtOH g. dihydro-sesquicitronellene, b.p. ₁₂ = 131-3°, d ₂₀ = 0.832, n _D = 1.480. (527).
999	268-72 ⁵²⁷ 128-32 ⁵⁶⁹ (11 mm.)	0.923 (15) ^{527, 569}	1.505 (20) ^{527, 569}	1,5-Dimethyl-8-methoethenyl-bicyclo-(0,4,4)-decene-(4), L-Selinene, C ₁₅ H ₂₄ . -- Natural Selinene is a mixt. of L + β-Selinene. The structure of β-Selinene has not been elucidated. (527). - (L) _D : +49°30'. (527). - Occurs up to 20% in celery seed oil. (527). - Both L + β-cpds. in ether soln. tested w. HCl diluted w. 3 pts. air g. dihydrochloride, m.p. = 72-4°. (527, 569). - Htd. w. S g. eudalene (3-

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				isopropyl-5-methyl-naphthalene) b.p. ₂₂ = 130-50°, picrate: m.p. = 90-1°, styphnate, yellow, m.p. = 119-20°. (527). - O ₃ in AcOH g. diketone, C ₁₃ H ₂₀ O ₂ b.p. ₁₁ = 178-80°, d ₂₀ : 1.057, n _D : 1.500, (∞) _D : 15°, disemicarbazone, m.p. = 228°, + only small amts. of acid products. (569). - (Selinene regenerated from the dihydrochloride g. chiefly acid oxid. products.). (569).
1001	269-70 ⁴⁶⁸	0.964 (20) ⁴⁶⁸	1.512 (20) ⁴⁶⁸	Dodecahydro-phenanthrene, C ₁₄ H ₂₂ . -- Warm HNO ₃ nitrates. (468). - Conc. H ₂ SO ₄ , cold, g. yellow-red color, warming g. wine-red color wh. w. long htg. g. green-black soln. (468). - Conc. H ₂ SO ₄ + CrO ₃ g. green-black soln. - "G. no picrate." (468).
1003	130-2 ⁴⁴⁷ (11 mm.)	0.868 (20) ⁴⁴⁷	1.495 (20) ⁴⁴⁷	1-Methyl-4-methoethenyl-6-isoamyl-cyclonhexadiene-(1,5), or an isomer, C ₁₅ H ₂₄ . -- D-form shows (∞) _D ²⁰ : 18°30'. (447). - Does not form solid cpd. w. HCl nor undergo condensation to a bicyclic cpd. - Adds 6 atoms of H g. isoamyl-methane, b.p. ₁₄ = 131-3°, d ₂₂ : 0.825, n _D ²² : 1.456, (∞) _D ¹² : -1.5. (447).
1005	122-5 ⁶⁹⁴ (7 mm.)	0.856 (20) ⁶⁹⁴	1.484 ⁶⁹⁴	Dihydrozingiberene, C ₁₅ H ₂₆ . -- (∞) _D : -37°. (694).
1007	131-3 ⁴⁸⁸ (12 mm.)	0.832 (20) ⁴⁸⁸	1.480 ⁴⁸⁸	Dihydrosesquictronellene, C ₁₅ H ₂₆ .

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
1009	269-70 ⁶⁹⁵	0.873 (20) ⁶⁹⁶	1.494 (20) ⁶⁹⁶	<p>1-Methyl-4-(4¹,4⁵-dimethohexadiene(4²,4³)-yl)-cyclohexene-(6), "Zingiberene", C₁₅H₂₄. -- Occurs in ginger oil. (694) - (α)_D²⁰: -73.38°. (696). Nitrosite, needles fm. hot MeOH, m.p. = 97-8°. (697). - Nitrosate, m.p. = 86-8° (dec.), yellow powder by pptn. fm. EtOAc by EtOH. (698). - Nitrosochloride, white powder fm. EtOAc + EtOH, m.p. = 96-7° (dec.). (698). - Ether soln. of hy. + HCl g. dihydrochloride of isozingiberene, m.p. = 169-70°. (527). - Na + EtOH reduces to monocyclic dihydrozingiberene, No. 1005, b.p.₇ = 122-5°. (527). - Pt + H g. monocyclic hexahydrozingiberene, b.p.₁₁ = 128-30°. (527). - Htd. w. S g. cadalene (527), cf. under bisabolene, No. 957. - 50% H₂SO₄ in glac. AcOH for 6 hrs. at 60-5° g. isozingiberene. (694).</p>
1011	270-80 ⁷¹⁵	0.898 (20-20) ⁷¹⁵	1.500 (20) ⁷¹⁵	<p>C₁₅H₂₄, fm. C₁₅H₂₆O of oil of <i>Hedeoma pulegioides</i>. (715). - (α)_D¹⁵: +1°4' (l=10 cm.). (715).</p>
1013	272-5 ⁴⁶⁷	0.912 (15) ⁴⁶⁷	532, 533, 536	<p>C₁₅H₂₄, Heavy sesquiterpene fm. citronella oil. (α)_D¹⁵: +5°50' (l=10 cm.). (467).</p>
1015	274-5 ^{532, 535}	0.919 (20-4) ^{533, 535, 536, 537}	1.509 (20) ⁵³⁷	<p>2,8-Dimethyl-5-isopropylbicyclo-(0,4,4)-decadiene-(4,7), "α-Cadinene", or 2,8-Dimethyl-5-isopropylbicyclo-(0,4,4)-decadiene-(4,8), "β-Cadinene", or both, "Cadinene", C₁₅H₂₄. -- Very widely distributed. (527). - The l-form occurs in various turpentine, cedar, cade, cypress, (530).</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				cubeb (531), etc. oils. - The d-form occurs in Atlas cedar oil (532), turpentine oil fm. pinus contorta (527), etc. - L-form, (∞) _D : -110.96° (533). - D-form, (∞) _D : +47°55' (532). - Nitrosate, white powder, m.p. = 105-10° (dec.) (534). - Nitrosocchloride, white powder, m.p. = 93-4° (dec.) (534). - Adds Br. (537). - Ether soln. sat'd. w. HCl at 0° g. dihydrochloride, colorless needles fm. AcOEt, m.p. = 117-8.5° (527, 538). - <u>Note</u> : through rearrangements caused by HCl, other related sesquiterpenes can give the dihydrochloride of cadinene (527). - 70 g. hyd. heated w. 33 g. powdered S at 200° for 6 hrs. + finally at 250°, followed by distn. in vacuo after all H ₂ S has been evolved g. 50 g. yellow oil, cadalene, b.p. ₁₂ = 148-58° (538), for whose characterization cf. under "Bisabolene", No. 957.
1017	155 ^{430*} (15 mm.)	0.797 (20) ⁴³⁰		Hexadecine-(1)*, C ₁₆ H ₃₀ . -- M.p. = 15°.* (430). - Cf. Div. A, Sect. 1, No. 23.
1019	275-6 ⁴⁶⁸	0.993 (20-4) ⁴⁶⁸	1.534 (20) ⁴⁶⁸	Decahydrophenanthrene, C ₁₄ H ₂₀ . -- M.p. = (-20) - (-18°). (468). - Conc. H ₂ SO ₄ + K ₂ Cr ₂ O ₇ g. black-green soln. (468). - Sol. In warm conc. H ₂ SO ₄ w. wine-red color. (468).
1021	148-50 (10 mm.)	0.899 (20-4)	1.485 (20)	Dodecanydroretene, C ₁₈ H ₃₀ . -- Very stable toward hot HNO ₃ . - Slowly sol. in H ₂ SO ₄ w. yellow color g+ brown-green on mtg.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
1023	153-5 ⁴⁶⁵ (11 mm.)	0.831 (21) ⁴⁶⁵	1.483 ⁴⁶⁵	Heptadecatetraene- (χ, χ, χ, χ), "Aplotaxene", $C_{17}H_{28}$. -- In ethereal oil of Costus root to about 20%. (465). - Optically inactive. (465). - Na + EtOH g. dihydroaplotaxene. - H + Pt g. n-heptadecane. (465).
1025	160 ⁴³⁰ (15 mm.)	0.804 (20-4) ⁴³⁰ (liquid)		Hexadecine-(2), $C_{16}H_{30}$. -- M.p. = 20°. (430). Cf. Div. A, Sect.1, No. 33.
1027	142-7 ⁴⁰⁰ (6 mm.)	0.815 (18-4) ⁴⁰⁰	1.456 (18) ⁴⁰⁰	Hexadecadiene-(1,15), $C_{16}H_{30}$. -- M.P. = (-14°) - (-12°). (400).
1029	154-7 ⁴⁶⁵ (11 mm.)	0.818 (21) ⁴⁶⁵	1.471 ⁴⁶⁵	Heptadecatriene-(χ, χ, χ) "Dihydroaplotaxene", $C_{17}H_{30}$. -- By aplotaxene + Na + EtOH. (465).
1031	155-8 ⁴⁵⁷ (10 mm.)	0.934 (20-4) ⁴⁵⁷	1.515 (20) ⁴⁵⁷	Decahydroretene, $C_{18}H_{28}$. -- Odor like petroleum. (457). - Nitrated by hot HNO_3 . (457). - Slowly sol. in H_2SO_4 w. brown color. (457).
1033	163-4 ⁴⁸⁹ (13 mm.)	0.942 (20-4) ⁴⁸⁹	1.510 (20) ⁴⁸⁹	4-Cyclohexyl-bicyclo-(0,4,4)-decene-(3), $C_{16}H_{26}$.
1035	162-3 ⁴⁸⁹ (12 mm.)	0.955 (20-4) ⁴⁸⁹	1.518 (20) ⁴⁸⁹	2-Cyclohexyl-bicyclo-(0,4,4)-decene-9(10), $C_{16}H_{26}$.
1037	163-5 ⁴⁵⁷ (10 mm.)	0.958 (20-4) ⁴⁵⁷	1.530 (20) ⁴⁵⁷	Octahydroretene, $C_{18}H_{26}$. -- Unattacked by cold, attacked by hot HNO_3 . (457). - Sol. in conc. H_2SO_4 w. red-brown color, deep red by transmitted light, changing to black-green on htg. (457).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
1039	169-71 ⁴⁸² (11 mm.)	lighter than ⁴⁸² H ₂ O		<u>2,2'-Dimethyl-5,5'-di-(methoethenyl)-1,4,5,6,1',4',5',6'-octahydro-diphenyl, "Biscarvene", C₂₀H₃₀.</u> -- Light yellow. (482). - Faint odor of cautchouc. (482). - AcOH-H ₂ SO ₄ or conc. H ₂ SO ₄ g. deep red color wh. disappears on addn. of H ₂ O. (482).
1041	188-90 ⁵⁵¹ (20 mm.)			<u>Diisocarvestrene, C₂₀H₃₂.</u> - Br in CHCl ₃ g. yellow color, changing to purple + finally blue. (551).
1043	170 ^{505,506} (10 mm.)	0.972 (20-4) ^{505,506}	1.539 (20) ^{505,506}	<u>C₁₈H₂₄, Hexameride of propadiene-(1,2).</u> -- Nearly odorless. (505).
1045	170-3 ⁵⁵⁰ (10 mm.)	0.928 (20) ⁵⁵⁰	1.518 (20) ⁵⁵⁰	<u>Dicarvenene, C₂₀H₃₂.</u> -- By boil. carvenene w. alc. H ₂ SO ₄ . (550).
1047	abt. 290 ⁴⁹¹ (dec.) 210 ⁴⁹¹ (100 mm.)	0.939 (20) ⁴⁹¹	1.507 ⁴⁹¹	<u>Dicyclopentyl-cyclopentene C₁₅H₂₄.</u>
1049	186-7 ^{472,473} (13 mm.)	0.826 (0-4) ⁴⁷³		<u>"Phytadiene", C₂₀H₃₈.</u> -- Misc. w. MeOH, AcOH, pet. ether. (473). - Decolorizes KMnO ₄ in AcOH. (473).
1051	193-7 ⁵⁴³ (19 mm.)	0.903 (21) ⁵⁴³	1.503 ⁵⁴³	<u>Iso-<i>L</i>-camphorene, C₂₀H₃₂.</u>
1053	180-6 ⁴⁶³ (14 mm.)	0.859 (21) ⁴⁶³	1.468 ⁴⁶³	<u>Hexahydro-iso-<i>L</i>-camphorene, C₂₀H₃₈.</u>
1055	183-95 (16 mm.) 147-52 (3 mm.)	0.912 (20)	1.503	C ₁₅ H ₂₄ , fm. geraniol by htg. w. 20% H ₂ SO ₄ at 200-210° for 6 hrs. or w. 65% H ₂ SO ₄ at 60°, together w. other products.
1057	310-5 ⁷²⁰	0.899 ⁷²⁰	1.480 (20) ⁷²⁰	<u>Dimethene, C₂₀H₃₂.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
1059	310-6 ⁷¹⁹			Paracajeputene, C ₂₀ H ₃₂ ; fm. cajeput oil + P ₂ O ₅ . (719). - Lemon yellow. (719). - Blue fluorescence (719).
1061	318-20 ⁴⁷⁵ 210-2 ⁴⁷⁸ (40 mm.)	0.939 (25) ⁴⁷⁶	1.514 ⁴⁷⁹	C ₂₀ H ₃₂ , "Diterpilene", "Colophene", "Dicamphene", a diterpene fm. pinene or closely related cpds. - Thick yellow oil. (478). - Distn. at atm. press. g. camphene + other products. (477).
1063	328-33 ⁴⁸⁰			C ₂₀ H ₃₂ , "Dicinenene", Diterpene fm. wormseed oil + P ₂ O ₅ . -- Yellow. (480). - Weak blue fluorescence. (480).
1065	186-6.5 ⁴⁹⁶ (12 mm.)	0.952 (20-4) ⁴⁹⁶		(2,3),(4,5)-Diendotetramethylene-bicyclo-(0,4,4)-decene-(1), " $\Delta^{9(14)}$ " Hexadecahydro-9,10-benzophenanthrene", C ₁₈ H ₂₈ . -- Immediately decolorizes KMnO ₄ . (496). - Ni + H g. perhydro-9,10-benzophenanthrene, whl b.p. ₂₃ = 175-6°, d ₄ : 0.943, n _D : 1.521, entirely stable to KMnO ₄ . (496).
1067	185-8 ⁴⁴⁵ (10 mm.)	0.845 (0) ⁴⁴⁵		2-Methyl-heptadecadiene-(2,3)(?), C ₁₈ H ₃₄ .
1069	210 ⁴³³ (25 mm.)	0.952 (15) ⁴³³		Dicamphenylidenebutane, C ₂₄ H ₄₀ . -- Will combine w. Br, but cannot be titrated (433).
1071	204-7 ⁴⁶⁹ (15 mm.)			1,3-Dicyclohexyl-cyclohexene-(1), C ₁₈ H ₃₀ .
1073	abt. 175 ⁴⁷⁰ (2.5 mm.)	abt. 0.818 ⁴⁷¹ (24) (detnd. for an isomer)		Eikosadiene-(1,19), C ₂₀ H ₃₈ . -- Xtals. fm. pet. ether + absolute alc. (470). - M.p. = 20.3°. (470).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
1075	335-6 ⁴⁸¹ u.c.			Dodecahydrotetene, C ₁₈ H ₃₀ . -- Colorless oil w. blue fluorescence. (481). - Distn. w. Zn dust g. a little retene. (481). - Br + fum. HNO ₃ attack it in the cold, CrO ₃ , in AcOH on hgt. (481).
1077	340-5 ⁴⁸³	0.973 (18-9) ⁴⁸³	1.537 (n) ⁴⁸³	C ₁₉ H ₃₀ , "Abietene", "Colophene", "Diterebentyl". -- (∞) _D : +92.9°. (483). - Htg. w. S g., among other products, retene. (483).
1079	abt. ⁴⁸⁴ 360			Chrysene-hexadecahydride, C ₁₈ H ₁₈ . -- Unattacked by Br or cold HNO ₃ (d:1.48). (484).
1081	228-30 ⁶⁵⁷ (3 mm.)	0.924 (20-20) ⁶⁵⁷	1.510 (20) ⁶⁵⁷	Tetracyclosqualene, C ₃₀ H ₅₀ . -- Htd. w. S g. distillate wh. g. orange-red picrate, m.p. = 139-40°, wh. latter w. NH ₄ OH g. C ₁₃ H ₁₄ , b.p.: 145°, m.p. = 33.5°, d ₂₀ : 1.010, n _D : 1.611. (657).
1083	230-2 ⁴³³ (3 mm.)	0.919 (15) ⁴³³		8-Propyl ^{en} -camphenylidene, C ₁₃ H ₂₀ . -- K ₂ Cr ₂ O ₇ g. camphenylidene-acetic ac. + a little corresp. aldehyde. (433).
1085	260-2 ^{654b} (9 mm.)	0.860 (18-18) ^{654b, 655, 656}	1.497 (20) ^{654b, 655, 656}	2,6,26-Trimethyl-heptaicosahexaene-(2,6,10,18,22,26)(?), "Squalene", "Spinacene", C ₃₀ H ₅₀ . -- Occurs up to 90% in various liver oils, as that fm. Centrophorus granulosa + Scymnus lichia (deep sea fish), etc. (655). - Absorbs O fm. air. (655). - Dries, like linseed oil, very slowly in thin layers. (655). - C ₅ H ₁₁ ONO + slow addn. of HNO ₃ in AcOH at -15° g. nitrosate, yellow powder, dec. at 85° (655). - HCl in Me ₂ CO g. hydrochloride, sepd. by xtaln. fm. Me ₂ CO into 3

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				<p>isomers, m.p. = 107-8°, 144-5°, 113-4°. (654b). - Hexabromide, m.p. = 118-20°, together w. higher m. isomers. (654b). - In Et₂O w. dry HCl g. C₃₀H₅₀·6HCl, sinters at 108°, begins to m. at 110°, evolves HCl at higher temps. (655). - Br in Et₂O g. dodecabromide, purified by addn. of EtOH to its soln. in C₂H₂Cl₄, darkens abt. 160°, m. abt. 185° w. dec. (655). - Din nitrosochloride-mononitro-1-piperidide, C₃₀H₅₀(NOCl)-NONC₅H₁₀, buff-colored, sinters abt. 110°, m.p. abt. 146° w. dec. (655). - Not reduced by Na + C₅H₁₁OH or C₈H₁₇OH. (654b).</p>

DIVISION B

.....
Liquid Hydrocarbons

Section 4

.....
Acyclic Olefines

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
2	20-1 ^{1,2,3}	0.632 (15-4) ¹	1.388 (15) ¹	2-Methyl-butene-(3)*, C ₅ H ₁₀ . -- I. at 0° in 2 vols. conc. H ₂ SO ₄ + 1 vol. aq. (4). - Aq. HBr g. chiefly sec. + some ter. bromide. (2,5,6). - Cold KMnO ₄ g. chiefly isopropyl-ethylene glycol + some AcOH, Me ₂ CO, isobutyraldehyde. (7). - HI in 60% AcOH g. Me ₂ CH·CHI·Me. (5).
4	30-1 ^{8*}	0.641 (21) ^{8,9}	1.371 (21) ^{8,9}	Pentene-(1)*, C ₅ H ₁₀ . -- HI g. 2-iodopentane. (9). - KMnO ₄ g. formic ac., succinic ac., + some butyric ac. - Dibromide, b.p. = 184°, D ¹⁸ : 1.668, n _D ²¹ : 1.509. (8). - Hg deriv. m.p. = 118.5-9°* (Test 927).
6	31-2 ¹⁰	0.667 (0) ¹¹	1.378 (16) ¹¹	2-Methyl-butene-(1), C ₅ H ₁₀ . -- Sat'd. aq. HBr at 0° g. 2-brom-2-methyl-butane. (2). - S. at 0° in 2 vols. conc. H ₂ SO ₄ + 1 vol. aq. (12,13).
8	36-7 ^{1,14}	0.651 (20-4) ^{1,14}	1.381 (20) ^{1,14}	Pentene-(2)*, C ₅ H ₁₀ . -- HI g. 2-iodopentane. (15). - KMnO ₄ g. (COOH) ₂ , AcOH, EtCO ₂ H. (15). - HBr g. 3-brom-pentane, b.p. 74.5 = 117-9°, n _D ²⁰ : 1.444. (16). B.b.no. = 227° (Test 925). (121).
10	37.5-8.5 ^{17,18,19}	0.663 (20-4) ¹⁷	1.386 (14) ¹²⁰	2-Methyl-butene-(2)*, C ₅ H ₁₀ . -- KMnO ₄ g. trimethyl-ethylene-glycol, Me ₂ CO, MeCHO, AcOH. (5). + 60% HI in Hac g. ter. iodide, (CH ₃) ₂ CIC ₂ H ₅ . (5). - B.b.no. = 227° (Test 925). (121).
12	41-2 ²⁰	0.655 (18-4) ²⁰	1.377 (18) ²⁰	2,2-Dimethyl-butene-(3), C ₆ H ₁₂ . -- KMnO ₄ oxid. g. trimethyl acetic ac. (20).
14	cf. 54.5-5.5 ^{27*}	0.7 (25/4)*	1.38 (25)*	4-Methyl-pentene-(1)*, C ₆ H ₁₂ .

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
16	56-9 ²¹	0.680 (0) ²¹		2,3-Dimethyl-butene-(1), <u>C₆H₁₂</u> . -- HgO + 1 g. iodo- hydrin wh. g. w. powdered KOH, an isomer of pinnacolin, b.p. = 100.5°-101.5°, D°: 0.8413. (22).
18	57-9²³	0.671 (20-4)²³	1.388 (20)²³	2-Methyl-pentene-(3), C₆H₁₂. -- KMnO₄ oxid. g. isobutyric ac. (23).
20	58-9 ^{23*}	0.669 (25-4 vac.) ^{23*}	1.387 (25) ^{23*}	4-Methyl-pentene-(2), <u>C₆H₁₂</u> . -- KMnO ₄ oxid. g. isobutyric ac.(22)
22	63-4^{24,26*}	0.679 (15-4) ²⁴	1.382 (20) ²⁵	Hexene-(1), <u>C₆H₁₂</u> . -- KMnO ₄ oxid. g. formic + valeric acs. (15). - Dibromide, b.p. ₁₆ = 86.5-87.5°, d ₄ ¹⁵ : 1.587, n _D ¹⁹ : 1.501 g. hexene- (1) w. alc. KOH. (24). - Hg deriv. (Test 927) m.p. = 93-4°* - B.b. no. = 192 ^{24*} (Test 925).
24	64-6 ²⁵			2-Methyl-pentene-(1), <u>C₆H₁₂</u> . -- Sol. in 85% H ₂ SO ₄ w. formation of a ter. alc. (?). (25).
26	65-6 ²⁶	0.722 (15) ²⁶	1.400 (20) ²⁶	3-Methyl-pentene-(2), isomer 1, <u>C₆H₁₂</u> . -- See below, No. 34.
28	66.5- 7.5^{28,29}	0.691 (15-4) ³⁰	1.403 (15) ³⁰	2-Methyl-pentene-(2), <u>C₆H₁₂</u> . -- CrO ₃ oxid. g. Me ₂ CO, AcOH, EtCO ₂ H. (28). - HI g. iodide, b.p. = 142°, wh. g. dimethyl-propyl- carbinol w. Ag ₂ O. (28). - Dibromide, b.p. ₁₄ = 70°, D ₄ ¹⁵ : 1.583, n _D ¹⁵ : 1.506, m.p. = (-149°)-(-54°). (30). - Chlornitrosite, m.p. = 78- 9°. - Nitrosate, m.p. = 105° dec. (122).
30	67-8 ³¹	0.694 (20.5) ³¹	1.404 (20.5) ³¹	2-Ethyl-butene-(1), <u>C₆H₁₂</u> . -- Dibromide, b.p. ₅₀ = 108-10°. (31).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
32	68-9 ²⁴	0.686 (15-4) ²⁴	1.398 (15) ²⁴	Hexene-(2)*, C ₆ H ₁₂ . -- CrO ₃ oxid. in AcOH g. n-butyric ac. (24). - Dibromide, b.p. 118.5 = 82.6°, d ₄ ¹⁵ : 1.594, wh. g. w. alc. KOH hexene-(2). (24).
34	69-70 ^{33,35}	0.695 (20-4) ^{28,32,33}	1.402 (20) ^{32,33}	3-Methyl-pentene-(2), isomer 2, C ₆ H ₁₂ . -- CrO ₃ oxid. g. MeCOEt. (28). - HI g. 3-iodo-3-methyl-pentane. (34). - Dibromide, b.p. 115 = 83°, 161° w. dec. (760 mm.) D ₂₀ : 1.575, n _D ²⁰ : 1.507. (32). Cf. No. 26.
36	72-3 ³⁵	0.698 (20-0) ³⁵	1.407 (20) ³⁵	2,3-Dimethyl-butene-(2)*, C ₆ H ₁₂ . -- CrO ₃ oxid. (cold) g. Me ₂ CO (37), AcOH + trimethyl acetic ac. (38). - HI g. Me ₂ CH.CiMe ₂ . (37). - N ₂ O ₅ g. chiefly dinitrate, m.p. = 210-11°. (39).
38	72-3 [*]	0.688 (25-4 [*] vac.)	1.391 (25) [*]	4,4-Dimethyl-pentene-(1)*, C ₇ H ₁₄ . -- B.b.no. = 177* (Test 925). - Hg deriv. m.p. = 108.5-12°. (Test 927).
40	75-80 ⁴⁰	0.710 (15-4)		2-Methyl-hexene-(4) or (5), C ₇ H ₁₄ .
42	78-80 ^{41,42}	0.710 (15-4) ⁴³	1.406 (15) ⁴³	2,2,3-Trimethyl-butene-(3), C ₇ H ₁₄ . -- 1% KMnO ₄ g. 2,3,3-trimethylbutanediol-(3,4). (42). - 4% KMnO ₄ oxid. g. pinacolin, formic ac., trimethyl acetic ac., + (H ₃ C) ₃ C.C(CH ₂)(OH)COOH. (42). - HI g. pentamethylethyl-iodide. (41). - HBr g. pentamethyl-brom-ethane. (43). - Dibromide, b.p. 14 = 98-9°, m.p. = 38-9° (fm. ether). (42). - For identification by preparation of (H ₃ C) ₃ C.CH ₂ .C:C.COOH see Opel, Faworski X 50, 67; C.(1923) III, 668.

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
44	83-4 ^{44,45*}	0.696 (20-4) ⁴⁵	1.402 (20) ⁴⁵	2,4-Dimethyl-pentene-(2)*, C ₇ H ₁₄ . -- KMnO ₄ oxid. g. 2,4-dimethylpentanol-(2)-one-(3), Me ₂ CO, AcOH+ isobutyric ac. (46). - HI (in cold) g. dimethyl-isobutyl carbinol-iodide. (47). - Prepared fm. dimethyl-isobutyl-carbinol or its halogen derivatives. - See below, No. 66. - B.b.no. = 224°* (Test 925).
46	84-6 ⁴⁸	0.722 (20-4) ⁴⁸		4,4-Dimethyl-pentene-(2), C ₇ H ₁₄ . -- Oxid. g. AcOH + trimethyl acetic ac. (48). - Dibromide, b.p. ₁₅ = 86-8°, d ₂₀ : 1.5303, wh. g. ter. butyl-allene w. xs. alc. KOH. (48).
48	85-6 ^{25*}	0.690 (25-4 ^{cf. 25*} vac.)	1.394 (25)*	5-Methyl-hexene-(1)*, C ₇ H ₁₄ . -- B.b.no. = 164° (Test 925). - Hg deriv. m. p. = 88-9° (Test 927)*.
50	86.5- [*] 7.5	0.711 (25-4 [*] vac.)	1.399 (25)*	4-Methyl-hexene-(1)*, C ₇ H ₁₄ . -- B.b. no. = 159* (Test 925). + Hg deriv. m. p. = 87.5-8.5°* (Test 927).
52	85-90 ⁴⁹	0.730 (25-4) ⁴⁹	1.413 (20) ⁴⁹	3-Methyl-hexene-(2), C ₇ H ₁₄
54	90-3 ^{32,50}	0.72 (22) ^{32,50}	1.413 (22) ^{32,50}	2,3-Dimethyl-pentene-(2), C ₇ H ₁₄ . -- Oxid. g. Me ₂ CO, MeCOEt, HOCMe ₂ CMeEt(OH). (51).
56	93.5- ⁵¹ 4.5			3-Methyl-hexene-(3), C ₇ H ₁₄ . -- KMnO ₄ oxid. g. MeCOEt + EtCO ₂ H. (51). - For identification thru addn. of HI cf. Faworski, Zaleski-Kibardine, Bull. Soc. chim. (4) 37, 1227 (1925).

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
58	50, 52, 120 94-6*	52, 53 0.702 (20-4)	52, 53 1.404 (20)	Heptene-(3)*, C ₇ H ₁₄ . -- Dibromide, b.p. ₁₁ = 98-9°. (52). - Dichloride, b.p. = 179-81°, d ₄ ²⁰ : 1.0260, n _D ²⁰ : 1.450. (52). - Heptandiol-(3,4), by sapon. of semi-acetate, b.p. = 212° (761 mm.), m.p. = 98-9°; see Mathus + Gibon, Bull. Soc. Chim. Belg. 34, 303-13 (1925). - B.b.no. = 162* (Test 925).
60	54* 94-5	0.700 (19) ^B	1.400 (19) ^B	Heptene-(1)*, C ₇ H ₁₄ . -- KMnO ₄ oxid. g. caproic ac. (55). - Dibromide, b.p. ₁₂ = 98-9°, d ₁₉ ¹⁹ : 1.509, n _D ¹⁹ : 1.5020. (5). - Hg deriv. (Test 927), m.p. = 60-61°. (56). - B.b.no. = 157* (Test 925).
62	50 95-6	50, 57 0.725 (17)	50 1.417 (17)	3-Ethyl-pentene-(2), C ₇ H ₁₄ . -- KMnO ₄ oxid. g. EtCO ₂ H, HAc, + triethyl-carbinol. (57).
64	45 96-7			2,3-Dimethyl-pentene-(3), C ₇ H ₁₄ .
66	50 96-7	50 0.724 (17)	50 1.413 (17)	2,4-Dimethyl-pentene-(2), C ₇ H ₁₄ . -- Fm. diisopropyl-carbinol. See above, No. 44.
68	58 97-8	58 0.718 (20-4)	58 1.411 (20)	3-Methyl-hexene-(2) or (3), C ₇ H ₁₄ . -- CrO ₃ oxid. g. AcOH + EtCO ₂ H. (58).
70	59, 60* 98-9			Heptene-(2)*, C ₇ H ₁₄ . -- K ₂ Cr ₂ O ₇ + H ₂ SO ₄ oxid. g. AcOH + valeric ac. (59, 60). - B.b.no. = 182* (Test 925).
72	61 102-3	61 0.715 (15)		Diisobutylene*, C ₈ H ₁₆ . -- CrO ₃ oxid. (cold) g. Me ₂ CO, trimethyl-acetic ac., AcOH, octylic ac. + Me ₃ C.CH ₂ CO.Me (61). - HI + HCl at 100° g. Me ₃ C.CH ₂ .CHlgMe ₂ . (61).

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
74	110-1 ⁶²	0.713 ²⁵	1.399 ²⁵	<u>2,2,3-Trimethyl-pentene-(3)</u> or <u>2,2-Dimethyl-3-methylene-</u> <u>pentane, C₈H₁₆.</u> -- Forms constant boil. mixt. w. EtOH at about 80°. (62).
76	111-2 ²⁵	0.713 ²⁵ (20/4)	1.399 ²⁵ (20)	<u>2-Methylheptene-(6), C₈H₁₆.</u>
78	113-4 ⁶³			<u>3,4-Dimethyl-hexene-(3),</u> <u>C₈H₁₆.</u>
80	113.5- [*] 4.5	0.719 (25-4 [*] vac.)	1.410 (25) [*]	<u>4-Methyl-heptene-(2),</u> <u>C₈H₁₆.</u>
82	114.5- ⁶⁴ 6.5 (741 mm.)			<u>2-Methyl-3-ethyl-pentene-</u> <u>(2), C₈H₁₆.</u>
84	119-20 ^{58, 55}	0.736 (20-0) ⁵⁸		<u>3-Ethyl-hexene-(2), C₈H₁₆.</u> -- CrO ₃ oxid. g. hexanone- (3) + AcOH. (58).
86	120-1 ⁵⁸	0.731 (20-0) ⁵⁸	1.417 (25) ⁴⁹	<u>4-Methyl-heptene-(3),</u> <u>C₈H₁₆.</u> -- CrO ₃ oxid. g. AcOH + EtCO ₂ H. (58).
88	121-2 ⁸	0.716 (19) ⁸	1.409 (19) ⁸	<u>Octene-(1), C₈H₁₆.</u> -- Di- bromide, b.p. ¹⁴ = 116-8°, d ¹⁹ : 1.453, n _D ¹⁹ : 1.4961. (8). -- Hg deriv. m.p. = 79-80° (Test 927).
90	123-5 ²⁶	0.725 (20-0) ²⁶	1.412 ²⁶ (20)	<u>2-Methyl-heptene-(2)(?),</u> <u>C₈H₁₆.</u> -- Nitrosocnloride, m.p. = 48-51°. (121).
92	cf. 125-6 ^{66*}	0.720 ⁶⁶ (20/4)	1.413 ⁶⁶ (20)	<u>Octene-(2),*</u> C ₈ H ₁₆ . -- G. octylene oxide w. benzoyl hydroperoxide, see Pril- eschajew, Ber. 42, 4812. - B.b.no. = 126 [*] (Test 925).
94	128.5- [*] 9.5	0.738 (25-4 [*] vac.)	1.420 (25) [*]	<u>4,5,5-Trimethyl-hexene-(2)</u> <u>C₉H₁₈.</u>
96	129-30 [*]	0.724 (25-4 [*] vac.)	1.414 (25) [*]	<u>4,6-Dimethyl-heptene-(2),*</u> <u>C₉H₁₈.</u> -- B.b.no. = 133 [*] (Test 925).
98	132-3 ⁶⁷			<u>2-Methyl-4-methylene-</u> <u>heptane (?), C₉H₁₈.</u>

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
100	132-4 ⁶⁸			<u>2,6-Dimethyl-heptene-(3), C₉H₁₈.</u>
102	133-8 ⁴⁹	0.739 (25-4) ⁴⁹	1.418 (25) ⁴⁹	<u>4-Methyl-octene-(4) or (5), C₉H₁₈.</u>
104	134.5- [*] 5.5	0.743 (25-4 [*] vac.)	1.422 (25) [*]	<u>4,5-Dimethyl-heptene-(2)[*], C₉H₁₈.</u>
106	137-8 [*]	0.729 (25-4 [*] vac.)	1.416 (25) [*]	<u>4-Methyl-octene-(2)[*], C₉H₁₈</u> -- B.b. no. = 127 [*] (Test 925).
108	139-40 ⁶⁷			<u>2-Methyl-5-methylene-heptane (?), C₉H₁₈.</u>
110	139-40 ⁵⁸	0.743 (20-0) ⁵⁸		<u>Nonylene, C₉H₁₈.</u> -- Fm. EtPr ₂ Cl + Alc. KOH. (58).
112	140-1 ⁶⁹	0.748 (20/4) ⁶⁹	1.428 (20) ⁶⁹	<u>3-Ethyl-heptene-(3), C₉H₁₈</u>
114	141.5-3 ⁷⁰			<u>2-Methyl-octene-(1), C₉H₁₈</u>
116	141.5- ⁷¹ 3.5			<u>4-Methylene-octane (?), C₉H₁₈.</u>
118	144-5 ⁷²	0.742 (21) ⁷²	1.426 (21) ⁷²	<u>Nonylene, C₉H₁₈.</u> -- fm. EtMgBr + brom-valer-aldehyde.
120	145-6 [*]			<u>Nonene-(1)[*], C₉H₁₈.</u> -- B.b. no.: 128 [*] (Test 925). - Hg deriv., m.p. = 68-9 ^o * (Test 927).
122	147-8 ^{53*}	0.732 (18) ⁵³	1.421 (18) ⁵³	<u>Nonene-(4)[*], C₉H₁₈.</u> -- KMnO ₄ oxid. g. butyric + n-valeric acs. (53). - Dibromide, b.p. ₁₈ = 119-20 ^o , D ¹⁷ : 1.410, n _D ¹⁷ : 1.499. (53). - B.b.no.: 126 [*] (Test 925).
124	147-8 ^{73,74}			<u>Nonene-(2), C₉H₁₈.</u> -- 4% KMnO ₄ (cold) oxid. g. AcOH + cenanthic ac. (73,74).
126	149-50 ⁷⁵	0.754 (15-15) ⁷⁵		<u>Nonene-(2) or (3), C₉H₁₈.</u> -- Fm. 3-iodononene + alc. KOH.

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
128	152-7 ⁴⁹	0.746 (25-4) ⁴⁹	1.419 (25) ⁴⁹	<u>2,5-Dimethyl-octene-(4) or (5), C₁₀H₂₀.</u> -- Fm. 4-brom-2,5-dimethyloctane + pyridine.
130	152-5 ⁷⁶	0.744 (19) ⁷⁶	1.421 (19) ⁷⁶	<u>2,6-Dimethyl-octene-(X)₂, C₁₀H₂₀.</u>
132	154-6 ⁷⁷ 78, 79 160	0.772 ⁷⁷ (20/4)		<u>Diamylene, C₁₀H₂₀.</u>
134	155-7 ⁸⁰	0.757 ⁸⁰ (20/4)	1.430 ⁸⁰ (20)	<u>2-Methyl-3-ethyl-heptene-(2) or-(3), or 2-Methyl-3-ethylidene-heptane, C₁₀H₂₀.</u>
136	157-8 ⁸¹	0.773 (21-21) ⁸¹		<u>3,3,5-Trimethyl-heptene-(4), C₁₀H₂₀.</u>
138	157-8 ⁸² (750 mm.)	0.752 (11.4-4) ⁸²	1.427 (11.4) ⁸²	<u>2-Methyl-5-ethyl-heptene-(5), C₁₀H₂₀.</u> -- 0.3 g. MeCHO + small amts. of Et iso-amyl-ketone. (82).
140	159.5-61 ⁸³	0.758 (19) ⁸³	1.432 (19) ⁸³	<u>2,3,6-Trimethyl-heptene-(5) or (6), C₁₀H₂₀.</u>
142	162-3 ^{84, 85}	0.749 ⁸⁴ (20/4)	1.427 ⁸⁴ (20)	<u>2,6-Dimethyl-octene-(6), C₁₀H₂₀.</u> -- CrO ₃ or KMnO ₄ oxid. g. AcOH + 2 Me-heptanone-(6). (84, 85).
144	164-5 ⁸⁶	0.752 (20-0) ⁸⁶	1.432 ⁸⁶ (20)	<u>2,6-Dimethyl-octene-(X), C₁₀H₂₀.</u>
146	165-9 ⁴⁹	0.753 (25-4) ⁴⁹	1.428 (25) ⁴⁹	<u>2,6-Dimethyl-nonene-(4) or (5), C₁₁H₂₂.</u>
148	168-9 ⁸⁷	0.777 (22.5) ⁸⁷	1.448 (22.6) ⁸⁷	<u>2,6-Dimethyl-octene-(1) or (2) or both (?), C₁₁H₂₂.</u> -- Identity very doubtful.
150	159-62 ⁸⁸ (650 mm.)	0.748 (20-0) ⁸⁸		<u>2,7-Dimethyl-octene-(2), C₁₀H₂₀.</u>
152	170-1 ^{89*}	0.740 (30) ⁸⁹	1.431 (16) ⁹⁰	<u>Decene-(1)*, C₁₀H₂₀.</u> -- Hg deriv. (Test 927) m.p. = 81-2°* (91). - B.d.no. = 116* (Test 925).

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
154	65 ⁹² (10 mm.)			<u>2,6-Dimethyl-4-isopropylidene-heptane, C₁₂H₂₄.</u>
156	177.5 ⁹³ - 8.5	0.774 (0) ⁹⁴		Triisobutylene,* C ₁₂ H ₂₄ . -- CrO ₃ oxid. g. AcOH, trimethyl acetic ac., Me-diter-butyl-acetic ac., acetone + inactive residual oil. (94).
158	180-3 ⁹⁵	0.771 (15) ⁹⁵	1.433 (15) ⁹⁵	<u>2-Methyl-4-propyl-heptene-(3) or-(4), C₁₁H₂₂.</u>
160	76-8 ⁹⁶ (15 mm.)	0.763 (16-4) ⁹⁶	1.434 (16) ⁹⁶	Undecene, C ₁₁ H ₂₂ . -- of unknown structure.
162	188-90 ⁹⁷	0.763 (20/4) ⁹⁷	1.428 (20) ⁹⁷	Undecene-(1), C ₁₁ H ₂₂ .
164	188-90 ⁹⁸ (772 mm.)	0.778 (20-0) ⁹⁸	1.444 (20) ⁹⁸	<u>2,6-Dimethyl-3-isopropyl-heptene-(2) or-(3), C₁₂H₂₄,</u> probably both.
166	191-2 ^{95,99}	0.767 (21) ^{95,99}	1.434 (21) ^{95,99}	<u>2-Methyl-5-Propyl-octene-(4) or (5), C₁₂H₂₄.</u>
168	192-3 ⁷³	0.774 (15-15) ¹⁰⁰	1.433 (20) ¹⁰⁰	Undecene-(2), C ₁₁ H ₂₂ . -- 4% KMnO ₄ g. pelargonic ac. (74).
170	74-6 ¹⁰¹ (9 mm.)	0.768 (12.3-4) ¹⁰¹	1.435 (12.3) ¹⁰¹	<u>2,5,8-Trimethyl-nonene-(4), C₁₂H₂₄.</u>
172	197-8* vac.)	0.756 (25-4*) vac.)	1.429 (25)*	<u>4-Butyl-octene-(2),* C₁₂H₂₄.</u>
174	204-6 ¹⁰²			<u>2-Methyl-undecene-(4) or (5), C₁₂H₂₄.</u>
176	210.5-2 ²⁵	0.759 (20/4) ²⁵	1.427 (20) ²⁵	<u>2-Methyl-undecene-(2), C₁₂H₂₄.</u> -- CrO ₃ oxid. g. acetone. (25).
178	96-7 ¹⁰³ (15 mm.)	0.762 (15-4) ¹⁰³		Dodecene-(1), C ₁₂ H ₂₄ . -- M.p. = (-31.5°). (103).
180	215.5- ¹⁰⁴ 6.5	0.775 (20/4) ¹⁰⁴	1.438 (20) ¹⁰⁴	<u>5-n-Butyl-nonene-(4), C₁₃H₂₆.</u>
182	228-31 ¹⁰⁵	0.798 (20/4) ¹⁰⁵	1.449 (20) ¹⁰⁵	Tridecene-(X), C ₁₃ H ₂₆ . -- By dry dist'n. of sodium oleate.

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
184	232-3 ¹⁰⁶	0.845 ¹⁰⁶ (20/4)		Tridecene, C ₁₃ H ₂₆ . -- Of unknown structure fm. Burmese petroleum.
186	105-7 ¹⁰⁷ (12 mm.)	0.780 ¹⁰⁷ (20/4)		3-Methyl-dodecene-(3), C ₁₃ H ₂₆ . -- KMnO ₄ oxid. g. pelargonic ac. ⁽¹⁰⁷⁾ .
188	245-8 ¹⁰⁹	0.814 ¹⁰⁹ (20/4)		Triamylene, C ₁₅ H ₃₀ .
190	250-1 ¹⁰⁸	0.798 (0-0) ¹⁰⁸		Tetradecene, C ₁₄ H ₂₈ . -- Of unknown structure.
192	116-22 ¹⁰⁷ (13 mm.)	0.786 ¹⁰⁷ (20/4)		2,3-Dimethyl-dodecene-(3), C ₁₄ H ₂₈ . -- KMnO ₄ oxid. at 17° g. pelargonic ac. (107)
194	127-8 ¹⁰³ (15 mm.)	0.775 (15-4) ¹⁰³		Tetradecene-(1), C ₁₄ H ₂₈ . -- M.p. = (-12°). (103).
196	114-5 ¹¹⁰ (10 mm.)			2,8-Dimethyl-5-(5 ³ -Metho-butyl)-nonene-(4), C ₁₆ H ₃₂ .
198	115-7 ¹⁰⁷ (10 mm.)	0.788 ¹⁰⁷ (20/4)		4-Methyl-tridecene-(4), C ₁₄ H ₂₈ . -- KMnO ₄ oxid. g. pelargonic ac. (107).
200	274-5 ¹¹¹	0.784 (15-4) ¹⁰³	1.442 (19) ¹¹²	Hexadecene-(1), C ₁₆ H ₃₂ . -- M.p. = 4°.* (103). - See Genus IX, Div. A, Sect. 1, No. 5. - B.b.no.: 69.9.* (Test 925).
202	275-85 ²⁵	0.825 (17) ¹¹³		Dicaprylene, C ₁₆ H ₃₂ .
204	160-1 ¹¹⁴ (9.5 mm.)	0.798 (10) ¹¹⁴		Heptadecene-(8)*, C ₁₇ H ₃₄ .
206	290-1 ¹¹⁵ (724 mm.)	0.790 (20-4) ¹¹⁵		Heptadecene of unknown structure, C ₁₇ H ₃₄ .
208	179-80 ¹⁰³ (15 mm.)	0.788 (22-4) ¹⁰³		Octadecene-(1), C ₁₈ H ₃₆ . -- M.P. = +18°. (103). - Cf. Div. A, Sect. 1, No. 31.
210	190-1 ¹¹⁶ (15 mm.)	0.797 (20-4) ¹¹⁶	1.449 (20) ¹¹⁶	trans(?) - Octadecene-(9), C ₁₈ H ₃₆ . -- M.p. = abt. (-15°). (116).
212	around ¹¹⁶ 190-200 (15 mm.)	0.792 (19) ¹¹⁶	1.448 (19) ¹¹⁶	cis(?) - Octadecene-(9), C ₁₈ H ₃₆ . -- M.p. = 2°. (116).

DIVISION B SECTION 4

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
214	390-400 ¹⁰⁹	0.871 (0) ¹⁰⁹		<u>Tetra-amylene, C₂₀H₄₀.</u>
216	167-8 ¹¹⁷ (7.5 mm.)	0.817 (0-4) ¹¹⁷		<u>Phytene, C₂₀H₄₀.</u>
218	201-2 ¹¹⁸ (11 mm.) (thermometer immersed to 110°)	0.802 (20-4) ¹¹⁸		<u>Heneicosene-(9), C₂₁H₄₂.</u> -- M.p. = abt. +3°. (118). -- Identity doubtful.
220	175-6 ¹¹⁹ (1 mm.)			<u>cis + trans Tricosene-(11),</u> <u>C₂₃H₄₆.</u> -- M.p. = 17-18°. (119). -- See Genus IX, Div. A, Sect. 1.

DIVISION B

Liquid Hydrocarbons

Section 5

Naphthenes

DIVISION B, SECTION 5.

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
1	21-2 ¹	0.660(20/4) ¹	1.366(20) ¹	<u>1,1-Dimethyl-cyclopropane, C₅H₁₀.</u> -- Stable to 1% KMnO ₄ . (1). Sol. at 0° in H ₂ SO ₄ (2 vol. H ₂ SO ₄ :1 vol. H ₂ O). (1). Shaking w. fum. HI g. ter. amyl iodide. (1). Br g. trimethyl ethylene dibromide. (1,2).
3	32-3 ³	0.681(20/4) ³	1.382(10) ³	<u>1,2-Dimethyl-cyclopropane, C₅H₁₀.</u> -- 1% KMnO ₄ oxid. completely in 1/2 hr. (3) Difficultly sol. in H ₂ SO ₄ . (3). Reacts only slowly w. Br. (3).
5	36-7 ^{4,5}	0.683(20/4) ^{4,5}	1.379(20) ^{4,5}	<u>Ethyl-cyclopropane, C₅H₁₀.</u> -- Very stable. (5). Unattacked by prolonged contact w. KMnO ₄ . (5). Fum. HBr g. an amyl bromide, b.p. ₄₅ :118.5-9.5°, d ₂₀ ⁴ =1.217, n _D ²⁰ =1.443 wh. loses HBr, when htd. w. H ₂ O to 100°, g. pentene-(2). (5). Unattacked by Br in diffused light. (5). In direct light and in sealed tube rapidly attacked by Br. (5).
7	36-7 ^{4,6}	0.695(18/4) ⁴	1.385(18) ⁴	<u>Methyl-cyclobutane, C₅H₁₀.</u> -- Odor like HCCl ₃ . (4).
9	49.5-50.5 ^{7,8,9}	0.751(20/4) ^{8,9}	1.404(20) ^{8,9}	<u>Cyclopentane, C₅H₁₀.</u> -- C.S.T. in PhNH ₂ :18°(7,10). HNO ₃ g. nitro-cyclopentane and glutaric ac. (11). Htg. w. Al(NO ₃) ₃ in a sealed tube g. C ₅ H ₉ NO ₂ , colorless, charact. odor of sec. nitro cpds., b.p. ₄₀ =90-1°, d ₂₃ ⁸ :1.078; n ₂₃ ⁸ =1.452. (12).
11	52.5-3.5 ¹³	0.695(20/0) ¹³	1.387 ¹³	<u>1,1,2-Trimethyl cyclopropane, C₆H₁₂.</u> - Only slowly affected by alk. KMnO ₄ . (13). Easily disvd. by HNO ₃ (d=1.52) w. evoln.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
13	¹⁴ 65-7 7,15,16,	¹⁴ 0.695(18/4)	¹⁴ 1.395(18)	<u>1,2,3-Trimethyl-cyclopropane, C₆H₁₂.</u>
15	^{15,16,17} 71.5-25	^{15,16,17} 0.749(20/4)	^{15,16,17} 1.410(20)	<u>Methyl-cyclopentane, C₆H₁₂.</u> C.S.T. in PhNH ₂ :35°. (7,10) Al(NO ₃) ₃ in a sealed tube g. 1-nitro-1-methyl-cyclopentane + 1-nitro-2-methyl cyclopentane. (20). HNO ₃ g. either these cpds. or oxid. it to glutaric ac., succinic ac., AcOH, + HCOOH. (15,17,19,21,22).
17	²³ 72-3	²³ 0.745(20/4)	²³ 1.408(20)	<u>Ethyl-cyclobutane, C₆H₁₂.</u> Oxid. w. HNO ₃ g. succinic ac., (23), C ₂ H ₂ O ₄ . (24). Unaffected by KMnO ₄ , H ₂ SO ₄ , HBr. (24). ²⁶
19	^{25,26} 79-80	²⁵ 0.818(20/4)	²⁵ 1.432(n_{20})	<u>Bicyclo-[0,1,3]-hexane, C₆H₁₀.</u>
21	²⁷ 79.5- 80.5	²⁷ 0.825(19/4)	²⁷ 1.448(19)	<u>Bicyclo-(0,2,2)-hexane, C₆H₁₀.</u> -- Easily oxid. by KMnO ₄ . (27). Adds Br g. the liquid dibromide. (27). EtOH w. conc. H ₂ SO ₄ g. neither red-violet nor blue color. (27).
23	^{28,29,30} ^{31,32} 80.5- 1.5	²⁸ 0.783(15/4 vac.)	²⁸ 1.429(15)	<u>Cyclohexane, * C₆H₁₂.</u> -- M.P.:6.4°. (28,30,31,32). C.S.T. in PhNH ₂ :51°. (7,10,33). C.S.T. in MeOH:49.1°. (34). Passed over finely divided Ni at 270-80° g. C ₆ H ₆ +H ₂ . (35). Stable to KMnO ₄ . (36). HNO ₃ g. either nitro- cyclohexane or adipic ac., succinic ac., + glutaric ac. (37,38,39). Long htg. w. Br at 150-200° g. 1,2,4,5-tetrabrombenzene. (31).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
25	80.5- ⁴⁰ 1.5	0.710(20/0) ⁴⁰	1.393(n_{20}) ⁴⁰	<u>1-Methyl-2-isopropyl-cyclopropane, C₇H₁₄.</u> -- Stable to KMnO ₄ .(40). Only slowly unites w. Br. (40). Violently attacked by HNO ₃ g. a heavy oil. (40).
27	87-8 ⁴¹	0.755(20/0) ⁴¹	1.413(20) ⁴¹	<u>1,1-Dimethyl-cyclopentane, C₇H₁₄.</u> -- Unattacked by Br, HNO ₃ (conc.), + aq. KMnO ₄ . (42).
29	^{44,45} 90.5-1.5	0.747(20) ⁴⁵	1.408(21) ⁴⁵	<u>1,3-Dimethyl-cyclopentane, C₇H₁₄.</u> -- C.S.T. in PhNH ₂ :48.8°. (45).- Absorbs O spontaneously even at pressures of 60mm., final products:5-methyl-hexanone(2)+4-methyl-hexanone(2). (45,46).
31	^{41,43} 92-3	0.753(20/0) ^{41,43}	1.413(20) ^{41,43}	<u>1,2-Dimethyl-cyclopentane, C₇H₁₄.</u> --
33	⁴⁷ 100-1	0.744(19/4) ⁴⁷	1.412(19) ⁴⁷	<u>n-Propyl-cyclobutane, C₇H₁₄</u> Does not decolorize alk. KMnO ₄ or Br. (47). Slowly dissolves in H ₂ SO ₄ ctg. 10% SO ₃ . (47).
35	^{7,8,28,48,49,50} 100-1	0.773(15/4) ²⁸	1.425(15) ^{28,51}	<u>Methyl-cyclohexane, C₇H₁₄</u> C.S.T. in PhNH ₂ :41°. (7,10) HNO ₃ (d:1.2) or Al(NO ₃) ₃ g. prim. + sec. cpds. septd. by sol. in aq.KOH; 1-nitro-1-methyl-cyclohexane, b.p. ₄₀ =109-10°, d ₂₀ ²⁰ =1.038, n ₂₀ =1.458, wh. w. HNO ₃ g. succinic ac., or wh. w. Sn+HCl g. 1-amino-1-methyl-cyclohexane, b.p. ₇₄₄ =143°, d ₁₉ ¹⁹ =0.857, n ₁₉ ¹⁹ =1.454, whose benzoyl deriv. m.p.:100.5-101°; this nitro cpd. sept'd. frm. its isomers by greater sol. in dil. aq. NaOH.(52). Dil. HNO ₃ nitrates + oxid., g. chiefly succinic ac. (53). Un-

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
37	100.5 ⁸ - 1.5	0.761(20/4 ⁸ vac.)	1.418(20) ⁸	attacked by HNO ₃ -H ₂ SO ₄ at 80°. (53). Br in presence of AlBr ₃ g. pentabromtoluene. (53,54). <u>Ethyl-cyclopentane, C₇H₁₄.</u>
39	108.5 ¹³ - 9.5	0.738(20/0) ¹³	1.410 ¹³	<u>1-Methyl-1,2-diethyl-cyclopropane, C₈H₁₆.</u> -- Reacts w. alk. KMnO ₄ noticeably only after 4-5 hrs. (13). Not vigorously reactive w. Br in AcOH soln. (13).
41	110-11 ⁵⁵	0.740(20/4) ⁵⁵	1.409 ⁵⁵	<u>1-Methyl-2-isobutyl-cyclopropane, C₈H₁₆.</u>
43	56, 57, 58 59 113-4	0.773(18/4) ⁵⁶	1.424(18) ⁵⁶	<u>1,1,2-Trimethyl-cyclopentane, C₈H₁₆.</u> -- Odor like camphor. (57). Oxid. w. dil. HNO ₃ g. α,α-dimethyl-glutaric ac. (57).
45	114-5 ⁵⁶	0.769(19/4) ⁵⁶	1.423(19) ⁵⁶	<u>1,2,3-Trimethyl-cyclopentane, C₈H₁₆.</u> -- Camphor-like odor. (57). Oxid. w. HNO ₃ g. C ₂ H ₂ O ₄ . (57).
47	114.5- ⁶⁰ 5.5	0.813(20/4) ⁶⁰		<u>3,5-Dimethyl-bicyclo[0,1,3]hexane.</u>
49	115.6 ⁵⁵	0.770(20/4) ⁵⁵	1.422 ⁵⁵	<u>1,1,3-Trimethyl-cyclopentane, C₈H₁₆.</u>
51	55, 61 115-6	0.813(20/4) ⁶¹	1.439(20) ⁶¹	<u>3,3-Dimethyl-bicyclo[0,1,3]-hexane, C₈H₁₄.</u> Readily reacts w. HNO ₃ . (55). Unattacked by H ₂ SO ₄ . (55).
53	62, 63 118-9	0.811(20/4) ⁶³	1.445(20) ⁶³	<u>Cycloheptane, C₇H₁₄.</u> -- M.P.: (-12°). (63). Unattacked by cold conc. HNO ₃ . (64). Htg. w. HNO ₃ (d:1.4) in sealed tube at 100° g. pimelic ac. + other acids. (64). Br + a little AlBr ₃ in a sealed tube g. pentabromtoluene. (65).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
55	^{8,66,67} 118.5- 9.5	^{66,67} 0.772(20/4)	^{8,66,67} 1.425(20)	<u>trans-1,3-Dimethyl-cyclohexane, C₈H₁₆.</u> - See No. 63.
57	^{8,67,70} 118.5- 9.5	^{8,67} 0.769(20/4)	^{8,67} 1.421(20)	<u>trans-1,4-Dimethyl-cyclohexane, C₈H₁₆.</u> -- See No. 65.
59	^{72,73} 118.5- 2.0	⁷² 0.782(20/4)	⁷² 1.434(18)	<u>1,1-Dimethyl-cyclohexane, C₈H₁₆.</u> -- Does not dehydrogenate at 300° w. most active platinized asbestos remains completely unchanged. (72). Odor like geraniums. (73).
61	⁷⁴ 120.5- 1.5	⁷⁴ 0.767(16/4)	⁷⁴ 1.421(16)	<u>1-Methyl-3-ethyl-cyclopentane, C₈H₁₆.</u> -- Active form shows [α] _D =+4.34°. (74).
63	^{7,8,66,67} 121-2	^{8,66,67} 0.774(20/4)	^{8,66,67} 1.427(20)	<u>cis-1,3-Dimethyl-cyclohexane, C₈H₁₆.</u> -- See No. 55. C.S.T. in PhNH ₂ : 49.7°. (7,10). Conc. HNO ₃ -H ₂ SO ₄ does not attack cold, (69), warming g. trinitro-m-xylene. (68,69). Stable to KMnO ₄ . (69). Br + a little AlBr ₃ g. tetrabrom-m-xylene. (69)g
65	^{8,67} 121-2	⁶⁷ 0.773(20/4)	^{8,67} 1.423(20)	<u>cis-1,4-Dimethyl-cyclohexane, C₈H₁₆.</u> -- See No. 57. C.S.T. in PhNH ₂ =48°. (7). Odor like fennel oil. (32). Easily sol. in HNO ₃ -H ₂ SO ₄ by gentle warming. (71). Br in presence of AlBr ₃ g. tetrabrom-p-xylene. (71).
67	^{8,67} 124-5	^{8,67} 0.780(20/4)	^{8,67} 1.430(20)	<u>trans-1,2-Dimethyl-cyclohexane, C₈H₁₆.</u> -- See No. 73.
69	⁷⁵ 124-5			<u>1-Methyl-2-ethyl-cyclopentane, C₈H₁₆.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
71	125-6 ⁷⁶			^{1,1,3,3} ^{2,4} <u>2,2,4,4-Tetramethyl-1,3-diethyl-cyclobutane, C₁₂H₂₄</u> Stable to cold KMnO ₄ , cold conc. H ₂ SO ₄ , + Br. (76). The reported b.p. of this hy. is probably considerably in error. -R.L.W.
73	^{8,87} 126.5- 7.5	0.786(20/4) ^{8,87}	1.431(20) ^{8,87}	<u>cis-1,2-Dimethyl-cyclohexane, C₈H₁₆</u> . -- See No. 67. C.S.T. in PhNH ₂ :42.1° (7).
75	128-9 ⁸	0.772(20/4 ⁸ vac.)	1.425(20) ⁸	<u>Isopropyl-cyclopentane, C₈H₁₆</u> .
77	⁸ 129.5- 30.5	0.772(20/4 ⁸ vac.)	1.425(20) ⁸	<u>n-Propyl-cyclopentane, C₈H₁₆</u> .
79	^{8,32} 129.5- 30.5	0.784(20/4 ⁸ vac.)	1.433(20) ⁸	<u>Ethyl-cyclohexane, C₈H₁₆</u> .
81	132-3 ⁷⁷	0.797(20/4) ⁷⁷	1.435(20) ⁷⁷	<u>1,4-Dimethyl-s-spiroheptane, C₉H₁₆</u> . -- By polymerization of allene, and then hydrogenation. (77).
83	132-4 ⁷⁸	0.773(19) ⁷⁸	1.425(19) ⁷⁸	<u>1-Methyl-3-isopropyl-cyclopentane, C₉H₁₈</u>
85	⁷⁹ 132.5- 3.5	0.782(14) ⁷⁹		<u>1,1,2,3-Tetramethyl-cyclopentane, C₉H₁₈</u> .
87	134-5 ⁸⁰	0.798(18/4) ⁸⁰	1.439(18) ⁸⁰	<u>Methyl-cycloheptane, C₈H₁₆</u> .
89	^{81,82} 137-8	0.790(20/4) ⁸¹	1.436(20) ⁸¹	<u>1,1,3-Trimethyl-cyclohexane, C₉H₁₈</u> .
91	138-9 ⁸	0.772(20/4 ⁸ vac.)	1.427(20) ⁸	<u>trans-1,3,5-Trimethyl-cyclohexane, C₉H₁₈</u> . -- See No. 99.
93	138-9 ⁸³	0.882(21/4) ⁸³	1.466(20) ⁸³	<u>Bicyclo-[0,3,3]-octane, C₈H₁₄</u> .

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
95	138.5- ^s 9.5	0.781(20/4 ^s vac.)	1.431(20) ^s	<u>trans-1,2,4-Trimethyl-cyclohexane, C₉H₁₈.</u> -- See Nos. 103, 105, 119.
97	139.5- ^{s3} 40.5	0.860(20/4) ^{s3}	1.461(20) ^{s3}	<u>Bicyclo-[0,x,x]-octane, C₈H₁₄.</u> -- Stable to KMnO ₄ , colored by a little Br. (s3).
99	140-1 ^s	0.777(20/4 ^s vac.)	1.430(20) ^s	<u>cis-1,3,5-Trimethyl-cyclohexane, C₉H₁₈.</u> -- See No. 91.
101	140.5- ^{s6} 1.5	0.822(18.5) ^{s6}	1.447(18.5) ^{s6}	<u>2,6,6-Trimethyl-bicyclo-[0,1,3]-hexane, C₉H₁₆.</u> -- Does not decolorize KMnO ₄ . (s6). Dissolves in fuming HNO ₃ w. evolution of ht. (s6).
103	141-2 ^s	0.785(20/4 ^s vac.)	1.433(20) ^s	<u>cis-1,2,4-Trimethyl-cyclohexane, C₉H₁₈.</u> -- See Nos. 95,105. Cf. especially No. 119.
105	141.5- ^{s7} 2.5	0.786(20/4) ^{s7}	1.432(20) ^{s7}	<u>1,2,4-Trimethyl-cyclohexane, C₉H₁₈.</u> -- See Nos. 95,103,119. Br+ AlBr ₃ g. tribrom-pseudo-cumene. (s7). HNO ₃ -H ₂ SO ₄ g. a little trinitro-pseudo-cumene. (s7).
107	142-3.5 ^s	0.791(20/4 ^s vac.)	1.436(20) ^s	<u>trans-1,2,3-Trimethyl-cyclohexane, C₉H₁₈.</u> -- See No. 117.
109	142-3 ^{s6} 90,91,92	0.779(20/0) ^{s6}	1.428(20) ^{s6}	<u>1-Methyl-2-isopropyl-cyclopentane, C₉H₁₈.</u>
111	93,207 142-3	90,91,92 0.859(20/4)	90,91 1.448(20)	<u>2,2,6-Trimethyl-3,6-endo-methylene-bicyclo-[0,1,3]-hexane, or 1-Methyl-3,6-endo-(dimethyl-methylene)-bicyclo-[0,1,3]-hexane, "Cyclo-fenchane", "β-Pinolene", C₁₀H₁₆.</u> -- At (-15°) in dry ether (s1) g. a hydrochloride, m.p.= 26-8°. (90,91,93). KMnO ₄ g. isophthalic ac. (90).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
113	142.5- ⁸⁹ 3.5	0.855(20/4) ⁸⁹	1.456(20) ⁸⁹	<p>Hydrobromide, m.p.=4°, b.p.₁₈=92-3°, d²⁰:1.239; n_D²⁰:1.506. (9⁴)</p> <p><u>2,2-Dimethyl-bicyclo-[1,2,2]-heptane, Camphenilane, C₉H₁₆.</u> -- M.P.=15-16°. (89) 1 vol. hy. + 3 vols. HNO₃ (d=1.075) htd. 16 hrs. at 140-5° g.; fm. 180 g. camphenilane 22 g. α-nitro-camphenilane + 3 g. 2-nitro-camphenilane + cis-apo-fencho-camphoric ac. (in ac. layer). (89). α-Nitro camphenilane, b.p.₂₀=124.5-5°, m.p.=18-20°; wh. w. Zn+HCl g. α-camphenyl amine, b.p.:189-9.5, m.p.=10-14°, d²⁰=0.928, n₂₀=1.482, whose benzoyl deriv., m.p.:141-3°; + wh. w. oxid. g. α-iso-camphenilone, b.p.₇₄₆:196-6.5°, m.p.=63-5°, whose semi-carbazone, m.p.=63-5°, whs semicarbazone, m.p.:192-3°. (89). 2-nitro-camphenilane m.p.:89-91°. (89). Cis-apo-camphoric ac., m.p.:144.5-5°, 100 cc. H₂O at 19° dissolves 0.72 g.; whose anhydride, m.p.:136-7°, whose anilide, m.p.:155-7° by htg. ac. w. XS. PhNH₂; whose dianilide, m.p.:148-50° by ac. + PCl₅, then poured into PhNH₂ in Et₂O. (89). d²⁰=1.239; n_D²⁰=1.506. (9⁴).</p>
115	143-4 ⁹⁴	0.854(20/4) ⁹⁴	1.454(20) ⁹⁴	<p><u>1-Methyl-3-isopropyl-cyclopentane, "β-Apofenchanane", "β-Fencho-camphorane", C₉H₁₈.</u> - Cf. Div. A, Sect. 1, No. 27. - M. p.: 17-17.5°.</p>
117	144-6 ⁸	0.793(20/4 ⁸ vac.)	1.437(20) ⁸	<p><u>cis-1,2,3-Trimethyl-cyclohexane, C₉H₁₈.</u> -- See No. 107.</p>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
119	^{66,67} 145-6	0.790(20/4) ⁶⁷	1.433(20) ⁶⁷	<u>1^c,2^c,4^c-Trimethyl-cyclohexane, C₉H₁₈.</u> -- Does not decolorize Br. (66). See Nos. 95,105. Cf. especially No. 103.
121	abt. 145 50-2 ⁸⁴ (17mm.)	0.856(21) ⁸⁴	1.496(21) ⁸⁴	<u>Tricyclo-octane, C₈H₁₂.</u> -- Adds no Br. Polymerizes by long htg. under pressure. (85).
123	⁹⁶ 145-6.5	0.760(20/4) ⁹⁶	1.420(20) ⁹⁶	<u>1,1,2-Trimethyl-3-isopropyl-cyclobutane, C₁₀H₂₀.</u> Unaffected by KMNO ₄ . (96).
125	⁹⁷ 146-8	0.786(16) ⁹⁷	1.434(16) ⁹⁷	<u>1,2-Dimethyl-3-isopropyl-cyclopentane, C₁₀H₂₀.</u> --
127	¹⁰⁰ 146-7	0.788(15/4) ¹⁰⁰	1.435(15) ¹⁰⁰	<u>1-Methyl-4-ethyl-cyclohexane, C₉H₁₈.</u>
129	¹⁰¹ 146-8.5	0.784(20/0) ¹⁰¹	1.431 ¹⁰¹	<u>1,2-Diethyl-cyclopentane and 1,3-Diethyl-cyclopentane, mixture of both, C₉H₁₈.</u> -- Reacts w. HNO ₃ (d=1.52) w. evolution of ht. (101).
131	^{32,102} 146-8	0.812(0/4) ³²		<u>Isopropyl-cyclohexane, C₉H₁₈.</u> of #155
133	¹⁰³ 147.5-9	0.785(20/0) ¹⁰³	1.430(20) ¹⁰³	<u>1,3-Diethyl-cyclopentane, C₉H₁₈.</u>
135	⁸ 148-9	0.780(20/4 ⁸ vac.)	1.430(20) ⁸	<u>Isobutyl-cyclopentane, C₉H₁₈.</u>
137	⁷⁴ 148.5- 9.5	0.790(17/4) ⁷⁴	1.435(17) ⁷⁴	<u>1-Methyl-3-ethyl-cyclohexane, C₉H₁₈.</u> -- L-form shows $[\alpha]_D = -2.9^\circ$. (74).
139	⁹⁵ 149-50	0.86(22/22) ⁹⁵	1.461 ⁹⁵	<u>2,6,6-Trimethyl-bicyclo-[1,1,3]-heptane, "Nopinane" C₁₀H₁₈.</u>
141	¹⁰⁴ 149-51	0.875(20) ¹⁰⁴	1.469 ¹⁰⁴	<u>2-Methyl-bicyclo-[1,2,3]-octane, C₉H₁₆.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
143	^{99,99} 149.5- 50.5	0.839(20/4) ⁹⁸	1.459(20) ⁹⁸	<u>Cyclooctane, C₈H₁₆.</u> -- M.P.:14°.(98). Odor like camphor. (99). Htg. w. HNO ₃ g. much suberic ac. (99).
145	⁹⁷ 150-2	0.781(17) ⁹⁷	1.432(17) ⁹⁷	<u>1-Methyl-2-3-diisopropyl-</u> <u>cyclopentane, C₁₂H₂₄.</u> -- The reptd. b.p. of this hy. is probably consider- ably in error. - R.L.W.
147	^{105,106} 150-2	0.784(20) ¹⁰⁵	1.432(20) ¹⁰⁵	<u>1-Methyl-2-ethyl-cyclo-</u> <u>hexane, C₉H₁₈.</u>
149	¹⁰¹ 150.5- 1.5	0.803(20/0) ¹⁰¹	1.439 ¹⁰¹	<u>1,1-Diethyl-cyclopentane,</u> <u>C₉H₁₈.</u> -- Does not react. w. fuming HNO ₃ . (101).
151	¹⁰³ 150.5- 1.5	0.795(19/0) ¹⁰³	1.433(19) ¹⁰³	<u>1-Ethyl-1-n-propyl-cyclo-</u> <u>butane, C₉H₁₈.</u>
153	¹¹⁵ 151-2	^{115,116} 0.833(20/4)	^{115,116} 1.446(20)	<u>1,3,3-Trimethyl-bicyclo-</u> <u>[1,2,2]-heptane, (117)</u> <u>"Fenchane", C₁₀H₁₈.</u>
155	⁸ 152-3	0.790(20/4 ⁸ vac.)	1.436(20) ⁸	<u>Isopropyl-cyclohexane,</u> <u>C₉H₁₈.</u> <i>cf #131</i>
157	¹⁰⁷ 153-4			<u>1,2-Diethyl-cycloheptane,</u> <u>C₁₁H₂₂.</u>
159	¹⁰⁸ 153-5.5	0.810(0/4) ¹⁰⁸		<u>Sec.-Butyl-cyclopentane,</u> <u>C₉H₁₈.</u>
161	^{8,109} 154.5- 5.5	0.790(20/4 ⁸ vac.)	1.436(20) ⁸	<u>n-Propyl-cyclohexane,</u> <u>C₉H₁₈.</u> -- Fum. HNO ₃ at 0° has no action. (109). HNO ₃ (d=1.53) dissolves it slowly at ord. temp.(109). Br + AlBr ₃ g. small amt. of C ₉ H ₉ Br ₃ , m.p.=230°.(110).
163	⁷⁷ 155-6	0.773(20/4) ⁷⁷	1.424(20) ⁷⁷	<u>1,2-Dimethyl-3,4-diethyl-</u> <u>cyclobutane, C₁₀H₂₀.</u>
165	⁹⁵ 156.5- 7.5	0.816(22/21) ⁹⁵	1.441 ⁹⁵	<u>Tanacetane, C₁₀H₁₈.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
167	^{111,112} ¹¹³ 157-8	0.817(20/0) ¹¹³	1.441(20) ¹¹³	<u>4-Methyl-1-isopropyl-bicyclo-[0,1,3]-hexane</u> , "Thujane", "Sabinane", C ₁₀ H ₁₈ . -- [α] _D ²⁰ = 69.29°. (113). Odor of turpentine. (112). Gives an unstable bromide w. Br. (112). Conc. HBr g. a bromide wh. on boiling w. K ₂ CO ₃ g. an unsat'd. hy., C ₁₀ H ₁₆ , b.p. = 162-5°, d ₂₀ ²⁰ : 0.814, n _D ²⁰ : 1.451. (112) ²⁰ . Dibromide + alc. KOH C ₁₀ H ₁₆ , unsat'd., b.p.: 162-4°, d ₂₀ ²⁰ : 0.816, n _D ²⁰ : 1.452, [α] _D ²⁰ = 23.62°. (112).
169	¹¹⁴ 157-8			<u>1,2,3-Trimethyl-4-isopropyl-cyclopentane</u> , C ₁₁ H ₂₂ .
171	⁹⁶ 157-8.5	0.776(20/4) ⁹⁶	1.428(20) ⁹⁶	<u>1,2-Diisopropyl-cyclobutane</u> , C ₁₀ H ₂₀ .
173	⁸¹ 160.5- 1.5	0.785(20/4) ⁸¹	1.434(20) ⁸¹	<u>1,2,4,5-Tetramethyl-cyclohexane</u> , C ₁₀ H ₂₀ . -- See No. 197, 211.
175	⁶⁷ 161-2	0.792(20/4) ⁶⁷	1.439(20) ⁶⁷	<u>trans-1-Methyl-4-isopropyl-cyclohexane</u> , "trans-p-Menthane", C ₁₀ H ₂₀ . -- See No. 201, 203.
177	⁸ 162-4	0.814(20/4 ⁸ vac.)	1.447(20) ⁸	<u>trans-1,2,3,5-Tetramethyl-cyclohexane</u> , "trans-Hexahydroisodurene", C ₁₀ H ₂₀ . -- See No. 199.
179	⁷⁷ 163-4.5	0.799(20/4) ⁷⁷	1.441(20) ⁷⁷	<u>1,3-Dimethyl-3-ethyl-cyclohexane</u> , C ₁₀ H ₂₀ .
181	^{119,125,} ¹²⁶ 163.5- 4.5	^{119,125} 0.879(16/4)	¹²⁵ 1.471(16)	<u>Bicyclo-[0,3,4]-nonane</u> , "Indane", Indene-octahydride", C ₉ H ₁₆ .
183	^{127,128} 163.5- 4.5	^{127,128} 0.858(20/4)	^{127,128} 1.459(20)	<u>2,7,7-Trimethyl-bicyclo-[1,2,2]-heptane</u> , "Isobornylane", C ₁₀ H ₁₈ . -- 10 g. htd. w. 30 ccs. HNO ₃ (d:1.075) for 6 hrs. at

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
				130-5° in a sealed tube g. ter.-nitro-isobornylane, b.p. ¹¹² :112-3°, d_{20}^{20} =1.058, n_D^{20} =1.481, $[\alpha]_D^{20}$ =-26.7° (in alc.); wh. w. Zn+HCl g. the amine, b.p. ¹⁷⁵ =199-9.5°, d_{20}^{20} =0.917, n_D^{20} =1.480, $[\alpha]_D^{20}$ =6.2° (in alc.) whose benzyl deriv., m.p.:134-5°. (127,128). HNO ₃ also g. isobornylone, b.p. ⁷³² =197.5-8°, d_{20}^{20} =0.968, n_{20} =1.469, whose semicarbazone, m.p.:174-5° (127,128). HNO ₃ also g. very small amt. of sec.-nitro-isobornylane, b.p. ¹¹² =89-90°, d_{20}^{20} =1.0466, n_{20} =1.481. (127,14). HNO ₃ also g. some cis-apocamphoric ac. (127,128).
185	164-5 ¹¹⁸	0.815(20/0) ¹¹⁸		Ethyl-cycloheptane, ^{C₉H₁₈} C₉H₁₈ . Red fum. HNO ₃ g. pimelic ac., oxalic ac., + AcOH. (118).
187	164.5-5.5 ¹²⁹	0.855(20/4) ¹²⁹	1.461(20) ¹²⁹	2,7,7-Trimethyl-bicyclo-[1,1,3]-heptane, (129) "Pinocamphane", "Dihydro-d-pinene", C ₁₀ H ₁₈ .
189	165-6 ⁹⁵	0.838(20/20) ⁹⁵	1.458 ⁹⁵	3,7,7-Trimethyl-bicyclo-[0,1,4]-heptane, (95), "Carane", C ₁₀ H ₁₈ .
191	166-7 ¹³⁰	0.797(24/0) ¹³⁰	1.44 ¹³⁰	dl-1-Methyl-3-isopropyl-cyclohexane, C ₁₀ H ₂₀ .
193	32,131 166-7	0.861(15/15) ¹³¹		2,6,6-Trimethyl-bicyclo-[1,1,3]-heptane, (129), "Pinane", "Dihydro-l-pinene", C ₁₀ H ₁₈ . -- D-form shows $[\alpha]_D^{20}$ =+22.7°. (131). L-form shows $[\alpha]_D^{20}$ =-21.3°. (131). D-form freezes at abt. -45°. (131)
195	166-7 ¹³²	0.831(16/4) ¹³²	1.456(16) ¹³²	ter-Butyl-cyclohexane, C ₁₀ H ₂₀ . --

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
197	166-8 ^s	0.810(20/4 ^s vac.)	1.444(20) ^s	<u>trans-1,2,4,5-Tetramethyl-cyclohexane, "trans-Hexahydrodurene", C₁₀H₂₀.</u> -- See Nos. 173, 211.
199	168-70 ^s	0.817(20/4) ^s	1.448(20) ^s	<u>cis-1,2,3,5-Tetramethyl-cyclohexane, "cis-Hexahydroisodurene", C₁₀H₂₀.</u> -- See No. 177.
201	168.5- ⁶⁷ 9.5	0.816(20/4) ⁶⁷	1.451(20) ⁶⁷	<u>cis-1-Methyl-4-isopropyl-cyclohexane, C₁₀H₂₀.</u> -- See Nos. 175, 203.
	32, 133, 134, 135, 136			
203	169-71 ¹³⁶	0.790(20) ¹³⁶	1.438(21) ¹³⁴	<u>1-Methyl-4-isopropyl-cyclohexane, *p-Menthane, C₁₀H₂₀.</u> -- See Nos. 175, 201.
205	169-70 ^s	0.795(20/4 ^s vac.)	1.439(20) ^s	<u>Isobutyl-cyclohexane, C₁₀H₂₀.</u>
207	169-71 ¹³⁸	0.796(22/4) ¹³⁸	1.439(20) ¹³⁸	<u>1,3-Diethyl-cyclohexane, C₁₀H₂₀.</u>
209	170-2 ¹⁴¹	0.773(16/4) ¹⁴¹	1.433(16) ¹⁴¹	<u>Cyclononane, C₉H₁₈.</u> -- Scarcely reacts w. KMnO ₄ . (141). Br substitutes. (141).
211	8, 137 171-2	0.812(20/4 ^{s, 137} vac.)	1.446(20) ^{s, 137}	<u>cis-1,2,4,5-Tetramethyl-cyclohexane, "cis-Hexahydrodurene", C₁₀H₂₀.</u> -- See Nos. 197, 173.
213	130, 140 171-2	0.814(21/0) ¹³⁰	1.447 ¹³⁰	<u>1-Methyl-2-isopropyl-cyclohexane, C₁₀H₂₀.</u>
215	139 171.5- 3	0.877(17/4) ¹³⁹	1.468(17) ¹³⁹	<u>2,2,-Dimethyl-bicyclo-[1,2,3]-octane, "Dihydro-endocamphene", C₁₀H₁₈.</u>
217	142 176-7	0.804(18) ¹⁴²	1.444(18) ¹⁴²	<u>n-Butyl-cyclohexane, C₁₀H₂₀.</u>
219	143 176.5- 8.5	0.842(20/4) ¹⁴³	1.453(20) ¹⁴³	<u>1-Methyl-bicyclo-[1,3,3]-nonane, C₁₀H₁₈.</u>
221	144 183-4	0.818(18/4) ¹⁴⁴	1.450(18) ¹⁴⁴	<u>n-Propyl-cycloheptane, C₁₀H₂₀.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
223	183-4 ¹⁴⁵	0.885(20) ¹⁴⁵	1.469 ¹⁴⁵	<u>1,2-Dimethyl-2-ethyl-3,6-endomethylene-bicyclo-[0,1,3]-hexane(?) "Nortri-cycloeksantalene", C₁₁H₁₈.</u>
225	148,149 150 187-8	148,149, 150 0.876(20/4)	148,149, 150 1.471(20)	<u>cis-Bicyclo-[0,4,4]-decane,* "cis-Decahydro-naphthalene", C₁₀H₁₈.</u> -- See No. 231.
227	146,147 188-9	0.860(20/4) ¹⁴⁶	1.465(20) ¹⁴⁶	<u>Dicyclopentyl, C₁₀H₁₈.</u> -- Indifferent to Br (146). Slowly oxid. by KMnO ₄ . (146). Does not undergo catalytic dehydrogenation. (146).
229	191-2 ^{132*}	0.823(16/4) ¹³²	1.454(16) ¹³²	<u>ter-Amyl-cyclohexane,* C₁₁H₂₂.</u>
231	148,149 150 193-4	148,149, 150 0.894(20/4)	148,149, 150 1.480(20)	<u>trans-Bicyclo-[0,4,4]-decane,* "trans-Decahydro-naphthalene," C₁₀H₁₈.</u> -- See No. 225. KMnO ₄ in acid soln. g. only phthalic ac. (151). 100 g. refluxed 6 hrs. w. 250 g. HNO ₃ (d=1.2) g. 17 g. NO ₂ cpds. ctg. 9-nitro-decalin(I), 9,10-dinitro-decalin(II), 1-nitro-decalin(III). (152) (I), b.p. ₂ :96-7°, d ₂₀ ^o =1.085, n ₂₀ ^o =1.494; wh. w. Zn dust + AcOH g. the amine, b.p. ₁₅ =98°, d ₂₀ ^o =0.9444, n ₂₀ ^o =1.493 whose benzoyl deriv. m.p.=148-9°. (152). (II), m.p.=164°(dec.). (152). (III) light yellow liquid, b.p. _{1.4} =108-9°, d ₂₀ ^o =1.083, n ₂₀ ^o =1.498, ⁴ sol. in alkalis; 7 g. in cold alkali w. calcd. amt. of KMnO ₄ g. 3 g. of a decalone, b.p. ₁₁ =107°, d ₂₀ ^o =0.993, n ₂₀ ^o =1.488, whose semicarbazone after 4 xtains. fm. EtOH, m.p.=

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
233	193-5 ³⁸	0.823(0/4) ³⁸		205-8°. (152). <u>1,3-Dimethyl-5-isobutyl-cyclohexane, C₁₂H₂₄.</u>
235	195- ¹⁵³ 200.5			<u>1,3-Dimethyl-bicyclo-[1,3,3]-nonane, C₁₁H₂₀.</u>
237	Abt. 203 77-8 ⁷⁷ (13.5 mm.)	0.868(20/4) ⁷⁷	1.468(20) ⁷⁷	<u>C₁₂H₂₀.</u>
239	204-5 ¹⁰⁵	0.812(17) ¹⁰⁵	1.454(17) ¹⁰⁵	<u>1-Methyl-2-isoamyl-cyclohexane, C₁₂H₂₄.</u>
241	206-7 ¹³²	0.837(16/4) ¹³²	1.467(16) ¹³²	<u>2-Methyl-2-cyclohexyl-pentane, C₁₂H₂₄.</u>
243	207-8 ¹³²	0.831(16/4) ¹³²	1.457(16) ¹³²	<u>3-Methyl-3-cyclohexyl-pentane, C₁₂H₂₄.</u>
245	207-8 ¹⁵⁵	0.878(21) ¹⁵⁵	1.473 ¹⁵⁵	<u>s-Spiroundecane, C₁₁H₂₀.</u>
247	208-9 ¹⁵⁴	0.815(20/0) ¹⁵⁴		<u>1-Methyl-3-ethyl-4-isopropyl-cyclohexane, C₁₂H₂₄.</u> --L-form shows [α] _D = -12.25°. (154).
249	abt. 210 106-7 ¹⁶³ (27 mm.)	0.804(17/4) ¹⁶³	1.446(17) ¹⁶³	<u>1,1,2,2-Tetramethyl-3,4-diisopropyl-cyclobutane, C₁₄H₂₈.</u>
251	213-5 ¹⁴⁶	0.848(20/4) ¹⁴⁶	1.458(20) ¹⁴⁶	<u>3,3'-Dimethyl-dicyclopentyl, C₁₂H₂₂.</u> -- See No. 253. Cannot be catalytically dehydrogenated. (146).
253	214-5 ¹⁵⁶	0.878(20/4) ¹⁵⁶	1.476(20) ¹⁵⁶	<u>3,3'-Dimethyl-dicyclopentyl, C₁₂H₂₂.</u> -- See No. 251. Boiling w. HNO ₃ (d=1.45) g. PrCO ₂ H.
255	220-2 ¹⁵⁸	0.881(20/4) ¹⁵⁸		<u>Dicyclohexyl, C₁₂H₂₂.</u> -- See Nos. 265, 273, 277.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
257	¹³² 220-1	0.830(16/4) ¹³²	1.458(16) ¹³²	<u>2,4-Dimethyl-2-cyclohexyl-pentane, C₁₃H₂₆.</u>
259	¹³² 222-3	0.839(16/4) ¹³²	1.466(16) ¹³²	<u>3-Ethyl-3-cyclohexyl-pentane, C₁₃H₂₆.</u>
261	¹³² 224-6	0.841(16/4) ¹³²	1.465(16) ¹³²	<u>3-Methyl-3-cyclohexyl-hexane, C₁₃H₂₆.</u>
263	¹⁵⁷ 225-7	0.881(21/4) ¹⁵⁷	1.477(21) ¹⁵⁷	<u>Cyclohexyl-cyclopentane, C₁₁H₂₀.</u>
265	^{158,159} 227.5- 8.5	0.880(20/4) ¹⁵⁸		<u>Dicyclohexyl, C₁₂H₂₂.</u> -- See Nos. 255, 273, 277.
267	¹⁶² 228-9			<u>4-Cyclohexyl-heptane, C₁₃H₂₆.</u>
269	^{164,165} 230-4	0.937(0) ¹⁶⁴		<u>2,10-Endoethylene-bicyclo-[0,4,4]-decane, "Acenaphthene-decahydride", C₁₂H₂₀</u>
271	¹⁶⁶ 232-3	0.864(20/4) ¹⁶⁶	1.466(20) ¹⁶⁶	<u>9-Methyl-3-isopropyl-bicyclo-[1,3,3]-nonane, C₁₃H₂₄.</u> -- Terpene-like odor. (166).
273	¹⁵⁸ 235.5- 7.5	0.882(20/4) ¹⁵⁸		<u>Dicyclohexyl, C₁₂H₂₂.</u> -- See Nos. 255, 265, 277.
275	abt. ²³⁷ 95-6 ⁷⁷ (13.5 mm.)	0.883(20/4) ⁷⁷	1.483(20) ⁷⁷	<u>C₁₂H₂₀.</u> -- Oxid. g. formic ac., C ₂ H ₂ O ₄ , + succinic ac. (77).
277	¹⁴⁶ 239-40	0.885(20/4) ¹⁴⁶	1.480(20) ¹⁴⁶	<u>Dicyclohexyl, C₁₂H₂₂.</u> -- See Nos. 255, 265, 273. M.P.=40: (160). Un- attacked by KMnO ₄ . (161). Slowly dissolves in fum. HNO ₃ (161). Unattacked by HNO ₃ -H ₂ SO ₄ . (161). Catalytically dehydrogen- ated to Ph ₂ . (146).
279	^{166,167,} ¹⁶⁸ 250-2	0.875(20/4) ¹⁶⁷	1.475(20) ¹⁶⁷	<u>Dicyclohexyl-methane, C₁₂H₂₄.</u> -- Pt. charcoal at 300° g. fluorene. (167)

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
281	^{164,170} 253-8	¹⁷⁰ 0.920(22)	¹⁷⁰ 1.486(22)	<u>2,2'-Endomethylene-dicyclohexyl, "Fluorene-dodecahydride", C₁₃H₂₂.</u>
283	abt.254 116-8 ⁷⁷ (23mm.)	⁷⁷ 0.838(20/4)	⁷⁷ 1.464(19)	<u>C₁₅H₂₈.</u>
285	¹⁷² 256-7	¹⁷² 0.927(20)	¹⁷² 1.511(40)	<u>1,1-Dicyclohexyl-ethane, C₁₄H₂₆.</u>
287	¹⁶⁹ abt.259 131-3.5 (20mm.)	¹⁶⁹ 0.906(20/4)	¹⁶⁹ 1.497(n)	<u>1-Methyl-2-cyclohexyl-cyclohexane, C₁₈H₃₄.</u>
289	¹⁷² 265-4			<u>1,2-Dicyclohexyl-ethane, C₁₃H₂₄.</u> -- See No. 299.
291	¹⁶¹ 264-5	¹⁶¹ 0.879(20/0)		<u>3,3'-Dimethyl-dicyclohexyl, C₁₄H₂₆.</u> -- L-form shows: $[\alpha]_D = -3^{\circ}44'$. (161).
293	¹³² abt.269 134-5 (30mm.)	¹³² 0.851(19/4)	¹³² 1.469(23)	<u>2,5-Dimethyl-2-cyclohexyl-hexane, C₁₄H₂₈.</u>
295	¹⁶⁵ 270-5	¹⁶⁵ 0.933(20/20)		<u>2,2'-Endoethylene-dicyclohexyl, "Phenanthrene-tetradecahydride", C₁₄H₂₄.</u> --M.P.=-3°. (165). H ₂ SO ₄ , HNO ₃ , Br, do not attack in the cold. (165). CrO ₃ + HAc attack it only w. difficulty on boiling. (165).
297	¹⁷² 270-1	¹⁷² 0.889(23/0)	¹⁷² 1.485	<u>1,1-Dicyclohexyl-propane, C₁₅H₂₈.</u>
299	^{172,174} 270-5	¹⁷² 0.884(18)	¹⁷² 1.480(18)	<u>1,2-Dicyclohexyl ethane, C₁₄H₂₆.</u> -- See No. 299.
301	¹⁷² 272-3	¹⁷² 0.873(21/0)	¹⁷² 1.479	<u>1,2-Dicyclohexyl-propane, C₁₅H₂₈.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
303	¹⁷⁸ 273-4	0.900(23/0) ¹⁷⁸	1.490 ¹⁷⁸	<u>2,2-Dicyclohexyl-propane, C₁₅H₂₈.</u>
305	¹⁷⁹ 276-8	0.908(18/0) ¹⁷⁹	1.50(n ₁₈) ¹⁷⁹	<u>1,2-Dicyclohexyl-butane, C₁₆H₃₀.</u>
307	¹⁷⁹ 278-9	0.891(15/0) ¹⁷⁹	1.492(15) ¹⁷⁹	<u>2-Methyl-1,1-Dicyclohexyl-propane, C₁₆H₃₀.</u>
309	¹³² abt.279 114-6 (13mm.)	0.855(19/4) ¹³²	1.475(23) ¹³²	<u>3-Ethyl-3-cyclohexyl-hexane, C₁₄H₂₈.</u>
311	¹³² abt.279 115-6 (13mm.)	0.848(19/4) ¹³²	1.472(19) ¹³²	<u>4-Methyl-4-cyclohexyl-heptane, C₁₄H₂₈.</u>
313	¹⁷⁹ 280-2	0.884(16/0) ¹⁷⁹	1.485(n ₁₆) ¹⁷⁹	<u>1,1-Dicyclohexyl-butane, C₁₆H₃₀.</u>
315	¹⁹¹ abt.281 118-20 (14mm.)	0.874(18) ¹⁹¹	1.474(18) ¹⁹¹	<u>Tetrahydrobetene^{ul}, C₁₅H₂₈.</u>
317	^{178,180} 289-90	0.870(21/0) ^{178,180}	1.474(24) ¹⁸⁰	<u>1,3-Dicyclohexyl-propane, C₁₅H₂₈. - M.P.=-17°.(180).</u>
319	¹⁸⁴ 290-1	0.894(21/0) ¹⁸⁴	1.489(21) ¹⁸⁴	<u>4,4-Dicyclohexyl-2-methyl-butane, C₁₇H₃₂.</u>
321	¹¹⁸ 290-1 (728mm.)	0.907(20/0) ¹¹⁸		<u>Dicycloheptyl, C₁₄H₂₈. --</u> Very slowly attacked by alk. KMnO ₄ +fum. HNO ₃ . (182). Stable to HNO ₃ -H ₂ SO ₄ . (182). Unattacked by conc. H ₂ SO ₄ at 100°. (118). Br in presence of AlBr ₃ g. pentabromtoluene (118).
323	¹⁷⁹ 290-2	0.884(19/0) ¹⁷⁹	1.484(19) ¹⁷⁹	<u>2-Methyl-1,3-dicyclohexyl-propane, C₁₆H₃₀.</u>
325	¹⁷⁶ abt.295 114-6 (10mm.)	0.845(20) ¹⁷⁶	1.462(n _D)	<u>Hexahydroelemene, C₁₅H₃₀.</u> L-form shows [α] _D =4.8°. (176).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
327	296-7 ¹⁸⁴	0.885 (21/0) ¹⁸⁴	1.843 ¹⁸⁴	<u>1,3-Dicyclohexyl-2-ethylpropane, C₁₇H₃₂.</u>
329	300-15 ¹⁸⁴			<u>1-Methyl-4'-isopropyl-2,2'endoethylene-dicyclohexyl, "Retenetetradecahydride", C₁₈H₃₂.</u>
331	abt. 301 ¹⁹³ 122-3 (12 mm.)	0.871 (20) ¹⁹³	1.470 ¹⁹³	<u>Tetrahydrocaryophyllene, C₁₅H₂₈.</u> -- D-form shows [α] _D ²⁰ = +3°.
333	185,195 abt. 301 115-6 (9mm.)	0.866 (20) ^{185,195}	1.469 ^{185,195}	<u>Tetrahydro-santalene, C₁₅H₂₈.</u> -- D-form shows [α] _D = 7°30'. (195). Un-attacked by O ₃ +alk. KMnO ₄ . (195).
335	abt. 302 ¹⁷⁷ 118-20 (10mm.)	0.840 (21) ¹⁷⁷	1.458 (20) ¹⁷⁷	<u>Tetrahydroferulene, C₁₅H₃₀</u> -- D-form shows [α] _D ²⁰ = 4.2°. (177).
337	304-6 ¹⁷⁹	0.877 (21/0) ¹⁷⁹	1.475 (21) ¹⁷⁹	<u>1,4-Dicyclohexyl-butane, C₁₆H₃₀.</u> -- M.P. = 9°. (179).
339	abt. 306 ¹⁹⁰ 130.5- 1.5 (15mm.)			<u>γ-Dihydrocaryophyllene, "Dihydroisocaryophyllene", C₁₅H₂₈.</u>
341	abt. 308 ¹³² 129-30 (13mm.)	0.838 (19/4) ¹³²	1.460 (23) ¹³²	<u>4-Ethyl-4-cyclohexylheptane, C₁₅H₂₈.</u>
343	abt. 308 ¹⁷⁵ 125-8 (12 mm.)	0.916 (20/4) ¹⁷⁵	1.514 (20) ¹⁷⁵	<u>Isocadinene, C₁₅H₂₄.</u>
345	abt. 310 ¹⁷⁷ 131-3 (14mm.)	0.825 (22) ¹⁷⁷	1.456 (22) ¹⁷⁷	<u>1-Methyl-4-isopropyl-2-isoamyl-cyclohexane, "Isoamylmenthane, C₁₅H₃₀</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
347	abt. ¹³² 310 120-1 (10 mm.)	0.872(19/4) ¹³²	1.487(23) ¹³²	<u>2,5-Dimethyl-5-cyclohexyl-heptane, C₁₅H₃₀.</u>
349	abt. ¹⁶⁹ 310 141-2.5 (20 mm.)	0.913(20/4) ¹⁶⁹	1.496(n) ¹⁶⁹	<u>2-Ethyl-dicyclohexyl, C₁₄H₂₈.</u>
351	311-2 ¹⁸⁴	0.872(21/0) ¹⁸⁴	^{181,184} 1.479(21)	<u>1,5-Dicyclohexyl-pentane, C₁₇H₃₂.</u> -- Inert to fum. HNO ₃ and to KMnO ₄ . (181).
353	abt. ¹⁸⁸ 311 123-4 (10 mm.)	0.882(20) ¹⁸⁸	1.479 ¹⁸⁸	<u>Tetrahydroisozingiberene, C₁₅H₂₈.</u> -- D-form shows [α] _D = +4°36'. (188).
355	abt. ¹⁸⁹ 311 123-5 (10 mm.)	0.895(20/19) ¹⁸⁹	1.485	<u>Tetrahydro-calamene, C₁₅H₂₈</u>
357	abt. ¹⁸⁸ 315 128-30 (11 mm.)	0.826(20) ¹⁸⁸	1.456(n _D) ¹⁸⁸	<u>Hexahydrozingiberene, C₁₅H₃₀.</u> -- L-form shows [α] _D = -10°12'. (188).
359	abt. ¹⁹⁶ 317 125-8 (10 mm.)	0.884(21) ¹⁹⁶	1.480(21) ¹⁹⁶	<u>Tetrahydro-cadinene, C₁₅H₂₈</u>
361	abt. ¹⁸⁶ 319 126-8 (10 mm.)	0.888(20) ¹⁸⁶	1.483 ¹⁸⁶	<u>Tetrahydrocelinene, C₁₅H₂₈.</u> --D-form shows [α] _D = 7°. (186).
363	abt. ¹⁸⁷ 320 120-1 (8 mm.)	0.926 ¹⁸⁷	1.498 ¹⁸⁷	<u>Tricyclodihydrogurjunene, C₁₅H₂₆.</u>
365	abt. ¹³² 320 148-50 (10 mm.)	0.844(19/4) ¹³²	1.466(23) ¹³²	<u>2-Methyl-4-n-propyl-4-cyclohexyl-heptane, C₁₇H₃₄</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
367	abt. ¹³² 328 133-5 (11 mm.)	0.840(19/4) ¹³²	1.462(23) ¹³²	<u>2,4,6-Trimethyl-4-cyclohexyl-heptane, C₁₆H₃₂.</u>
369	abt. ¹³² 328 133-5 (11 mm.)	0.838(19/4) ¹³²	1.461(23) ¹³²	<u>4-Propyl-4-cyclohexyl-heptane, C₁₆H₃₂.</u>
371	abt. ¹⁹² 328 129-30 (10 mm.)	0.903(11) ¹⁹²	1.496(12) ¹⁹²	<u>Tetrahydroatractylene, C₁₅H₂₈.</u> -- D-form shows [α] _D ¹² =+36.99°. (192).
373	abt. ¹⁸⁵ 329 122-3 (7.5 mm)	0.889(20) ¹⁸⁵	1.483 ¹⁸⁵	<u>Tetrahydroendesmene, C₁₅H₂₈.</u> -- D-form shows [α] _D ¹² =+10°2'. (185).
375	abt. ⁷⁷ 330 123.5-4.5 (8 mm.)	0.915(20/4) ⁷⁷	1.496(20) ⁷⁷	<u>C₁₅H₂₄.</u>
377	²⁰³ 330-40	0.945(17/4) ²⁰⁴	1.514 ²⁰⁴	<u>Abietindihydride, C₁₉H₃₂.</u>
379	¹⁹⁹ 340-5	0.941(50) ²⁰⁰	1.492(50) ²⁰⁰	<u>Tricyclohexyl-methane, C₁₉H₃₄.</u> -- Br in CS ₂ g. substitution products. (201). Hot conc. H ₂ SO ₄ g. a brown color. (201). Hardly attacked by fum. HNO ₃ . (201).
381	abt. ¹³² 374 156-8 (12mm.)	0.844(21/4) ¹³²	1.467(20) ¹³²	<u>2,5,8-Trimethyl-5-cyclohexyl-nonane, C₁₈H₃₆.</u>
383	abt. ¹⁸³ 389 160.5-2 (12 mm.)	0.932(20/4) ¹⁸³	1.500(20) ¹⁸³	<u>3-Cyclohexyl-bicyclo-[0,4,4]-decane, "Perhydro-β-phenyl-naphthalene," C₁₆H₂₈.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
385	¹⁸³ abt. 391 162-2.5 (12mm.)	0.944(20/4) ¹⁸³	1.506(20) ¹⁸³	<u>2-Cyclohexyl-bicyclo-[0,4,4]-decane, "Perhydro-α-phenyl-naphthalene", C₁₆H₂₈.</u>
387	²⁰⁶ abt. 394 155-7 (10mm.)	0.956(17/4) ²⁰⁶	1.509 ²⁰⁶	<u>2,2'-Di[1,3,3-trimethyl-bicyclo-[1,2,2]-heptyl] "Difenchyl", "Hydrodifenchene", C₂₀H₃₄. --D-form shows $[\alpha]_D^{18} = 5^{\circ}30'$, (50 mm. tube).^D(206).</u>
389	¹³² abt. 399 162-4 (10mm.)	0.868(21/4) ¹³²	1.479(20) ¹³²	<u>2,8-Dimethyl-5-ethyl-5-cyclohexyl-nonane, C₁₉H₃₂.</u>
391	¹⁹⁷ abt. 416 193-4 (20 mm.)			<u>Chaulmoogrene, C₁₈H₃₄.</u>
393	¹³² abt. 421 190-2 (17 mm.)	0.842(21/4) ¹³²	1.465(20) ¹³²	<u>2,8-Dimethyl-5-n-propyl-5-cyclohexyl-nonane, C₂₀H₄₀.</u>
395	¹⁹⁴ abt. 421 183-6 (14 mm.)	0.833(20) ¹⁹⁴	1.460 ¹⁹⁴	<u>Octahydro-α-camphorene, C₂₀H₄₀.</u>
397	¹⁹⁸ abt. 431 200-1.5 (20mm.)	0.947(19/4 ¹⁹⁸ vac.)	1.507(20) ¹⁹⁸	<u>[2,3], [4,5]-Diendotetramethylene-bicyclo-[0,4,4]-decane, "Perhydro-9,10-benzophenanthrene, C₁₈H₃₀.</u>
399	¹³² abt. 455 162-3 (6 mm.)	0.880(21/4) ¹³²	1.491(20) ¹³²	<u>2,8-Dimethyl-5-isobutyl-5-cyclohexyl-nonane, C₂₁H₄₂.</u>
401	²⁰² abt. 496 192-3 (8.5mm.)	0.926(20/4) ²⁰²	1.500(20) ²⁰²	<u>3-(3²-cyclohexoethyl)-dicyclohexyl, C₂₀H₃₆.</u>

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
403	²⁰⁵ abt. 501 205-8 (11 mm.)			<u>Dicamphanyl-ethane, C₂₂H₃₈</u>

DIVISION B

.....
Liquid Hydrocarbons

Section 6

.....
Acyclic Paraffins

DIVISION B SECTION 6

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n_D)	Hydrocarbon
1	1, 2, 3, 4, 2 5, 6, 7, 8 27.5- 8.5	0.625 (15-4) ^{2, 5} vac.)	1.358 (15) ⁵	2-Methyl-butane, C_5H_{12} . -- C.S.T. in $PhNH_2$: 77° (7, 8) -- C.S.T. in $PhNO_2$: 32.2° (9). - Br g. 2-brom-2- methyl-butane (10); b.p. 75 = 108-9° w. dec., d_D^{20} : 1.216, n_D : 1.442 (11). - 1 mol. w. 1.5 mol. HNO_3 (d: 1.42) g. 2-nitro-2- methyl-butane; b.p. 74.8 = 149-50°, d^{20} : 0.974. (11).
3	8, 12, 13, 14, 15, 16 35.5- 6.5	0.630 (18-4) ¹⁶	1.360 (18) ¹⁶	Pentane, C_5H_{12} . -- C.S.T. in $PhNH_2$: 72° (7, 9).
5	17, 18, 19 49-50 *	0.650 (20-4) ¹⁹	1.372 (15) ^{20, 36}	2,2-Dimethyl-butane, * C_6H_{14} -- C.S.T. in $PhNH_2$: 80.75° (19). - HNO_3 (d.: 1.24) above 110° g. 3-nitro-2,2-dimethyl- butane, m.p. = 40°, b.p. = 167.5-8.5°, g. K cpd., glistening xtals. (20). - Cf. hexane, No. 13, for characterization.
7	19, 22, 23, 24, 25 * 58-9	0.662 (20-4) ¹⁹	1.381 (15) ^{20, 36}	2,3-Dimethyl-butane, * C_6H_{14} -- C.S.T. in $PhNH_2$: 72.3° (19, 25). - 5 hrs. htg. (at 125°) of 6 g. hy. w. HNO_3 (d.: 1.075) g. 2-nitro- 2,3-dimethyl-butane, m.p. 5-7°, b.p. = 170-4°, d_D^{20} : 0.961, insol. in conc. KOH (20). - For character- ization cf. hexane, No. 13.
9	19, 25, 26 60-1 *	0.654 (20-4) ^{8, 19, 25}	1.375 (15) ²⁰	2-Methyl-pentane, * C_6H_{14} . -- C.S.T. in $PhNH_2$: 74.7° (25, cf. 7, 8, 19). - For characterization cf. hexane, No. 13.

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
11	^{19,25} ^{28,29} 63-4 *	^{19,25} 0.665 (20-4)	^{19,25} 1.379 (15) ²⁰	3-Methyl-pentane*, C ₆ H ₁₄ ; -- C.S.T. in PhNH ₂ : 69.4° (19,25). -- C.S.T. in PhNO ₂ : 20.0°* (Test 923). -- HNO ₃ (d.: 1.4) at 60° g. 3-nitro-3-methyl-pentane, b.p. = 168-70°, d ₂₀ : 0.968. (30). -- Stable toward cold KMnO ₄ . (31). Attacked by fum. HNO ₃ . (31). -- For characterization cf. hexane No. 13.
13	^{9,19,22;} ^{32,33,34*} 69-70	^{32,35,36} 0.660 (20-4)	³² 1.375 (20)	Hexane*, C ₆ H ₁₄ . -- C.S.T. in PhNH ₂ : 69°* (7,8,19,38). C.S.T. in PhNO ₂ : 19.8°* (Test 923) (Cf. 9,37,38). -- Htg. w. dil. HNO ₃ (d.: 1.075) at 130-40° g. principally 2-nitro-hexane, b.p. = 176° corr., d ₂₀ : 0.936, easily sol. in boil. conc. KOH; g., w. Zn dust + alc. AcOH, 2-amino-hexane + methyl butyl ketone. (39). -- For a very complete and highly satisfactory procedure for characterizing all five of the isomeric hexanes + distinguishing between them, as well as for a consideration of their mixtures and their preparation cf. R+L. Berry, B.S. Thesis, Course V, 1930, M.I.T.
15	^{40,41;} ^{42,43} 78.5- 9.5	^{41,42,43} 0.674 (20-4)	^{41,42,43} 1.382 (20)	2,2-Dimethyl-pentane, C ₇ H ₁₆ ; --M.p. = -137°. (41). -- C.S.T. in PhNH ₂ : 77.7° (41,42,43). -- HNO ₃ (d.: 1.24) at 110-5° g. 2,2-dimethyl-3-nitro-pentane, b.p. ₄₀ = 89-90°, d ₂₀ : 0.940, g. a light blue pseudo-nitrole reaction. (40).

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
17	^{41,42,43} 80.5- ^{43,44} 1.5	^{41,42,44} 0.673 (20)	^{41,42,43,44} 1.381 (20)	2,4-Dimethyl-pentane, C ₇ H ₁₆ . -- M.p. = -119.4° (^{41,44}). - C.S.T. in PhNH ₂ : 78.6° (^{41,42,43}). - HNO ₃ (d:1.11) in sealed tube at 110-15° for 12-25 hrs. g. 2-nitro-2,4-dimethyl-pentane, b.p. = 182-3°, d ₂₀ ³⁰ : 0.931, n _D ³⁰ : 1.424 (^{45,46}), + 2,4-dinitro-2,4-dimethyl-pentane, m.p. = 81-2° (⁴⁵).
19	^{42,43,47} 80.5- 1.5	^{42,43,47} 0.690 (20-4)	^{42,43,47} 1.389 (20)	2,2,3-Trimethyl-butane, C ₇ H ₁₆ . -- M.p. = -25° (^{42,43,47}). - C.S.T. in PhNH ₂ : 72.4° (^{42,43}).
21	^{42,43,48} 86-7	^{42,43} 0.690 (20-4)	^{42,43} 1.391 (20)	3,3-Dimethyl-pentane, C ₇ H ₁₆ . -- C.S.T. in PhNH ₂ : 71-0° (^{42,43}). - M.p. = -135.0° (⁴²).
23	^{42,43,44} 89.5- 90.5 ^{8,42,50}	^{42,43} 0.695 (20-4)	^{42,43} 1.392 (20)	2,3-Dimethyl-pentane, C ₇ H ₁₆ . -- C.S.T. in PhNH ₂ : 68.1° (^{42,43}).
25	^{51,52} 90-1	^{8,42,43} 0.679 (20-4)	^{42,43} 1.385 (20)	2-Methyl-hexane, C ₇ H ₁₆ . -- C.S.T. in PhNH ₂ : 72.8° - 74.1° (^{7,8,42,43}).
27	^{41,42} ^{43,53} 91-2	^{41,42,43,53} 0.687 (20-4)	^{42,43} 1.389 (20)	3-Methyl-hexane, C ₇ H ₁₆ . -- C.S.T. in PhNH ₂ : 70.5° (⁴¹). - For the d-form, (L) _D ²⁰ : +9.5° (⁵³). - C.S.T. in PhNH ₂ : 70.5° (^{42,43}).
29	^{42,43} 93-4 ^{8,42,43}	^{42,43} 0.698 (20-4)	^{42,43} 1.394	3-Ethyl-pentane, C ₇ H ₁₆ . -- C.S.T. in PhNH ₂ : 66° (^{42,43}).
31	^{55,56,57} 98-9	^{8,42,43} 0.684 (20-4)	^{42,43,55} 1.388 (20)	Heptane*, C ₇ H ₁₆ . -- C.S.T. in PhNH ₂ : 70.0° (^{7,8,42,43,58}). - C.S.T. in PhNO ₂ : 18-9°* (Test 923). - Boil. under reflux w. HNO ₃ (d: 1.42) g. 1-nitro-heptane, beside CO ₂ , AcOH, succinic ac., oxalic ac. (⁵⁹). - 1-nitro-heptane, b.p. = 193-5°, d ₁₅ ¹⁷ : 0.948, g. nitrolic

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
				ac. reaction, + w. Fe filings + AcOH g. heptyl amine. (s.).
33	98.5- ⁶⁰ 9.5	0.692 (20-4) ⁶¹	1.392 ⁶¹	2,2,4-Trimethyl-pentane,* <u>C₈H₁₈</u> : -- C.S.T. in PhNO ₂ : 29-30°* (Test 923).
35	105-11 ⁶⁷			3,3-Dimethyl-hexane, C ₈ H ₁₈ .
37	^{23, 51, 63, 64, 65, 66} 108.5- 9.5	0.698 (17-4) ⁶⁶	1.394 (17) ⁶⁶	2,5-Dimethyl-hexane, C ₈ H ₁₈ .
39	109-10 ⁶²	0.708 (15-15) ⁶²	1.399 (25) ⁶²	2,4-Dimethyl-hexane, C ₈ H ₁₈ .
41	110.5- ⁶⁸ 111.5	0.722 (15-15) ⁶⁸	1.416 (25) ⁶⁸	2,2,3-Trimethyl-pentane, <u>C₈H₁₈</u> .
43	113.5- ⁷⁰ 4.5	0.724 (15-15) ⁷⁰	1.408 (25) ⁷⁰	2,3-Dimethyl-hexane, C ₈ H ₁₈ .
45	114-5 ⁶⁹	0.708 (15) ⁶⁹	1.400 (25) ⁶⁹	3-Methoethyl-pentane, C ₈ H ₁₈ .
47	^{31, 71, 72} 116-8	0.699 (20-0) ³¹	1.394 (20) ³¹	2-Methyl-heptane, C ₈ H ₁₈ . -- C.S.T. in PhNH ₂ : 74° (7).
49	cf. ⁷³ 118-9	0.717 (25-4) ⁷³	1.404 (25) ⁷³	3,4-Dimethyl-hexane,* <u>C₈H₁₈</u> . -- C.S.T. in PhNO ₂ : 13.7°* (Test 923). -- Fum. HNO ₃ at 100° g. a ter-nitro cpd. (73).
51	118-9 ⁷⁴	0.718 (15-15) ⁷⁴	1.399 (25) ⁷⁴	3-Ethyl-hexane, C ₈ H ₁₈ .
53	118-9 ⁷⁵	0.722 (15) ⁷⁵	1.398 (25) ⁷⁵	4-Methyl-heptane, C ₈ H ₁₈ .
55	120-2 ⁷⁶	0.707 (20-4) ⁷⁶	1.398 (20) ⁷⁶	3-Methyl-heptane, C ₈ H ₁₈ .
57	^{8, 35, 51, 66, 77} 125-6	0.705 (18-4) ⁶⁶	1.399 (18) ⁶⁶	Octane,* <u>C₈H₁₈</u> . -- C.S.T. in PhNH ₂ : 72° (7,8). - C.S.T. in PhNO ₂ : 14.7°* (Test 923). - Boil. w. HNO ₃ (d.: 1.14) g. 1-nitro-octane, b.p. = 206-10° w. slight dec., d ₂₀ : 0.935; + 1,1-dinitro- octane. (s.). - HNO ₃ (d.: 1.075) at 130° for 1 hr. g. 2-nitro-octane, b.p. ₄₀ = 123-4°, b.p. ₇₆₀ = 210-2° w. dec., d ₂₀ : 0.936. (26, 78).

cf. Sheppard, Henne, Mudgett, JACS 53 1931 et seq (1931)
Main Bu. Stds. J. Res. 9 471 (1922)

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No.	Boiling Point (C°)	Specific Gravity	Refractive Index [n_D]	Hydrocarbon
59	125-6 ⁷⁹	0.708 (20-0) ⁷⁹	1.399 ⁷⁹	<u>2,2,5-Trimethyl-hexane, C₉H₂₀.</u>
61	^{79,80,81} 132-3	0.713 (20-0) ^{79,80}	1.403 ⁷⁹	<u>2,6-Dimethyl-heptane, C₉H₂₀.</u> -- 10 hrs. htg. w. HNO ₃ (d.: 1.11) at 120° g. 2,6-dinitro-2,6-dimethyl-heptane, needles, m.p. = 74-4.5°, dec. by distn. at ord. press. (s ₂).
63	⁸³ 133-4	0.721 (15-15) ⁸³	1.401 (25) ⁸³	<u>2,4-Dimethyl-heptane, C₉H₂₀.</u>
65	⁸³ 135-6	0.719 (15-15) ⁸³	1.402 (25) ⁸³	<u>2,5-Dimethyl-heptane, C₉H₂₀.</u> -- The d-form (ctg. 97% active substance) shows (α) _D ¹⁶ : +9.48°. (s ₄).
67	⁸⁵ 138-9	0.741 (20) ⁸⁵	1.416 (20) ⁸⁵	<u>4-Ethyl-heptane, C₉H₂₀.</u>
69	⁸⁶ 139-40	0.752 (20-4) ⁸⁶	1.421 (18) ⁸⁶	<u>3,3-Diethyl-pentane, C₉H₂₀.</u>
71	⁸⁷ 141-2	0.732 (15-15) ⁸⁷	1.403 (25) ⁸⁷	<u>4-Methyl-octane, C₉H₂₀.</u>
73	⁸⁴ 142.5-3.5	0.721 (17) ⁸⁴		<u>3-Methyl-octane, C₉H₂₀.</u> -- D-form shows (α) _D ¹⁷ : 9.38° (s ₄).
75	⁸⁸ 150-1	0.722 (15-15) ⁸⁸	1.403 (25) ⁸⁸	<u>Nonane, C₉H₂₀.</u> -- Boil. w. HNO ₃ (d.: 1.080) g. 1-nitro-nonane, light yellow liquid, p.p. = 215-8°, w. dec., d ₁₇ ¹⁷ : 0.923 (s ₉); + 1,1-dinitro-nonane, m.p. = 164° w. dec. (s ₉).
77	⁹⁰ 151-2	0.722 (20-4) ⁹⁰	1.409 (20) ⁹⁰	<u>2,2,6-Trimethyl-heptane, C₁₀H₂₂.</u>
79	^{66,93,94,95} 159-60	0.728 (18-4) ⁶⁶	1.409 (18) ^{66,91}	<u>2,7-Dimethyl-octane, C₁₀H₂₂.</u> " <i>Diisoamy</i> " -- C.S.T. in PhNO ₂ : 25-6° (Test 925). -HNO ₃ (d.: 1.075) at 120-5° for 40 hrs. g. 2,7-dinitro-2,7-dimethyl-octane, m.p. = 101.5-2 (s ₆) + 1-nitro-2,7-dimethyl-octane, b.p. = 235-7° w. dec., d ₂₁ ²¹ : 0.925, n _D ²¹ : 1.443. (s ₆).

DIVISION B SECTION 6

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
81	159 ¹⁰¹ -60	0.724 (20-4) ¹⁰¹	1.408 (20) ¹⁰¹	2-Methyl-nonane, C ₁₀ H ₂₂ .
83	159-60 ⁹²	0.740 (15) ⁹²	1.415 ⁹²	3,6-Dimethyl-octane, C ₁₀ H ₂₂ . -- Active form shows (α) _D ¹³ : +16.85°. (84).
85	159.5-60.5 ^{97,98,99}	0.729 (20-4) ^{99,100}	1.411 (20) ^{99,100}	2,6-Dimethyl-octane, C ₁₀ H ₂₂ .
87	161-2 ¹⁰¹	0.736 (20-4) ¹⁰¹	1.414 (20) ¹⁰¹	4-Propyl-heptane, C ₁₀ H ₂₂ .
89	161.5-2.5*			4,5-Dimethyl-octane,* C ₁₀ H ₂₂ . -- C.S.T. in PhNO ₂ : 19-20°.*(Test 923).
91	165-6 ^{76,102}	0.732 (20-4) ⁷⁶	1.412 (20) ⁷⁶	5-Methyl-nonane, C ₁₀ H ₂₂ . -- KMnO ₄ at 37° in 16 hrs. appreciably oxid. this hy. (76). C.S.T. PhNH ₂ 77.9
93	165-6 ⁷⁶	0.735 (20-4) ⁷⁶	1.413 (20) ⁷⁶	3-Methyl-nonane, C ₁₀ H ₂₂ . -- KMnO ₄ at 37° in 16 hrs. does not appreciably oxid. this hy. (76). C.S.T. PhNH ₂ 78.25
95	173-4 ¹⁰³	0.730 (20) ¹⁰³	1.409 ¹⁰⁴	Decane,* C ₁₀ H ₂₂ .
97	186-8 ¹⁰⁵	0.766 (20) ¹⁰⁵	1.432 ¹⁰⁵	2,6-Dimethyl-3-isopropyl-heptane, C ₁₂ H ₂₆ .
99	189-90 ¹⁰⁶	0.754 (14) ¹⁰⁶	1.425 ¹⁰⁶	2-Methyl-5-propyl-octane, C ₁₂ H ₂₆ .
101	71 ¹⁰⁷ (16 mm.)	0.751 (19-4) ¹⁰⁷	1.421 (20) ¹⁰⁷	5-Ethyl-nonane, C ₁₁ H ₂₄ .
103	194-5 ^{103,107,108}	0.742 (20-4) ¹⁰⁷	1.419 (20) ¹⁰⁷	Undecane, C ₁₁ H ₂₄ .
105	204-5 ⁷⁶	0.756 (20-4) ⁷⁶	1.423 (20) ⁷⁶	5-Propyl-nonane, C ₁₂ H ₂₆ .
107	208-10 ¹⁰⁹			2,4,5,7-Tetramethyl-octane, C ₁₂ H ₂₆ .
109	214.5-5.5 ¹⁰³ *	0.751 (20-4) ¹⁰³	1.421 ¹⁰⁴	Dodecane,* C ₁₂ H ₂₆ .
111	217.5-8.5 ¹¹⁰	0.764 (19-4) ¹¹⁰	1.427 (19) ¹¹⁰	5-Butyl-nonane, C ₁₃ H ₂₈ .

DIVISION B SECTION 6

No.	Boiling Point (C°)	Specific Gravity	Refractive Index (n _D)	Hydrocarbon
113	220-1 ¹¹¹	0.775 (25) ¹¹¹	1.434 (n ₂₅) ¹¹¹	4,5-Dipropyl-octane, <u>C₁₄H₃₀</u> .
115	225.5- ⁷⁶ 7.0	0.758 (20-4) ⁷⁶	1.424 (20) ⁷⁶	5-Methyl-dodecane, C ₁₃ H ₂₈ .
117	234-5 ¹⁰³	0.757 (20-4) ¹⁰³		Tridecane, C ₁₃ H ₂₈ . -- M.p. = -6.2° (103).
119	252.5- ^{103,112} 3.5	0.765 (20-4) ¹¹²		Tetradecane, *C ₁₄ H ₃₀ . -- M.p. = 5.5° * (112).
121	115.7 ¹¹³ (9 mm.)	0.779 (20) ¹¹³	1.435 ¹¹³	Octahydrosesquicitronellene, <u>C₁₅H₃₂</u> .
123	267.5- ¹¹⁴ 9.5	0.792 (14) ¹¹⁴		7,8-Dimethyl-tetradecane, <u>C₁₆H₃₄</u> . -- Liquid at -30°. (114).
125	125-7 ¹¹⁹ (18 mm.)	0.789 ¹¹⁹	1.445 ¹¹⁹	4,7-Dipropyl-decane, C ₁₆ H ₃₄ . <i>Bp 274.5-50</i>
127	270-1 ¹⁰³	0.769 (20-4) ¹⁰³		Pentadecane, C ₁₅ H ₃₂ . -- M.p. = 10° (103).
129	169-70 ^{115,116} (9.5 mm.)	0.803 (0-4) ^{115,116}		Phytane, C ₂₀ H ₄₂ . -- By phytol (115) or phytene (115,116) + H + Pt black.
131	263 ¹¹⁷ (10 mm.)	0.812 (15-4) ¹¹⁷	1.453 (15) ¹¹⁷	Dodecahydrosqualene, C ₃₀ H ₆₂ . <i>Bp ~ 425°</i>

DIVISION C

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Gaseous Hydrocarbons

DIVISION C

No.	Boiling Point (C°)	Hydrocarbon
2	-162 ¹	<u>Methane, CH₄.</u>
4	-102.7 ⁵	<u>Ethene, C₂H₄.</u>
6	-88.5 ¹²	<u>Ethane, C₂H₆.</u>
8	-82.4 ¹	<u>Ethine, "Acetylene", C₂H₂.</u>
10	-47.5 ⁵	<u>Propene, C₃H₆.</u>
12	-44.5 ²	<u>Propane, C₃H₈.</u>
14	abt. 18 -34.5	<u>Cyclopropane, "Trimethylene", C₃H₆.</u>
16	-32 ⁸	<u>Propadiene, "Allene", C₃H₄.</u>
18	-23.5 ^{8,9}	<u>Propine, "Allylene", C₃H₄.</u>
20	-10.5 ³	<u>2-Methyl-propane, "Isobutane", C₄H₁₀.</u>
22	-6.8 ^{20,21}	<u>Methyl-propene, "Isobutene", C₄H₈.</u>
24	-(6.7- ^{5,22} 6.5)	<u>Butene-(1), C₄H₈.</u>
26	-(5-4) ¹¹ (513 mm.)	<u>Butadiene-(1,3), "Erythrene", C₄H₆.</u>
28	+0.5 ³	<u>Butane, C₄H₁₀.</u>
30	1-1.5 ⁷	<u>Butene-(2), low boil. form, C₄H₈.</u>
32	1.5-2 ¹⁷ (729 mm.)	<u>Cyclobutene, C₄H₆.</u>
34	2-2.7 ⁷	<u>Butene-(2), high boil. form, C₄H₈.</u>
36	2-3 ¹⁴ (729 mm.)	<u>Butene-(1)-ene-(3), C₄H₆.</u>
38	4-5 ¹⁶	<u>Methyl-cyclopropane, C₄H₈.</u>
40	8.5 ¹³	<u>Butene-(1), C₄H₈.</u>
42	9.5 ⁴	<u>2,2-Dimethyl-propane, C₅H₁₂.</u>
44	9.5-10 ¹⁹	<u>Butadiene, C₄H₂.</u>
46	11-2 (726 mm.)	<u>Cyclobutane, "Tetramethylene", C₄H₁₀.</u>

DIVISION C

No.	Boiling Point (C°)	Hydrocarbon
48	18-9 ¹⁰	<u>Butadiene-(1,2), C₄H₆.</u> -- Cf. Div. B, Sect. 3, No. 1.
50	20-1	<u>2-Methyl-butene-(3), C₅H₁₀.</u> -- Cf. Div. B, Sect. 4, No. 1.
52	21	<u>1,1-Dimethyl-cyclopropane, C₅H₁₀.</u> -- Cf. Div. B, Sect. 5, No. 1.

D

Numbered Specific
and Semi-Specific Tests for
Species of Hydrocarbons

NUMBERED SPECIFIC AND SEMI-SPECIFIC TESTS FOR
SPECIES OF HYDROCARBONS.

Tests 901-921, inclusive, are taken from Mulliken, Identification of Pure Organic Compounds, John Wiley and Sons, Inc., (First Edition), exactly as there given.

901. Bromine Test for Unsaturation.

This test for unsaturation finds many applications, but is most frequently employed in connection with the species of Genera IX and III.

Dissolve or suspend 0.1 gm. of the pure compound - finely powdered, if it is an insoluble solid - in 2 cc. of dry carbon tetrachloride in a three-inch test-tube. Add three drops of a bromine solution⁺ prepared by dissolving 2.0 cc. of bromine in 50 cc. of carbon tetrachloride. If decolorization does not take place at once, stopper the tube loosely, and allow to stand for three minutes in the cold, shaking occasionally if the body is insoluble. If the solution becomes colorless before the end of two minutes, drop in more bromine solution until a color that is permanent for a minute or two is produced. Then blow sharply across the mouth of the tube, and notice whether a white cloud (hydrated hydrobromic acid) makes its appearance.

If no signs of action in the cold are observed, hold

the tube high above a small flame and boil very gently for two minutes. If decolorization results, drop in more bromine until the coloration remains permanent for nearly a minute when the solution is again boiled. Test for hydrobromic acid as before by blowing across the mouth of the tube.

Complete decolorization in either part of this test (either in the cold or after heating), if unaccompanied by evolution of hydrobromic-acid gas, shows that the compound under examination is unsaturated; that is, that it can add bromine.

The presence of double or triple bondings in hydrocarbons may in the great majority of cases be detected by use of the test in the cold only; but there are a few unsaturated hydrocarbons like stilbene which require short heating, and in tetraphenylethylene we have one which remains unchanged even when heated. Among the unsaturated acids, maleic and fumaric acids* also show an exceptional behavior in not decolorizing the tetrachloride solution after two minutes' boiling. Some other unsaturated acids, like aconitic, do not decolorize the solution until it has been heated, but the number of such species is not large.

Decolorization in either part of the test when accompanied by a copious evolution of hydrobromic acid always indicates substitution; but since addition may, or may not, have taken place at the same time, satisfactory inferences as to the

existence of unsaturation in such cases can not be drawn. The appearance of scanty traces of hydrobromic acid towards the end of an experiment in which a considerable quantity of bromine has been consumed, may, however, be due to minor secondary reactions and may be disregarded.

In the heat, the number of compounds in Order I that are attacked by the treatment with bromine is greatly increased. The saturated hydrocarbons of the marsh-gas series, (C_nH_{2n+2}) , with unbranched carbon skeletons, and the members of the acetic-acid series, $(C_nH_{2n}O_2)$, are conspicuous examples of compounds unaffected under these circumstances. Some paraffin hydrocarbons like diisoamyl with branched carbon skeletons are, on the contrary, quite readily attacked in the heat, although not in the cold. Many of the aromatic hydrocarbons like mesitylene and anthracene are so easily substituted that decolorization occurs within a fraction of a minute in the cold; but pure benzene is so comparatively unreactive that it does not cause decolorization within the two minutes' limit on boiling.

Most phenols, and many aldehydes and ketones, cause decolorization cold within a few seconds. Whenever decolorization takes place readily in consequence of addition or substitution in a homogeneous compound, if the experiment is continued after the first disappearance of color, it will be found that the quantity of bromine eventually consumed will be at least several times greater than what was added at the beginning of the experiment.

+ Carbon tetrachloride is given the preference as the solvent, because bromine solutions prepared by its use may be kept for weeks without spoiling; because such solutions do not entirely lose their orange-yellow color on heating unless boiled for more than twice the time prescribed in the test procedure; and because the tetrachloride is such a poor solvent for hydrobromic acid that the gas escapes as soon as formed, and thus is easily detected by the fumes.

+ Fumaric or maleic acids will, however, decolorize hot bromine water. (Bromine water is as a rule a very unsatisfactory substitute for the carbon tetrachloride reagent, since it is frequently decolorized by acting as an oxidizing agent, holds back hydrobromic acid, and loses its color rather quickly on boiling.)

902. Action of Fuming Sulphuric Acid.

Support a three-inch test-tube containing 1 cc. of fuming sulphuric acid (sp. gr. 1.89) by means of a small clamp in a nearly vertical position, but so that it shall be slightly inclined away from the operator. Drop in slowly from a medicine-dropper about five drops of the compound. If there are no immediate signs of solution or chemical action, shake the mixture cautiously for about one minute. Then allow to stand for a short time, and notice whether the compound added separates apparently unchanged as an upper layer.

This test, as well as 903, may be applied for confirmatory evidence. Aromatics, especially unsaturated aromatics, and other unsaturated hydrocarbons are attacked. Condensed ring naphthenes, and some naphthenes with side chains are also attacked. The paraffins are most inert, and among these, the less branched the chain, in general, the less the reaction.

903. Action of Fuming Nitric Acid.

(This test is dangerous unless performed cautiously as directed.)

In a three-inch test-tube, supported as in Test 902, place 1 cc. of fuming nitric acid of specific gravity 1.48. Then add from a medicine-dropper, held at arm's length, a single drop of the compound to be tested. A violent reaction

often ensues, and there may be a slight explosion, or the substance may even ignite. If there are no signs of action, cautiously add a few more drops of the substance, and shake gently.

Div. B, Sect. 6,

The liquid paraffins, although they are unattacked, and do not dissolve, always dissolve oxides of nitrogen so as to acquire a color much like that of the nitric acid. The presence of two layers after shaking may, therefore, be easily overlooked in a hasty observation. *Cf. conclusion to Test 902.*

904. Colorations with Aluminium Chloride.

Drop a hard lump of sublimed aluminium chloride weighing about 0.2-0.3 gm. into a clean 6-8-inch test-tube that has just been taken from a hot drying oven. Stopper the tube loosely. Hold it in a nearly horizontal position, and by means of a small flame placed under one end slowly sublime the chloride until it forms a thin light-yellow coating covering a considerable portion of the glass surface. Allow to cool. Drop in 0.5 cc. of a solution containing 0.05 gm. of the hydrocarbon dissolved in a 2.5 cc. of chloroform. Stopper the tube tightly. Lay it on its side upon a sheet of white paper that rests upon and partly covers the color standard. Then roll it back and forth so that the solution shall flow over and wet all parts of the sublimate. Observe the color after a few seconds, and again after 15-20 minutes.

Most aromatic hydrocarbons give colorations when thus treated. The colors are often very intense, and some-

times admit of employment as minor preliminary or confirmatory tests; but since the hue may be much modified by the presence of small quantities of impurities, too great importance ought not to be attached to the indications obtained by their use.

The initial colorations given by the liquid homologues of benzene approximate orange; e.g. pseudocumene, RO; m-xylene, O; benzene, OY (after five minutes). After standing fifteen minutes these colors will either remain unchanged, or will change by about one hue of the standard in the direction of the red end of the spectrum. The initial coloration with diphenylmethane and triphenylmethane is YO, darkening within a few minutes to YOTI; with anthracene it is OYS2-YS2.

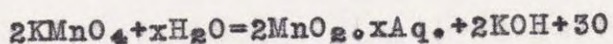
Initial colorations of great intensity which persist unchanged for more than twenty minutes and approximate blue, are given by several important solid hydrocarbons; e.g. blue (B), by diphenyl; blue to green-blue (GB-B), by phenanthrene; and blue-green (BG), by naphthalene.

905. Oxidation of Side Chains.

The oxidation of the side chains in aromatic hydrocarbons to carboxyl groups by hot aqueous solutions of potassium permanganate, chromic acid, or nitric acid, has been employed in determining the constitution of many species of Genus IX. The most serious difficulty encountered in adapting these methods for use as practical specific test arises from the extreme insolubility of all hydrocarbons in aqueous

solutions. This renders the oxidations very slow. During the oxidation period - which is seldom less than several hours - the oxidation product, which is itself never entirely stable, is exposed to the destructive action of the oxidant. Hence the yield, which even under favorable circumstances falls much under the theoretical, is often very poor indeed. Hydrocarbons which are themselves stable, but give unstable oxidation products, are therefore the most difficult to treat successfully. Whenever it is suggested in the tables that some particular oxidant may be used in the identification of a hydrocarbon, it does not always follow that the oxidant mentioned is the best that could have been selected for the purpose, or that the yield will be good, but merely that the product named has been obtained by its use. It should also be understood that the following general directions are given as suggestions rather than mandatory procedures; and that what is said refers more especially to aromatic hydrocarbons having one or two side chains.

1. (Oxidations with Potassium Permanganate.) - The oxidation with permanganate, when applicable, will usually be preferred to either of the other methods. The reagent is a neutral aqueous solution containing 61.6 grms. of potassium permanganate to the liter. In organic oxidations it is said to be reduced according to the equation



1 cc. of the solution accordingly contains 0.01 gm. of "available oxygen" and the alkali liberated is sufficient to combine with the full quantity of organic acid and carbon dioxide that will be produced in any ordinary oxidation. The latter fact makes it possible to perform these oxidations in closed vessels, and thus avoid the violent bumping that is one of the greatest objections to the use of permanganate when the oxidation is performed by boiling in flasks.

Calculate by aid of the equation given above how much permanganate solution will be theoretically needed to produce the desired effect, and place it in a strong wide flask or bottle of about one-liter capacity. If, as will sometimes happen, the hydrocarbon is lighter than water, and a liquid, the extended contact surface presented by the permanganate solution, which will be spread out in rather a thin layer, will do much to accelerate the reaction. When the oxidation product expected is benzoic, isophthalic, or terephthalic acid, about 1 gm. of the hydrocarbon should be enough for an experiment.

Suspend the bottle by a wire, so that the lower part will be immersed in a boiling water-bath; and, as soon as the air within has been expanded by the heat, and the hydrocarbon introduced, stopper tightly to prevent loss of substance by volatilization. Then heat until the red color of the permanganate is seen to have completely disappeared. This may require from two to eight hours, and some of the hydrocarbon

will always remain unattacked. Separate the colorless alkaline solution from the bulky brown precipitate of hydrated manganese oxide by filtration. Evaporate to a small volume. Filter if necessary, and cool. Acidify the solution with a moderate excess of hydrochloric acid, and shake vigorously. Benzoic, isophthalic, and terephthalic acid will precipitate at this point. The two former may then be identified by their melting-points and specific tests, after a single crystallization from boiling water; the latter after being well washed with water. Phthalic acid being comparatively easily oxidized by hot permanganate, will not be detected, unless the hydrocarbon is one that oxidizes quite rapidly. The loss of benzoic acid in long-continued oxidations is also large, though less serious. In an oxidation of 1 gm. of ethylbenzene requiring six hours, the yield of pure benzoic acid was 0.20 gm. Benzoic acid is easily separated from any of the phthalic acids by treatment with chloroform, in which it is very soluble.

2. (Oxidations with Chromic-acid Mixture.) - Boil the hydrocarbon in a round-bottomed flask containing ebullator tubes with the quantity of chromic-acid mixture theoretically required to produce the desired effect, until the chromic acid is completely reduced. The apparatus, chromic-acid mixture, and general procedure for the oxidation are the same as have been more fully described in Test 702 for the oxidation of ketones and alcohols, except that longer heating will be necessary. As the action of hot chromic acid on most of the aromatic acids is even

more destructive than that of permanganate, it is advisable to use at least 2 grms. of the hydrocarbon for each experiment, and even larger quantities may sometimes be found necessary. Collect the insoluble residue of oxidation products, and unchanged hydrocarbon that separates from the well-cooled solution, on a small filter. Wash with a little cold water. Dissolve out the aromatic acids by boiling with a slight excess of sodium-carbonate solution. Reprecipitate with an excess of hydrochloric acid, and identify them by appropriate tests.

3. (Oxidations with Dilute Nitric Acid.) - Although nitric acid, being a milder oxidant than either permanganate or chromic acid, may be successfully employed in some cases in which the latter are inapplicable, and is occasionally mentioned in the tables, it has the disadvantage of being exceedingly slow in its action, and of giving products which sometimes consist largely of nitrosubstitution derivatives whose removal is troublesome. The proper procedure depends so much on the properties of the particular hydrocarbon to be oxidized, that in the few instances in which this method is referred to in the tables, it will always be best to consult the original literature relating to the subject before proceeding to the experiment. The following general statement and suggestions may, however, be of some assistance.

It is best to oxidize at least 2 grms. of the hydrocarbon with a large excess of acid. The nitric acid is usually a mixture of one part of concentrated commercial nitric acid with

three parts of water, though in some cases a stronger acid can be used, shortening the time without causing much substitution.⁺ The time of boiling varies from six to forty-eight hours. In general it is best to boil at least eight hours. If it is expected that a solid aromatic acid, not volatile with steam, will be formed, the excess of nitric acid should be removed by evaporation on a water-bath. The residue is next extracted with boiling sodium-carbonate solution, the solution filtered, and the organic acids precipitated from the filtrate by a moderate excess of hydrochloric acid. Nitro-acids may then be reduced by warming with tin and hydrochloric acid, so as to form soluble hydrochlorides of the corresponding amino-acids, which, upon filtration, will pass into the filtrate. Or, if the acid sought should also be soluble in dilute hydrochloric acid, it may be separated from the amino-acid by crystallization, after precipitating the tin with sulphuretted hydrogen.

⁺ Thus Fileti (G. 21, I, 5 and 22) used one part of acid to one of water in oxidizing p-propyl-isopropylbenzene, obtaining terephthalic acid with only a trace of a nitro-acid.

906. Test for Triple-bonding in Compounds Containing the (-C≡CH) Group.

Dilute 1 cc. of ammoniacal cuprous chloride solution,⁺ in a test-tube, with 5 cc. of cold water. Add a few drops of the hydrocarbon, if a liquid, and shake. If the hydrocarbon

is a gas, conduct it directly into the copper solution. Collect the precipitate on a filter. Wash with cold water and observe the color.

The hydrogen atoms in compounds containing the $\text{C}\equiv\text{CH}$ group are usually replaceable by copper when thus treated. These copper compounds appear as insoluble flocculent precipitates, varying in color, according to the body from which they are obtained, from a dark brick-red to a greenish yellow. When washed with alcohol and ether and dried with proper precautions, they often explode violently when struck a sharp blow or when strongly heated.

Alcoholic AgNO_3 (prepared by diluting with $\text{C}_2\text{H}_5\text{OH}$ a saturated aqueous AgNO_3 soln.) is an exceptionally fine reagent for distinguishing small amounts of $-\text{C}\equiv\text{C}-\text{H}$ (white ppt.)

+ Ammoniacal Cuprous Chloride Reagent. - This is the reagent used in gas analysis for the absorption of carbon monoxide. - It is prepared from an acid cuprous chloride solution as required for use. To prepare the acid solution, cover the bottom of a bottle with a layer of powdered copper oxide 1 cm. deep. Place in the bottle a number of pieces of rather stout copper wire, reaching from top to bottom, sufficient to make a bundle an inch in diameter, and fill the bottle with common hydrochloric acid of 1.10 sp. gr. Stopper, and allow to stand with occasional shaking for some days, or until the solution becomes nearly or quite colorless. When about to make a test, decant a little of the clear acid solution, and add ammonia to it until about to make a test, decant a little of the clear acid solution, and add ammonia to it until present in slight excess, i.e., until the mixture has a distinctly ammoniacal odor. The space left in the stock bottle after every withdrawal of solution should be immediately filled with more hydrochloric acid (1.10 sp. gr.) and the bottle always be kept tightly stoppered to prevent absorption of oxygen from the air.

907. Saturated Ethers

Drop 1 cc. of the compound slowly into 2 cc. of ice-cold sulphuric acid (sp. gr. 1.84) contained in a five-inch

test-tube standing in a beaker of ice-water. Without removing the test-tube from the ice-water, shake briskly for half a minute or more. Then, after allowing to stand for a minute or two, observe whether the compound has dissolved completely to a colorless or nearly colorless solution. In case such a solution has been formed, pour it slowly into a second test-tube containing 3 cc. of cold water, shaking and cooling meanwhile, just as was done during the preparation of the acid solution. If the mixture on standing separates into two layers, remove and reject the lower layer, which will consist of dilute sulphuric acid, with the aid of a long capillary-pointed medicine-dropper. Wash the upper layer by shaking with 2 cc. of sodium-carbonate solution. If an emulsion forms, hasten the separation into layers by warming. Remove the carbonate solution as before by the aid of the dropper, and transfer the organic liquid to a dry three-inch test-tube. Add a small fragment of solid potassium carbonate, and heat nearly to boiling to hasten the drying action. Then after a few minutes, in order to ascertain whether the product obtained is identical with the original substance, determine the boiling-point of the clear dried liquid by Siwoloboff's method. For a more detailed description of the manipulations involved in the washing and drying, and in the boiling-point determination, read the latter half of paragraph i. on the identification of soluble alcohols obtained in saponification tests, (cf. p. 115, Mulliken, Identification etc., Vol. I.)

Any species of Genus IX that dissolves in sulphuric acid in this test to a clear, nearly colorless solution, which, upon dilution, gives a liquid identical in boiling-point with the original substance, is probably the oxide of a saturated hydrocarbon radical or, possibly, an "unsaponifiable ester." Unsaturated ethers and unsaturated hydrocarbons may also dissolve completely in the cold acid, or may be entirely decomposed by the reagent; but when a clear solution does result, dilution with water can not be expected to yield the original substance. Saturated hydrocarbons, even the aromatic ones, do not dissolve in the acid to any considerable extent.

911. Acenaphthene.

Dissolve 0.05 gm. of the hydrocarbon and 0.10 gm. of picric acid in 2.5 cc. of boiling 95 per cent alcohol in a dry test-tube. Allow the solution to cool down to the temperature of the laboratory gradually. Acenaphthene under these conditions yields a beautifully crystallized orange-colored picric-acid compound, $C_{12}H_{10} \cdot C_6H_3(NO_2)_3O$, whose slender flat needles shoot from the bottom of the tube to the surface of the solution. Collect on a small filter, and wash with 3 cc. of cold alcohol. Dry for fifteen minutes on a piece of porous tile at 100° , and determine the melting-point.

The color of the dry crystals is nearly the orange of the color standard(O-YO). They melt at 161° - 162° (uncorr.)

912. Anthracene.

Place in a six-inch test-tube 0.05 gm. of the hydrocarbon, 1.5 grms. of chromic acid (CrO_3), 4 cc. of glacial

acetic acid, and 1 cc. of water. Support the tube by a clamp so that its lower end shall rest in a circular perforation in a piece of asbestos board arranged as in Test 312-2, and boil for ten minutes over a small flame, so gently that the vapors shall all condense on the sides of the tube. Pour into 20 cc. of cold water. Collect the **flocculent** precipitate on a filter. Wash thoroughly with much water, and finally with 5 cc. of cold alcohol. Transfer the precipitate to a dry test-tube and boil with 10 cc. of strong alcohol. Cool. Collect the nearly white precipitate on a small filter. Wash with 5 cc. of cold alcohol. Boil up a second time with 10 cc. of strong alcohol, and again cool. Filter, and wash with 5 cc. of cold alcohol. Dry the residue fifteen minutes at 100° on a piece of porous tile, and determine the melting-point.

Anthraquinone, the product obtained in this test, is a pale yellowish compound, crystallizing from alcohol in minute needles which melt at 279°-280° (uncorr.). For other characteristic properties of anthraquinone see Test 1011.

913. Benzene.

Mix in a dry test-tube three drops of the hydrocarbon, 1 cc. of nitric acid (sp. gr. 1.42), and 1 cc. of sulphuric acid (sp. gr. 1.84). Heat the mixture until it begins to boil, and maintain it at this temperature for half a minute. Then pour slowly into 10 cc. of cold water. Cool quickly. Shake. Collect the bulky flocculent precipitate on a small filter, and wash until the washings are no longer colored. Dissolve in 8 cc. of boiling

dilute alcohol (1:1). Allow to stand until the solution has assumed the room temperature. The liquid will become filled with long, fine, nearly white, needles of m-dinitrobenzene. Collect on a small filter. Wash with 5 cc. of cold dilute alcohol (1:1). Drain on a piece of porous tile and dry fifteen minutes at 50°.

The dinitrobenzene formed in this test melts at 89°-89.5° (uncorr.).

914. Mesitylene.

Allow one drop of the hydrocarbon to fall into a mixture of 2 cc. of sulphuric acid (sp. gr. 1.84) and 1 cc. of fuming nitric acid (sp. gr. 1.48) contained in a dry test-tube. Shake, and then boil very gently for one minute over a small flame. Break up any hard lumps that may form with a stirring-rod, and pour into 10-12 cc. of cold water. Collect the solid nitro-compound on a very small filter and wash well with cold water. Then wash once with 5 cc. of cold strong alcohol. Transfer to a test-tube and boil gently with 15 cc. of 95 per cent alcohol⁺ until all dissolves. (The compound dissolves quite slowly.) Allow to cool. Shake vigorously. Collect the crystalline precipitate in the point of a very small filter. Wash with 5 cc. of cold 95 per cent alcohol⁺⁺. Drain on a piece of porous tile; dry for fifteen minutes at 100°, and determine the melting-point.

The product in this test, trinitromesitylene, is obtained in the form of minute colorless needles melting at

235° (uncorr.).

915. Naphthalene.

Dissolve 0.05 gm. of the hydrocarbon and 0.10 gm. of picric acid in 2 cc. of boiling 95 per cent alcohol. Allow the solution to cool gradually. Collect the long, hair-like yellow needles of the picric-acid compound, $C_{10}H_8 \cdot C_6H_3O$, on a small filter, and wash with 1 cc. of strong alcohol. After draining, transfer to a piece of porous tile, and press out adhering mother-liquor. Form the crystals into a little mound on a dry part of the tile; rinse them off with 5-10 drops of strong alcohol. Repeat the washing with alcohol twice more in the same manner, pressing out the adhering alcohol on a dry part of the tile each time with a small spatula. Spread out the crystals on a bit of dry tile and dry for 15-20 minutes at 50°. Then determine the melting-point.

The picric-acid compound of naphthalene, thus purified melts at 150.5° (uncorr.). (Long continued drying at a high temperature is inadmissible since it causes a gradual loss of naphthalene.)

916. Phenanthrene.

Dissolve 0.10 gm. of the hydrocarbon and 0.20 gm. of picric acid in 5.0 cc. of boiling 95 per cent alcohol. Allow to stand until quite cold. The picric-acid derivative of phenanthrene that forms separates in crystals. Collect on a filter, and allow to drain well without washing. Transfer to a piece of porous tile to absorb the last of the mother-liquor.

Redissolve in 1 cc. of boiling alcohol. Allow to cool slowly as before. Collect the crystals on a piece of tile to absorb the mother-liquor, and wash with five drops of strong alcohol. When the alcohol has nearly all disappeared, place on a fresh piece of tile; dry fifteen minutes at 100°, and determine the melting-point.

The picric-acid compound of phenanthrene, $C_{14}H_{10} \cdot C_6H_3(NO_2)_3$ obtained in this test, forms long, hair-like needles which are orange-yellow when dry, and melt at 143°. (uncorr.)

917. Pseudocumene.

Nitrate two drops of the hydrocarbon by the procedure of Test 914 for mesitylene. Do not increase the quantities of acids and solvents prescribed, but follow the directions given literally, except that more than usual care must be taken not to overheat during nitration. During the operation the test-tube should be held at some distance above the flame, and the heating should be interrupted before the expiration of the minute if the mixture shows signs of darkening, or if a sublimate should begin to appear on the sides of the tube.

The trinitro-pseudocumene formed in this test is a nearly white crystalline compound melting at 184°. (Uncorr.)

918. Toluene.

Dissolve three drops of the hydrocarbon in 1.5 cc. of the strongest fuming nitric acid. Then add at once, without cooling, 1.5 cc. of fuming sulphuric acid (concentrated sulphuric acid containing in solution about 10 per cent of

sulphuric anhydride - the same reagent that is used for Test 902). After half a minute pour the mixture into 10 cc. of cold water in a test-tube. Cool well with running water. Close the tube with the thumb and shake vigorously and persistently until the nitro-compound separates in yellow-white flocks, leaving the solution clear.

Collect the precipitate on a very small filter and wash with cold water. Dissolve in 8 cc. of boiling 50 per cent alcohol. Cool in running water. Shake vigorously. Filter. Wash the precipitate with 5 cc. of cold 50 per cent alcohol. Redissolve the washed precipitate a second time in 8 cc. of boiling 50 per cent alcohol. Cool. Shake. Wash with 5 cc. of 50 per cent alcohol. Dry, and determine the melting-point.

2,4-Dinitrotoluene, the product in this test, is a nearly white precipitate of crystalline structure melting at 70°-71° (uncorr.) (If the solution, in making the last crystallization, is allowed to cool slowly, the compound will separate out in the form of delicate white needles.)

919. m-Xylene.

Nitrate two drops of the hydrocarbon by the procedure of Test 914 for mesitylene. Do not increase the quantities of acids or solvents prescribed, but follow the directions given literally in every detail, except that the precipitate referred to at the point marked by the double asterisk (++) should receive one additional crystallization from 10 cc. of boiling 95 per cent alcohol before being dried.

The trinitro-*m*-xylene formed in this test is a nearly white crystalline compound melting at 181°-181.5° (uncorr.)

920. p-Xylene.

Nitrate two drops of the hydrocarbon by the procedure of Test 914 for mesitylene. Do not increase the quantities of acids or solvents prescribed, but follow the directions given literally in every detail, except that the quantity of 95 per cent alcohol used in crystallizing at the point marked by the single asterisk(+) should be reduced from 15 cc. to 5 cc.

The trinitro-*p*-xylene formed in this test is a nearly white crystalline compound melting at 138.5°-139°. (Uncorr.)

921. o-Xylene.

This hydrocarbon is easily distinguished from the meta and para compounds by the fact that when nitrated by the procedure prescribed in Tests 919 and 920, it gives an oil instead of a solid high-melting nitro-derivative. The following test may also be applied.

Sulphonate 0.25 cc. of the hydrocarbon by persistently shaking in a test-tube with 1 cc. of sulphuric acid (sp. gr. 1.84). During the shaking the tube should be gently warmed from time to time by dipping it for a second or two into boiling water. When the hydrocarbon has all dissolved (this will require 3-5 minutes), cool, and pour slowly into 10 cc. of a saturated solution of common salt. Cool well, and shake vigorously. The mixture will soon become pasty from the separation of a heavy precipitate of sodium *o*-xylenesulphonate. Filter, and wash with 10 cc. of a cold saturated salt solution. Press on a tile, and dry 10

minutes at 125°. Crush.

Mix 4 parts by weight of phosphorus pentachloride with 3 parts of the dry sulphonate in a test-tube, and heat for 10 minutes at about 100°. Cool, and pour in 5 cc. of ice-cold water. Shake. Allow to settle. Decant the water through a wet filter. Wash again by shaking with 5 cc. of cold water, followed by decantation. Return any precipitate that may have collected on the filter to the test-tube. Add 2 cc. of the most concentrated ammonia. Boil gently until the ammonia odor has almost disappeared. Dilute with 10 cc. of water. Heat to boiling. Filter hot. Cool the filtrate well with ice water. Shake vigorously, and collect the precipitate of the sulphonamide on a small filter. Wash with 5 cc. of cold water. Redissolve in 5 cc. of boiling water, and cool. Shake, filter, and wash as before. Repeat these operations twice more. Then dry for 15 minutes at 110° and determine the melting-point.

o-Xylenesulphonamide, the product of this test, crystallizes in pearly-white scales which melt at 143.5°-144°.

922. Determination of Solubility in
Various Organic Solvents.

This test, modified with respect to temperature and solvent, finds several applications in the Sectional Tests of this Genus. To carry out the test it is advisable to prepare a dropper from a long capillary tube, about 35 cm. long and 0.5 mm. in diameter. From a ~~graduated~~ one cc. pipette, ^{graduated to 0.1 cc.} deliver 0.1 cc. of water into a small container and then suck this completely into the dropper, allowing no air spaces in the **stem**. Mark the dropper at the meniscus of the water. It is well to repeat this procedure using 0.15 cc. of water and 0.2 cc. The use of this dropper permits the more ready handling of these small amounts of material than is otherwise possible, although a ~~graduated~~ one cc. pipette, ^{graduated to 0.1 cc.} may be used. A small glass-stoppered test tube is also necessary. This may be prepared in a few minutes by grinding a two cm. length of stirring rod with emery powder and linseed oil, into the fire polished open end of a piece of six mm. soft glass tubing, four or five cm. long, closed at the other end. To conduct the test measure out from the dropper described above 0.1 cc. of the hydrocarbon at room temperature (or, if solid, weigh out about 0.08 gm., corresponding approximately to 0.1 cc.) into

the glass stoppered test tube. Then add 0.1 cc. of solvent, nitromethane, aniline, or benzyl alcohol, or 0.2 cc of hydrobromic acid, as the case may be, shake, and note whether two layers separate. When nitromethane is the solvent it will frequently be found advantageous to add a crystal or two of picric acid, as the partition coefficient for this substance between nitromethane and hydrocarbons insoluble in nitromethane is high, hence the line separating the two liquids is distinct although the refractive indices of the two immiscible liquids may be identical. Temperatures around 20° are most easily attained by using water in a small beaker and cooling with ice if necessary, adding the latter bit by bit. Higher temperatures are readily attained by using nujol in a small beaker. A temperature of -17° may readily be reached by using crushed ice and hydrochloric acid (d: 1.19). For convenience in determining temperatures the tube may be strapped to the bulb of a thermometer by a small elastic band. The solubility of a compound should always be determined by heating the contents of the tube at least ten degrees higher than the temperature at which it is desired to note the solubility. After thorough shaking, if solubility has occurred, the tube should be allowed

to cool gradually. If haziness, followed by separation of two layers, appears above the temperature under consideration, the compound is insoluble. The word "soluble" as here used means that the two liquids are completely miscible at the specified temperature. "Insoluble" means that the two liquids are not completely miscible. The solvents used in this test are prepared as follows:

nitromethane -- Eastman, pure

aniline -- dried by standing over stick,
C.P., potassium hydroxide for
some time, then distilled from
a flask, the first third of the
distillate being discarded, and
the next, constant boiling third
being collected and preserved in
a brown, glass-stoppered bottle.

benzyl alcohol -- Eastman, pure

hydrobromic acid -- colorless, constant
boiling, d. : 1.48.

923. Determination of the Critical Solu-
tion Temperature of Various Hydro-
carbons in Nitrobenzene.

This test is conducted as the solubility tests

in Test 922 except that the water bath in which the test tube containing 0.15 cc. of hydrocarbon and 0.10 cc. of nitrobenzene is cooled gradually with small pieces of ice, or warmed gradually with a small flame while the tube, attached to the thermometer, is agitated constantly. The bath must first be heated above the C.S.T. of the hydrocarbon being studied and then, after the contents of the tube are well mixed, it is allowed to cool slowly. The C.S.T. is the temperature at which cloudiness first appears in the tube. THE C.S.T.'s listed were determined using nitrobenzene dried with calcium chloride and collected from 210.5 - 210.8°. They differ by a few tenths of a degree from those obtained with technical nitrobenzene.

924. Bromide-Bromate Titration for the

Qualitative Detection of Unsaturation. Cf. Test 925 !!

Into a 1 oz. glass-stoppered narrow mouth bottle run two cc. of approximately 0.5 Normal bromide-bromate solution and add 0.15 cc. of hydrocarbon and quickly 1.5 cc. of 10% sulphuric acid. Stopper and **shake** vigorously for two minutes. If at the end of this time there is no color in the solution, add more bromide-bromate solution, one cc. at a time, until

after two minutes of shaking a light yellow color remains. Then add one cc. of 15% potassium iodide solution and a few drops of starch indicator solution. Titrate with 0.2 N. sodium thiosulfate. If titration shows that less than one cc. of bromide-bromate solution has reacted, then the compound is saturated, if more than one cc. has reacted the compound has given a positive test and is unsaturated. In the latter event, save the contents of the tube for Test 927.

If the compound has given a positive test (i.e., added bromine) by means of the following formula it may be found whether it has added more than three atoms of bromine per molecule.

Formula 1.

If $\frac{VNM}{G} > 450$, then the compound has added more than three atoms of bromine per molecule; if $\frac{VNM}{G} < 450$, it has added less than three atoms per molecule.

- V = volume of the bromide-bromate solution
- N = normality of the bromide-bromate solution
- G = specific gravity of the hydrocarbon
- M = average molecular weight of diolefines listed in Div. B, Sect. 3 as having boiling points the same as that of the unknown compound.

925. The Quantitative Determination of
Unsaturation. - The "Bromide-
Bromate" Number.

To the boiling point of the hydrocarbon add 25°. Look through the compounds of Div. B, Sect. 4 which boil at about this temperature and select that one having the greatest molecular weight. This compound is taken as the basis for calculation of the amount of bromide-bromate solution to be used. (This is done to allow for possible errors in the determination of the boiling point, and to make certain that the amount of solution taken is not greater than that required to react with the compound at hand.) The amount of bromide-bromate solution required to titrate 0.5 cc. of this olefine is given in ccs. from the expression $\frac{1000 G}{MN}$, where G is the specific gravity of the hydrocarbon, M its molecular weight, and N the normality of the standard bromide-bromate solution. This quantity of solution is run from a burette into a four ounce glass-stoppered, narrow-mouth bottle. 0.5 cc. of hydrocarbon is added from a ~~graduated~~ 1 cc. ^{graduated to 0.1 cc.,} pipette, and 15 ccs. of ten per cent sulfuric acid are added quickly, the bottle tightly stoppered and shaken vigorously until color no longer develops immediately upon cessation of shaking. If the color

has all disappeared, more bromide-bromate solution is added, one cc. at a time, the bottle being shaken as before, until sufficient has been added to leave a faint yellow color (the faintest detectable) after the bottle has been shaken for two minutes. At this point 5 ccs. more of 10% sulfuric acid are added and the bottle shaken for two minutes longer, or until color disappears if this occurs in less than two minutes. The addition of standard solution is continued until color remains, as before, after two minutes of shaking. Then 5 ccs. of 15% potassium iodide solution are added, and the liberated iodine titrated with 0.2 N. thiosulfate, using starch indicator.

If the substance to be titrated is extremely volatile, it is advisable to put cracked ice in the bottle before beginning the titration. The mixture being titrated must warm up to room temperature before the titration is concluded, however, as the acid liberates bromine only very slowly from the cold bromide-bromate solution, so that a considerable excess of the latter may be added before any color appears at 0°. In some cases it may be necessary to find the approximate amount of solution required by means of a rough titration before a final, accurate titration

can be made. This will not often be the case, and never with compounds boiling above 40°.

The "bromide-bromate number" is the number of centigrams of bromine added by one gram of hydrocarbon. It is given by the expression $\frac{16VN}{G}$, where V is the volume of bromide-bromate solution, N its normality, and G the specific gravity of the hydrocarbon. For a discussion of this titration and the method of preparing and standardizing the solutions, see page 32 and 73.

This titration gives more accurate results than Test 924 and should be run in borderline cases, when the sectional location of a compound may be in doubt from the results of Test 924.

Save the product from this titration in case it is necessary to apply Test 927.

926. Preparation of Derivatives of Mono-substituted Acetylenes.

To an ice cold solution of 3 ccs. of Nessler's reagent (note) ^{cf.} add slowly, with stirring, three drops of the hydrocarbon dissolved in one ccl of reagent alcohol. Filter the precipitate and recrystallize from about 5 ccs. of hot alcohol. If necessary, add a few drops of water to the alcoholic solution to

hasten crystallization. Determine the melting point of the derivative. Derivatives of the higher acetylenes are frequently difficultly soluble in alcohol. They may be recrystallized from benzene, ethyl acetate, and in some cases, petroleum ether. This procedure gives mercury salts of true acetylenes, similar to the copper salts. They are remarkably stable, not exploding below their melting points, although some of them do explode several degrees above.

→ (NOTE) : Nessler's reagent is conveniently prepared after the manner of Johnson and McEwen, J. Am. Chem. Soc. 48, 469 (1926) as follows(

Dissolve 66 gms. (0.486 gm. equivalents) of HgCl_2 in a solution of 163 gms. (0.61 gm. equivalents) of KI in 163 ccs. of water, and add 125 ccs. of 10% NaOH solution (0.31 gm. equivalents). All reagents should be of the C.P. quality.

927. Preparation of Derivatives of Mono-
substituted Ethylenes. (Note 1)

Combine the products from Tests 924 and 925 and add about 10 ccs. of petroleum ether, shake thoroughly, decolorize with thiosulfate if necessary, and separate the petroleum ether solution of the dibromide. Dry this solution by shaking it for a few minutes with anhydrous sodium sulfate. Decant the

petroleum ether solution from the sulfate into a 50 cc. round bottom flask. Wash the sulfate twice with petroleum ether and add the washings to the original solution. Evaporate the petroleum ether from the dibromide by means of an air blast, add about two grams of finely powdered, fresh sodamide (Note ^{cf} 2) to the dibromide, and heat under a reflux in an oil bath held at 150-160° for fifteen or twenty minutes.

Immerse the flask in a freezing solution and slowly add a solution of 5 ccs. of HCl (d.:1.19) in 20 ccs. of water. This may be a violent reaction! Care should be observed! Shake until all of the solid matter is in solution and there is left an oily layer on top. Connect a pipette to an aspirator and draw off the bottom, aqueous layer. Wash the acetylene (oily) layer twice with 20-25 ccs. of water, drawing off the water each time by means of the pipette. To the acetylene remaining in the flask, add 14 ccs. of reagent alcohol, shake until solution is complete, and pour it slowly, with stirring, into an ice cold mixture of 5 ccs. of alcohol and 5 ccs. of Nessler's reagent. (Note ^{cf} 3)

Filter off the precipitate and dissolve it in about 20 ccs. of hot alcohol. Evaporate the

solution to five or ten ccs., filter off the precipitate, dry it on a porous tile and determine its melting point. Recrystallize until the melting point remains constant.

Benzene, ethyl acetate, or petroleum ether may be found more suitable as a solvent for derivatives obtained from high boiling olefines.

For a discussion of this reaction, see par. III.

→ (NOTE 1) Improved procedure of Charles W. Schroeder, developed by him in the course of his special problem in the Course V junior organic chemistry laboratory at M.I.T. in 1930.

→ (NOTE 2) Sodamide for this test may be preserved under xylene, as is sodium, a piece being taken out and wiped off as desired. It loses ammonia rapidly in a glass stoppered bottle, giving a product unsuitable for this test. Only the gray lumps should be used. That portion which is white has hydrolyzed, and that which is yellow is said to be dangerously explosive; it should be carefully destroyed.

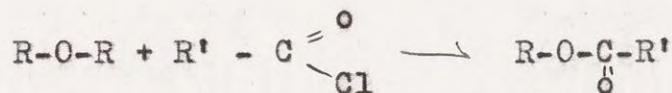
→ (NOTE 3) Nessler's reagent is conveniently prepared after the manner of Johnson and McEwen, J. Am. Chem. Soc. 48, 469 (1926) as follows:

Dissolve 66 gms. (0.486 gm. equivalents) of

HgCl₂ in a solution of 163 gms. (0.61 gm. equivalents) of KI in 163 ccs. of water, and add 125 ccs. of 10% NaOH solution (0.31 gm. equivalents). All reagents should be of the C.P. quality.

928. Preparation of Derivatives of Sym-
metrical Ethers (Note)

To 0.5 cc. of the ether add 0.1-0.15 gm. of anhydrous zinc chloride and 0.3 gm. of 3,5-dinitrobenzoyl chloride in a three inch test tube. Heat under a reflux for 40-60 minutes. Cool and add five ccs. of 10% sodium carbonate solution to the reaction mixture. Filter, wash on the filter with 5 ccs. of 10% sodium carbonate solution and twice with five cc. portions of water. Recrystallize the ester from five ccs. of alcohol and determine its melting point. This test depends upon the following reaction which is general for symmetrical aliphatic esters:



- N.B. 1. If the amount of ester permits, a third recrystallization is advisable+
2. It is necessary to cool the filtrates with ice in order to obtain sufficient ester for recrystallization.

3. If the amount of ether permits the use of one cc., the procedure is easier. In such a case, use 0.5 gm. of dinitrobenzoyl chloride.

(NOTE) This procedure has been developed by Mr. Gilbert C. Toone, in the course of work done with Dr. H. W. Underwood, Jr. for the degree of Master of Science, at M.I.T. It is adopted through their courtesy.

929. Solubility and Color Reactions with Sulfuric Acid.

1. Solubility in sulfuric acid is used for two purposes. The first of these is to distinguish aliphatic ethers of Div. B, Sect. 2. This test is applied as the other solubility tests of Test 922. The tube is kept at 0°, and the acid added is at 0°. If a complete solution of the compound occurs, and remains for two minutes it is probably an aliphatic ether, if reaction does not appear to have taken place. The solution is then diluted, cold, with cold water. If the original compound is obtained again, by dilution, after complete solution, it is an aliphatic ether, and belongs in Div. B, Sect. 2. If the result of this test is doubtful, Test 907 should

be applied.

2. The second purpose for which solubility in sulfuric acid is used is to distinguish compounds like the cyclohexylacetylenes from the aromatics having saturated side chains. In applying this test, the hydrocarbon (0.1 cc.) is cooled down in the tube used in Test 922 to 0°, and 0.1 cc. of sulfuric acid (d.: 1.84) at room temperature is added to the tube while it is surrounded with ice water. The tube is then shaken, and if complete solution has not occurred, it is removed from the ice bath and shaken for a minute at room temperature. If the gravity of the compound tested lies between 0.84 and 0.87 and complete solution has occurred in this test, it is not an aromatic, and does not belong in Div. B, Sect. 1.

In this test chemical reaction, accompanied by marked color formation will frequently be met. The development of color has nothing to do with the term solubility as used here, although compounds which are soluble will always react to give a deep red solution. The solution must be clear, not an emulsion. Aromatic hydrocarbons with saturated side chains frequently give emulsions, they do not give clear solutions. To determine whether a clear solution has resulted in this test, it will frequently be necessary to hold

the tube horizontally to let the reaction mixture spread out, then to hold it toward the light and look through it. If the compound is "soluble" in the acid, not only will heat have been evolved and a deep red color formed, but there will be no sign of a second layer. On dilution of the reaction mixture, in such a case, with water, the oil coming to the top will be found to have a sweet odor different from that of the original hydrocarbon, more resembling that of a ketone; the presence of SO_2 will also frequently be noted. A slight coloration not resulting in complete miscibility should not be mistaken for an affirmative test, neither should the test be considered negative because the reaction mixture develops a high viscosity. Since this test is introduced to provide for very rare compounds of the nature of cyclohexylpropine, it may be expected to give a negative result.

3. The procedure described in the second paragraph of Test 929 may be applied frequently to compounds already found to be in Div. B, Sect. 3 for the purpose of gaining some idea as to the relative location of the double bonds. Acetylenes go completely into solution in the acid, and on dilution with water give rise to an oil of a pleasant (ketone-like)

odor; allenes are violently attacked and may go into solution; conjugated diolefines are vigorously attacked in the cold, giving a red intersurface, on warming they form a thick red paste which on dilution with water leaves a heavy red oil in suspension; diallyl type diolefines ($R-CH=CH-CH_2-CH_2-CH=CH-R'$) are much less attacked than those previously mentioned, they frequently form only unstable, colored emulsions with acid, and on dilution with water they leave a light yellow oil on top of the aqueous layer. For a further discussion of this method of distinguishing highly unsaturated acyclic hydrocarbons, see page 109.

APPENDIX I

Bibliography to the Descriptive Matter

The bibliography given in the next few pages, listed as Appendix I, is divided into four parts, corresponding to the four principal divisions of the descriptive matter of this thesis. To find the reference desired it is necessary first to note the heading of the section in which the allusion to the reference is made, and then to locate it in this appendix under this very same sectional heading.

Only the most important references of those consulted have been included in this bibliography.

The abbreviations of titles of journals given in this appendix accord with those given in the "List of Periodicals" published by "Chemical Abstracts" in 1926, except for titles of discontinued journals, which are abbreviated according to the 1910 Beilstein.

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APPENDIX I

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APPENDIX I

Chapter II, Experimental Work

B. - Methods of Dividing the Hydrocarbons into Groups

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APPENDIX II

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The bibliography given in this appendix is divided into segments corresponding to the Divisions and Sections of the Tables, to which the references belong. To find the reference wanted, first note the Division and Section of the Tables in which the reference number occurs, then turn to exactly the same Division and Section of this appendix and find the number.

The abbreviations of titles of journals given in this appendix accord with those given in the "List of Periodicals" published by "Chemical Abstracts" in 1926, except for titles of discontinued journals, which are abbreviated according to the 1910 Beilstein.

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APPENDIX II.

DIVISION B, SECTION 2.

1. The constants, names, and descriptions of the ethers in this section have all been taken from Mulliken, Identification of Pure Organic Compounds, Vol. I, Genus IX, Division B. No ethers in addition to those listed in this source are tabulated in the tables of this thesis.

The melting points of the 35-dinitro-benzoates are the only exceptions of the above statement. They have been taken from the sources indicated.

The refractive indices of the ethers here listed are not given in the International Critical Tables, except for that of di-isoamyl ether. Other sources have not been consulted for indices of refraction.

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APPENDIX II.

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Biographical Note

The writer was born in Boston in the September of 1905. After receiving his elementary education in the vicinity of Boston he graduated from the Dedham High School in the June of 1922. In the Fall of that year he entered the Massachusetts Institute of Technology. Four years later, due largely to generous scholarship aid granted him by the Institute, he received the degree of Bachelor of Science in Chemistry. In the fall of 1926 he entered the graduate school, receiving in the next June the degree of Master of Science, having presented to the Faculty of the Institute, in partial fulfillment of the requirements for that degree, a thesis which has since been incorporated in a paper by Professor H.W. Underwood and himself, published in the Journal of the American Chemical Society in January, 1930 (page 387), entitled "Catalysis in Organic Chemistry I. Reactions of Ethers with Acid Chlorides, Acids, and Anhydrides". During his years in the graduate school he assisted in the undergraduate organic chemistry laboratory until June of 1929, at which time he was awarded the Bolles Fellowship. In June of 1930 he presented to the Faculty of the Department of Chemistry of the Massachusetts Institute of Technology, in partial

fulfillment of the requirements for the degree of Doctor of Philosophy, a thesis entitled "A New Procedure for the Identification of Hydrocarbons". In this same month he was made the Moore Traveling Fellow for the following academic year.