melted at 113–114° and were shown to be tetrabromostearic acid by the mixed melting point method.

Micro-anal. Calcd. for $C_{18}H_{32}O_2Br_4$: Br, 53.29. Found: Br, 53.08.

The bromides which were soluble in the brominating solution were evidently largely dibromostearic acid.

Micro-anal. Calcd. for $C_{18}H_{34}O_2Br_2$: Br, 36.15. Found: Br, 37.27.

The above data on the liquid acids establish the presence of oleic and linoleic acids in the acetone-soluble fat and indicate that no higher unsaturated acids occur.

Acknowledgment.—The present work was supported in part by grants from the Duke University Research Council and the John and Mary R. Markle Foundation. We wish also to express our gratitude to Drs. D. T. Smith and Donald S. Martin of the Duke University Medical School for advice on growing the fungus.

Summary

1. The lipids of *Monilia albicans* soluble in alcohol and ether and in chloroform have been investigated. These lipids make up 5.3% of the weight of the cells.

2. The lipids were separated into 3% phosphatide fraction and 97% acetone-soluble fraction.

3. The phosphatide had a nitrogen:phosphorus ratio of 1.2:1. On saponification, glycerophosphoric acid was isolated and identified. A mixture of fatty acids was isolated but the individual acids were not identified. The water-soluble portion gave a positive Molisch test, indicating the presence of carbohydrates.

4. The acetone soluble fraction probably consisted of a mixture of glycerides, free fatty acids, and free and bound sterols. After saponification of this fraction, ergosterol, glycerol, and palmitic, stearic, oleic and linoleic acids were isolated from the resulting mixture and identified.

5. The data obtained were compared with the lipids of *Blastomyces dermatiditis*.

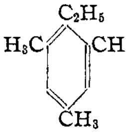
DURHAM, N. C. RECEIVED NOVEMBER 25, 1938

[CONTRIBUTION FROM THE SCHOOL OF CHEMISTRY OF THE UNIVERSITY OF MINNESOTA]

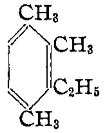
Polymethylbenzenes. XXIII. The Preparation and Physical Properties of 3- and 5-Ethylpseudocumenes and of Ethylmesitylene[1]

BY LEE IRVIN SMITH AND MATTHEW A. KIESS[2,3]

In connection with the studies on the Jacobsen rearrangement, three of the six ethyltrimethylbenzenes have been prepared in quantity and certain of their physical properties have been determined. The three hydrocarbons investigated are ethylmesitylene (I), 3-ethylpseudocumene (II) and 5-ethylpseudocumene (III).



I



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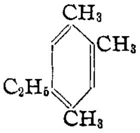

III

Ethylmesitylene (I).—This hydrocarbon was first synthesized by Jannasch and Wigner[4] in 20% yields from bromomesitylene, ethyl bromide and sodium. A short time later, Töhl[5] repeated this synthesis, and he also varied it by using iodomesitylene and ethyl bromide, as well as bromomesitylene and ethyl iodide. Klages[6] obtained a mixture of mesitylene and ethylmesitylene by reducing mesitylmethylcarbinol with hydriodic acid, while Klages and Keil[7] obtained the hydrocarbon in good yield by reduction of vinylmesitylene. Von Auwers[8] reduced acetomesitylene with zinc and hydrochloric acid, and Willgerodt and Scholz[9] reduced the ketone with ammonium sulfide, both reductions giving the hydrocarbon.

In the present work, the hydrocarbon I was synthesized by two methods. Conversion of bromomesitylene to the Grignard reagent and ethylation of the latter produced the hydrocarbon in over-all yields of slightly better than 40% (based upon the mesitylene used). Conversion

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[1] Paper XXII: Nightingale and Smith, THIS JOURNAL, **61**, 101 (1939).

[2] Abstracted from a thesis by Matthew A. Kiess, presented to the Graduate Faculty of the University of Minnesota, in partial fulfilment of the requirements for the Ph.D. degree, November, 1938.

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[4] Jannasch and Wigner, *Ber.*, **28**, 2028 (1895).

[5] Töhl, *ibid.*, **28**, 2459 (1895).

[6] Klages, *ibid.*, **35**, 2256 (1902).

[7] Klages and Keil, *ibid.*, **36**, 1644 (1903).

[8] Von Auwers, *Ann.*, **419**, 120 (1919).

[9] Willgerodt and Scholz, *J. prakt. Chem.*, [2] **81**, 386 (1910).

of mesitylene to acetomesitylene and reduction of the ketone by the method of Clemmensen produced the hydrocarbon in over-all yields of 66%.

5-Ethylpseudocumene (III).—Töhl and Karchowski[10] first synthesized this hydrocarbon from 5-bromopseudocumene, ethyl bromide and sodium. Other syntheses were those of Klages and Keil,[7] who reduced 5-vinylpseudocumene; of von Auwers,[8] who reduced 5-acetopseudocumene with zinc and hydrochloric acid, and of Willgerodt and Scholz,[9] who reduced the ketone with ammonium sulfide.

In the present work, the hydrocarbon III was prepared by the two methods used for the synthesis of I. The action of ethyl sulfate upon the Grignard reagent from 5-bromopseudocumene gave the hydrocarbon in over-all yields of about 35%, while the Clemmensen reduction of 5-acetopseudocumene gave III in over-all yields of about 70%, both yields based upon the pseudocumene actually used in the reactions.

3-Ethylpseudocumene (II).—This hydrocarbon is new. It was prepared by converting 3-bromopseudocumene to the Grignard reagent, which was then ethylated using ethyl sulfate. The yield was 50%, based upon the bromo compound used.

Purification.—When prepared from bromo compounds by ethylation of the Grignard reagents, the crude hydrocarbons were refluxed with sodium ethoxide in ethanol. Water was added and the hydrocarbon was removed by extraction with ether. The solvent was evaporated and the residual material was refluxed for three hours over metallic sodium. After cooling, the sodium was filtered off and the hydrocarbon was fractionated. When prepared by reduction of the ketones, the hydrocarbons were simply washed, dried, and fractionated. The column used was one of the Fenske[11] type, the column proper having the dimensions 30 × 1 cm. It was packed with one-turn helices $^3/_{32}$″ (2.4 mm.) in diameter, made from number 30 B. & S. steel wire. The column was equipped with an electrically heated jacket, and with the arrangement for controlling the reflux ratio and take-off as described by Fenske. One fractionation of the crude hydrocarbons through this column regularly gave materials with boiling ranges of 2° or less, but for the determination of physical properties the materials were refractionated several times and only middle fractions having constant boiling ranges of 0.1° were used.

Vapor pressures were determined by observing the boiling points under different pressures in an apparatus essentially like that of MacDougall and Smith.[12] Thermometers were calibrated, and were totally immersed in the vapor. The manometer was of the closed type with a range up to one atmosphere. Refractive indices were determined by means of an Abbe refractometer. The freezing points were obtained by plotting the cooling curves, using the apparatus and method of Smith and MacDougall.[13] The cooling bath was composed of carbon tetrachloride (m. p. −22.6°) to which dry-ice was added until the temperature of the bath was about 10° lower than the freezing point of the hydrocarbon. Temperature control was maintained easily at any desired point by merely adding small amounts of dry-ice from time to time. The temperature was read every minute on an accurate thermometer graduated to 0.1°.[14]

Results

The *boiling points* of the three hydrocarbons under various pressures are given in Table I. The curves are not given, for those for ethylmesitylene and 5-ethylpseudocumene practically coincide throughout the whole range of pressures, and the curve for 3-ethylpseudocumene follows the other two very closely. The relationships between the pressure and boiling point for the three hydrocarbons are given by the following equations, which hold fairly well at the lower pressures (up to 130 mm.). At higher pressures, the deviations are quite large ($u = 1000/T$).

Ethylmesitylene: $\log_{10}p = 5.329 - 0.226u - 0.4656u^2$
5-Ethylpseudocumene: $\log_{10}p = 4.928 + 0.136u - 0.548u^2$
3-Ethylpseudocumene: $\log_{10}p = 4.303 + 0.671u - 0.664u^2$

TABLE I

VAPOR PRESSURE DATA

Ethylmesitylene		5-Ethylpseudocumene		3-Ethylpseudocumene	
Press., mm.	B. p., °C.	Press., mm.	B. p., °C.	Press., mm.	B. p., °C.
5	70	6	76.2	5	74.5
10.5	85	11	85.8	10	87
16.5	95	18.5	98.3	16	95.5
20	100	29.5	108	31	110
36.5	112.5	61.5	125.4	57	125
63	125.5	94.5	136	76	133.2
91	133.5	171	157	96	139.2
173	156.8	232	166.5	140	148.4
233	166	337.5	179.5	178	154
339	178	448	190.5	235.5	166.9
440	190	654	205	306	172
649	203.5	725	210	393	182.2
725	210			404	185.8
				490	191.6
				563	200
				729	214.5

Indices of refraction were determined every two degrees for the range 0 to 30°, using an Abbe

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(10) Töhl and Karchowski, *Ber.*, **25**, 1530 (1892).

(11) Tongberg, Lawroski and Fenske, *Ind. Eng. Chem.*, **29**, 957 (1937), where other references are also given.

(12) MacDougall and Smith, THIS JOURNAL, **52**, 1998 (1930).

(13) Smith and MacDougall, *ibid.*, **51**, 3005 (1929).

(14) Thermometer made by Rascher and Betzold, number 14165, range +3 to −50°, reading to 0.1° directly. This thermometer was not certified as to accuracy beyond the statement that it was within the limits of accuracy for calibration by the Bureau of Standards.

refractometer. When the values for n_{D} are plotted against temperature, the curves are practically straight lines and those for 5-ethylpseudocumene and ethylmesitylene coincide. The *average* change in n_{D} per degree over the range 0 to 30° is -0.00043 for each of the hydrocarbons.

TABLE II

INDICES OF REFRACTION

Temp., °C.	3-Ethyl-pseudocumene	5-Ethyl-pseudocumene	Ethyl-mesitylene
0	1.5218	1.5161	1.5161
4	1.5202	1.5145	1.5147
8	1.5185	1.5128	1.5129
12	1.5167	1.5113	1.5110
16	1.5150	1.5094	1.5091
18	1.5142	1.5084	1.5082
20	1.5133	1.5075	1.5074
22	1.5124	1.5066	1.5066
24	1.5115	1.5057	1.5057
26	1.5106	1.5048	1.5048
28	1.5098	1.5039	1.5039
30	1.5088	1.5029	1.5028

Freezing points were obtained by plotting the cooling curves, and good "plateaus" were obtained for ethylmesitylene and 5-ethylpseudocumene. 3-Ethylpseudocumene remained liquid at −50°. The *densities* were determined in the usual way. The results are given in Table III, which contains also, for comparison, some results of previous investigators.

TABLE III

PHYSICAL PROPERTIES OF ETHYLTRIMETHYLBENZENES

	Ethylmesitylene	5-Ethylpseudocumene	3-Ethylpseudocumene
Boiling point	210 (725 mm.),[a] 212–214,[4,9] 210–212,[8] 207–209,[5] 207–208[7]	210 (725 mm.),[a] 206–208,[10] 211[8]	214 (725 mm.)[a]
Freezing point	−15.56,[a] liquid at −20[5]	−13.58[a] liquid at −20[10]	Liq. at −50[a]
$n^{20}\text{D}$	1.5074,[a] 1.5130 (16.3°),[8] 1.5091 (18.5°)[7]	1.5075,[a] 1.5077 (18°),[7] 1.5100 (15.7°)[8]	1.5133[a]
d^{20}_{20}	0.894,[a] 0.8885 (16.3°),[8] 0.8907 (18.3°)[7]	0.889,[a] 0.889 (14°),[7] 0.8866 (15.8°)[8]	0.900[a]

[a] Results of the present study.

Experimental

5- and 3-Bromopseudocumenes.—The preparation of 5-bromopseudocumene already has been described, as has the Jacobsen rearrangement of the 5-bromo compound to the 3-bromo compound.[15] But it was discovered that when pseudocumenesulfonic acid-5[16] was brominated in dilute aqueous hydrochloric acid solution, 5-bromopseudocumene separated out, while 3-bromopseudocumene sulfonic acid-5 remained in solution. By hydrolysis of this sulfonic acid, pure 3-bromopseudocumene could be obtained. This method represents a decided saving in time and leads to both bromo compounds in a pure state directly from the hydrocarbon.[17] The sulfonic acid from 160 g. of pseudocumene was dissolved in water (100 cc.) containing hydrochloric acid (100 cc.). To the cooled solution there was added gradually and with stirring a mixture of bromine (223 g.), water (50 cc.) and hydrochloric acid (50 cc.). Crude 5-bromopseudocumene (156 g., 60%) separated at once and was removed by filtration. The filtrate was concentrated and cooled; 3-bromopseudocumenesulfonic acid-5 (56 g., 37%), melting at 106°, was obtained. Hydrolysis of the sulfonic acid (757 g. recrystallized, m. p. 116°) was effected by slowly dropping an aqueous solution of it into 50% sulfuric acid at 175–180° while steam was passed through the mixture. The distillate contained pseudocumene (78 g.) and 3-bromopseudocumene (229 g., b. p. 85.5–86.5° at 5 mm., $n^{20}\text{D}$ 1.5575), while a mixture of polybromo compounds and tars remained in the distillation flask.

Bromomesitylene was prepared from mesitylene[18] in 72% yield; it boiled at 102–103° under 13 mm.

The Grignard Reactions.—As the procedure used was essentially the same for the three bromo compounds, only one example will be described in detail. 5-Bromopseudocumene (200 g.) was dissolved in dry ether (1000 cc.) and the solutiom was dropped slowly onto magnesium (26 g.). The reaction was started by adding a few drops of ethyl bromide, after which 10 cc. of ethyl bromide was mixed with the remainder of the solution of the bromo compound. Most of the metal had dissolved after five hours, but the mixture was heated on the steam-bath for an hour longer, then thoroughly cooled. Ethyl sulfate (308 g.) dissolved in ether (200 cc.) was added slowly with cooling and with rapid stirring until the mass became too thick to stir. After standing for twenty-four hours, the mixture was decomposed with iced hydrochloric acid. The ether layer was separated, washed with water and dried over calcium chloride. After removal of the ether, the residual hydrocarbon was refluxed for thirty minutes with sodium ethoxide in alcohol (30 g. of sodium, 500 cc. of alcohol). The mixture was cooled, ether (300 cc.) was added, and the alkali and alcohol were washed out with water. The

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[15] Smith amd Moyle, THIS JOURNAL, **58**, 8 (1936).

[16] Preparation by Smith and Cass, THIS JOURNAL, **54**, 1606 (1932).

[17] It had been reported previously (ref. 15, p. 8, lower half of second column) that bromination of an aqueous solution of pseudocumenesulfonic acid-5 by alcoholic bromine gave only 5-bromopseudocumene, while bromination of an alcoholic suspension of the sodium sulfonate by bromine in chloroform gave only 3-bromopseudocumenesulfonic acid-5. All subsequent attempts to duplicate these two preparations have failed; the sodium sulfonate remains practically inert, and we do not know what caused the reaction to succeed in the earlier work.

[18] *Org. Syntheses*, **11**, 24 (1931). John Wiley & Sons, Inc., New York.

TABLE IV

PRODUCTS AND YIELDS IN THE PREPARATION OF ETHYLTRIMETHYLBENZENES FROM BROMOTRIMETHYLBENZENES

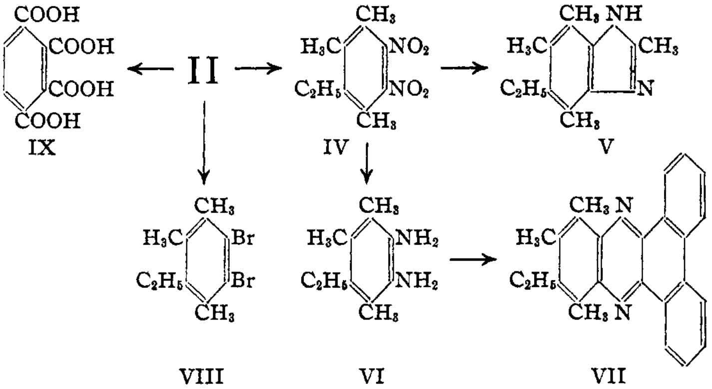
A = 5-bromopseudocumene; B = 3-bromopseudocumene; C = bromomesitylene; D = 5-ethylpseudocumene; E = 3-ethylpseudocumene; F = ethylmesitylene; G = pseudocumene; H = mesitylene.

Bromo compd., g.	Ethyltrimethylbenzene G.	%	B. p., °C.	P., mm.	Trimethylbenzene G.	%	B. p., °C.	P., mm.	Residue G.
100 A	23 D	31	93–99	21.5	15 G	25	67–97	32	15
200 A	66 D	44	94–99	22	33 G	27	60–86	22	16
450 A	176 D	52	92–95	16	60 G	22	59–70	16	38
553 A	132 D	33	92–97	18	116 G	35	60–72	17	60
129 B	50 E	51	92–101	18	11 G	14	60–72	22	20
120 C	53 F	49	93	17	18 H	21	58–59.5	19.5	13
287 C	80 F	38	....	..	42 H	24	....	..	27
355 C	98 F	37	....	..	63 H	29	....	..	15
148 C	54 F	49	....	..	19 H	21	....	..	13

ether solution was dried over calcium chloride and the ether evaporated. The residue was heated with sodium (30 g.) on the steam-bath for three hours, then cooled, filtered and the filtrate was fractionated. The products are shown in Table IV.

Hydrocarbons from the Ketones.—Acetomesitylene and 5-acetopseudocumene were prepared as described previously[19] and were reduced to the hydrocarbons by the method of Clemmensen. 5-Acetopseudocumene (296 g.) was refluxed (one hundred and seventy hours) with amalgamated zinc (950 g.), hydrochloric acid (1200 cc.) and water (1200 cc.). Smaller quantities of acid or zinc, or a shorter period of refluxing proved insufficient for complete reduction. The washed and dried material was fractionated, yielding 5-ethylpseudocumene (208.5 g., 77.2%) boiling at 99.5° under 21 mm., $n^{22}D$ 1.5067. Since the ketone can be obtained from pseudocumene in 90% yields, this method of preparing the hydrocarbon gives over-all yields of about 70%, as compared with 35% yields when the Grignard reaction is used. A similar reduction (one hundred and seventy hours) of acetomesitylene (253 g.) with amalgamated zinc (800 g.), hydrochloric acid (1200 cc.) and water (1200 cc.) yielded ethylmesitylene (171 g., 74%) boiling at 97–98° under 22 mm., $n^{18.5}D$ 1.5082. Acetomesitylene also can be obtained in 90% yields from the hydrocarbon, hence the over-all yield from mesitylene to ethylmesitylene by this method is about 66% as compared with 43% when the Grignard reaction is used.

3-Ethylpseudocumene. Derivatives and Proof of Structure.—*Anal.* Calcd. for $C_{11}H_{16}$: C, 89.19; H, 10.81. Found: C, 89.10; H, 10.75. The structure of the new hydrocarbon was proved by the transformations shown.



5,6-Dinitro-3-ethylpseudocumene (IV).—The hydrocarbon (11.5 g.) was dissolved in chloroform (64 cc.) and the solution was thoroughly stirred with sulfuric acid (47 cc.) at 5°. Fuming nitric acid (8 cc., d., 1.5) was added dropwise during fifteen minutes. The chloroform layer was removed, washed with carbonate and then with water several times, after which it was dried over calcium chloride. The chloroform was removed by evaporation and the residue (7.5 g., 41%) after crystallization from alcohol, weighed 5 g. (28%) and melted at 79–80°.

Anal. Calcd. for $C_{11}H_{14}O_4N_2$: C, 55.46; H, 5.88; N, 11.76. Found: C, 55.65; H, 6.00; N, 11.77.

5,6-Diamino-3-ethylpseudocumene (VI).—The dinitro compound (1 g.) dissolved in hot ethanol (10 cc.) was refluxed for three hours with stannous chloride (8 g.) and hydrochloric acid (10 cc.). The double salt, obtained on cooling the mixture, was removed and decomposed with excess sodium hydroxide. The amine was crystallized twice from a little ethanol. The solution regularly remained supersaturated when cooled, but scratching or inoculation induced crystallization at once. The melting point was 84–85°.

Anal. Calcd. for $C_{11}H_{18}N_2$: C, 74.15; H, 10.11; N, 15.74. Found: C, 74.01; H, 10.35; N, 15.54.

5-Ethyl-2,4,6,7-tetramethylbenzimidazole (V).—The dinitro compound (1 g.) was refluxed for two hours with acetic acid (10 cc.), stannous chloride (10 g.) and hydrochloric acid (10 cc.). The imidazole (about 1 g.) which crystallized on cooling was removed and recrystallized from dilute ethanol (25 cc., 50%). It melted at 205.5°.

Anal. Calcd. for $C_{13}H_{18}N_2$: N, 13.86. Found: N, 13.9.

11-Ethyl-10,12,13-trimethylphenanthrophenazine (VII).—Phenanthraquinone (0.28 g.) dissolved in hot acetic acid (12 cc.) was added to a solution of the diamine (0.2 g.) in warm alcohol (8 cc.). The phenazine precipitated quantitatively at once. It was crystallized twice from

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[19] (a) Noller and Adams, THIS JOURNAL, **46**, 1889 (1924); (b) Smith and Guss, *ibid.*, **59**, 804 (1937).

chloroform, then once from chloroform-alcohol. The melting point was 242°.

Anal. Calcd. for $C_{25}H_{22}N_2$: N, 8.0. Found: N, 7.95.

5,6-Dibromo-3-ethylpseudocumene (VIII).—The hydrocarbon (3 g.) was sulfonated by shaking it vigorously with sulfuric acid (8 cc.) and 20% oleum (4 cc.) for three minutes. The clear solution was poured over ice (300 g.) and bromine (10 g.) was added in 0.5-g. portions with shaking. A yellow emulsion formed immediately, which after standing for three hours at room temperature deposited a yellow solid. Excess bromine was removed by sodium bisulfite, and the solid (6 g.) was then washed thoroughly with alkali. The solid was crystallized twice from alcohol, when it formed white needles which melted at 65–66°.

Anal. Calcd. for $C_{11}H_{14}Br_2$: C, 42.25; H, 4.57; Br, 52.18. Found: C, 42.64; H, 4.68; Br, 52.28.

Prehnitic Acid (IX).—The hydrocarbon (5 g.) was oxidized with 400 cc. of aqueous potassium permanganate (20 g.) containing potassium carbonate (12 g.). After shaking the mixture at room temperature for five hours, it was heated on the steam-bath for thirty hours, when the color had disappeared. The solution was made slightly acid with acetic acid, and lead acetate was added. The lead salt was removed, thoroughly washed, then suspended in water and the suspension was saturated with hydrogen sulfide. The lead sulfide was filtered off and the filtrate was concentrated to about 3 cc. After addition of a few drops of hydrochloric acid, the solution was cooled. It deposited the tetracarboxylic acid in the form of needles. These were removed, dried, extracted with ether and the ether soluble part was recrystallized from a little water containing a few drops of hydrochloric acid. The yield of pure material was only about 100 mg. The acid melted at 236°.[20] In a similar fashion, pyromellitic acid was obtained from 5-ethylpseudocumene, although the yield (about 100 mg. from 5 g. of the hydrocarbon) was again poor. It melted at 270–272°.[20]

Mellophanic acid could not be obtained from ethylmesitylene by the above procedure. The hydrocarbon (10 g.) was heated for two weeks on the steam-bath with a solution of potassium permanganate (50 g.) and sodium hydroxide (10 g.) in water (1500 cc.). Sodium bisulfite (about 1 g.) was added, the solution was neutralized with acetic acid, and filtered to remove manganese dioxide. The organic acid was then isolated via the lead salt as described above. The yield was about 5 g. After drying at 110° for an hour, the acid melted at 238–267° with anhydride formation. When crystallized from acetone, the acid melted at 238–240°.[20] Neutral equivalent: calcd., 80; found, 80.

Summary

1. Three ethyltrimethylbenzenes, namely, 3- and 5-ethylpseudocumenes and ethylmesitylene, have been prepared in quantity and certain of their physical properties have been determined.

2. Since 3-ethylpseudocumene is a new hydrocarbon, its structure has been proved and several derivatives of it have been described.

3. Each of the hydrocarbons has been oxidized to a benzenetetracarboxylic acid. Each gives only the one tetracarboxylic acid to be expected from its structure, though the yield is poor.

[20] Smith and Byrkit, THIS JOURNAL, **55**, 4305 (1933).

MINNEAPOLIS, MINNESOTA RECEIVED NOVEMBER 28, 1938

[CONTRIBUTION FROM THE SCHOOL OF CHEMISTRY OF THE UNIVERSITY OF MINNESOTA]

Prehnitic Acid (Benzene-1,2,3,4-tetracarboxylic Acid)

BY LEE IRVIN SMITH AND EARL J. CARLSON[1]

Some time ago, Smith and Byrkit[2] prepared two of the isomeric benzene tetracarboxylic acids, namely, pyromellitic acid (I) (1,2,4,5) and mellophanic acid (II) (1,2,3,5) and definitely established the orientation of the carboxyl groups in these acids by relating them to compounds of known orientation. This work also established by difference the orientation of the carboxyl groups in the third acid, prehnitic (III) as 1,2,3,4. In the previous work, satisfactory methods were developed for the preparation of acids I and II in quantity from readily accessible materials; these methods consisted largely of improvements in procedures already described in the literature.

At the time of the earlier work, there was no satisfactory method for preparing prehnitic acid (III) in any quantity from common materials. The oxidation of prehnitene—itself rare and costly—was extremely difficult and produced the acid in minute amounts only, and it is generally true that oxidation of the polymethylbenzenes does not constitute a good preparative method for obtaining the corresponding polycarboxylic acids. Freund and Fleischer[3] prepared III in good yield (60%) from 4,7-dimethyl-2,2-diethyl-

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[1] Abstracted from a thesis by Earl J. Carlson, presented to the Graduate Faculty of the University of Minnesota, in partial fulfilment of the requirements for the M.S. degree, June, 1938.

[2] Smith and Byrkit, THIS JOURNAL, **55**, 4305 (1933).

[3] Freund and Fleischer, *Ann.*, **411**, 14 (1916).