configuration.[19-21] Nickel acetylacetonate has been judged to be tetrahedral since it is paramagnetic.[22] However, it has been pointed out by Calvin and Melchior[23] that one can conceive of paramagnetic, square complexes of nickel(II). However, the specific case for which such a hybrid configuration was suggested, nickel disalicylaldehyde, was shown beyond reasonable doubt to have the tetrahedral configuration since it had an X-ray powder pattern resembling that of the corresponding zinc complex but very different from that of the square copper disalicylaldehyde.[24] All of the other metals included in the present study almost certainly form tetrahedral complexes. There is a possibility that the discontinuity in the relationship between the linear free energy relationship is associated with the tendency of copper to form square complexes with β-diketones. Lateral interference between the two ligands may be more ser-

(19) E. G. Cox and K. C. Webster, *J. Chem. Soc.*, 731 (1935).

(20) E. A. Shugam, *Doklady Akad. Nauk S.S.S.R.*, **81**, 853 (1951).

(21) H. Koyama, Y. Saito and H. Kuroyo, *J. Inst. Polytech Osaka City Univ., Sec. C*, **4**, 43 (1953); *C. A.*, **48**, 3097 (1954).

(22) F. P. Dwyer and F. P. Mellor, THIS JOURNAL, **63**, 81 (1941).

(23) M. Calvin and N. C. Melchior, *ibid.*, **70**, 3270 (1948).

(24) D. H. Curtiss, F. K. C. Lyle and E. C. Lingafelter, *Acta Cryst.*, **5**, 388 (1952).

ious in the planar configuration although models do not indicate that the tertiary butyl groups of DPM would be uncomfortably close together in square planar chelates. Alternatively, the sterically hindered ligands may give serious interference with the normal interaction of the metal in a square complex with nucleophilic solvent molecules. This line of argument also must be considered with reservation since coördination compounds of nickel, *rather than copper*, are notoriously prone to accept solvent molecules and change to an octahedral configuration.[25] Since Q_1/Q_2 for the copper–acetylacetone system is only a little larger than for other metals, the effect is almost certainly steric in origin. Since the first formation constants for copper fit the linear free energy relationship well it is unlikely that effect can be attributed to errors in measurement.[26]

(25) A. E. Martell and M. Calvin, ref. 13, p. 283 ff.

(26) K_1 for copper was determined by measuring K_1K_2 and K_2; see Experimental.
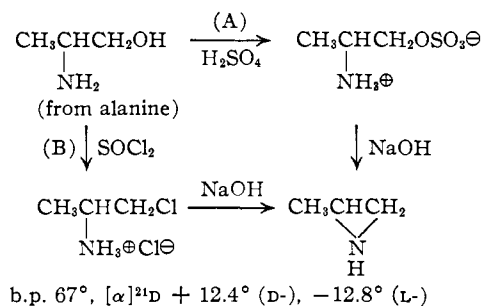
AMES, IOWA

Preparation and Polymerization of D- and L-Propylenimine and N-Methylpropylenimine[1]

BY YUJI MINOURA, MATSUJI TAKEBAYASHI AND CHARLES C. PRICE

D- and L-Propylenimine have been prepared from D- and L-alanine. N-Methylpropylenimine has been prepared from propylene oxide and from alanine. Polymerization of the DL-monomers by a wide variety of catalysts has so far produced only oily polymers. The D- and L-monomers gave solid, optically active polypropylenimine of relatively low molecular weight.

Our interest in propylene oxide polymerization[2] has stimulated an interest in analogous polymers from propylenimine and related monomers. We wish here to report on synthetic work designed to prepare optically active propylenimine and its N-methyl derivative, as well as attempts, so far unsuccessful, to prepare high molecular weight crystalline polymers from these monomers.

b.p. 67°, $[\alpha]^{21}D$ + 12.4° (D-), −12.8° (L-)

Propylenimine has been prepared in racemic form by many investigators. The successful reduction of alanine esters to alaninol with lithium aluminum hydride[3] suggested the following routes (A and B) for the preparation of D- and L-propylenimine. Cyclization through the sulfate ester gave superior yields and purer imine.

N-Methylpropylenimine has apparently not been reported in the literature. We have prepared it by the following alternate routes.
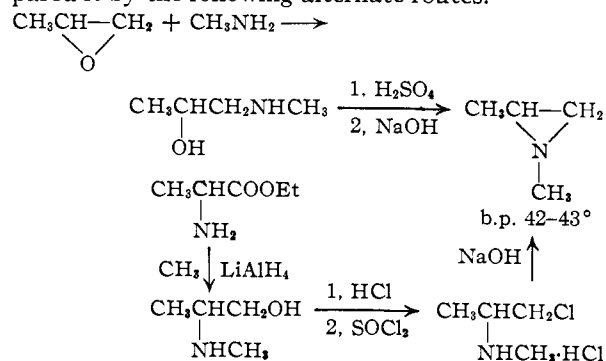
(1) Presented in part at the Delaware Valley Regional Meeting, Amer. Chem. Soc., Feb., 1958, and 135th American Chemical Society Meeting, Boston, Mass., April, 1959. Supported in part under Contract No. 36-039-SC72410, U. S. Army Signal Corps.

(2) C. C. Price and L. E. St. Pierre, THIS JOURNAL, **78**, 3432 (1956); C. C. Price and M. Osgan, *ibid.*, **78**, 4787 (1956).
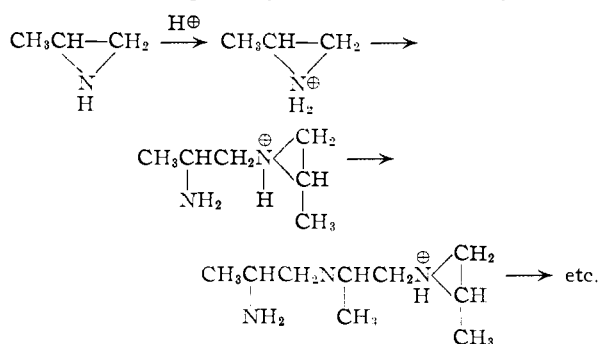
(3) P. Karrer, P. Portmann and M. Suter, *Helv. Chim. Acta*, **31**, 1617 (1948).

Since both starting materials are available in optically active form, both procedures can presumably be adapted to the synthesis of optically-active product.

As has been found earlier for ethylenimine,[4] strong Lewis and mineral acids are the most effective catalysts, while bases, such as potassium hydroxide and carbonate, actually slightly retard autopolymerization of propylenimine. Aluminum alkyls and azoisobutyronitrile are also ineffective as catalysts. Increased amounts of boron fluoride and hydrogen chloride lead to increased conversion to polymer of lower molecular weight.

Polymerization of D- and L-propylenimine (Table II) gave solid polymers, softening about 60–80°, with strong (ca. ±100°) optical rotation of opposite sign to that of the monomer. The high degree of optical rotation and the considerable increase in softening point compared with the DL-isomer suggest a fairly high degree of retention of asymmetry during polymerization. This would be most reasonably explained by S_N2 type ring opening at the primary carbon atom.[4] By such a

$$CH_3CH—CH_2 \xrightarrow{H^\oplus} CH_3CH—CH_2 \longrightarrow$$
$$\underset{\underset{H}{|}}{N} \qquad \underset{\underset{H_2}{|}}{N^\oplus}$$

$$CH_3CHCH_2\underset{\underset{NH_2}{|}}{N}^\oplus\underset{\underset{H}{|}}{\overset{CH_2}{\diagdown}}\overset{|}{\underset{CH_3}{\underset{|}{CH}}} \longrightarrow$$

$$CH_3CHCH_2\underset{\underset{NH_2}{|}}{N}CHCH_2\underset{\underset{H}{|}}{N}^\oplus\underset{\underset{CH_3}{\overset{|}{CH}}}{\overset{CH_2}{\diagdown|}} \longrightarrow etc.$$
$$\qquad\quad \underset{CH_3}{}$$

procedure, no bonds at the asymmetric carbon atom would be made or broken during the polymerization.

Termination could occur by opening the immonium ring by some other nucleophilic reagent than another monomer molecule, e.g., a water molecule, to terminate the polymer chain with a hydroxyl group.

Experimental

Alaninol.—L-Alanine ethyl ester[5] (23.5 g., b.p. 60–61° (24 mm.), $n^{26}D$ 1.4204) in 200 ml. of ether was added dropwise with stirring to 15 g. of lithium aluminum hydride in 300 ml. of ether.[3] After adding 10 ml. of water, the ether was evaporated and the residual mass extracted by alcohol in a Soxhlet apparatus for 10 hours. The alcoholic solution was filtered, and evaporated in vacuum. The residue was mixed with 100 ml. of absolute alcohol and 100 ml. of ether and left standing overnight. A small amount of inorganic precipitate was removed and the filtrate was fractionated under reduced pressure to give 10–13 g. of L-alaninol, b.p. 84° (23 mm.), 91° (26 mm.), $n^{19}D$ 1.4493–1.4495, $[\alpha]^{26}D$ +21.8° (0.294 g./10 ml. in ethanol). D- and DL-alaninol were produced by the same method as mentioned above; D-alaninol, $[\alpha]^{25}D$ −21.8° (0.275 g./10 ml. in ethanol).

2-Aminopropylsulfuric Acid.—A cold mixture of 200 g. of concentrated sulfuric acid and 200 g. of water was added drop by drop, with agitating and with cooling by an ice-bath, to a solution of 150 g. (2 moles) of alaninol in 120 ml. of water in a 2-l. flask. After 300–320 ml. of water had been distilled from the mixture (at 30 mm. pressure), the liquid

was transferred to a plate and crystallized by cooling. The solidified product was powered in a mortar and washed with 100 ml. of methanol. The white crystalline 2-amino-propylsulfuric acid so obtained weighed 255 g. (90% yield), m.p. 231° (DL-). In the same way, D- and L-2-amino-propylsulfuric acid were also obtained; m.p. 246°(L-), 247° (D-).

Anal. Calcd. for $C_3H_9NSO_4$: C, 23.22; H, 5.85; N, 9.07; S, 20.67. Found: (DL-) C, 23.46; H, 5.90; N, 8.88; S, 20.35. (L-) C, 23.57; H, 5.13; N, 8.70; S, 20.61. (D-) C, 23.80; H, 6.27; N, 8.79; S, 19.87.

Propylenimine. Method A.—White solid sulfate from the above operations was placed in a 2-l. distilling flask and a cold solution of 200 g. (5 moles) of sodium hydroxide in 800 ml. of water was added to the solid in the flask; heat was applied by means of an oil-bath and 450 to 500 ml. of distillate was collected. The head temperature ranged from 80 to 100°. The distillate was cooled and saturated with 200 g. of alkali; the organic layer which formed was separated and dried over potassium hydroxide pellets (25 g.) in a refrigerator overnight. The organic layer was separated from the drying agent and distilled from a few fresh pellets of potassium hydroxide through a column. Propylenimine distilled at 66–7°, yield 68 g. (60%), $n^{27.5}D$ 1.4156, $d^{27.5}$ 0.7934.

Anal. Calcd. for C_3H_7N: C, 63.15; H, 12.27; N, 24.56. Found: (DL-) C, 62.86; H, 12.23; N, 24.24.

D- and L-Propylenimine were obtained in a similar way; b.p. 66–67°, m.p. −44° (D- and L-), $[\alpha]^{25}D$ +12.4° (D-) (0.258 g./10 ml. in ethanol), −12.8° (L) (0.227 g./10 ml. in ethanol).

Method B.—L-Alaninol (7.5 g., 0.1 mole) was converted to the hydrochloride by treating with a slight excess of 36% hydrochloric acid (10 ml.) in alcohol solution and removing the excess acid and solvent *in vacuo*. The hydrochloride, which crystallized in the form of a white mass, was mixed with 40 ml. of chloroform by heating gently and then 18 g. of thionyl chloride was added slowly to the well-stirred hot mixture. After the reaction was over, the solvent and volatile reaction products were removed by suction at 70°. The product crystallized on cooling; yield, 12 g. (83%).

A concentrated, warm, aqueous solution of β-chloroiso-propylamine hydrochloride was added to 30 ml. of warm, 40% sodium hydroxide solution. Reaction occurred immediately, and the distillate was collected. The distillate was mixed with 30 ml. of ether and the mixture was shaken with anhydrous potassium carbonate. The ether solution was fractionated by using an efficient column. The fraction (1.6 g.) boiling at 62–64° was collected. The product, L-propylenimine, was distilled again, b.p. 64°,[6] yield 0.9 g. (19%).

1-Methylamino-2-propanol was prepared essentially according to Krassuski.[7] In a flask, provided with a dropping funnel and a mechanical stirrer and cooled by a mixture of ice and salt, was placed 500 g. (6.4 moles) of 40% aqueous methylamine solution. After the stirred mixture has cooled to −5°, 87 g. (1.5 moles) of propylene oxide was added slowly without allowing the temperature to rise above 5° during the course of about three hours, and the stirring was continued overnight. Then the excess of methylamine was driven out under reduced pressure, and the residue was shaken with anhydrous potassium carbonate in order to separate the product. After distillation twice using an efficient column, 87 g. (66%) of 1-methylamino-2-propanol, boiling at 73–74° (26 mm.), m.p. 17°, was obtained.

Anal. Calcd. for $C_4H_{11}ON$: C, 53.89; H, 12.44; N, 15.72. Found: C, 53.33; H, 12.54; N, 15.67.

N-Methylpropylenimine. Method A.—The imine was prepared by the method of Wenker.[8] An equimolar mixture of methylaminopropanol (90 g.) and 98% sulfuric acid (100 g.) was heated rapidly over a free flame until charring began. About 80% of one mole of water was expelled, and the product began to turn brown. On cooling, the product changed to a brown mass. After addition of 250 g. of 40% sodium hydroxide solution, it was distilled. On addition of

(4) See, *e. g.*, G. D. Jones, A. Langsjoen, M. M. C. Neumann and J. Zomlefer, *J. Org. Chem.*, **9**, 125 (1944); G. D. Jones, *ibid.*, **9**, 484 (1944).

(5) E. Fischer, *Ber.*, **34**, 442 (1901).

(6) This material had $[\alpha]^{25}D$ +17°, presumably from contamination with polymer. If any chloroamine is distilled with the imine, polymerization by hydrogen chloride catalysis could occur.

(7) K. Krassuski, *Chem. Zentr.*, **97**, I, 617 (1926).

(8) H. Wenker, THIS JOURNAL, **57**, 2328 (1935); *Org. Syntheses*, **30**, 38 (1950).

solid potassium hydroxide to the distillate, 40 g. of crude imine separated. The base was dried repeatedly with potassium hydroxide, then with metallic sodium, and fractionated. The fraction boiling at 42–43° was N-methylpropylenimine; yield 21–25 g. (30–35%).

Anal. Calcd. for C₄H₉N: C, 67.55; H, 12.75; N, 19.70. Found: C, 67.46; H, 12.71; N, 19.66.

Ethyl α-Methylaminopropionate.—In a flask fitted with a reflux condenser, a stirrer and a separatory funnel were placed 23 g. (0.2 mole) of alanine ethyl ester, 25 g. of potassium carbonate and 50 ml. of alcohol. A mixture of 28 g. (0.2 mole) of methyl iodide and 40 ml. of alcohol was run in slowly from the separatory funnel, vigorous agitation being maintained. Since the reaction took place with evolution of heat, the flask was surrounded by water (27°), and the stirring was continued overnight. The reaction mixture was filtered, and the filtrate was fractionated under reduced pressure, b.p. 78–80° (60 mm.), yield 5.5 g. (20%).[9]

N-Methylalaninol.—The experimental procedures were similar to those in the case of alaninol. α-Methylaminopropionate (13 g., 0.1 mole) was reduced by 8 g. of lithium aluminum hydride in ether solution. The product, boiling at 48–51° (23 mm.), was obtained in 4 g. (45%) yield.

N-Methylpropylenimine. Method B. N-Methylalaninol (4 g., 0.045 mole) was converted to the hydrochloride and then to methyl-β-chloroisopropylamine hydrochloride by the action of thionyl chloride (6. g.) in chloroform. The resulting hydrochloride (5 g.) was heated with 8 ml. of 40% sodium hydroxide solution, giving rise to N-methylpropylenimine, b.p. 42–43°, yield 0.5 g. (20%). The infrared spectra of this material is summarized in Table I.

TABLE I

INFRARED SPECTRA OF 1-METHYLAMINO-2-PROPANOL AND
N-METHYLPROPYLENIMINE (FILMS)

1-Methylamino-2-propanol		N-Methylpropylenimine		N-Methylpropylenimine	
λ, μ	% Abs.[a]	λ, μ	% Abs.[b]	λ, μ	% Abs.[b]
3.0–3.2	91	2.32	27	7.16	93
3.4–3.6	93	2.95	37	7.33	75
6.9	89	3.03	35	8.02	91
7.29	86	3.29	83	8.40	88
7.5	83	3.4–3.5	95	8.77	80
7.7	76	3.60	89	8.99	90
8.0	73	4.65	24	9.20	74
8.49	79	5.0	21	9.53	90
8.8	89	5.13	20	10.03	42
9.05	90	5.40	20	10.76	77
9.45	86	6.10	39	11.48	70
10.25	77	6.53	42	13.2–13.3	90
10.7	80	6.8–6.9	92		
11.0	81				
11.5–12.0	83				

[a] Base line absorption, 42%. [b] Base line absorption, 12%.

General Technique of Polymerization.—Propylenimine (usually 3 ml., 2.4 g.) was placed in a heavy hard glass tube, which was cooled in a Dry Ice–acetone-bath, avoiding entrance of moisture, and catalyst was added. After being flushed with dry nitrogen, the glass tube was immediately sealed. The sealed tube was agitated and placed in a constant temperature bath (at 80 ± 0.5°), or left standing at room temperature for the appropriate times. After the required reaction time, the tube was opened and rinsed out with absolute ethanol. Unreacted imine and alcohol were evaporated, and the polymer was dried to constant weight at 60° *in vacuo* for three days. The polymers were refined by dissolving in absolute ethanol and precipitating by pouring into anhydrous ether.

Monomer alone at 80° for twenty days, or with added bases such as potassium carbonate or hydroxide, gave less than 1% conversion to polymer. Addition of *ca.* 1% of a number of compounds such as water, carbon dioxide, triphenylphosphine or its oxide, triphenylarsine and acetic acid (about 1%) gave 10–20% conversion to polymer.

Strong acids, such as aluminum chloride, ferric chloride, boron fluoride or its etherate, hydrochloric, sulfuric or nitric acids and p-toluenesulfonic acid gave essentially quantitative conversion to polymer. The polymers were all yellow or brown viscous oils or gums.

Intrinsic viscosities were calculated from the viscosities determined in alcohol at 30 ± 0.01°, using an Ubbelohde viscometer. The values of [η] were between 0.09 and 0.13. For hydrogen chloride and boron fluoride, decreased catalyst concentration gave decreased conversion to polymer but of increased viscosity.

Polymerization of D- and L-propylenimine was carried out as described above and the polymers were refined by precipitation. The results are shown in Table II. These polymers were very hygroscopic. The softening point of these optically-active polymers was considerably higher than the racemic polymer.

TABLE II

POLYMERIZATION OF D- AND L-PROPYLENIMINE (3 ML.,
2.4 G.) AT 80°

Imine	Cat.	Cat. wt.	Yield of polymer, g.	[η]	Softening point, °C.[θ]	[α]²⁵D
D	HCl	[a]	2.41	0.119	67	− 93.6°
D	BF₃	[a]	2.48[b]	.128	65	− 99.32
D	BF₃·Et₂O	0.2 ml.	2.41	.152	73	−101.46
L	HCl	[a]	1.60[c]	.101	73	+ 81.50
L	BF₃	[a]	1.56	.112	60	+102.78
L	BF₃·Et₂O	0.1 ml.	1.49[d]	.166	83	+109.27
DL	HCl	[a]	2.48[e]	.113	15	0
DL	BF₃	[a]	2.49	.121	11	0
DL	BF₃·Et₂O	0.2 ml.	2.42[f]	.147	17	0
D	p-Toluene sulfonic acid	0.05 g.	1.02	.091	..	−93.33

[a] Gas bubbled in for two minutes. *Anal.* Calcd. for C₃H₇N: C, 63.15; H, 12.27; N, 24.56. Calcd. for C₃H₇N·H₂O: C, 47.98; H, 12.07; N, 18.77. Found: [b] C, 53.84; H, 10.98; N, 21.12. [c] C, 50.50; H, 10.60; N, 19.50. [d] C, 52.38; H, 10.63; N, 20.25. [e] C, 52.76; H, 10.80; N, 20.70. [f] C, 57.32; H, 11.61; N, 21.70. [θ] The approximate temperature of transition from an opaque resin to a clear viscous oil.

They were soluble in methanol, ethanol and water, and insoluble in ether, acetone, benzene and dioxane.

Polymerization of N-Methylpropylenimine was studied with addition of 1–3% of such compounds as copper amide, aluminum isopropoxide and anhydrous ferric chloride at 35 and 80° in an atmosphere of nitrogen. A trace of moisture seemed to retard the polymerization. Ferric chloride was removed from polymer by washing a benzene solution of the product with a saturated aqueous solution of sodium phosphate; boron fluoride was removed by shaking with dilute alkaline solution; and the other catalysts were removed with dilute hydrochloric acid solution. In the latter case, the benzene solution of polymer was then neutralized by sodium carbonate.

Boron fluoride gave quantitative conversion to polymer in two weeks at room temperature. Ferric chloride gave quantitative conversion in two weeks at 80° when more than 2% was used; 1% gave only about 65% conversion after a month. In a month, cupric amide[10] gave no polymer; cupric cyanide and aluminum isopropoxide gave about 15% conversion.

The polymer produced by the catalytic action of ferric chloride was dissolved in hot acetone to make an approximately 2% solution, cooled to about −30°, and the precipitated polymer was collected by decantation while cold. The operation was repeated three times, and the pale precipitate (*ca.* 20% of original crude polymer), which melted at about −10°, was dried *in vacuo* at room temperature. The intrinsic viscosity of these polymers in benzene was found to be 0.09–0.12. The cyanate test indicated this polymer to be free of ferric ion. Cryoscopic molecular weight determinations in benzene indicated these fraction-

(9) N. Zelinski, *J. Russ. Phys. Chem. Soc.*, **43**, 1091 (1911), prepared this ester from N-methylalanine and reported b.p. 75–76° (65 mm.).

(10) F. F. Fitzgerald, THIS JOURNAL, **29**, 656 (1907).

ated polymers had molecular weights of not more than 2000–4000.

In the same way, the polymer produced by boron fluoride was fractionated in acetone solution. Nearly colorless polymers (gummy at room temperature) were obtained in 20–25% yield.

The polymerization of N-methylpropylenimine in the presence of triisobutylaluminum failed, while triethylaluminum at 80° in the atmosphere of nitrogen gave 10–20% conversion in one month.

In other experiments, copper phenyl[11] and silver phenyl[12] were used as possible catalysts for the polymerization of N-methylpropylenimine. However, the compounds showed practically no effect on polymerization other than to turn the monomer suspensions dark brown or black.

(11) R. Reich, Compt. rend., 177, 322 (1923).
(12) E. Krause and B. Wendt, Ber., 56, 2064 (1923).

PHILADELPHIA 4, PENNA.

The Structure of the Alcohol Obtained by the Hydration of Myrcene

BY WILLIAM J. HOULIHAN,[1] JOSEPH LEVY AND JOSEF MAYER

Evidence is brought forward that myrcenol is most likely 3-methylene-7-methyl-1-octen-7-ol. Tetrahydromyrcenol has been shown by synthesis to be identical with DL-2,6-dimethyl-2-octanol.

The alcohol, $C_{10}H_{18}O$, obtained by the sulfuric acid-catalyzed addition of acetic acid to myrcene (I) and subsequent hydrolysis of the intermediate ester, has been the subject of investigation by a number of workers.[2–6] Power and Kleber,[2] the first of these, concluded that this alcohol was identical with linalool. Barbier,[3] however, maintained that the alcohol was not linalool but a new isomeric alcohol called myrcenol. From a study of the products from dichromate oxidation he incorrectly assigned structure[7] IX to myrcenol.

IX   X   XI
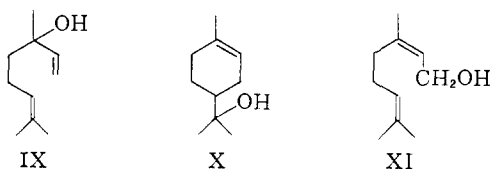
A reinvestigation of the alcohol by Semmler[4b] and co-workers led them to conclude that myrcenol was not a single alcohol but a mixture of linalool and α-terpineol (X). However, Enklaar[5] isolated a phenylurethane from myrcenol that differed from that of linalool, α-terpineol or the isomeric geraniol (XI), thereby indicating that myrcenol was a distinct alcohol of unknown structure. Recently, Milas[6] in a patent states that the acid-catalyzed addition of acetic acid to myrcene furnishes a mixture of the acetates of linalool and geraniol.

Since myrcenyl acetate has value as an odorant it became of interest to determine the structure of this substance. We wish to present evidence that myrcenol is 3-methylene-7-methyl-1-octen-7-ol (III).

(1) Department of Chemistry, Seton Hall University, South Orange, N. J.
(2) Power and Kleber, Pharm. Rundschau (N. Y.), 13, 60 (1895).
(3) P. Barbier, Bull. soc. chim. France, [III] 25, 688 (1901).
(4) (a) O. Tiemann and F. W. Semmler, Ber., 28, 2126 (1895); (b) F. W. Semmler, ibid., 34, 3128 (1901); (c) F. W. Semmler and E. W. Mayer, ibid., 44, 2009 (1911).
(5) P. Enklaar, Rec. trav. chim., 26, 167 (1907).
(6) N. A. Milas, U. S. Patent 2,467,330, April 12, 1949.
(7) This structure has been rigorously proved (by synthesis) to be the correct structure for linalool; cf. L. Ruzicka and P. Fornasir, Helv. Chim. Acta, 2, 182 (1919).

The treatment of myrcene with a cold acetic–sulfuric acid mixture gave an acetate fraction in ca. 25% yield. Careful distillation of this material gave a monoacetate of composition $C_{12}H_{20}O_2$. The ultraviolet spectrum of this compound was identical with the spectrum of myrcene. Saponification of the acetate with ethanolic sodium hydroxide afforded an alcohol, $C_{10}H_{18}O$, in 75% yield. Its ultraviolet spectrum also exhibited the same characteristic myrcene curve. The infrared spectrum showed a band at 8.35 μ which can be assigned[3] to a tertiary C–O absorption. The treatment of this alcohol with active manganese dioxide under controlled conditions failed to furnish an aldehyde or ketone. Geraniol (XI) under identical conditions gave a 69% yield of citral. On the basis of the above physical and chemical information the structure of the $C_{10}H_{18}O$ alcohol, myrcenol, can best be represented by III and that of myrcenyl acetate as II.

The gross skeleton of myrcenol was established by catalytic reduction to tetrahydromyrcenol which was identical with DL-2,6-diethyl-2-octanol (IV).

I   II   III   IV

V   VI   VII   VIII
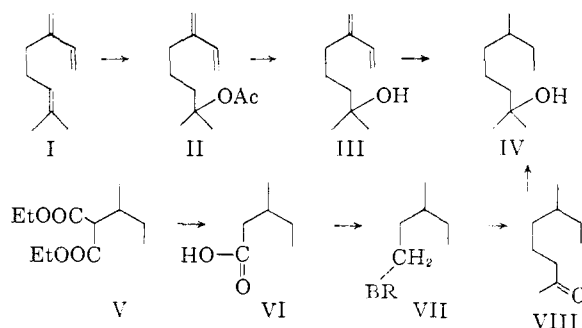
The synthesis of IV was accomplished by starting with diethyl sec-butyl malonate (V). The saponification and decarboxylation of this compound gave DL-3-methylpentanoic acid (VI). Lithium aluminum hydride reduction of VI gave

(8) The C–O absorption band for t-butyl alcohol is reported as 8.3 μ; cf. H. H. Zeiss and M. Tsutsui, THIS JOURNAL, 75, 897 (1953).