(B) **Product from 2-Butene.**—The chlorooctane (b.p. 58–59° at 21 mm., n^{20}_D 1.4427–1.4491) obtained by the condensation of *t*-butyl chloride with 2-butene in the presence of ferric chloride (expt. 17) was converted to octene by heating with water at 200°. Experiments in which 90–135 g. of the chloride was heated with 150 cc. of water in a sealed tube yielded 65–100 g. of product which still contained chlorine; distillation of a sample indicated that about 15% of the product was unreacted. The material was therefore further dehydrochlorinated by heating at 225° with an aqueous suspension of calcium hydroxide: two runs were made in each of which 220–240 cc. of the olefin–chlorooctane mixture was treated with 10 g. of calcium oxide and 50 cc. of water. The product which contained only traces of chlorine boiled chiefly at 103–112°, n^{20}_D 1.4195–1.4265. Hydrogenation yielded octane boiling at 110–112°, n^{20}_D 1.4036–1.4051. It was shown by infrared analysis to consist of 52% 2,2,3- and 48% 2,3,4-trimethylpentane.

Acknowledgment.—The authors wish to express their gratitude to Dr. W. S. Gallaway[13] and Mr. Edmond Baclawski of the Physics Division, Universal Oil Products Company, for the infrared analysis of the reaction products. We are also indebted to Prof. H. C. Brown of Purdue University for the sample of 3-chloro-2,2,3-trimethylpentane and to Prof. E. B. Reid of The Johns Hopkins University for the semicarbazone of 5,5-dimethyl-3-hexanone.

[13] Beckman Instrument, Incorporated, South Pasadena, California.

Riverside, Illinois

[Contribution from the Ipatieff High Pressure and Catalytic Laboratory, Department of Chemistry, Northwestern University]

Studies in the Terpene Series. XVII.[1] The Thermal Isomerization of Pinane at High Pressure[2,3]

By V. N. Ipatieff,[4] William D. Huntsman[5] and Herman Pines

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Pinane was isomerized at 400° and 815 atmospheres. The major product appeared to be 1-*trans*-2-dimethyl-*cis*-3-isopropenylcyclopentane. The yield of this compound and double bond isomers of it totaled 34% of the pinane converted. In addition, an unidentified isomeric bicyclic hydrocarbon, other alkenylcyclopentanes and higher boiling hydrocarbons were formed.

The non-catalytic isomerization of terpenes and hydroterpenes at high temperatures has been the subject of a number of investigations.[6] However, the effect of pressure on these reactions has not been reported. For that reason, a study of the isomerization of pinane at 815 atmospheres was undertaken.

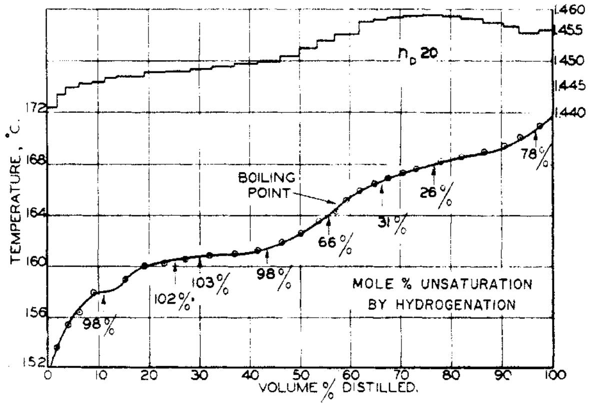

Fig. 1.

The reaction was carried out in a copper tube packed with copper pellets, which has been shown to exhibit no catalytic activity. A temperature of 400° was found to be adequate for reaction at 815 atmospheres, although pinane underwent no appreciable change at 400° and atmospheric pressure.

The product, obtained in 99% yield, comprised a mixture of liquid hydrocarbons of which about 3% distilled below 152°, 69% distilled at 152–172°, and the remainder boiled above 172°. The 152–172° fraction was investigated in some detail.

A fractionation curve of this material is shown in Fig. 1, where the percentage unsaturation for several of the fractions, as determined by hydrogenation, is also shown. A well-defined plateau is indicated at 160–162° and, in fact, infrared spectra of first, center and last cuts in this boiling range were identical. This material absorbed hydrogen equivalent to one double bond per mole and its infrared spectrum had a strong band at 880 cm.$^{-1}$ indicative of a disubstituted terminal double bond, $R_1R_2C{=}CH_2$.[7] It was identified as 1-*trans*-2-dimethyl-*cis*-3-isopropenylcyclopentane (I) by chemical degradation to known hydrocarbons shown.

Ozonization gave formaldehyde and a ketone, $C_9H_{16}O$ (III), which gave a positive iodoform test. Oxidation of the ketone with sodium hypobromite gave an acid, $C_8H_{14}O_2$ (IV). Reduction of this acid with lithium aluminum hydride yielded a primary alcohol, $C_8H_{16}O$ (V). Hydrogenolysis of this primary alcohol at 160–180° in the presence of nickel–kieselguhr[8] gave practically pure 1-*trans*-2-dimethylcyclopentane.

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[1] For the previous paper of this series, see V. N. Ipatieff, F. J. Pavlik and H. Pines, This Journal, **75**, 3179 (1953).

[2] This work was made possible, in part, by the financial assistance of the Universal Oil Products Company.

[3] Presented before the Organic Division of the American Chemical Society Meeting at Los Angeles, Calif., March 16–19, 1953.

[4] Deceased, November 29, 1952.

[5] Postdoctoral Research Associate, 1950–1951.

[6] (a) B. A. Arbuzov, *Ber.*, **67B**, 1946 (1934); (b) G. Dupont and R. Dulou, *Compt. rend.*, **201**, 219 (1935); (c) R. E. Fuguit and J. E. Hawkins, This Journal, **67**, 242 (1945); (d) L. A. Goldblatt and S. Palkin, *ibid.*, **63**, 3517 (1941); **66**, 655 (1944); (e) L. Rummelsburg, *ibid.*, **66**, 1718 (1944); (f) T. R. Savich and L. A. Goldblatt, *ibid.*, **67**, 2027 (1945).

[7] H. W. Thompson and O. H. Whiffen, *J. Chem. Soc.*, 1412 (1948).

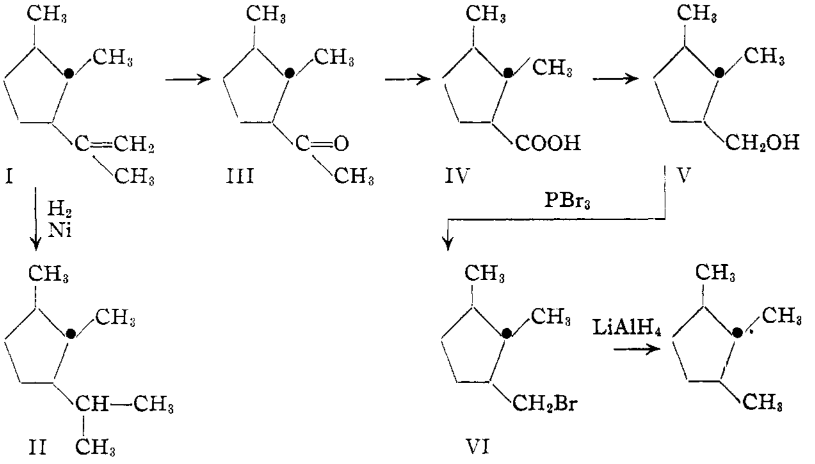
[8] V. N. Ipatieff, G. J. Czajkowski and H. Pines, This Journal, **73**, 4098 (1951).

TABLE I

ANALYSIS OF 162–172° FRACTION FROM THERMAL ISOMERIZATION OF PINANE

Fraction	1	2	3	4	5
Boiling range, °C.	161.9–164.3	164.3–166.5	166.5–167.6	167.6–169.4	169.4–171.7
Weight, g. (pinane) %	44 (6.6)	42 (6.3)	39 (5.8)	78 (11.6)	44 (6.6)
Olefins by hydrogenation, %	85	53	30	33	69
Composition by chromatography					
Monocyclic olefins,[a] %	88	52	21	18	70
B.p. after H_2-ation, °C.	159–160	159–161	160–162.5	164–166[b]	166–168
$n^{20}D$ after H_2-ation	1.4340	1.4368	1.4348	1.4362	1.4389
Compound II yield, %	95	90	75	50	0–10[c]
Bicyclic saturates, %	12	48	79	82	30
Boiling range, °C.	162–163	164–165	165–166[d]	164–165.5	164–166.5
$n^{20}D$	1.4574	1.4600	1.4610	1.4590	1.4550
Pinane content, %	35	55	60	75	70

[a] Includes aliphatic diolefins. [b] *Anal.* Calcd. for $C_{10}H_{20}$: C, 85.63; H, 14.37. Found: C, 85.78; H, 14.50. [c] The hydrogenation product was mainly 1-*cis*-2-dimethyl-*cis*-3-isopropylcyclopentane. [d] *Anal.* Calcd. for $C_{10}H_{18}$: C, 86.91; H, 13.08. Found: C, 86.76; H, 13.10.



The product from ozonolysis also yielded an acidic fraction containing acid IV. In order to establish the position from which the –CH_2OH group was removed, part of the alcohol V was converted to a bromide, $C_8H_{15}Br$ (VI). Reduction of this bromide with lithium aluminum hydride in refluxing tetrahydrofuran[9] yielded the known 1-*trans*-2-*cis*-3-trimethylcyclopentane of 90% purity.

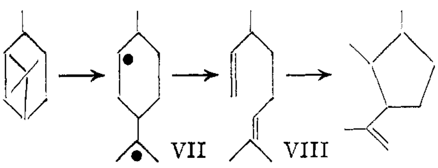
This degradation sequence established the structure and stereochemical configuration of the major component of the 160–162° plateau. In order to determine quantitatively the amount of this compound I and its double bond isomers produced in the isomerization reaction, a carefully purified center cut from the 160–162° plateau was hydrogenated to obtain a specimen of 1-*trans*-2-dimethyl *cis*-3-isopropylcyclopentane (II) for use as an infrared standard. A composite of the material from the 160–162° plateau was then hydrogenated and analyzed for II by infrared spectrophotometry. In this way it was found that the 160–162° plateau comprised 95% of compound I and, possibly, double bond isomers of I which would also furnish II on hydrogenation.

For analysis of the material boiling 162–172° (Fig. 1), it was divided into five fractions, each with a boiling range about 2°, and these fractions were passed over silica gel[10] to separate monocyclic olefins from bicyclic saturated hydrocarbons. The saturated hydrocarbons were subjected directly to infrared analysis; the unsaturated hydrocarbons were hydrogenated before being analyzed. The analytical data so obtained are summarized in Table I.

The analytical data summarized in Table I and those obtained on the 160–162° fraction showed that the material boiling between 160 and 172°, amounting to 55% of the pinane charged, contained 42% of compound I and its double bond isomers, 6% of monocyclic olefins yielding 1-*cis*-2-dimethyl-*cis*-3-isopropylcyclopentane[11] on hydrogenation, 8% of an unidentified bicyclic dihydroterpene and 16% of unreacted pinane. The stability of the bicyclic dihydroterpenes to pyrolysis and to dehydrogenation indicated the absence of a three- or four-membered ring.

The total yield of compound I and its double bond isomers, including some found in the material boiling 152–160° (Fig. 1), was calculated to be 34% of the pinane converted.

Formation of I from pinane may involve a biradical VII and diolefin VIII as



Such a mechanism is analogous to that proposed for the thermal isomerization of α-pinene.[12] The cyclization reaction can be envisaged as proceeding by any one of a number of free radical chain mechanisms.

(9) J. E. Johnson, R. H. Blizzard and H. W. Carhart, *ibid.*, **70**, 3664 (1948).

(10) B. J. Mair and A. F. Forziati, *J. Research Natl. Bur. Standards*, **32**, 151, 165 (1944).

(11) H. Pines, N. E. Hoffman and V. N. Ipatieff, unpublished work.

(12) F. O. Rice and R. K. Rice, "The Aliphatic Free Radicals," The Johns Hopkins Press, Baltimore, Md., 1935, p. 164; R. L. Burwell, Jr., THIS JOURNAL, **73**, 4461 (1951).

Experimental

High Pressure Reaction.—The high pressure thermal reaction was conducted in an apparatus previously described.[13] The reaction tube was lined with copper and packed with 50 ml. of 1/8 inch copper pellets. The pinane charged was prepared by hydrogenating redistilled α-pinene at 50° and 100 atmospheres pressure in the presence of nickel–kieselguhr and possessed the following constants: b.p. 167.5–168°, $n^{20}D$ 1.4620, d^{20}_4 0.8580, $[\alpha]^{21}D$ +10.28°. Pinane, 740 g., was charged to the reaction tube at a rate of 124 ml./hr., while maintaining the temperature at 396–405° and the pressure at 815 atm. The liquid product recovered amounted to 731 g.; no condensable gases were formed and the non-condensable material amounted to 1400 ml. Mass spectrometric analysis of this material showed it to consist largely of air with small amounts of methane and hydrogen. A rough distillation of the liquid product through a 26-inch, wire-spiral packed column gave: 18 g., b.p. 36–150°; 526 g., b.p. 150–180°, and a residue of 165 g.

Fractionation.—The data obtained by fractionating 497 g. of the product, b.p. 150–180°, through an efficient Podbielniak Hypercal column at a reflux ratio of approximately 80 to 1 are shown in Fig. 1. A total of 462 g. of distillate was collected; the residue and holdup amounted to 35 g.

Analysis. 1-*trans*-2-Dimethyl-*cis*-3-isopropylcyclopentane (II).—A center cut, 12 g., b.p. 160.8–160.9°, $n^{20}D$ 1.4485, of the material from the major plateau was hydrogenated at 50° and 100 atmospheres pressure in the presence of nickel–kieselguhr. Upon fractionation through a Piros–Glover micro-column,[14] 75% of the product distilled at 159.7–161.3°, $n^{20}D$ 1.4325, d^{20}_4 0.7882.

Anal. Calcd. for $C_{10}H_{20}$: C, 85.63; H, 14.37. Found: C, 85.67; H, 14.45.

This material was used as the infrared reference sample of 1-*trans*-2-dimethyl-*cis*-3-isopropylcyclopentane (II). A hydrogenated composite of the material from the 160–162° plateau had the following constants: b.p. 157–159°, $n^{20}D$ 1.4330; infrared analysis showed this material to contain 95% II. In addition, the product obtained by hydrogenation of the material, b.p. 156.5–158.1, was found to contain 20% of the same compound. By interpolation, it was assumed that the isomer I was present to the extent of 80% in the material b.p. 158.1–160.0°. From this information, along with that presented in Table I, it was calculated tha the isomer I and double bond isomers of I which gave II upon hydrogenation were formed to the extent of approximately 34% based on the total product obtained. The characteristic infrared bands of compounds I and II are given in Table II.

TABLE II

CHARACTERISTIC INFRARED BANDS OF COMPOUNDS I AND II

Compd.	λ, microns							
I	6.07	6.88	7.27	7.73	8.07			
				8.82	9.16	9.39	10.24	11.25
II	6.86	7.29	7.56	8.34	8.52	8.72	9.43	
	9.87	10.13	10.35	10.61	10.84	11.56	12.12	12.95

Separation of Monocyclic and Bicyclic Fractions.—The fractions shown in Table I were chromatographed at 10–12° over 100–200 mesh, high grade silica gel[15] through a jacketed column of the type previously described.[6] The samples charged were diluted with a volume of pentane such that the olefin content of the resulting solution was approximately 30–40%. Absolute ethanol was used as the eluant. In most cases, it was a simple matter to follow the olefin fraction through the column by virtue of the fact that it formed a translucent band. Refractive index and testing with 2% permanganate provided an additional check on the product composition.

Monocyclic Fractions.—After distillation through a short Vigreux column, a sample (0.3–0.7 g.) of the monocyclic fractions was hydrogenated in the presence of Adams platinum oxide catalyst at atmospheric pressure. Acetic acid, 0.5–1.0 ml., was added to the reaction mixture. The various fractions absorbed 99–100% of the theoretical amount of hydrogen calculated for one double bond. For infrared analysis, approximately 10-g. samples were hydrogenated at 50° and 100 atmospheres pressure in the presence of nickel–kieselguhr using an equal volume of pentane as the solvent. Aromatic hydrocarbons are not hydrogenated under these conditions, and since aromatics were not detected in the hydrogenated products, they can be assumed to be absent in the original product.

Bicyclic Fractions.—The data obtained by infrared analysis of the bicyclic fractions are presented in Table I. Thirty-two grams of this material was passed through a glass tube packed with 70 ml. of copper pellets over a period of 1 hour. The temperature was maintained at 498–502°. No gases were evolved during the reaction and distillation of the liquid product gave 31 g. of material, b.p. 148–164.5°, $n^{20}D$ 1.4525. Chromatographic separation furnished 17.5 g. of olefinic material and 10 g. of a bicyclic hydrocarbon, b.p. 164–164.5°, $n^{20}D$ 1.4583.

Anal. Calcd. for $C_{10}H_{18}$: C, 86.91; H, 13.09. Found: C, 87.15; H, 13.20.

This material was stable to 2% permanganate and solidified completely in Dry Ice.

Degradation Experiments. Ozonolysis of the Isomer I Methyl *trans*-2-*cis*-3-Dimethylcyclopentyl Ketone (III).—A solution of 15.0 g. of the isomer I in 165 ml. of dry carbon tetrachloride was cooled to 0° and a stream of oxygen containing 3–4% ozone was passed through the solution at a rate of approximately 250 ml. per min. The ozonide was decomposed by adding it slowly to a vigorously stirred solution of 30 ml. of 10% sodium carbonate and 30 ml. of hydrogen peroxide. The mixture was then refluxed for 3 hours.

A composite from six such runs was worked up in the following manner. After adding 25 g. of sodium carbonate, the mixture was steam distilled. The organic layer in the distillate was separated and the aqueous layer extracted with two 100-ml. portions of carbon tetrachloride. The aqueous layer was fractionated through a 15-plate column and the distillate (15 ml.) collected until it no longer decolorized 2% permanganate. Five ml. of this distillate was added to 5 ml. of ethanol containing 0.4 g. of dimethyldihydroresorcinol and two drops of piperidine.[16] After standing overnight, the crystalline product, *ca.* 0.2 g., was filtered and recrystallized from dilute methanol, m.p. 188–189°, mixed m.p. with the corresponding derivative of formaldehyde[17] 188–189°.

After stripping the carbon tetrachloride from the organic layer, the product was distilled through a short Vigreux column and furnished 47.5 g. of distillate, b.p. 77–132° (92 mm.), and 11.5 g. of black, viscous residue. Careful fractionation of the distillate through a Piros–Glover micro-column yielded the ketone III as the main fraction, 29.2 g., b.p. 112–115.5° (98 mm.), $n^{20}D$ 1.4390–1.4400, d^{20}_4 0.9011.

Anal. Calcd. for $C_9H_{16}O$: C, 77.10; H, 11.50. Found: C, 75.00, 75.01; H, 11.37, 11.38.

Semicarbazone, m.p. 133.5–135.0°. *Anal.* Calcd. for $C_{10}H_{19}N_3O$: C, 60.88; H, 9.71; N, 21.30. Found: C, 60.83; H, 9.63; N, 20.96.

In addition, 8.4 g. of a lower boiling fraction, b.p. 83.5–89.2° (98 mm.), $n^{20}D$ 1.4370–1.4423, was obtained. This material was unstable to 2% permanganate and gave a positive aldehyde test with Tollens reagent.

Anal. Calcd. for $C_7H_{12}O$: C, 74.93; H, 10.78. Found: C, 74.23; H, 11.80.

Semicarbazone, m.p. 206–206.5°. *Anal.* Calcd. for $C_8H_{15}N_3O$: C, 56.78; H, 8.94; N, 24.83. Found: C, 56.67; H, 9.00; N, 24.73.

Seven grams of this low-boiling fraction was mixed with 65 ml. of 40% sodium bisulfite solution and shaken for 2.5 hours. The crystalline bisulfite compound (4.6 g.) was filtered and washed with ether. The washings were combined with the ether extracts and dried. Evaporation of the ether and distillation of the residue furnished 3 g. of product, b.p. 142–154°, $n^{20}D$ 1.4379–1.4440. This material was not investigated further. The bisulfite addition compound was dissolved in water, 100 ml. of 10% sodium car-

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(13) V. N. Ipatieff, G. S. Monroe and L. E. Fischer, *Ind. Eng. Chem.*, **40**, 2059 (1948).

(14) Manufactured by H. S. Martin and Company, Evanston, Ill.

(15) Davison Chemical Corporation, Baltimore 3, Md., Grade 923.

(16) R. L. Shriner and R. C. Fuson, "The Systematic Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1948, p. 172.

(17) E. H. Huntress and S. P. Mulliken, "Identification of Pure Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1941, p. 50.

bonate was added, and the mixture steam distilled. The distillate was saturated with sodium carbonate, extracted with ether, and the extracts dried over sodium sulfate. Distillation gave 1.1 ml. of product, b.p. 148–151°, n^{20}D 1.4315.

Anal. Calcd. for $C_7H_{12}O$: C, 74.93; H, 10.78. Found: C, 73.50; H, 11.12.

The semicarbazone of this material was identical with the one described above.

trans-2-*cis*-**3-Dimethylcyclopentanecarboxylic Acid (IV).**—A solution of sodium hypobromite was prepared by adding 83.3 g. (0.52 mole) of bromine dropwise to a solution of 57.2 g. (1.4 moles) of sodium hydroxide in 485 ml. of water in a one-liter, three-neck flask equipped with stirrer, dropping funnel, reflux condenser and thermometer well. The flask was surrounded by an ice-bath during the addition, and the temperature was maintained below 5°. The solution was allowed to warm to room temperature and 24.3 g. of the ketone III was added dropwise with stirring. The temperature was maintained at 30–35° by cooling the flask occasionally. The mixture was stirred for an additional two hours and then allowed to stand overnight at room temperature. The bromoform layer (25 g.) was separated and the aqueous layer extracted with ether. The aqueous solution was then cooled in an ice-bath and acidified by adding an ice-cold solution of 70 ml. of concentrated sulfuric acid in 70 ml. of water with shaking. The mixture was extracted with five 100-ml. portions of ether and the extracts dried over sodium sulfate. After stripping the ether, distillation gave 15.7 g. of *trans*-2-*cis*-3-dimethylcyclopentanecarboxylic acid, b.p. 123.5–125.5° (13 mm.), n^{20}D 1.4490 neut. equiv. 143, calcd. for $C_8H_{14}O_2$, 142.

Anal. Calcd. for $C_8H_{14}O_2$: C, 67.56; H, 9.92. Found: C, 67.12; H, 10.00.

The amide melted at 171.5–172.5 after recrystallization from benzene. *Anal.* Calcd. for $C_8H_{15}NO$: C, 68.04; H, 10.71; N, 9.92. Found: C, 68.08; H, 10.71; N, 9.95.

trans-2-*cis*-**Dimethylcyclopentylmethanol (V).**—The usual procedure for lithium aluminum hydride reductions was followed here.[18] From the reaction of 12.0 g. (0.084 mole) of the acid IV with 4.2 g. (0.11 mole) of lithium aluminum hydride in 340 ml. of anhydrous ether, there was obtained 9.8 g. (91% yield) of *trans*-2-*cis*-3-dimethylcyclopentylmethanol (V), b.p. 91.5–92.5° (21 mm.), n^{20}D 1.4510, $d^{20}{}_4$ 0.8962.

Anal. Calcd. for $C_8H_{16}O$: C, 74.95; H, 12.58. Found: C, 75.15; H, 12.78.

Hydrogenolysis of *trans*-2-*cis*-**3-Dimethylcyclopentylmethanol.**—Hydrogenolysis of 4.6 g. (0.036 mole) of the alcohol V in the presence of 1.5 g. of nickel–kieselguhr at 160° and an initial hydrogen pressure of 60 atm. gave 1.8 g. (50% yield) of liquid product, b.p. 91–92°, n^{2}D 1.4105. Infrared analysis of this product showed it to be practically pure 1-*trans*-2-dimethylcyclopentane.[19] Mass spectrometric analysis of the gas from the reaction showed an 87% yield of methane.

trans-2-*cis*-**3-Dimethylcyclopentylcarbidyl Bromide (VI).**—Phosphorus tribromide, 3.5 g. (0.013 mole), was added dropwise with shaking to 4.05 g. (0.031 mole) of the alcohol V while the temperature was maintained at 60–75°. The mixture was kept at 70° for 2 hours with frequent shaking and then warmed on the steam-bath overnight. The mixture was poured over ice, the organic layer was taken up in ether, washed with water, 5% sodium carbonate, again with water and dried over calcium chloride. Distillation furnished 4.67 g. (77% yield) of *trans*-2-*cis*-3-dimethylcyclopentylcarbinyl bromide, b.p. 84–85° (24 mm.), n^{20}D 1.4760.

Anal. Calcd. for $C_8H_{15}Br$: Br, 41.82. Found: Br, 41.87.

Reduction of *trans*-2-*cis*-**3-Dimethylcyclopentylcarbinyl Bromide.**—The procedure described in the literature[18] for the reduction of alkyl halides with lithium aluminum hydride was followed. The reaction of 4.5 g. (0.023 mole) of the bromide VI with 1.0 g. (0.026 mole) of lithium aluminum hydride in 25 ml. of refluxing tetrahydrofuran gave 3.4 ml. of product, b.p. 74–111°, which was unstable to 2% permanganate and contained some tetrahydrofuran. Upon quantitative hydrogenation, this material absorbed 0.00072 mole of hydrogen. The catalyst was filtered and the product taken up in pentane. The solution was washed with two 10-ml. portions of cold 50% sulfuric acid, 10 ml. of water, 10 ml. of 5% potassium carbonate and again with water. After drying over calcium chloride, distillation gave 0.9 ml. of product, b.p. 107–108.5°, n^{20}D 1.4090. Infrared analysis showed this product to be 1-*trans*-2-*cis*-3 trimethylcyclopentane of approximately 90% purity.

Acidic Fraction from the Ozonolysis.—The resinous product remaining from the steam-distillation of the ozonolysis product was extracted with ether and the aqueous layer evaporated to dryness. The residue was dissolved in the minimum quantity of water, cooled in an ice-bath and acidified with 90 ml. of 25% sulfuric acid. The mixture was extracted with four 50-ml. portions of ether. The extracts were dried over sodium sulfate. The aqueous layer gave a positive test for formic acid with mercuric oxide.[20] After removing the ether from the extracts, distillation gave 6.5 g. of acidic material, b.p. 121–130° (68 mm.), n^{20}D 1.4459–1.4508; neut. equiv., 159 (calcd. for $C_8H_{14}O_2$, 142). This material darkened rapidly during distillation and was unstable to 2% permanganate. The amide melted at 165–165.6°; mixed m.p. with the amide of IV, 170–171.5°.

Anal. Calcd. for $C_8H_{15}NO$: C, 68.04; H, 10.71; N, 9.92. Found: C, 67.64; H, 11.11; N, 9.80.

Acknowledgment.—The authors wish to thank Bruno Kvetinskas and E. E. Meisinger of the Research Laboratories, Universal Oil Products Company, for the pyrolysis experiments made under pressure, and Edmond Baclawski of the Physics Department, Universal Oil Products Company, for infrared spectral analyses.

EVANSTON, ILLINOIS

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[18] R. Nystrom and W. G. Brown, THIS JOURNAL, **69**, 1197 (1947).

[19] Catalogue of Infrared Spectra, American Petroleum Institute Project 44.

[20] Reference 16, page 179.