

*Anal.* Calcd for  $C_{22}H_{16}O_6$ : C, 70.21; H, 4.29. Found: C, 70.03; H, 4.50.

**2,3-Methylenedioxy-9-methoxy[2H-1]benzopyrano[3,4-b][1]-benzopyran-12-one (14b).**—The sample for analysis was recrystallized from chloroform-benzene: mp 225° dec; ir ( $CHCl_3$ ) 3.56 (methylenedioxy), 6.05 (C=O), and 10.55  $\mu$  (methylenedioxy).

*Anal.* Calcd for  $C_{18}H_{12}O_6$ : C, 66.67; H, 3.73. Found: C, 66.83; H, 3.86.

**1,2,12,12a-Tetrahydro-2-isopropenyl-8,9-methylenedioxy[1]-benzopyrano[3,4-b]furo[2,3-h][1]benzopyran-6(6aH)-one (Isomillettone, 1b)** and **1,2,12,12a-Tetrahydro-2-isopropenyl-8,9-methylenedioxy[1]benzopyrano[3,4-b]furo[2,3h][1]benzopyran (18).**—The preparation was similar to a general method published previously<sup>15</sup> and the crude product was chromatographed on neutral alumina. Compound 18 was recrystallized from benzene-cyclohexane: mp 180°; ir ( $CHCl_3$ ) 3.60 (methylenedioxy), 6.13 (C=C), 6.18 (C=C), 10.64 (methylenedioxy), and 11.00  $\mu$  (isopropenyl).

*Anal.* Calcd for  $C_{22}H_{18}O_6$ : C, 72.92; H, 5.01. Found: C, 72.74; H, 4.99.

Isomillettone (1b) was recrystallized from ethanol: mp 166–167.5°; ir ( $CHCl_3$ ) 3.61 (methylenedioxy), 5.97 (C=O), 6.21 (the strongest band), 10.64 (methylenedioxy), and 11.04  $\mu$  (isopropenyl); uv (EtOH) 237.5 ( $\epsilon$  10,800) and 297.5  $\mu\mu$  ( $\epsilon$  15,000); nmr ( $CDCl_3$ ) see text;  $[\alpha]_D^{24} -87.5^\circ$  (c 2.10, benzene).

*Anal.* Calcd for  $C_{22}H_{18}O_6$ : C, 69.83; H, 4.80. Found: C, 69.76; H, 5.08.

**Methylenedioxy Analog of Munduserone. 2,3-Methylenedioxy-9-methoxy-6,6a-dihydro[1]benzopyrano[3,4-b][1]benzopyran-12(12H)-one (2b).**—Eight hundred milligrams of 14b was converted into 2b in the usual manner.<sup>15</sup> Without the use of chromatography, the product crystallized from 20 ml of benzene to give rise to 245 mg of colorless, long needles, mp 210–211.5°. From the mother liquor a second crop of 151 mg, mp 202°, was obtained. The final mother liquor deposited more crystalline product on standing for a few days. Total yield was at least 396 mg (49.2%). The first crop was used for analysis: ir ( $CHCl_3$ ) 3.59 (methylenedioxy), 5.94 (C=O), 6.19 (the strongest band), and 10.65  $\mu$  (methylenedioxy); uv (MeOH) 231.5 ( $\epsilon$  14,880), 275.5 ( $\epsilon$  15,760), and 305  $\mu\mu$  ( $\epsilon$  11,750).

*Anal.* Calcd for  $C_{18}H_{14}O_6$ : C, 66.25; H, 4.32. Found: C, 66.07; H, 4.43.

**Registry No.**—1b, 22256-05-9; 2b, 22256-06-0; 3, 13668-86-5; 4, 22297-78-5; 5, 22252-99-9; 6, 22253-00-5; 7, 22253-01-6; 8, 22253-02-7; 9, 22253-03-8; 10, 22253-04-9; 11, 22253-05-0; 12, 22253-06-1; 14a, 22256-07-1; 14b, 22297-79-6; 18, 22256-08-2.

**Acknowledgment.**—The author is indebted to Mr. C. R. Dorn for preparation of 1-tubaic acid and Mr. C. H. Yen for large-scale preparation of 9. The author expresses thanks to Dr. R. R. Burtner and Dr. F. B. Colton for valuable discussion and to Mr. C. R. Dorn for assistance in the preparation of this manuscript.

(15) M. Miyano and M. Matsui, *Chem. Ber.*, **91**, 2044 (1958); **93**, 54 (1960).

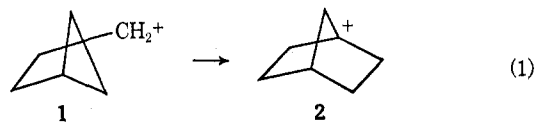
## Studies on Reactions of Isoprenoids. VII.<sup>1</sup> Acid-Catalyzed Hydrolysis of 5,5-Dimethylbicyclo[2.1.1]hexane-1-epoxyethane

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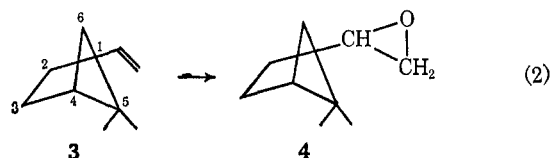
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Although facile ring expansion of the bicyclo[2.1.1]-hexane-1-carbinyl cation (1) to the 1-norbornyl cation



(2) (eq 1) is documented as one of the general carbocyclic ring expansion reactions of Wagner-Meerwein type rearrangements,<sup>2</sup> the initial cationic species seems to be restricted mostly to the primary cation.<sup>3</sup> In this paper we wish to describe the somewhat difficult ring expansion of a bicyclo[2.1.1]hexane-1-carbinyl to a 1-norbornyl system as the result of acidic hydrolysis of the title epoxide, in which the ring-enlargement aptitude is diminished by the intervention of a secondary cationic species and also by a possible cyclobutane ring fission facilitated by the presence of a *gem*-dimethyl group at C-5.

The title epoxide 4 was readily prepared by oxidation of 5,5-dimethyl-1-vinylbicyclo[2.1.1]hexane (3)<sup>4</sup> with perbenzoic acid (eq 2). The epoxide 4, however



was shown to be an inseparable mixture (55:45) of two stereoisomers by vpc analysis<sup>5</sup> and by the nmr spectrum (100 MHz), in which partly overlapping sets of multiplets in the typical ABX pattern were observed at  $\tau$  7.00–7.67 for three protons assignable to protons of the HC–O–CH<sub>2</sub> moiety and also two singlet signals at  $\tau$  8.73 and 8.77 for three protons assignable to one of the methyl protons at C-5 were observed in a ratio of ca. 4:6. The formation of two isomeric epoxides is reasonable, because a new asymmetric carbon is produced on the epoxidation of 3, though their separation was not successful.

Hydrolysis of 4 was carried out by refluxing an ether solution containing 5% sulfuric acid, and the reaction was followed on vpc (PG-6000). After 11 days, the conversion was 83%; then the products were purified by chromatography on a silica gel column to give 14% of recovered 4, 7.5% of oily vinyl ketone 5, and 72.5% of a semicrystalline glycol mixture. The recovered 4 had a different isomer ratio from the starting material (32:68 as compared with the original 55:45),<sup>5</sup> indicating that one of the isomers reacted faster than the other.<sup>6</sup>

The structure of 5 was established as 3-(1,1-dimethyl-2-propenyl)cyclopentanone on the basis of the analytical data and the following spectral evidence: ir (neat) 1740 (cyclopentanone), 1643, 980, and 920  $cm^{-1}$  (vinyl); nmr ( $CCl_4$ )  $\tau$  3.60–5.10 (m of a typical ABX

(1) Part VI of this series: T. Sasaki, S. Eguchi, M. Ohno, and T. Oyobe, *Bull. Chem. Soc. Jap.*, in press.

(2) K. B. Wiberg and B. R. Lowry, *J. Amer. Chem. Soc.*, **85**, 3188 (1963).

(3) For a review about bicyclo[*n*.1.1]alkanes, see J. Meinwald and Y. C. Meinwald, "Advances in Alicyclic Chemistry," Vol. I, Academic Press, New York, N. Y., 1966, pp 2–51.

(4) R. H. Liu and G. S. Hammond, *J. Amer. Chem. Soc.*, **89**, 4936 (1967).

(5) The major isomer had a slightly longer retention time than that of the minor one on vpc (PG-6000).

(6) We could not obtain further detailed information on the stereoselectivity in the reaction of such conformationally mobile epoxide without pure isomers. For similar reactions which have been more extensively explored, see C. J. Cheer and C. R. Johnson, *J. Amer. Chem. Soc.*, **90**, 178 (1968).

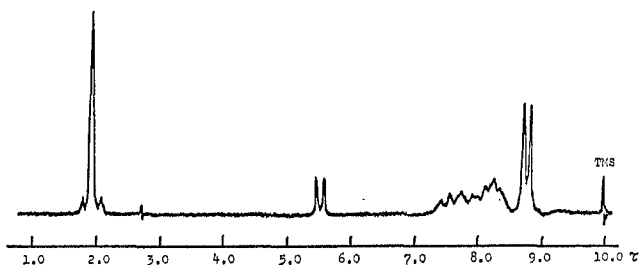
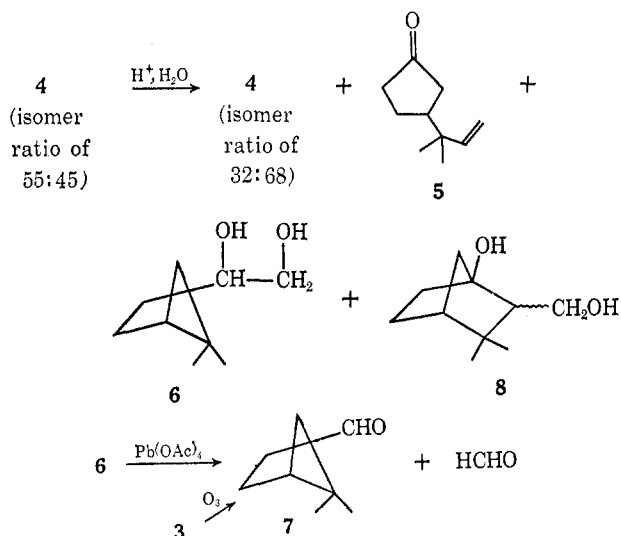


Figure 1.—Nmr spectrum ( $\text{CDCl}_3$ , 60 MHz) of the di-*p*-nitrobenzoate of **8**.

pattern, 3 H,  $\text{CH}=\text{CH}_2$ ),<sup>7</sup> 7.75–8.50 (broad and complex m, 7 H, methine and methylene protons), and 8.75 and 8.84 [each s, 6 H,  $\text{C}(\text{CH}_3)_2$ ]; mass spectrum *m/e* (rel intensity, ion) 152 (4.5,  $\text{M}^+$ ), 137 (2.1,  $\text{M}^+ - \text{CH}_3$ ), 123 (27.6,  $\text{M}^+ - \text{C}_2\text{H}_5$ ), 96 (13.1,  $\text{C}_7\text{H}_{12}^+$ ), 82 (20.3,  $\text{C}_5\text{H}_6\text{O}^+$ ), 69 (60.3,  $\text{C}_5\text{H}_9^+$ ), and 55 (100,  $\text{C}_3\text{H}_3\text{O}^+$ ).

The glycol mixture obtained as the main product was treated with dichloromethane to give colorless prisms, mp 114–115°, which had the following spectral data: ir (KBr) 3440 (OH), 1108 (secondary OH), and 1045  $\text{cm}^{-1}$  (primary OH); nmr ( $\text{DMSO}-d_6$ )  $\tau$  5.77 (t, 1H,  $J = 5.5$  Hz,  $\text{CH}_2\text{OH}$ ), 5.92 (d, 1 H,  $J = 4.5$  Hz,  $\text{CHOH}$ ), 6.45 (double t, 1 H,  $J = 7.0$  and 4.5 Hz,  $\text{CHOH}$ ), 6.75 (q, 2 H,  $J = 7.0$  and 5.5 Hz,  $\text{CH}_2\text{OH}$ ), 8.86 and 9.28 [each s, 3 H,  $\text{C}(\text{CH}_3)_2$ ], 9.12 (d, 1 H,  $J = 7.0$  Hz, the characteristic signal of an *endo* proton at a C-6 methylene bridge),<sup>4</sup> and 7.90–8.70 (broad m, 6 H, other methine and methylene protons). These data indicated this product to be a 1,2-glycol (**6**) produced by simple hydrolytic ring opening of **4**. The structural assignment of **6** was further confirmed by its chemical conversion to 5,5-dimethylbicyclo[2.1.1]hexane-1-aldehyde (**7**) and formaldehyde on treatment with lead tetraacetate. Compound **7** was identified as its 2,4-dinitrophenylhydrazone by comparison with a specimen prepared by direct ozonization of **3**.



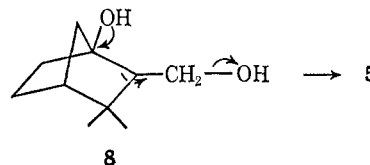
The remaining noncrystalline glycol mixture was treated with *p*-nitrobenzoyl chloride in pyridine and the crude *p*-nitrobenzoate was purified on a silica gel column to give two crystalline products. The first

(7) Appearance of this signal in the typical ABX pattern ( $J_{\text{AB}} = 2.3$  Hz,  $J_{\text{BC}} = 17.0$  Hz,  $J_{\text{AC}} = 10.5$  Hz) excluded the possibility of 2,2-dimethyl-3-allylcyclopentanone as the structure of **5**.

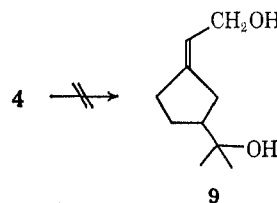
was identified as the di-*p*-nitrobenzoate of **6**, mp 170–171°, by mixture melting point determination and the complete superimposition of the ir spectrum on that of an authentic specimen from **6**.

The other product, with mp 158–160°, was identified as the di-*p*-nitrobenzoate of 3,3-dimethyl-2-hydroxymethylbicyclo[2.2.1]heptan-1-ol (**8**), a ring-expanded product, on the basis of analytical and spectral data.<sup>8</sup> In the nmr spectrum (Figure 1), only one doublet at  $\tau$  5.58 (2 H,  $J = 6.0$  Hz) appeared in the  $\tau$  3.0–7.0 region, which was assignable to the  $\text{CHCH}_2\text{OH}$  moiety. No signal characteristic of an *endo* proton of a bicyclo[2.1.1]hexane ring was observed at *ca.*  $\tau$  9.1.<sup>4</sup>

The relative yield of the 1,2-glycol **6** to the 1,3-glycol **8** was determined as *ca.* 64:36 on the basis of the amounts of the corresponding di-*p*-nitrobenzoates and the free glycol **6** which were isolated. However, the ratio of nonrearranged product **6** to ring-expanded product **8** should actually be *ca.* 1:1, since vinyl ketone **5** can be regarded as a dehydration product of **8**.



Surprisingly an unsaturated alcohol like **9** which might be the result of cyclobutane ring fission of **4** could not be isolated. Thus, the ring-enlargement aptitude of **4** under hydrolytic conditions is shown to be relatively small compared with the almost exclusive ring enlargement of bicyclo[2.1.1]heptyl-1 tosylate under acetolysis conditions.<sup>2</sup>



#### Experimental Section<sup>9</sup>

**5,5-Dimethyl-1-vinylbicyclo[2.1.1]hexane (3).**—This was prepared by the modified procedure of the method of Liu and Hammond.<sup>4</sup> A 0.3 *M* solution of myrcene in benzene containing a small amount of benzophenone was irradiated with a 100-W high-pressure mercury lamp through a Pyrex filter and the crude product was treated with maleic anhydride in order to remove the remaining myrcene and distilled through a 40-cm Widmer column to give **3** in 35–50% yield: bp 150–151°;  $n_{\text{D}}^{21}$  1.4654 (lit.<sup>4</sup> bp 151°;  $n_{\text{D}}^{21}$  1.4641). The structure was further confirmed by the ir and nmr spectra.

**5,5-Dimethylbicyclo[2.1.1]hexane-1-epoxyethane (4).**—To an ice-cooled solution of benzoic peracid (3.4 g, 0.025 mol) in 60 ml of chloroform was added 2.72 g (0.02 mol) of **3** with stirring and the mixture was allowed to stand for 24 hr at 20°. After removal of

(8) From the spectral data, the possibility of 7,7-dimethyl-2-hydroxymethylbicyclo[2.2.1]heptan-1-ol which might be produced by the alternate mode of ring expansion could not be ruled out. However, isolation of **5** supported the assigned structure.

(9) All boiling and melting points are uncorrected. Ir spectra were measured with a JASCO Model IR-S ir spectrophotometer. Nmr spectra were determined with a Varian Model A-60 spectrometer at 60 MHz, unless otherwise specified, and with a Varian Model HA-100 spectrometer at 100 MHz using TMS as an internal standard. Vpc analyses were carried out with a Yanagimoto gas chromatograph, Model GCG-220. Microanalyses were performed by Mr. Akiho Ishihara at our laboratory with a Yanagimoto C. H. N. Corder Model MT-1.

the solvent, the oily residue was distilled to give 2.2 g of **4** as an oil with a camphor-like odor: bp 82–83° (19 mm);  $n_D^{21}$  1.4699; ir (neat) 895, 967, and 805  $\text{cm}^{-1}$  (epoxide ring); nmr ( $\text{CDCl}_3$ )  $\tau$  7.17 (unsymmetrical double q, 1 H, X portion of an ABX pattern,  $\text{HC-O-CH}_2$ ), 7.29–7.75 (complex m, 2 H, AB portion of an ABX pattern,  $\text{HC-O-CH}_2$ ), 8.73 and 8.77 (each s, 3 H,  $\text{CH}_3$ ), 9.18 (s, 3 H,  $\text{CH}_3$ ), ca. 9.13 (d, ca. 1 H,  $J = \text{ca. } 7.0 \text{ Hz}$ , partly overlapped with the signal at  $\tau$  9.18, an *endo* proton of C-6 methylene bridge), and 7.90–8.60 (broad m, ca. 6 H, other methylene and methine protons). Vpc analysis on a 2-m PG-6000 column at 150° revealed two close but separated peaks, indicating this epoxide to be a 55:45 mixture (estimated from relative peak area) of two isomers.

*Anal.* Calcd for  $\text{C}_{10}\text{H}_{16}\text{O}$ : C, 78.89; H, 10.59. Found: C, 78.68; H, 10.99.

**Hydrolysis of 5,5-Dimethylbicyclo[2.1.1]hexane-1-epoxyethane (4).**—A mixture of 4.0 g (0.0262 mol) of **4**, 1.0 ml of 5% sulfuric acid, and 30 ml of ether was refluxed for 11 days. The organic layer was washed with 10 ml of water and 10 ml of 5% aqueous sodium bicarbonate and dried ( $\text{Na}_2\text{SO}_4$ ), and the solvent was removed to give 4.5 g of oily residue which was purified on a silica gel (Mallinckrodt, 100 Mesh) column (2 × 80 cm) by elution with dichloromethane, chloroform, and ethyl acetate, successively. From the first fraction, an oil was obtained after removal of the solvent, which amounted to 0.558 g (14.0%) and was identified as the recovered **4** on vpc (but with an isomer ratio of 32:68) and by the ir and nmr spectral comparisons.

From the second fraction, a volatile oil (0.30 g, 7.5%) of 3-(1,1-dimethyl-2-propenyl)cyclopentanone (**5**) was obtained.

*Anal.* Calcd for  $\text{C}_{10}\text{H}_{16}\text{O}$ : C, 78.89; H, 10.59. Found: C, 78.99; H, 10.55.

The third fraction afforded a sticky semicrystalline residue (3.46 g, 72.5%) after removal of the solvent *in vacuo*, which was assumed to be a glycol mixture from its ir spectrum (3440 and ca. 1110  $\text{cm}^{-1}$ ). Three recrystallizations from dichloromethane afforded colorless prisms (300 mg, 6.2%) of **6**, mp 114–115°.

*Anal.* Calcd for  $\text{C}_{10}\text{H}_{18}\text{O}_2$ : C, 70.54; H, 10.66. Found: C, 70.57; H, 10.88.

**Di-*p*-nitrobenzoate of 6.**—A mixture of 34 mg (0.187 mmol) of **6** and 90 mg (0.485 mmol) of *p*-nitrobenzoyl chloride in 5 ml of dry pyridine was stirred for 24 hr at room temperature. Addition of water to the reaction mixture resulted in precipitation. The precipitates were filtered, washed with water, and dissolved in 30 ml of benzene. Benzene solution was washed with 5% aqueous sodium bicarbonate and water and dried ( $\text{Na}_2\text{SO}_4$ ). Removal of the solvent *in vacuo* afforded faintly yellowish needles which were recrystallized from dichloromethane-*n*-hexane to afford 70 mg (80%) of fine needles of the di-*p*-nitrobenzoate of **6**: mp 170–171°; ir (KBr) 1725 ( $\text{C}=\text{O}$ ), 1610, and 728  $\text{cm}^{-1}$  (phenyl); nmr ( $\text{CDCl}_3$ , 100 MHz)  $\tau$  1.60–1.98 (m, 8 H, phenyl protons), 4.15 and 4.20 (two overlapped t, 1 H,  $J = \text{ca. } 5.5 \text{ Hz}$ ,  $\text{OCOCH}_2\text{OCO}$ ), 5.43 and 5.45 (2 H, two overlapped d, 2 H,  $J = \text{ca. } 5.5 \text{ Hz}$ ,  $\text{OCOCH}_2\text{OCO}$ ), 7.89 and 8.23 (each broad s, 6 H, methine and methylene protons), 8.68 and 8.70 (two overlapped s, 3 H,  $\text{CH}_3$ ), 9.08 and 9.21 (each s, 3 H,  $\text{CH}_3$ ), and 8.90 and 8.95 (two overlapped d, 1 H,  $J = \text{ca. } 7.0 \text{ Hz}$ , *endo* proton of C-6 methylene bridge). All of these signals could be explained by assuming that the product was a mixture of two stereoisomers (ca. 1:1 ratio estimated from relative signal height).

*Anal.* Calcd for  $\text{C}_{24}\text{H}_{24}\text{O}_8\text{N}_2$ : C, 61.53; H, 5.16; N, 5.98. Found: C, 61.78; H, 5.08; N, 6.07.

**Di-*p*-nitrobenzoate of 8.**—A 2.39-g portion of the glycol mixture from the mother liquid of the third fraction as described above was treated with 5.5 g of *p*-nitrobenzoyl chloride in 20 ml of dry pyridine for 2 days at room temperature. After addition of water, the mixture was extracted with five 40-ml portions of chloroform and the combined chloroform extracts were washed successively with two 30-ml portions of 5% sulfuric acid, 30 ml of water, two 30-ml portions of 5% aqueous sodium bicarbonate, two 30-ml portions of water, and dried ( $\text{Na}_2\text{SO}_4$ ). After removal of the solvent *in vacuo*, the crude product (3.5 g) was purified on a silica gel column (2 × 75 cm) by elution with benzene. From the first fraction, 1.07 g of faintly yellowish needles were obtained, mp 170–171° (dichloromethane-*n*-hexane), which was identified as the di-*p*-nitrobenzoate of **6** by mixture melting point determination and by the absolute superimposition of the ir spectrum on that of a specimen prepared directly from **6**. From the second fraction, 1.23 g of faintly yellowish prisms, mp 158–160° (di-

chloromethane-*n*-hexane) was obtained, which was characterized as a di-*p*-nitrobenzoate of **8**, though the stereochemistry of a hydroxymethyl group is uncertain: ir (KBr) 1725 ( $\text{C}=\text{O}$ ), 1603, and 715  $\text{cm}^{-1}$  (phenyl); nmr, see Figure 1.

*Anal.* Calcd for  $\text{C}_{24}\text{H}_{24}\text{O}_8\text{N}_2$ : C, 61.53; H, 5.10; N, 5.98. Found: C, 61.54; H, 5.15; N, 5.95.

Further fractions eluted with chloroform and ethyl acetate afforded *p*-nitrobenzoic acid and unidentified dark brown tar.

**Lead Tetraacetate Oxidation of 1,2-Glycol 6.**—Into a 50-ml, three-necked flask fitted with a dropping funnel and nitrogen inlet and outlet, which was immersed in a solution of 2,4-dinitrophenylhydrazine (2,4-DNP) reagent,<sup>10</sup> was placed 443 mg (1.0 mmol) of lead tetraacetate (commercial, used directly). After addition of 91 mg (0.5 mmol) of 1,2-glycol **6** in 20 ml of dry benzene, a slow stream of dry nitrogen was passed into the reaction mixture at ca. 60° for 1 hr. Precipitates in the 2,4-DNP solution were filtered and recrystallized from aqueous ethanol to give 40 mg (38%) of yellow needles, mp 166–168°, which was identified as the 2,4-dinitrophenylhydrazone (2,4-DNP) of formaldehyde by mixture melting point and superposition of the ir spectrum on that of an authentic sample.

The reaction mixture in the flask was washed with water and treated with 2,4-DNP reagent to afford yellow precipitates, which were collected by filtration and dried. Purification on a silical gel column by elution with dichloromethane gave 50 mg (31.4%) of the 2,4-DNP of **7** as fine yellow needles, mp 171–172°, identified by mixture melting point and superposition of the ir spectrum on that of an authentic sample.<sup>12</sup> Although **7** has been described in the literature,<sup>12</sup> its 2,4-DNP is not reported. It was prepared as follows: 1.0 g of **3** in 100 ml of methanol was cooled at –70° and ozone was passed through the solution until **3** had disappeared on thin layer chromatography. Nitrogen was bubbled into the solution, for a short time, 1.0 ml of dimethyl sulfide was added, and the mixture was stirred for 5 hr at room temperature. Removal of the solvent and excess dimethyl sulfide afforded an oil, aldehyde **7**, a portion of which was treated with semicarbazine in the usual way to give the known semicarbazone of **7**, mp 193–194° (lit.<sup>12</sup> mp 192–193.2°). Another portion of the oily **7** was converted to its 2,4-DNP, mp 171–172° (aqueous ethanol).

*Anal.* Calcd for  $\text{C}_{15}\text{H}_{13}\text{O}_4\text{N}_4$ : C, 56.59; H, 5.70; N, 17.60. Found: C, 56.39; H, 5.53; N, 17.26.

**Registry No.**—**4**, 21990-90-9; **5**, 21990-91-0; **6**, 21990-92-1; di-*p*-nitrobenzoate of **6**, 21990-95-4; 2,4-DNP of **7**, 22037-79-2; di-*p*-nitrobenzoate of **8**, 22002-75-1.

**Acknowledgment.**—We are grateful to Dr. T. Nishida and Mr. I. Miura of Nippon Electric Varian, Ltd., for taking the nmr spectra.

(10) R. L. Shrine, R. C. Fuson, and D. Y. Curtin, "The Systematic Identification of Organic Compounds," 4th ed, John Wiley & Sons, Inc., New York, N. Y., 1956, p 219.

(11) Reference 10, p 283.

(12) K. Ebisu, L. B. Batty, J. M. Higaki, and H. O. Larson, *J. Amer. Chem. Soc.*, **88**, 1995 (1966).

### A Stereospecific Synthesis of *trans*-3-(*exo*-5-*exo*-Isocamphyl)cyclohexanol

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Reports of the synthesis of substances possessing a sandalwood odor from terpenophenol ethers first appeared about 20 years ago.<sup>1,2</sup> Subsequently, Kheifits,

(1) J. R. Byers, *J. Amer. Perfumer*, **49**, 483 (1947).

(2) R. Hüttel and G. Keicher, German Patent 834,593 (1952); *Chem. Abstr.*, **47**, 5446i (1953).