

Method C. Attempted Half Hydrolysis of Succinylcholine (I). (1) **Water.**—A solution of 5.5 g. (0.01 mole) of I in 15 cc. of water was heated for five hours at 100° (initial pH 4–5, final pH 3.5–4). Upon chilling 4.5 g. (80%) of white crystals separated (unchanged I), m.p. 256–258°. Another 1 g. of I was obtained from the aqueous filtrates upon the addition of excess acetone; m.p. 254–256°. Total recovery of unchanged I was 5.5 g. (100%).

(2) **Aqueous Alkali.**—A solution of 11 g. (0.02 mole) of I in 15 cc. of hot water (80°, pH of solution is 4) was mixed quickly with 4.1 cc. of 4.8 N aqueous sodium hydroxide solution (= 0.02 mole of NaOH, pH < 11). The pH of the solution immediately after mixing was 7.5–8. After one or two minutes the pH was 7. After ten minutes at 70–80°, 2.5 cc. (4+ g.) of concentrated hydriodic acid solution (sp. g. 1.7) was added to the reaction solution bringing the pH to 3–4. Addition of excess acetone (300 cc.) and stirring gave a white crystalline precipitate. When chilled the product was collected by filtration and weighed 4.5 g. (40% of starting I), m.p. 248–252°. After recrystallization from 6 cc. of hot water, 4 g. (35%) of I was recovered melting at 254–256°.

Reworking the aqueous acetone filtrates gave another 0.5–1 g. of I and 1–2 g. of choline iodide.

(3) **Aqueous Alkali.**—To a solution of 5.5 g. (0.01 mole; initial pH 4) of I in 15 cc. of water at 100° was added dropwise 5 cc. of 10% aqueous sodium hydroxide (0.01 mole, initial pH > 11). Upon the first addition of alkali the pH rose at once to 7 and remained at 7 throughout the entire

addition of alkali over a 10–15 minute period. When all the alkali had been added the solution was evaporated to dryness *in vacuo* leaving 5.5 g. of white solid insoluble in acetone, and soluble in water. After washing several times with acetone by decantation, the 5.5 g. of white insoluble residue was taken up in 7 cc. of cold water and 1.3 g. (0.01 mole) of hydriodic acid was added to give a pH of 2–3. Addition of excess acetone gave 1 g. of white crystals (I) melting at 235–250°. Evaporation of the aqueous acetone filtrates to dryness and addition of more acetone gave another 1 g., m.p. 235–250°. This process was repeated again. The total yield was 2.3–2.5 g. (40–45% of the starting I). After boiling this 2.3–2.5 g. up with hot methanol (in which it was insoluble) there was recovered 2–2.3 g. (35–40%) of the unchanged starting compound I, m.p. 254–256°.

The acetone filtrates from the recovered I were evaporated to dryness and the residue was extracted repeatedly with dry ether. Evaporation of the ether extracts gave 0.35–0.4 g. of tan solid melting at 183–185°. This represents a 30–35% recovery of I as succinic acid.

The ether-insoluble residue weighed about 4 g. When this was extracted several times with acetone, the acetone extracts on evaporation gave 1.5 g. of white crystals melting > 320°. This fraction is mainly sodium iodide.

There remained 2 g. of white solid insoluble in acetone, melting at 121–122° and as yet unidentified; this can be reprecipitated from methanol solution with excess ether.

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The Stereochemistry of the 10-Methyl-2-decalols¹

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cis-10-Methyl-2-*cis*-decalol has been synthesized by stereospecific reactions which leave no doubt of its configuration. An isomer having the same m.p., 66–67°, has been formed by a different route and has been shown by mixed m.p. and m.p. of derivatives to be the epimer, *trans*-10-methyl-2-*cis*-decalol.

The stereochemistry of some of the angularly substituted methyl-octahydro- and methyl-decahydro-naphthalene compounds is of interest because this ring system is found in steroids. We are attempting to establish the configuration of the four geometric isomers of 10-methyl-2-decalol (I) to serve as points of reference for studies of stereochemical aspects of reactions and synthesis procedures pertaining to these compounds. Impure 10-methyl-2-*cis*-decalol has been synthesized previously,² but no claims have been made for the configuration of the hydroxyl group. The synthesis and the configuration of two of these isomers, *cis*-10-methyl-2-*cis*-decalol (Ia)³ and *trans*-10-methyl-2-*cis*-decalol (Ib)³ are reported here.

The first of these has been synthesized from 2-carbomethoxycyclohexanone by a sequence of nine steps, as represented in the equations. The second isomer has been isolated by a chromatographic procedure from a partial reduction of the previously described 10-methyl-2-*cis*-decalone (XVII)^{2,4} over

platinum oxide. Surprisingly, both isomers melt at 66–67°, but there can be no question of their non-identity because the m.p. of mixtures may be as low as 48° and the 3,5-dinitrobenzoate derivatives melt at 110–111° and 97–98°, respectively. Both show intense absorptions of almost equal intensities at 2.8, 3.5, 6.9 and 7.3 μ , but there are considerable differences beyond 8.5 μ in the fine structure region.

The assignment of a *cis-cis* configuration to Ia is based upon the following evidence. The keto-ester III was converted to 9-*cis*-decalincarboxylic acid (IV) by Wolff-Kishner reduction; thence by lithium aluminum hydride reduction of IV and then of the *p*-toluenesulfonate VI of the carbinol V to the known 9-methyl-*cis*-decalin (VIIIa).⁵ For confirmation, VIIa was isomerized to 9-methyl-*trans*-decalin (VIIb).⁵ A *cis* relationship of methyl and hydroxyl in Ia is a consequence of the requirement for a *cis* relationship of carboxyl to hydroxyl in the hydroxyacid IX to permit lactone formation in the precursor VIII.

A *trans-cis* configuration can be assigned to Ib because the parent 10-methyl-2-*cis*-decalone (XVII) from which it is derived by catalytic hydrogenation, has been demonstrated to have a *cis* ring fusion.⁴ Therefore, Ib must differ from Ia only in the configuration at C-2.

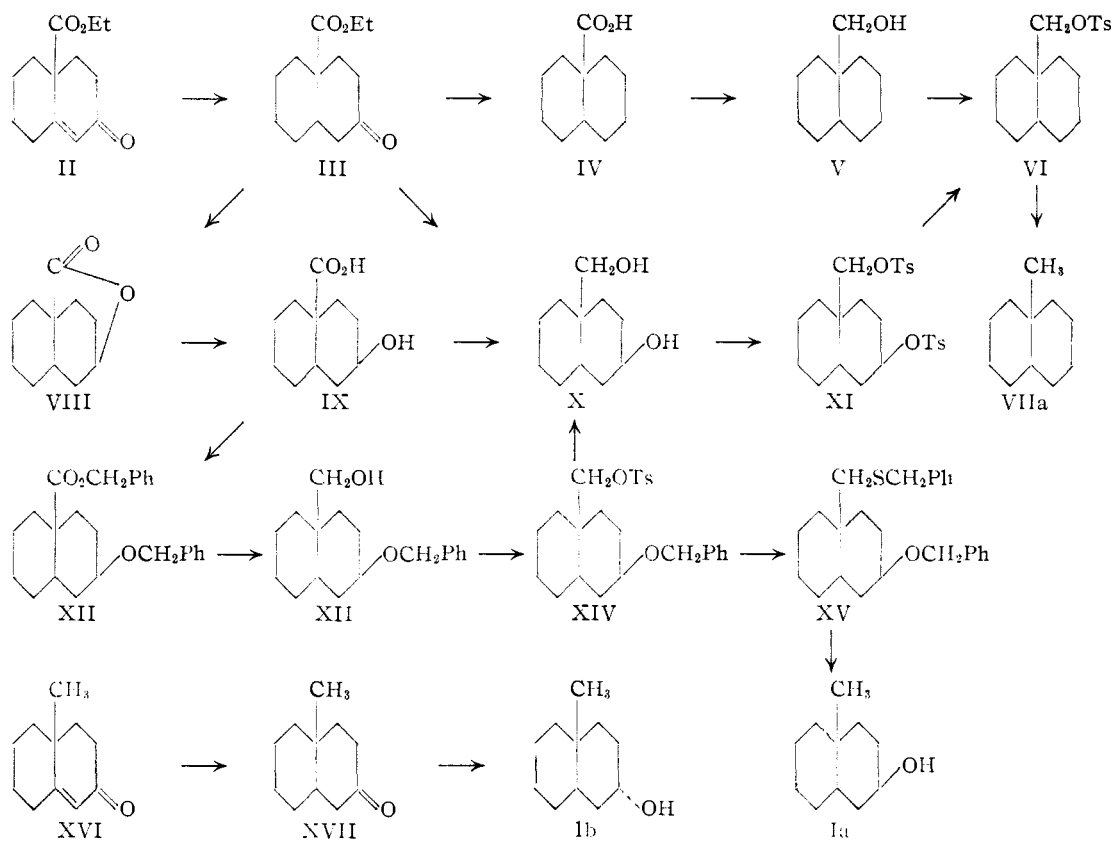
(1) This research was made possible by a special grant from the Research Corporation and, in part, by funds from the Graduate School of Northwestern University.

(2) V. C. E. Burnop and R. P. Linstead, *J. Chem. Soc.*, 720 (1940); R. B. Woodward, *This Journal*, **62**, 1208 (1940).

(3) The relationship of angular and peripheral substituents is expressed as a prefix of the name; the nature of the ring fusion as a prefix to the root of the name.

(4) E. C. du Feu, F. J. McQuillin and R. Robinson, *J. Chem. Soc.*, 53 (1937).

(5) R. P. Linstead and D. C. Hibbit, *ibid.*, 470 (1936); R. P. Linstead, A. F. Milledge and A. L. Walpole, *ibid.*, 1140 (1937).



The reduction of the sterically related ketones XVII and III by catalytic hydrogenation and with aluminum isopropoxide, respectively, is stereospecific in both instances but leads to opposite configurations. The *trans* configuration with catalytic hydrogenation is in accord with the reported formation of epicoprostanol from coprostanone⁶ as well as with the equatorial character and therefore greater stability⁷ of the *trans*-hydroxyl group. The reversibility of aluminum isopropoxide reduction, however, would permit the *trans*-hydroxy ester, if formed, finally to epimerize completely because the stable *cis*-lactone VIII can subsequently be formed.

However, there is evidence that factors other than lactone formation may also be influencing the steric course of chemical reduction of the carboxy ketone III. The hydroxy acid IX reduces smoothly with lithium aluminum hydride in butyl ether to give *cis*-10-hydroxymethyl-2-*cis*-decalol (X). The same diol is formed, and in only slightly lower yield, when ketoester III is reduced with this reagent in ethyl ether. The lactone VIII cannot be reduced with lithium aluminum hydride and therefore cannot be a sterically influencing intermediate in the reduction of ketoester to a *cis*-diol.

Originally we had hoped to convert the hydroxy acid IX to the configurationally related methyldecalol Ia by the same sequence of steps used in converting 9-*cis*-decalincarboxylic acid (IV) to 9-methyl-*cis*-decalin (VIIa), but we were unable to convert the diol X to a mono-*p*-toluenesulfonate derivative. Treatment of the silver salt of the hydroxy acid IX

with benzyl chloride and moist silver oxide in toluene furnished a mixture of benzyl ether-acid and benzyl ether-benzyl ester XII which was then reduced with lithium aluminum hydride to the benzyl hydroxymethyldecalyl ether XIII. The *p*-toluenesulfonate derivative XIV could be reduced with lithium aluminum hydride, but the only product isolated after hydrogenolysis of the benzyl group was the diol X. This was formed by reduction at sulfur rather than at carbon as has been observed previously.⁸

This difficulty was overcome by conversion of the *p*-toluenesulfonate derivative XIV to the benzyl thioether XV by the excellent procedure of Bordwell and co-workers,⁹ and the latter with an active Raney nickel catalyst lost both S- and O-benzyl groups to form Ia. The removal of O-benzyl groups in this way has been observed previously.¹⁰

Experimental^{11,12}

10-Carboxy-2-octalone (II).—When 2-carboxycyclohexanone¹³ and 4-diethylamino-2-butanone methiodide were heated at reflux in ethyl alcohol with 1.08 equivalents of sodium ethoxide instead of 0.87 equivalent as described,⁴ a 69–70% yield of 10-carboxy-2-octalone (b.p. 139–141° at 2 mm., n_D^{20} 1.5043) was realized.

10-Carboxy-2-*cis*-decalone (III).—The reduction of 40 g. of the unsaturated ketoester II in 150 cc. of ethyl alco-

(8) H. Schmid and P. Karrer, *Helv. Chim. Acta*, **32**, 1371 (1949).

(9) F. G. Bordwell, B. Pitt and M. Knell, *This Journal*, **73**, 5004 (1951).

(10) Private communication, Dr. W. A. Bonner, Stanford University.

(11) All m.p.'s corrected unless otherwise indicated.

(12) Microanalyses by M. Hines, V. Hobbs, J. Sorenson and C. White. Infrared spectra by E. Bowen.

(13) "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1948, p. 531.

(6) L. Ruzicka, *Helv. Chim. Acta*, **19E**, 90 (1936).

(7) D. Barton, *Experientia*, **6**, 316 (1950). For a recent interpretation see, S. Siegel, *This Journal*, **75**, 1317 (1953).

hol over platinum oxide under 3 atmospheres of hydrogen was complete within 20 minutes. Distillation gave 36 g. (90%) of 10-carbethoxy-2-*cis*-decalone, b.p. 146–148° at 2 mm., n_D^{25} 1.4790. A sample crystallized from petroleum ether melted at 3.4–4.0°. A 2,4-dinitrophenylhydrazone derivative melted at 167°, from ethyl alcohol.

Anal. Calcd. for $C_{19}H_{24}O_6N_4$: C, 56.42; H, 5.98; N, 13.86. Found: C, 56.5; H, 5.8; N, 13.7.

9-*cis*-Decalincarboxylic Acid (IV).—A mixture of 47 g. of 10-carbethoxy-2-*cis*-decalone, 330 cc. of diethylene glycol, 25 cc. of 85% hydrazine hydrate and 45 g. of potassium hydroxide was refluxed for 1 hour, distilled until the liquid temperature reached 200°, and then refluxed for 3 more hours.¹⁴ After dilution with water, then filtration and extraction with ether, acidification of the alkaline solution gave 31 g. (82%), of crude 9-*cis*-decalincarboxylic acid, m.p. 120–127°. A sample for analysis, twice recrystallized from ethyl alcohol, melted at 134.6–135.4°.

Anal. Calcd. for $C_{11}H_{18}O_2$: C, 72.49; H, 9.96; neut. equiv., 182.5. Found: C, 73.0; H, 10.2; neut. equiv., 183.

The solid material (1.5 g.) recovered by filtration prior to extraction with ether contained nitrogen and melted at 221–222° (uncor.), from alcohol. The nitrogen analysis agreed with that calculated for 1,2-bis-(9-*cis*-decalincarboxyl)-hydrazine. The amount of this material formed was materially reduced by making sure that excess hydrazine was removed before the temperature of the mixture was raised above 200°.

Anal. Calcd. for $C_{22}H_{30}O_2N_2$: N, 7.77. Found: N, 8.2.

The ether extract from above furnished 3.1 g. of a nitrogen-containing neutral compound, m.p. 144–145°, from alcohol. The nitrogen analysis agreed with that calculated for the product of intramolecular cyclization of the hydrazine.

Anal. Calcd. for $C_{11}H_{18}ON_2$: N, 14.57. Found: N, 14.5.

9-Hydroxymethyl-*cis*-decalin (V).—The reduction of 7.0 g. of 9-*cis*-decalincarboxylic acid in *n*-butyl ether solution with 2.8 g. of lithium aluminum hydride was complete after 2 hours at reflux. The reduction failed to proceed in ethyl ether. Sublimation of the crude product at atmospheric pressure gave 5.4 g. (83%) of 9-hydroxymethyl-*cis*-decalin, m.p. 84.2–84.6°.

Anal. Calcd. for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found: C, 79.0; H, 12.1.

Treatment of a pyridine solution of the alcohol with a small excess of *p*-toluenesulfonyl chloride and refluxing for 4 hours gave 93–95% of the *p*-toluenesulfonate derivative VI on dilution with cold water. A sample for analysis (from ethyl alcohol) melted at 141.0–141.6°.

Anal. Calcd. for $C_{18}H_{26}O_4S$: C, 67.14; H, 8.08. Found: C, 67.3; H, 8.4.

9-Methyl-*cis*-decalin (VII).—The reduction of 10.7 g. of the *p*-toluenesulfonate VI from above with 2.4 g. of lithium aluminum hydride in 75 cc. of *n*-butyl ether was carried out at reflux for 3 hours. The ether extracts of the neutralized reaction mixture were dried over potassium carbonate and carefully distilled. The product, collected over the range 80–83° at 11 mm., weighed 2.5 g. (47%), n_D^{25} 1.4802.⁵

Anal. Calcd. for $C_{11}H_{20}$: C, 86.84; H, 13.16. Found: C, 86.9; H, 13.2.

From the residue of the distillation 0.6 g. (6%) of 9-hydroxymethyl-*cis*-decalin (V) was isolated by crystallization from dilute alcohol followed by sublimation.

Isomerization of 9-Methyl-*cis*-decalin to 9-Methyl-*trans*-decalin.—A 2.0-g. sample of 9-methyl-*cis*-decalin, prepared as above, was allowed to stand over 0.25 g. of freshly sublimed aluminum chloride. The refractive index was observed over 6 days.

Time, days	n_D^{25}	Time, days	n_D^{25}
0	1.4802	4	1.4680
2	1.4718	5	1.4675
3	1.4690	6	1.4667

(14) M. L. Huang, *THIS JOURNAL*, **68**, 2487 (1946); **71**, 3302 (1949).

Distillation of the sample gave 1.5 g. of material distilling over the range 70–73° at 11 mm., n_D^{25} 1.4670.⁵ Treatment of this material with aluminum chloride caused no further change in physical properties.

***cis*-10-Carboxy-2-*cis*-decalol Lactone (VIII).**—A solution containing 124 g. of 10-carbethoxy-2-*cis*-decalone and aluminum isopropoxide from 10.0 g. of aluminum foil in 300 cc. of anhydrous isopropyl alcohol was refluxed for 20 hours with short periods of distillation to remove accumulated acetone.¹⁵ Removal of solvent and neutralization with ice-cold dilute sulfuric acid was followed by extraction with ether. From the material extracted by ether, 95 g. (95%) of lactone was distilled at 145–148° at 5 mm., n_D^{25} 1.4990. A redistilled sample (128–131° at 2 mm.) had n_D^{25} 1.5010.

Anal. Calcd. for $C_{11}H_{18}O_2$: C, 73.30; H, 8.95. Found: C, 73.0; H, 9.2.

***cis*-10-Carboxy-2-*cis*-decalol (IX).**—A mixture of 1.50 g. of the lactone VIII and 40 cc. of 10% potassium hydroxide was heated for 18 hours on the steam-bath. The crude *cis*-10-carboxy-2-*cis*-decalol was recrystallized from dilute alcohol to give 1.27 g. (85%) of pure acid, m.p. 149.2–150.6°.

Anal. Calcd. for $C_{11}H_{18}O_3$: C, 66.64; H, 9.15; neut. equiv., 198.3. Found: C, 67.0; H, 9.6; neut. equiv., 198.

The hydroxy acid IX, 5.00 g., readily formed the original lactone when refluxed four hours in benzene with 0.10 g. of *p*-toluenesulfonic acid. From the neutral product, 3.75 g. (87%) of lactone, b.p. 128–131° at 2.5 mm., n_D^{25} 1.5020, was isolated by vacuum distillation.

***cis*-10-Hydroxymethyl-2-*cis*-decalol (X).**—A solution of 1.5 g. of *cis*-10-carboxy-2-*cis*-decalol in 20 cc. of *n*-butyl ether with 0.50 g. of lithium aluminum hydride was refluxed for 3 hours and then carefully hydrolyzed with ice-water. The neutral product was extracted with several portions of ether, in which it was only slightly soluble. Removal of part of the ether caused the crystallization of 1.1 g. (85%), of *cis*-10-hydroxymethyl-2-*cis*-decalol, m.p. 141.8–142.6°.

Anal. Calcd. for $C_{11}H_{20}O_2$: C, 71.69; H, 10.94. Found: C, 71.8; H, 10.9.

Only unreduced *cis*-10-carboxy-2-*cis*-decalol lactone could be isolated when a similar reduction of VIII with lithium aluminum hydride was attempted.

The reduction of 15.0 g. of *cis*-10-carbethoxy-2-*cis*-decalone (III) in ethyl ether with 3.5 g. of lithium aluminum hydride at reflux for 3 hours gave 9.8 g. (66%) of diol X, m.p. 139–141°, no depression with the diol from above, together with 4.0 g. of unidentified, ether-soluble material, b.p. 125–145° at 2.5 mm.

Bis-*p*-toluenesulfonate Ester of *cis*-10-Hydroxymethyl-2-*cis*-decalol (XI).—A solution of 1.00 g. of the diol X in 2.5 cc. of pyridine with 6.0 g. of *p*-toluenesulfonyl chloride was diluted with 30 g. of ice after being refluxed for 2 hours. The crystalline precipitate was twice recrystallized from ethyl alcohol giving 2.22 g. (96%) of bis-*p*-toluenesulfonate ester, m.p. 96.2–97.4°.

Anal. Calcd. for $C_{25}H_{32}O_6S_2$: C, 60.96; H, 6.50. Found: C, 60.3; H, 6.8.

A solution of 3.80 g. of bis-*p*-toluenesulfonate ester XI and 0.68 g. of potassium hydroxide in 10 cc. of ethyl alcohol and 2 cc. of water was refluxed for 7 days. The white crystals which separated upon dilution with 50 cc. of water were recrystallized from benzene-petroleum ether to give 2.60 g. (88%) of unsaturated mono-*p*-toluenesulfonate, m.p. 104–106°.

Anal. Calcd. for $C_{18}H_{24}O_3S$: C, 67.47; H, 7.55. Found: C, 66.8; H, 7.9.

When reduced over platinum oxide, 0.379 g. of the unsaturated ester gave 0.345 g. (91%) of the *p*-toluenesulfonate ester of 9-hydroxymethyl-*cis*-decalin (VI) m.p. 136–138°, no depression when mixed with the ester formed from V.

Benzyl *cis*-10-Carboxy-2-*cis*-decalyl Ether and Benzyl Ester XII.—Treatment of an ammonia-free solution of the ammonium salt of *cis*-10-carboxy-2-*cis*-decalol with excess silver nitrate¹⁶ gave 91% of dry, silver nitrate-free silver salt. A suspension of 28.0 g. of silver salt, 15 g. of moist

(15) "Organic Reactions," Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1948, p. 198.

(16) F. Mathews, G. Warren and J. Mitchell, *Anal. Chem.*, **22**, 516 (1950).

silver oxide and 200 cc. of toluene was refluxed with good agitation for 1 hour. After adding 50 g. of benzyl chloride dropwise over two hours, reflux and agitation were continued for 30 hours. Silver oxide and silver chloride (32 g.) were separated by filtration and the toluene solution was distilled. Recovered benzyl chloride, b.p. 110–125° at 3 mm., n_D^{25} 1.5400, amounted to 19.5 g. The residue weighed 19.2 g. Triturating this oily residue with 50 cc. of acetone permitted the separation of 6.0 g. (23%) of benzyl *cis*-10-carboxy-2-decalyl ether, m.p. 137.2–138.5°, from 13.5 g. (37%) of acetone-soluble benzyl ester-ether contaminated with some *cis*-10-carbobenzoxy-2-*cis*-decalol.

Anal. Calcd. for $C_{18}H_{26}O_7$: C, 74.97; H, 8.39; neut. equiv., 288.4. Found: C, 75.3; H, 8.1; neut. equiv., 286.

Benzyl *cis*-10-Hydroxymethyl-2-*cis*-decalyl Ether (XIII).—A 19.0-g. sample of impure benzyl ether-ester XII prepared as above was reduced in 50 cc. of *n*-butyl ether with 2.0 g. of lithium aluminum hydride as described previously. From the viscous liquid product 3.0 g. of diol X, m.p. 140–141° (no depression), separated as crystals and was isolated easily by virtue of its low solubility in ether. The remainder of the oil was distilled at 215–225° at 2 mm. and gave 8.1 g. of XIII which crystallized after standing several days, m.p. 65–68°.

Anal. Calcd. for $C_{18}H_{26}O_3$: C, 78.79; H, 9.55. Found: C, 77.0; H, 9.6.

With 14.0 g. of *p*-toluenesulfonyl chloride in 35 cc. of pyridine this gave 12.5 g. (58% from XII) of *p*-toluenesulfonate ester XIV, m.p. 112.8–114.0°, from benzene-petroleum ether. Corrected for *cis*-10-carbobenzoxy-2-*cis*-decalol impurity in XII corresponding to diol X isolated above, this yield represents 78% of theory.

Anal. Calcd. for $C_{23}H_{32}O_4S$: C, 70.07; H, 7.53. Found: C, 70.7, 70.6; H, 7.6, 7.8.

Reduction of 12.0 g. of XIV with 2.4 g. of lithium aluminum hydride in 80 cc. of *n*-butyl ether gave 4.9 g. of colorless product which distilled at 170–173° at 2 mm. Without purification, this material was hydrogenated over platinum oxide in ethyl alcohol. The only product which could be isolated and identified was 1.2 g. (23%) of *cis*-10-hydroxymethyl-2-*cis*-decalol (X), m.p. 140–141°, no depression with an authentic sample.

***cis*-10-Methyl-2-*cis*-decalol (Ia).**—Sodium, 1.74 g., was added to a solution of 17.0 g. of the *p*-toluenesulfonate XIV and 7.2 g. of benzyl mercaptan in 100 cc. of diethylene glycol monomethyl ether and the mixture was refluxed for 4 hours after all of the sodium had reacted.⁹ Excess benzyl mercaptan was removed by steam distillation after acidification with hydrochloric acid. The oily product XV which separated on cooling weighed 12.4 g., n_D^{25} 1.5755, and without further purification this was refluxed in 400 cc. of ethyl alcohol with 80 g. of Raney nickel¹⁷ for 8 hours.¹⁸ The oily residue which was left after removal of catalyst and

solvent was distilled at 128–130° at 10 mm. to give 4.2 g. (63%) of colorless liquid, n_D^{25} 1.4990, which crystallized on standing. When recrystallized from petroleum ether, this material melted at 66.2–67.4°.

Anal. Calcd. for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found: C, 79.1, 79.0; H, 12.3, 12.2.

The 3,5-dinitrobenzoate derivative melted at 110.6–111.2° after recrystallization from petroleum ether.

Anal. Calcd. for $C_{18}H_{22}O_6N_2$: N, 7.73. Found: N, 7.9.

10-Methyl-2-octalone (XVI).⁴—A solution of 33.0 g. of 2-methylcyclohexanone and potassium *t*-butoxide (from 11.7 g. of potassium) in 150 cc. of anhyd. *t*-butyl alcohol was refluxed for 1 hour. A solution of 4-diethylamino-2-butanone methiodide (from 21.5 g. of 4-diethylamino-2-butanone and 21.5 g. of methyl iodide), in 100 cc. of anhyd. *t*-butyl alcohol was added slowly with good cooling. After 12 hours at room temperature, the mixture was heated to reflux for 1 hour and then acidified with ice-cold hydrochloric acid. The solvent was removed at the water-pump and the product was extracted with ether. Distillation of the dried ether extract gave 7.0 g. (28%) of 10-methyl-2-octalone at 142–145° at 15 mm., n_D^{25} 1.5060. The semicarbazone derivative⁴ melted at 203–205° (uncor.) from alcohol. With powdered sodamide in dry ether,⁴ the yield of distilled product was 10–15%.

10-Methyl-2-*cis*-decalone (XVII).⁴—The hydrogenation of 7.0 g. of 10-methyl-2-octalone over platinum oxide in ethyl alcohol slightly acidified with acetic acid was discontinued when one-half of the hydrogen necessary for reduction to the saturated ketone had been absorbed. The product, b.p. 94–96° at 3 mm., was separated by distillation from unreduced material (55%), b.p. 120–123° at 3 mm. Crystallization from petroleum ether gave 2.0 g. (29%) of 10-methyl-2-*cis*-decalone, m.p. 47.0–47.6°.^{2,4}

***trans*-10-Methyl-2-*cis*-decalol (Ib).**—A solution of 1.25 g. of 10-methyl-2-*cis*-decalone in ethyl alcohol slightly acidified with acetic acid was shaken with platinum oxide under two atmospheres of hydrogen for 3 hours. The product was deposited on a 20-cm. column of alumina from petroleum ether and eluted with successive portions of petroleum ether followed by benzene. The petroleum ether eluates furnished 0.51 g. (40%) of recovered ketone; the petroleum ether-benzene eluate, 0.40 g. (32%) of *trans*-10-methyl-2-*cis*-decalol, m.p. 66–67°. A sample recrystallized several times from petroleum ether melted at 66.2–67.0°.

Anal. Calcd. for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found: C, 78.5; H, 11.5.

The 3,5-dinitrobenzoate ester derivative melted at 97.5–98.5° when recrystallized from petroleum hexane.

Anal. Calcd. for $C_{18}H_{22}O_6N_2$: N, 7.73. Found: N, 8.2.

When mixed with isomeric methyldecalol Ia in proportions of 85, 50 and 10% Ib, the melting ranges observed were 56–65°, 48–64° and 59–67°, respectively.

Infrared Spectra.¹¹—The absorption of chloroform solutions (0.150 g. in 3.0 cc.; 0.1-mm. cell) of *cis*- and *trans*-10-methyl-2-*cis*-decalol from 2.6 to 11.0 μ , was measured in a Beckman IR2T recording spectrophotometer.

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(17) Prepared according to the procedure of *Org. Syntheses*, **21**, 15 (1941), except for heating at 50° for 1 hour rather than on the steam-bath for 8 hours.

(18) D. Papa, E. Schwenk and H. F. Ginsberg, *J. Org. Chem.*, **14**, 723 (1949).