

The analytical sample was obtained by short-path distillation.

Anal. Calcd for $C_6H_{12}O$: C, 71.9; H, 12.1. Found: C, 71.6; H, 11.9.

(*Z*)-2-Methyl-2-penten-1-ol (6).—Following the procedure for the preparation of alcohol 5, 15 g of the acid mixture consisting of 3 and 4 gave 9.6 g (74%) of a mixture of the alcohols 6 and 7, bp 71–74° (20 mm). The alcohols were separated by column chromatography on silver nitrate-alumina, prepared by adding 84 ml of saturated aqueous silver nitrate to 940 g of neutral alumina, activity I. The mixture was shaken vigorously until no lumps remained and allowed to stand for 48 hr; 9.7 g of the mixture of alcohols 6 and 7 was then applied. Elution with hexane-ether (1:4) yielded 7.7 g of alcohol 6 free of 7 by tlc.¹⁶ Distillation afforded analytically pure 6, bp 71–74° (15 mm).

Anal. Calcd for $C_6H_{12}O$: C, 71.9; H, 12.1. Found: C, 71.9; H, 12.5.

(*E*)-2-Methyl-2-pentenal (8).—To a solution of 1 g of alcohol 5 in 15 ml of methylene chloride was added 15 g of freshly prepared manganese dioxide.²⁸ After shaking for 18 hr at room temperature, the solid was filtered off and the solvent evaporated to give the crude aldehyde 8. Short-path distillation gave 0.81 g (83%) of 8 as a slightly yellow, volatile liquid that decomposes with ease under nitrogen at 3°: see footnote 22b for boiling point data; uv max (95% EtOH), 228 nm (ϵ 12,000); ir (CHCl₃), 2730 (CHO), 1685 (C=O), 1645 (C=C).

*Anal.*²⁹ Calcd for $C_6H_{10}O$: C, 73.4; H, 10.3. Found: C, 72.4; H, 10.2.

(*Z*)-2-Methyl-2-pentenal (9).—Following the procedure for preparation of aldehyde 8, 0.96 g of alcohol 6 in 60 ml of methylene

chloride was oxidized with 15 g of manganese dioxide to afford 0.62 g (64%) of the aldehyde 9. Spectroscopic data were determined on a freshly prepared sample: uv max (95% EtOH), 237 nm (ϵ 8900); ir (CHCl₃), 2743 (CHO), 1673 (C=O), 1656 (C=C). Short-path distillation under nitrogen followed by glpc²¹ afforded the analytical sample which was isomerically pure.

*Anal.*²⁹ Calcd for $C_6H_{10}O$: C, 73.4; H, 10.3. Found: C, 71.3; H, 10.2.

(*Z*)-2-Methyl-2-pentenoic Acid (3).—A 4.0-g sample of the freshly prepared aldehyde 9 was oxidized in the described manner³⁰ to yield 3.26 g (70%) of the acid 3, bp 102–103.5° (15 mm) [lit.¹² bp 94.0–94.4° (10 mm)].

Methyl (*E*)-2-Methyl-2-pentenoate (10).—To 0.8 g of acid 2 in 5 ml of methanol was added 0.1 ml of concentrated sulfuric acid. The mixture was refluxed for 1.5 hr and then poured into 15 ml of ice-water. The aqueous solution was extracted with three 10-ml portions of ether, and the combined ether extracts were washed twice with saturated sodium bicarbonate and then dried (MgSO₄). Evaporation of the solvent gave 0.62 g (69%) of the methyl ester 10.¹⁷ Short-path distillation gave the analytical sample.

Anal. Calcd for $C_7H_{12}O_2$: C, 65.6; H, 9.4. Found: C, 65.5; H, 9.3.

Methyl (*Z*)-2-Methyl-2-pentenoate (11).—A 0.9-g sample of acid 3 was esterified according to the procedure for the preparation of ester 10 to give 0.3 g (31%, 78% based upon the recovery of 0.56 g of 3) of the methyl ester 11.¹⁷ Short-path distillation gave the analytical sample.

Anal. Calcd for $C_7H_{12}O_2$: C, 65.6; H, 9.4. Found: C, 65.3; H, 9.3.

Registry No.—2, 16957-70-3; 3, 1617-37-4; 5, 16958-19-3; 6, 16958-20-6; 8, 14250-96-5; 9, 16958-22-8; 10, 1567-14-2; 11, 1567-13-1.

(30) A. A. Goldberg and R. P. Linstead, *J. Chem. Soc.*, 2343 (1928).

(28) J. Attenburrow, A. F. B. Cameron, J. H. Chapman, R. M. Evans, B. A. Hems, A. B. A. Jansen, and T. Walker, *J. Chem. Soc.*, 1094 (1952), or O. Mancera, G. Rosenkranz, and F. Sondheimer, *ibid.*, 2189 (1953).

(29) These aldehydes are unstable and, though isomerically pure, repeatedly gave low carbon analyses.

Diaxial Ring Opening of 1,2-Oxidocyclohexanes

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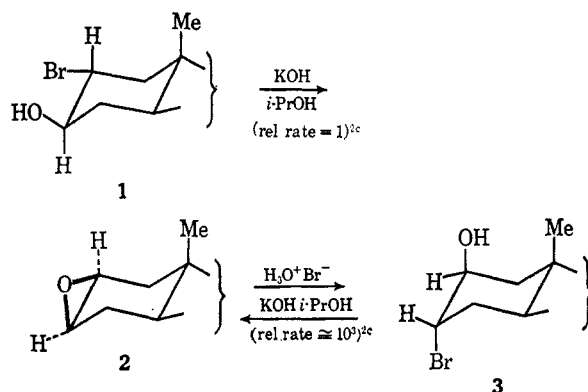
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The synthetic sequence (2-bromo-4,4-diphenylcyclohexanone \rightarrow bromohydrin \rightarrow epoxide \rightarrow new halohydrin \rightarrow 2-halo-5,5-diphenylcyclohexanone) has been carried out in 42% over-all yield. The same sequence also has been applied successfully to 2-bromo-4-methyl-4-phenylcyclohexanone. These results show that one of two possible diaxial ring openings for 4,4-disubstituted 1,2-oxidocyclohexanes occurs stereoselectively.

In 2-halo keto steroids it is sometimes possible to interchange the positions of the halogen and carbonyl functions by the following sequence of steps: (1) hydride reduction, (2) conversion of the resulting halohydrin into an epoxide, (3) cleavage of the epoxide with a hydrohalogen acid to form a new halohydrin in which the positions of the halogen and hydroxyl functions have been interchanged, and (4) oxidation to regenerate the carbonyl function. For example, 2 α -bromocholestan-3-one can be converted into 3 α -bromocholestan-2-one in this way.¹ (This also serves as a means of converting cholestan-3-one into cholestan-2-one.¹)

The success of this method depends on a marked preference for diaxial ring closure in the formation of the epoxides and a marked preference for diaxial ring opening in the cleavage of epoxides.^{1,2} Diaxial ring closure of 2 α -bromocholestan-3 β -ol (1) presumably proceeds via a boat conformation to form 2 β ,3 β -oxidcholestan-

(2), which accounts for the 10³ slower rate in this ring closure as compared to the formation of 2 by diaxial ring closure of 3 α -bromocholestan-2 β -ol (3).^{2c} Hydrobromic acid cleaves 2 to give the diaxial bromohydrin 3.^{2b}



(1) G. H. Alt and D. H. R. Barton, *J. Chem. Soc.*, 4284 (1954).

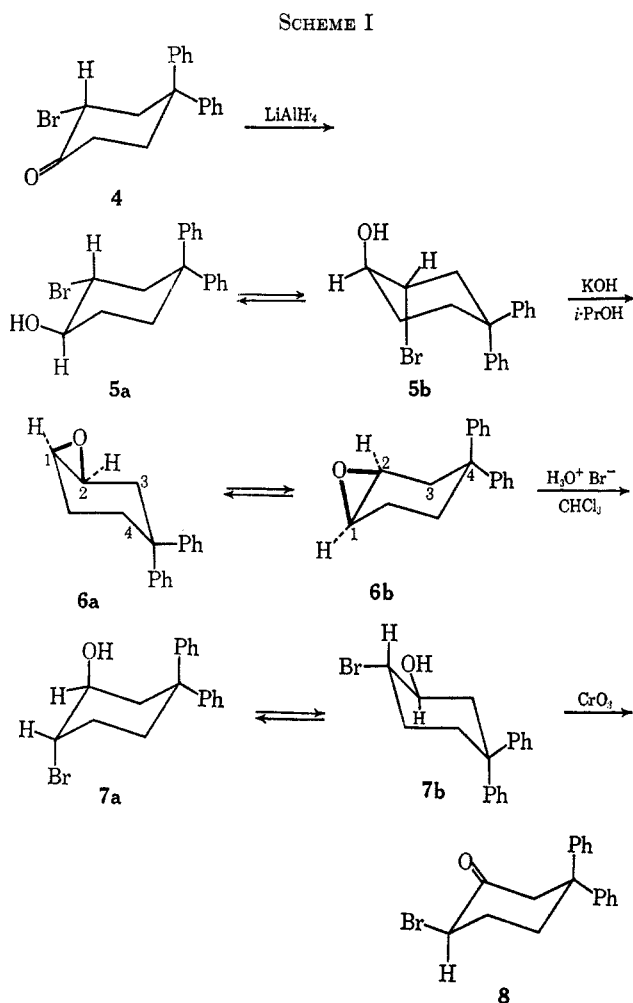
(2) (a) A. Fürst and R. A. Plattner, Abstracts, 12th International Congress of Pure and Applied Chemistry, New York, N. Y., 1951, p 409; (b) D. H. R. Barton, *J. Chem. Soc.*, 1027 (1953); (c) D. H. R. Barton and R. C. Cookson, *Quart. Rev.* (London), 10, 67 (1956); (d) R. E. Parker and N. S. Isaacs, *Chem. Rev.*, 59, 737 (1959).

It was of interest to see whether 2-halo-4,4-disubstituted cyclohexanones, which are readily available, could be transformed by a comparable series of steps to

2-halo-5,5-disubstituted cyclohexanones. The result here could not be predicted with confidence because the epoxide corresponding to **2** can undergo chair-chair type interconversion and can react in either of two chairlike conformations; one of these would give back the original bromohydrin (corresponding to **1**) by diaxial ring opening, whereas the other would give the rearranged bromohydrin (corresponding to **3**) by diaxial ring opening. Nevertheless, the sequence did proceed in the desired manner and therefore provided a useful synthetic route.

The synthetic sequence was first applied to the conversion of 2-bromo-4,4-diphenylcyclohexanone (**4**) into 2-bromo-5,5-diphenylcyclohexanone (**8**) (Scheme I).

Reduction of **4** was expected to give predominantly the *trans* (diequatorial) bromohydrin **5a**^{2b,3} (not isolated). Diaxial ring closure *via* conformation **5b** or, less likely, *via* a boat conformation (as for **1**) gave epoxide **6**. Diaxial ring opening by axial attack of bromide ion at C-1 of the conjugate acid of conformation **6b** of the epoxide gave **7a** (compare **2** → **3**). Di-

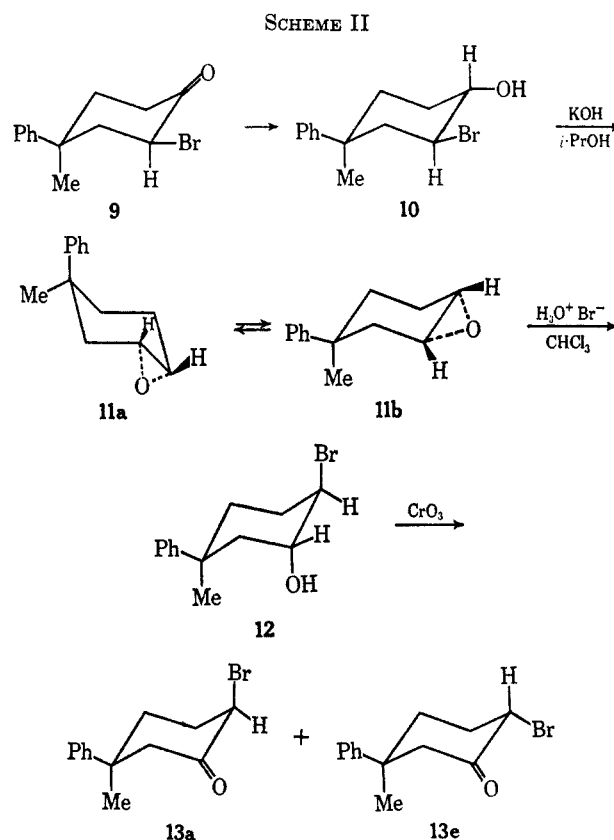


axial opening by attack at C-2 of the conjugate acid of conformation **6a** of the epoxide would have re-formed bromohydrin **5**. The success of the method depends, therefore, not only on the preference for diaxial ring opening,² but also on a preference for reaction *via* conformation **6b** rather than **6a**. This latter preference is

(3) L. F. Fieser and M. Fieser, "Steroids," Reinhold Publishing Corp., New York, N. Y., 1959, p 268.

understandable in view of the steric hindrance offered by the axial phenyl group at C-4 in the conjugate acid of **6a** (but not in that of **6b**) to axial attack by bromide ion.

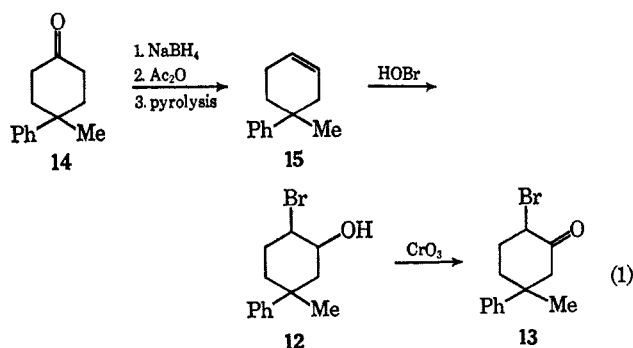
Application of this reaction sequence (Scheme II) to 2-bromo-4-methyl-*cis*-4-phenylcyclohexanone (**9**) gave



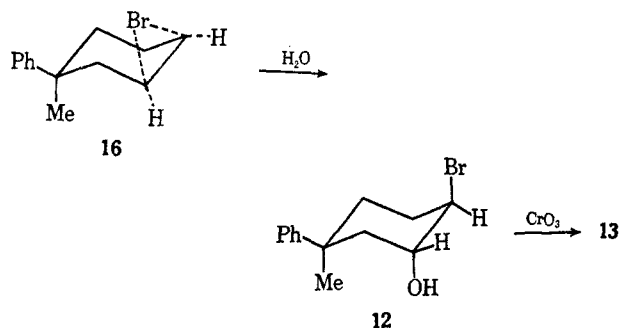
a mixture of 2-bromo-5-methyl-*cis*-5-phenylcyclohexanone (**13a**, bromine axial) and 2-bromo-5-methyl-*trans*-5-phenylcyclohexanone (**13e**, bromine equatorial) contaminated with a small amount of **9**. Similar results were obtained on opening epoxides **6** and **11** with hydrochloric acid.

Diaxial opening of **11** by attack of bromide ion at C-1 of **11b** should give bromohydrin **12**; on oxidation **12** should give **13a**, but not **13e**. Evidently the latter arises in the experiment by epimerization of **13a**. Indeed, the epimerization, **13a** → **13e** was observed to occur under the mild conditions used in processing (chromatography over silica gel).

The conversion of 4-methyl-4-phenylcyclohexanone (**14**) into a mixture of **13a** and **13e** also was accomplished by the alternative route shown in eq 1.



The success of this method depends on diaxial ring opening^{2c} of bromonium ion **16** (formed by "topside addition" to **15**) to form **12** by attack of a water molecule at C-2 in preference to diaxial opening of the isomeric bromonium ion (not shown; formed by "bottomside addition" to **15**) by attack of a water molecule at C-1. Here the mode of addition of bromine apparently controls the stereochemistry.



2-Bromo-5-methyl-*cis*-5-phenylcyclohexanone (**13a**) prepared by this route was obtained in pure form by crystallization, but the *trans* isomer (**13e**) was always contaminated with some **13a**.

Diaxial ring closures to form epoxides, as in the conversion of **3** into **2** or **5b** into **6a**, are clearly preferred on steric grounds to diequatorial ring closures such as **1** to **2** because they provide a path for inversion at the C-Br center *via* a *trans* coplanar transition state.² The "diequatorial ring closure" for **1** no doubt also proceeds *via* a *trans* coplanar transition state, but it is necessary in this instance for the bromohydrin to react in a boat conformation. It is not obvious why diaxial ring opening of an epoxide should occur in preference to diequatorial ring opening. Judging from the ground state it would appear that there is little to choose between attack by bromide ion at C-1 or C-2 on the conjugate acid of epoxide **6b**. It is only in the transition states that clearcut differences between the two modes of attack are manifested. In a diaxial ring opening the transition state can become nearly productlike in structure without greatly distorting the trigonal bipyramide type geometry (180° Br-C-O angle) preferred at the reacting center. On the other hand, the transition state for diequatorial ring opening must severely distort the Br-C-O angle of the trigonal bipyramide if it is to become productlike.⁴ (See Scheme III.)

Experimental Section⁵

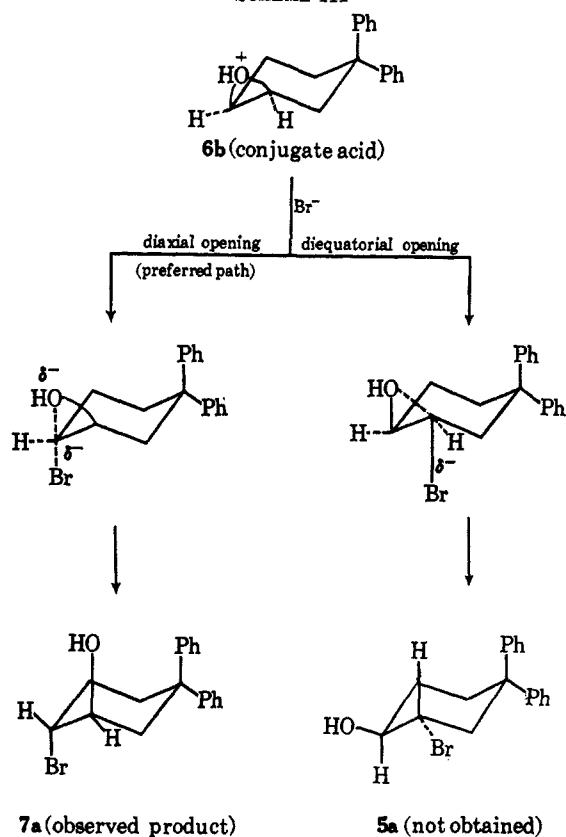
trans-2-Bromo-4,4-diphenylcyclohexanol (**5**).—2-Bromo-4,4-diphenylcyclohexanone⁶ (18 g, 54 mmol) suspended in 1 lb of anhydrous ether was treated with lithium aluminum hydride (0.382 g, 0.04 equiv) in one portion, and the mixture was stirred for 2 hr at room temperature. An additional 0.38 g (0.04 equiv) of lithium aluminum hydride was added and the solution was stirred for 2 hr. About 4 ml of water and 2.5 ml of 10% sodium hydroxide were added and, after stirring for 2.5 hr, the solution was diluted with 250 ml of chloroform. Filtration through diatomaceous earth, drying ($MgSO_4$), and concentration gave 16.8 g (51 mmol, 93%) of crude colorless **5**. This material was used without further purification in the next step of the synthesis. An analytical sample prepared by crystallizing from ether-

(4) See ref 2d for a discussion of this point and for examples of diaxial ring openings.

(5) Microanalyses were by Micro-Tech Laboratories, Skokie, Ill.

(6) F. G. Bordwell, R. R. Frame, R. G. Scamehorn, J. G. Strong, and S. Meyerson, *J. Amer. Chem. Soc.*, **89**, 6704 (1967).

SCHEME III



hexane melted at 135.5 – 137° : λ_{max}^{KBr} 6.19, 6.64, 6.84, 8.01, 9.38 (wide), 12.98, 13.24, 14.10, 14.50 μ .

Anal. Calcd for $C_{18}H_{19}BrO$: C, 65.26; H, 5.78. Found: C, 65.41; H, 5.86.

1,2-Oxido-4,4-diphenylcyclohexane (6).—A solution of 2-bromo-4,4-diphenylcyclohexanol (19.5 g, 59 mmol) and 39 g (690 mmol) of potassium hydroxide in 850 ml of isopropyl alcohol was stirred for 4 hr on the steam bath. Most of the alcohol was removed at reduced pressure, water was added, and the mixture was extracted with ether. Concentration of the ether solution yielded 10.4 g (41.4 mmol 70%) of **6** as yellow needles. This was used without further purification. Crystallization from ether-hexane gave an analytical sample of colorless needles: mp 98 – 99° ; λ_{max}^{KBr} 6.65, 6.86, 9.92, 10.63, 11.48, 12.43, 13.19, 14.20 μ (wide).

Anal. Calcd for $C_{18}H_{19}O$: C, 86.36; H, 7.24. Found: C, 86.34; H, 7.27.

2-Bromo-5,5-diphenylcyclohexanone (8).—1,2-Oxido-4,4-diphenylcyclohexane (10.4 g, 41 mmol) was dissolved in 350 ml of chloroform and 195 ml of aqueous 48% hydrobromic acid was added. This mixture was shaken in a separatory funnel for 1 hr. The layers were separated and the organic phase was extracted with two 100-ml portions of 5% sodium bicarbonate and a 100-ml portion of water. Drying ($MgSO_4$) and concentration, gave a viscous oil. This was oxidized according to the procedure of Djerassi.⁷ The solid obtained was triturated with 20 ml of 1:1 ether-benzene and enough additional ether to fully effect solution (*ca.* 5 ml). This solution was adsorbed onto a slurry-packed (2% ether-hexane) silica gel column (4.5 \times 65 cm) and eluted with ether-hexane as follows. Fractions 1 and 2 (750 ml, 3%; 750 ml, 6% ether) contained nothing. Fraction 3 (2250 ml, 8% ether) gave, after evaporation of most of the solvent, 8.5 g (26 mmol, 63%) of **8** as prisms, mp 96 – 96.5° . The mother liquors gave an additional 0.2217 g (0.7 mmol, 2%) of **8**: λ_{max}^{KBr} 5.80–5.85, 6.70, 6.92, 8.40 (wide), 9.70, 13.20 (wide), 14.00–14.40 μ .

Anal. Calcd for $C_{18}H_{17}BrO$: C, 65.66; H, 5.21. Found: C, 65.59; H, 5.24.

2-Chloro-5,5-diphenylcyclohexanone.—1,2-Oxido-4,4-diphenylcyclohexane (8.3 g, 33 mmol) was hydrolyzed with concentrated

(7) C. Djerassi, R. R. Engle, and A. Bowers, *J. Org. Chem.*, **21**, 1547 (1956).

hydrochloric acid and the chlorohydrin was oxidized as above to give 6 g (21 mmol, 64%) of 2-chloro-5,5-diphenylcyclohexanone. The mother liquors gave an additional 1.0 g (3.5 mmol, 11%) of product. Crystallization from methanol yielded prisms: mp 108.5–109.5°; $\lambda_{\text{max}}^{\text{KBr}}$ 5.78, 6.68, 6.89, 9.63, 10.13, 12.03, 12.90, 13.20, 14.18–14.30 μ .

Anal. Calcd for $\text{C}_{18}\text{H}_{17}\text{ClO}$: C, 75.91; H, 6.02. Found: C, 76.05; H, 6.12.

3,3-Diphenylcyclohexanone.—2-Bromo-5,5-diphenylcyclohexanone (1.4 g, 4.2 mmol) and 1.1 g of zinc dust (Fisher Certified Reagent) were stirred in a solution of 74 ml of absolute ether and 7 ml of acetic acid for 24 hr. At the end of this time the reaction mixture was decanted into a separatory funnel and diluted with 300 ml of ether. Addition of hexane to the ether and slow evaporation gave 0.83 g (3.3 mmol, 79%) of long colorless needles. The mother liquor gave an additional 10%. Crystallization from ether-hexane yielded 3,3-diphenylcyclohexanone as long needles: mp 114.5–115°; $\lambda_{\text{max}}^{\text{KBr}}$ 5.85, 5.90 (these are components of a partially resolved doublet), 6.65, 6.88, 7.02, 8.10, 8.34, 13.15, 14.17 μ .

Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{O}$: C, 86.36; H, 7.24. Found: C, 86.65; H, 7.22.

4-Methyl-4-phenylcyclohexyl Acetate.—The crude product (55 g, 0.29 mol) from the lithium aluminum hydride reduction of 4-methyl-4-phenylcyclohexanone⁶ in 200 ml of dry pyridine was mixed with 35.7 g (0.35 mol) of acetic anhydride and heated on the steam bath for 90 min. After cooling, the reaction mixture was diluted with 500 ml of ether, and the ether solution was washed with 2% sulfuric acid, with 5% sodium bicarbonate, and with saturated brine, dried over magnesium sulfate, and concentrated. The oily residue as distilled to give 48.3 g (0.21 mol, 72%) of a *cis* and *trans* mixture of the acetates: bp 111–112° (0.4 mm); $\lambda_{\text{max}}^{\text{film}}$ 5.86 (s), 8.19 (s, broad) μ .

Anal. Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$: C, 77.55; H, 8.68. Found: C, 77.80; H, 8.67.

Analysis by vpc using a 12-ft copper tube (0.25 in.) packed with 20% QF1 on 60–80 Chromosorb P operated at 170° and a helium flow rate of 80 cc/min indicated the presence of both isomers with the major one being eluted first.

4-Methyl-4-phenylcyclohexene (15).—Pyrolysis of 53.4 g (0.23 mol) of the isomeric 4-methyl-4-phenylcyclohexyl acetates using a 2 × 17 cm tube packed with 5-mm glass beads thermostated at 506 ± 2° and operated at a flow rate of 35 drops/min gave, after the removal of acetic acid, 28.5 g (0.17 mol, 74%) of 4-methyl-4-phenylcyclohexene. Analysis by glpc (using the same conditions as for the acetates) showed the olefin to be uncontaminated by starting acetate or by side-reaction products; it was used in the next step without further purification. Evaporative distillation afforded an analytical sample [bp ~100° (5.0 mm)] which was shown to be identical with that reported.⁸

2-Bromo-5-methyl-5-phenylcyclohexanol (12).—A 130-ml portion of 1 N sulfuric acid was added at 0° over 60 min to a stirred solution of 10.0 g (58.1 mmol) of 4-methyl-4-phenylcyclohexene and 11.4 g (63.9 mmol) of N-bromosuccinimide in 200 ml each of *t*-butyl alcohol and dioxane. The solution was stirred at room temperature for 3 hr and then concentrated to approximately 200 ml. The organic products were absorbed into ether, and the ethereal layer was washed with 5% sodium bicarbonate, with water, and with saturated brine, dried over magnesium sulfate, and concentrated. The oily residue (13.7 g) was applied to a slurry-packed (10% ether in hexane), silica gel (650 g) chromatography column (103 × 3 cm) and eluted with 1 l. of 10%, 3 l. of 15%, and 3 l. of 20% ether in hexane mixtures. Fractions (500 ml) number 8–14 contained 8.56 g (55%) of 2-bromo-5-methyl-5-phenylcyclohexanol of unknown stereochemistry: $\lambda_{\text{max}}^{\text{film}}$ 2.93 (s), 9.56 (s, broad) μ ; $\delta_{\text{TMS}}^{\text{CDCl}_3}$ 7.37 (5 H, broad), 41.8–3.43 (2 H, multiplet), 3.40 (1 H, broad singlet), 3.00–1.00 (9 H, multiplet).

Anal. Calcd for $\text{C}_{18}\text{H}_{17}\text{OBr}$: C, 58.00; H, 6.37. Found: C, 58.16; H, 6.48.

The isolated product is undoubtedly a mixture of stereoisomers for all attempts at crystallization failed and the coupling constants recorded in the nmr spectrum are inconsistent for a single product. The infrared and the elemental analyses do, however, indicate that the product is a bromohydrin.

2-Bromo-5-methyl-5-phenylcyclohexanone (13a) and 2-Bromo-5-methyl-*trans*-5-phenylcyclohexanone (13e).—A solution of 1.8 g (18.0 mmol) of chromium trioxide in a minimal volume of water was added with stirring at 0° over 20 min to a solution of 7.23 g (27.0 mmol) of the mixture of 2-bromo-5-methyl-5-phenylcyclohexanols in 200 ml of glacial acetic acid. The reaction mixture was stirred at room temperature for 2 hr and then poured into 100 ml of water. The organic products were extracted into three 100-ml portions of ether, and the combined ethereal solutions were washed with water, with 5% bicarbonate, and with saturated brine, dried over magnesium sulfate, and concentrated. The dark oil was applied to a slurry-packed (3% ether in hexane), silica gel (650 g) chromatography column (100 × 4 cm) and eluted with 8.0 l. of 3% ether in hexane. Fractions (500 ml) 7–9 contained 2.14 g of 13a contaminated by 13e; fraction number 10 contained 0.83 g of mixture of 13a and 13e; fractions number 11 and 12 contained 1.57 g of 13e contaminated by 13a. The total quantity isolated amounted to 4.54 g (17.0 mmol, 63%). Further elution (fractions number 12 and 14) yielded traces of 2-bromo-4-methyl-*cis*-4-phenylcyclohexanone identified by comparison with an authentic sample.

It was subsequently discovered that a repeated column chromatography over silica gel of each of the above separated fractions led only to mixtures of the isomeric α -bromo ketones, whereas a rapid thin layer chromatography with 5% ether in hexane gave a chromatogram with two clearly defined spots of R_f 0.59 and 0.51. This result indicates that the bromo ketones 13a and 13b are epimerized over silica gel within periods of time necessary for efficient column chromatographic separation.

The above fraction containing the majority of 13a was dissolved in enough pentane to give a slightly cloudy solution at 0°. The mixture was scratched and alternately cooled to –15° until crystals separated. Subsequent recrystallization from pentane afforded an analytical sample: mp 64–66°; $\lambda_{\text{max}}^{\text{KBr}}$ 5.81 (s) μ ; $\delta_{\text{TMS}}^{\text{CDCl}_3}$ 7.38 (5 H), 4.29 (1 H, an apparent triplet, $J_{\text{ae}} + J_{\text{ee}} = 7.8$ Hz), 3.08 to 1.83 (6 H), 1.33 (3 H, singlet).

Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{OBr}$: C, 58.44; H, 5.66. Found: C, 58.34; H, 5.67.

All attempts to crystallize 13e failed. Fractional freezing from pentane at Dry Ice-acetone temperatures did, however, afford a purified sample. The infrared and nmr spectra indicated that this method gave at best a product contaminated by 12% with 13a (determined from the nmr integrated intensities of the peaks corresponding to the C-5 methyl groups for 13a and 13e): $\lambda_{\text{max}}^{\text{film}}$ 5.78 μ (s, overlapped with 5.81; $\delta_{\text{max}}^{\text{CDCl}_3}$ 7.26 (5 H), 4.38 (1 H, doublet of doublets, $J_{\text{ae}} + J_{\text{aa}} = 15$ Hz), 3.50–1.70 (6 H), 1.35 and 1.26 (two singlets in a 12:88 integrated ratio).

Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{OBr}$: C, 58.44; H, 5.66. Found: C, 58.62; H, 5.71.

A sample (1.1 g) of epoxide 11, prepared from bromohydrin 12 by the method described for the conversion of 5 into 6, was opened with hydrobromic acid and chloroform. Oxidation of the resulting crude bromohydrin gave 13a and 13b containing a small amount of 9 (analysis by tlc). A similar result was obtained by opening the epoxide with hydrochloric acid and oxidation to the corresponding chloro ketones.

Registry No.—5, 17278-13-6; 6, 17245-73-7; 8, 17245-74-8; 2-chloro-5,5-diphenylcyclohexanone, 17245-75-9; 3,3-diphenylcyclohexanone, 17245-76-0; 4-methyl-4-phenylcyclohexyl acetate (*cis*), 17245-77-1; 4-methyl-4-phenylcyclohexyl acetate (*trans*), 1745-81-7; 12, 17245-78-2; 13a, 17245-79-3; 13e, 17245-80-6.

Acknowledgment.—We are grateful to the National Science Foundation for support of this work (GP-4208).

(8) A. O. Abdun-Nur, Ph.D. Dissertation, Northwestern University, 1965.