added gradually to a stirred mixture of 95.5 g. (0.375 mole) of technical grade *p*-bromobenzenesulfonyl chloride in 200 ml. of dry pyridine, cooled in an ice bath. The mixture was allowed to stir overnight at room temperature. An ice–water mixture (500 ml.) was added to the reaction flask, which was then stoppered and shaken vigorously to prevent lump formation. Gradually, a fine crystalline suspension separated. The mixture was stirred for an additional 2 hr. and then poured, with rapid stirring, into a mixture of 140 ml. of concentrated hydrochloric acid and 200 g. of crushed ice. The resulting slurry was filtered, washed thoroughly with water, and dried under vacuum at room temperature to give 81.9 g. (89.6%) of product. A small sample (0.5 g.) was dissolved in tetrahydrofuran and chromatographed on a deactivated alumina column with benzene as eluent. Recrystallization from benzene gave a white, crystalline solid, m.p. 146–147° dec. (lit.[11] m.p. 145–146°).

Anal. Calcd. for $C_{17}H_{18}Br_2O_6S_2$: C, 37.67; H, 3.35. Found: C, 37.68; H, 3.29.

Racemic 2,4-Pentanediol Di-*p*-bromobenzenesulfonate.—Starting from racemic 2,4-pentanediol, the method used for the synthesis of the *meso* isomer was applied. The racemic dibrosylate was obtained in 84% yield. Treatment of an analytical sample as described above yielded white crystals, m.p. 116.5–117°.

Anal. Found: C, 37.77; H, 3.30.

Reaction Product of *meso* 2,4-Pentane Di-*p*-bromobenzenesulfonate with Potassium Thiocyanate.—The dibrosylate, 25 g., was added to a solution of 62.5 g. of dry potassium thiocyanate in 120 ml. of dimethyl sulfoxide, and the reaction mixture was stirred under nitrogen for 50 hr. at 70–75° bath temperature. The reaction mixture was diluted with water to 1 l. and divided into two 500-ml. portions. Each portion was extracted with five 100-ml. portions of 3:1 pentane–methylene chloride. The organic extracts were combined, filtered to remove some insoluble yellow material, washed with water, dried, and concentrated to give a crude yellow oil 5.89 g. (68%). Distillation at 98° (0.2 mm.) yielded a pale yellow oil, $n^{25.5}\mathrm{D}$ 1.5313.

Anal. Calcd. for $C_7H_{10}N_2S_2$: C, 45.12; H, 5.41. Found: C, 45.22; H, 5.21.

Racemic 2,4-Dithiocyanopentane.—Starting from the racemic dibrosylate, the method used for the synthesis of the *meso* isomer was applied. The crude yellow oil was obtained in 67% yield. A small sample was distilled at 104–105° (bath) (0.35 mm.) to give a water-white oil which crystallized on standing. Two recrystallizations from ether–petroleum ether (b.p. 30–60°) yielded white crystals of racemic 2,4-dithiocyanopentane, m.p. 42.5–44°.

Anal. Found: C, 45.01; H, 5.70.

Reduction of the Dithiocyanate Obtained from the *meso* Dibrosylate.—The dithiocyanate, 9 g., in 45 ml. of ether was added dropwise to a stirred slurry of 3.69 g. of lithium aluminum hydride in 225 ml. of dry ether, cooled in an ice bath. The reaction mixture was heated to reflux for 2.5 hr., cooled to 0°, treated dropwise with water, and then acidified with 6 *N* hydrochloric acid until it became clear. The ether layer was separated, and the aqueous acid layer was extracted with four 100-ml. portions of ether. The ethereal solutions were combined, washed with a saturated salt solution, dried, and distilled under vacuum to give the dithiol, 4.88 g. (74%), as a slightly yellow oil, b.p. 74.5 (12 mm.).

Anal. Calcd. for $C_5H_{12}S_2$: C, 44.06; H, 8.88; S, 47.06. Found: C, 43.96; H, 8.81; S, 46.95.

Reduction of the Dithiocyanate Obtained from the Racemic Dibrosylate.—The above method was employed giving the dithiol in 83.3% yield as a pale yellow oil, b.p. 65° (11 mm.).

Acknowledgment.—The authors wish to thank Mr. R. Pitcher, Varian Associates at Pittsburgh Airport, for carrying out the 100 Mc./sec. n.m.r. spectra. Financial assistance from the Socony-Mobil Oil Company is also gratefully acknowledged.

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[11] H. B. Henbest and B. B. Millwood, *J. Chem. Soc.*, 3575 (1960).

The Diastereomers of 1,3-Diphenyl-1,2-propanediol

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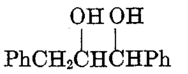
cis- and *trans*-1,3-diphenylpropene (I) isomers have been subjected to stereoselective hydroxylation reactions to determine the configurations of the diastereomeric 1,3-diphenyl-1,2-propanediols (II) resulting therefrom. *trans* benzoylation of *trans*-I, after saponification of the di-O-benzoate product, afforded *erythro*-II, m.p. 82–84°. *cis* hydroxylation of *cis*-I gave the same *erythro*-II isomer, while *cis* hydroxylation of *trans*-I produced *threo*-II, m.p. 62–64°. The diastereomeric diols obtained have been characterized both in the free state and as their di-O-benzoyl derivatives, with which they proved readily interconvertible. Stereochemical aspects of the production of the diastereomers of II by catalytic hydrogenations of phenylbenzylglyoxal and of benzalacetophenone oxide are briefly discussed.

In connection with another problem we have recently had occasion to prepare and characterize the *cis* and *trans* isomers of 1,3-diphenylpropene[2] (I). The same problem required a clean and quantitative method for degrading I into PhC- and PhCH$_2$C-fragments. As an approach to the latter objective we have undertaken a study of the hydroxylation of *cis*- and *trans*-I to 1,3-diphenyl-1,2-propanediol (II), with the idea that the latter might be quantitatively cleaved with periodic acid into benzaldehyde and phenylacetaldehyde.

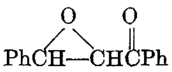
$\mathrm{PhCH_2CH{=}CHPh}$

I



$\mathrm{PhCH_2\overset{OH}{\overset{|}{C}}H\overset{OH}{\overset{|}{C}}HPh}$

II



$\mathrm{Ph\overset{O}{\overbrace{CH{-}CH}}\overset{O}{\overset{\|}{C}}Ph}$

III

We wish now to report the results of these hydroxylation experiments and the bearing they have on establishing the configurations of the diastereomers of II.

1,3-Diphenyl-1,2-propanediol has been reported in several isomeric forms. One form, m.p. 63–64°, was obtained by Levy[3] on hydrolysis of 1,3-diphenyl-1,2-dibromopropane (m.p. 110°, from 1,3-diphenylpropene[4]) with aqueous sodium carbonate. The same form, m.p. 65–66°, was obtained by Ruggli and Lutz by the Raney nickel catalyzed hydrogenation of phenylbenzylglyoxal,[5] and also by Trevoy and Brown[6] on lithium aluminum hydride reduction of benzalacetophenone oxide (III). The latter investigators confirmed the skeletal structure of their II product by periodate cleavage into benzaldehyde and phenyl-

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[1] National Science Foundation Science Teaching Faculty Fellow, 1962–1963.

[2] E. K. Raunio and W. A. Bonner, *J. Org. Chem.*, in press.

[3] J. Levy and Dvoleitzka-Gombinska, *Bull. soc. chim. France*, [4] **49**, 1765 (1931).

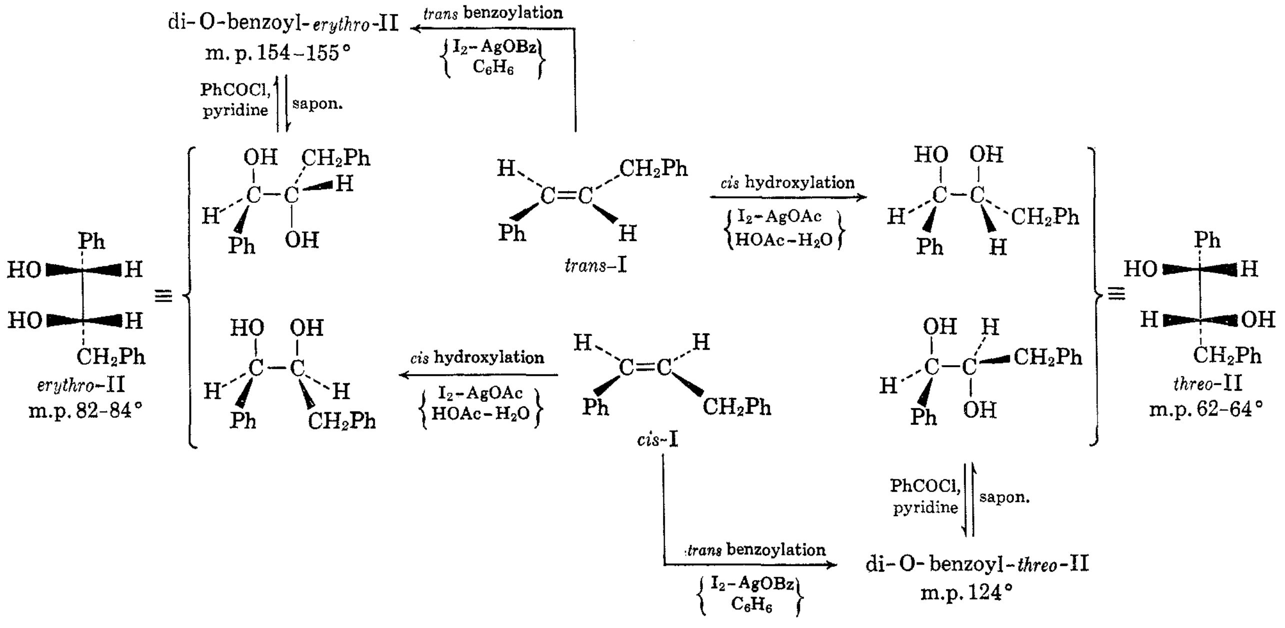
[4] W. Dieckmann and M. Kämmerer, *Ber.*, **39**, 3046 (1906).

[5] P. Ruggli and A. H. Lutz, *Helv. Chim. Acta*, **30**, 1070 (1947).

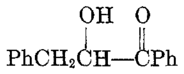
[6] L. W. Trevoy and W. G. Brown, *J. Am. Chem. Soc.*, **71**, 1675 (1949).

Chart I

Stereoselective Hydroxylations of *cis*- and *trans*-1,3-Diphenylpropene



acetaldehyde. In 1952 Herz[7] undertook the catalytic reduction of benzalacetophenone oxide (III), using platinum oxide catalyst in ethanol, and obtained an isomer of II having m.p. 84°. The structure of this compound was also confirmed by periodate cleavage into benzaldehyde and phenylacetaldehyde, and it proved convertible on benzoylation into a dibenzoate having m.p. 157°. Saponification of the latter regenerated the original II isomer, m.p. 84–84.5°. With platinum oxide in ether, III was hydrogenated to α-hydroxy-β-phenylpropiophenone (IV). When Herz


$PhCH_2CH(OH)—C(=O)Ph$

IV

performed the reduction of III with Raney nickel catalyst, an oily product was obtained. Its chromatography (alumina) and crystallization, however, provided a solid having m.p. >110°, from which fractional crystallization ultimately afforded 73 mg. of material having m.p. 128°. On the basis of its combustion analysis and "the characteristic odor of benzaldehyde" resulting on its treatment with periodic acid, Herz argued[7] that the latter 128° diol must be the diastereomer of his previously encountered isomer of II, m.p. 84°. By applying stereoselective hydroxylation reactions to the *cis*- and *trans*-1,3-diphenylpropenes we have also sought to shed light on the above ambiguity in the literature regarding the identity of the diastereomeric 1,3-diphenyl-1,2-propanediols.

The reaction of alkenes with iodine and silver acetate in moist acetic acid is believed to involve an orthoacetate intermediate and is known to result in stereoselective *cis* hydroxylation.[8,9] The action on alkenes of iodine and silver benzoate in benzene, on the other hand, proceeds in part by an S$_N$2 mechanism and results in *trans* benzoylation of the alkene.[9,10] We have now applied these and other stereoselective hydroxylation techniques to the geometrical isomers of 1,3-diphenylpropene, with the objective of elucidating the stereochemical configurations of the diastereomeric 1,3-diphenyl-1,2-propanediol products resulting therefrom. The stereochemical consequences of *cis* and *trans* hydroxylations of the two 1,3-diphenylpropenes are depicted in Chart I, which also summarizes the melting points of the products we obtained.

trans benzoylation[9,10] of *trans*-1,3-diphenylpropene (*trans*-I) with silver benzoate and iodine in benzene yielded the same dibenzoate, m.p. 154–155°, as proved obtainable by catalytic hydrogenation of III, followed by benzoylation of the crude product, after the method of Herz.[7] Hydrolysis of this ester afforded the diol, m.p. 82–84°, previously obtained by Herz.[7] As seen in Chart I, the stereochemistry of this *trans* benzoylation reaction requires that the final product be *erythro*-1,3-diphenyl-1,2-propanediol (*erythro*-II). *erythro*-II, m.p. 80–82°, was also obtained by the *cis* hydroxylation of *cis*-I using silver acetate, iodine, and moist acetic acid,[8,9] and the diol again yielded the 154–155° dibenzoate on benzoylation. When *trans*-I was subjected to a similar *cis* hydroxylation, a diol having m.p. 60–63° was obtained. Benzoylation of this diol afforded a previously undescribed di-O-benzoyl derivative, m.p. 124°, which regenerated the original diol on saponification. As Chart I indicates, the diol in question must be *threo*-1,3-diphenyl-1,2-propanediol (*threo*-II). The structure of *threo*-II, whose melting point accords closely with that reported by most previous workers,[3,5,6] was confirmed by cleavage with periodic acid, whereafter the resulting benzaldehyde and phenylacetaldehyde fragments were characterized as their 2,4-dinitrophenylhydrazones. *threo*-II, isolated as its di-O-benzoyl derivative, m.p. 124°, was also obtained by the *cis* hydroxylation of *trans*-I using potassium permanganate[11] as well as formic acid–hydrogen peroxide. The latter reagent, which may induce either *trans*

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[7] W. Herz, *J. Am. Chem. Soc.*, **74**, 2928 (1952).

[8] R. B. Woodward and F. V. Brutcher, *ibid.*, **80**, 209 (1958).

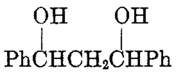
[9] E. L. Eliel, "Stereochemistry of Carbon Compounds," McGraw-Hill Book Co., New York, N. Y., 1962, pp. 358, 359.

[10] C. Prevost, *Compt. rend.*, **196**, 1129 (1933); **197**, 1661 (1933).

[11] Reference 9, p. 357.

or *cis* hydroxylation[12] here proceeded with predominately *cis* hydroxylation, since only a small quantity of *erythro*-II was obtained along with the more predominant *threo*-II isomer. When *cis*-I was subjected to conditions of *trans* benzoylation in an attempt to prepare di-O-benzoyl-*threo*-II, the reaction appeared to be less stereoselective in that the crude product had m.p. 105–115°. As shown in Chart I, knowing the geometry of the isomeric 1,3-diphenylpropenes, the above hydroxylations and interconversions establish the configurations of the diastereomers of 1,3-diphenyl-1,2-propanediol.

The question remains as to the identity of the diphenylpropanediol, m.p. 128°, suggested by Herz[7] to be one of the diastereomers of II. One possibility, of course, is that the 128° diol and *threo*-II are polymorphs. A second possibility is that our *threo*-II is stereochemically impure and is actually a mixture of the 128° diol and *erythro*-II. This we consider highly unlikely, however, not only because of the facts that our *threo*-II has been previously obtained by other investigators using widely different reactions[3,5,6] and has now been prepared by at least two highly stereoselective[8,9,11] *cis* hydroxylations, but also because of the presently observed interconvertibility of *threo*-II with its di-O-benzoyl derivative without changes in melting point. A third alternative is that Herz' 128° diol is actually 1,3-diphenyl-1,3-propanediol (V).



$$\mathrm{Ph\overset{OH}{\overset{|}{C}}HCH_2\overset{OH}{\overset{|}{C}}HPh}$$

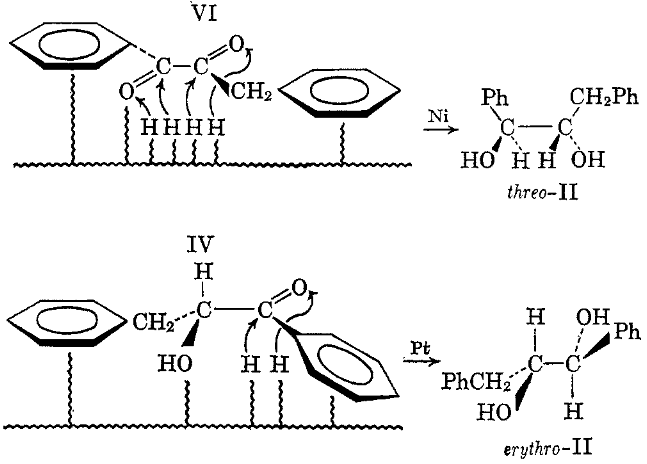
V

The racemic form of the latter is reported to have m.p. 130°,[13] 128–130°,[14] and 129°.[15] Interestingly, DL-V undergoes spontaneous resolution on recrystallization from methanol,[13] affording samples having m.p. >130°. *meso*-V, on the other hand, has been reported as having m.p. 108–109°[13] and, in presumably less pure form, 98–99°[16] and 93–97°.[17] Moreover, a diastereomer of V might well be expected to accompany II during the Raney nickel catalyzed hydrogenation of the epoxide III, since this catalyst has been observed on several occasions to open epoxides in both a "normal" and "abnormal" fashion.[18,19] We are thus inclined to believe that Herz' 128° diol, unfortunately obtained only in trivial yield, may actually have been a slightly impure sample of DL-1,3-diphenyl-1,3-propanediol, and that the "characteristic odor of benzaldehyde" which it produced on treatment with periodic acid may have been due to small amounts of residual *erythro*-II, IV,[7] or even III[18] present in it. As anticipated, a pure sample of V (m.p. 130°, prepared by the sodium borohydride reduction of dibenzoylmethane[13]) yielded no benzaldehyde odor on treatment with periodic acid.

The securement of *threo*-II, m.p. 65–66°, by the catalytic hydrogenation of phenylbenzylglyoxal[5] (VI), and of *erythro*-II, m.p. 84°, by the catalytic hydrogenation of benzalacetophenone oxide[7] (III), tempts us to speculate how these substrates might be adsorbed on the catalyst surface prior to reduction, assuming, by analogy with the usual pattern of alkene hydrogenation,[20] that reduction of their carbonyl group(s) occurs by *cis* addition of hydrogen. If VI is adsorbed on the catalyst surface in such a way as to orient the two polar carbonyl functions and the bulky phenyl and benzyl groups as far apart as possible, *cis* hydrogenation of the carbonyl groups would lead to *threo*-II, as seen in Chart II. If similar group-orientation criteria are applied to the substituents on α-hydroxy-β-phenylpropiophenone (IV), which seems a logical intermediate in the hydrogenation of III,[7] *cis* hydrogenation of the carbonyl function should afford *erythro*-II (Chart II).

CHART II

STEREOSELECTIVE CATALYTIC HYDROGENATIONS



Experimental Section

***trans* Benzoylation of *trans*-1,3-Diphenylpropene.**—A solution of *trans*-1,3-diphenylpropene (0.97 g., 0.005 mole) in benzene (25 ml.) was shaken with iodine (1.27 g., 0.01 mole) and silver benzoate (2.60 g., 0.0113 mole) until the iodine color had disappeared, whereupon the resulting suspension was stirred overnight and filtered. The filtrate was stripped of benzene under vacuum, the residue was dissolved in ether, and the solution was refiltered and again stripped of solvent. The residue was dissolved in ethanol (15 ml.) and chilled for several hours, affording 0.90 g. of crude product, m.p. 125–145°. Successive recrystallization from benzene–hexane and benzene–ethanol afforded a sample of di-O-benzoyl-*erythro*-1,3-diphenyl-1,2-propanediol having m.p. 154–155° and giving no mixture melting point depression with a sample prepared *via* the catalytic hydrogenation of benzalacetophenone oxide[21,22] according to the procedure of Herz.[7]

Saponification of Di-O-benzoyl-*erythro*-1,3-diphenyl-1,2-propanediol.—The above *erythro*-dibenzoate (0.55 g.) was heated in refluxing ethanol (5 ml.) containing potassium hydroxide (0.5 g.) for 30 min., whereupon the mixture was treated with water (25 ml.) and extracted with ether. Solvent removal yielded 0.29 g. of colorless oil which rapidly crystallized, m.p. 75–84°. Recrystallization from benzene–ligroin gave a sample of *erythro*-1,3-diphenyl-1,2-propanediol having m.p. 82–84°.

***cis* Hydroxylations of *trans*-1,3-Diphenylpropene.**—A suspension consisting of silver acetate (1.84 g., 0.011 mole), *trans*-1,3-diphenylpropene (0.97 g., 0.005 mole), and iodine (1.27 g., 0.01 mole) in acetic acid (33 ml.) was stirred vigorously at room temperature for 1.5 hr., then treated with water (0.2 ml.) in acetic acid (5 ml.), and heated under reflux for 1 hr., then cooled,

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[12] Reference 9, pp. 356, 102.

[13] J. Dale, *J. Chem. Soc.*, 911 (1961).

[14] H. E. Zimmerman and J. English, Jr., *J. Am. Chem. Soc.*, **76**, 2285 (1954).

[15] W. Perkin, Jr., and J. Stenhouse, *J. Chem. Soc.*, 1010 (1891).

[16] J. English, Jr., and A. D. Bliss, *J. Am. Chem. Soc.*, **78**, 4057 (1956).

[17] J. Sprague and H. Adkins, *ibid.*, **56**, 2669 (1934).

[18] R. E. Parker and N. S. Isaacs, *Chem. Rev.*, **59**, 743 (1959).

[19] M. S. Newman, G. Underwood, and M. W. Renoll, *J. Am. Chem. Soc.*, **71**, 3362 (1949).

[20] Reference 9, p. 350 ff.

[21] E. Weitz and A. Schaeffer, *Ber.*, **54**, 2329 (1921).

[22] E. P. Kohler, N. R. Richtmyer, and W. F. Hester, *J. Am. Chem. Soc.*, **53**, 205 (1931).

and filtered. Volatiles were removed from the filtrate at 100° under vacuum, and the residue was dissolved in cyclohexane. The solution was filtered and stripped of solvent to yield 1.36 g. of amber oil, which was dissolved in anhydrous ether (20 ml.) and stirred while adding lithium aluminum hydride (0.5 g.) in small portions. After being stirred for 10 min., the excess reagent was destroyed with dilute aqueous sodium hydroxide and the crude product, 1.09 g. of viscous amber oil, was isolated in the usual way. The oil was treated with hexane–ether, affording 0.83 g. of *threo*-1,3-diphenyl-1,2-propanediol, m.p. 60–63°. Benzoylation of this product, as described below, yielded di-O-benzoyl-*threo*-1,3-diphenyl-1,2-propanediol, m.p. 124°.

In another experiment the *trans* olefin (1.0 g.) in 98% formic acid (3 ml.) containing 30% hydrogen peroxide (0.75 ml.) was stirred for 1 hr., then poured onto 50 g. of ice containing water (10 ml.) and sodium hydroxide (4.5 g.). The mixture was extracted with ether and the extract was processed as usual and evaporated to give 1.20 g. of colorless, oily formate ester. The latter was hydrolyzed with refluxing aqueous (1 ml.) ethanolic (10 ml.) potassium hydroxide (0.5 g.) during 0.5 hr., and 1.06 g. of crude product was isolated as usual by ether extraction. It showed strong OH absorption at 3500 cm.$^{-1}$ and two spots in thin layer chromatography. The entire sample was heated briefly at 100° with pyridine (5 ml.) containing benzoyl chloride (1.5 ml.), whereupon the volatiles were stripped under vacuum and the residue was shaken with ether–water. The ether layer was washed thoroughly with dilute acid, dilute base, and water, then was evaporated to yield 2.37 g. of crude product which was dissolved in ethanol (20 ml.) and chilled, resulting in 1.53 g. of crude di-O-benzoyl-*threo*-1,3-diphenyl-1,2-propanediol, m.p. 118–124°. Several recrystallizations from ethanol afforded the pure product, m.p. 124°.

Anal. Calcd. for $C_{29}H_{24}O_4$: C, 79.79; H, 5.54. Found: C, 79.66; H, 5.61.

On standing in the refrigerator the filtrate from the initial crystallization above deposited 0.24 g. of crude *threo*-dibenzoate which, on recrystallizing repeatedly from ethanol, showed m.p. 154.5–156°.

In a final *cis* hydroxylation, a solution of potassium permanganate (1.1 g.) in water (30 ml.) was stirred into a solution of *trans*-1,3-diphenylpropene (1.94 g.) in ethanol (75 ml.) at such a rate as to maintain the temperature of the chilled ethanol solution below 6°. The mixture was then heated to coagulate the MnO_2, filtered, treated with 30% hydrogen peroxide (2 ml.), and warmed on the steam bath, whereupon the benzaldehyde odor disappeared. The solution was concentrated to about 30 ml. under vacuum, cooled, and extracted with ether. Customary processing of the extract yielded 1.5 g. of yellow liquid which smelled of starting alkene. The product was accordingly dissolved in 5 ml. of 1:10 benzene–petroleum ether and placed on a $^5/_8 \times 12$ in. column of silica gel G. Elution of the alkene forerun (discard) was accomplished with 75 ml. of 1:1 benzene–petroleum ether, and the glycol product was eluted with 50 ml. of ether. Removal of the solvent left 0.60 g. of viscous amber oil whose infrared spectrum was substantially identical with that of the above *threo*-1,3-diphenyl-1,2-propanediol. Benzoylation of this product with benzoyl chloride in pyridine yielded a sample of the *threo*-dibenzoate having m.p. 124° after two recrystallizations from ethanol.

Saponification of Di-O-benzoyl-*threo*-1,3-diphenyl-1,2-propanediol.—Three grams of the above *threo*-dibenzoate, m.p. 124°, was heated for 0.5 hr. in a refluxing mixture of potassium hydroxide (2 g.), water (2 ml.), and ethanol (15 ml.). Processing as described above afforded an oily product which, on dissolving in ether (4 ml.), treating with petroleum ether (25 ml.), and chilling, crystallized as long, thin needles of *threo*-1,3-diphenyl-1,2-propanediol, 1.2 g., m.p. 60–62°.

Periodate Cleavage of *threo*-1,3-Diphenyl-1,2-propanediol.—A solution of *threo*-1,3-diphenyl-1,2-propanediol (m.p. 62–64°, 0.50 g., 0.0022 mole) in ethanol (5 ml.) was added to 20 ml. of 0.14 *M* H_5IO_6 (containing 0.0028 mole H_5IO_6). The odor of benzaldehyde was immediately apparent. After 10 min. the solution was diluted with water (100 ml.) and extracted with ether. The extract was washed and evaporated, and the residue was treated with a hot solution of 2,4-dinitrophenylhydrazine (1 g.) in ethanol (40 ml.) containing concentrated hydrochloric acid (0.5 ml.). The solution was boiled for a few minutes and filtered hot, and the collected solid was rinsed with ethanol, m.p. 225–230°, 0.31 g. It was recrystallized twice from ethanol, whereupon it showed m.p. 232–233° and no mixture melting point depression with authentic benzaldehyde 2,4-dinitrophenylhydrazone. The above filtrate was chilled overnight, depositing 0.86 g. of a mixture of orange platelets and red, granular solid. The mixture was boiled with benzene (25 ml.), dissolving the platelets and leaving the dark red solid (0.29 g. of 2,4-dinitrophenylhydrazine, by melting point and mixture melting point). Concentration of the benzene filtrate to 5 ml., treatment with hexane to incipient cloudiness, and cooling yielded 0.40 g. of orange solid, m.p. 117–120°. Twice recrystallized from benzene–hexane, the solid had m.p. 118–120° and showed no mixture melting point depression with authentic phenylacetaldehyde 2,4-dinitrophenylhydrazone (lit.[23] m.p. 121°).

***cis* Hydroxylations of *cis*-1,3-Diphenylpropene.**—The above procedure employing silver acetate and iodine, followed by treatment with moist acetic acid, was utilized to perform the *cis* hydroxylation of 0.97 g. of *cis*-1,3-diphenylpropene. The crude product, 1.14 g. of thick oil, was treated as before with lithium aluminum hydride, ultimately affording 0.30 g. of *erythro*-1,3-diphenyl-1,2-propanediol, m.p. 80–82°. Mother liquors from the latter crystallization were stripped of solvent and benzoylated in the usual way, yielding 0.31 g. of crude *erythro*-dibenzoate (m.p. 145–148°), which showed m.p. 153–156° after two recrystallizations from ethanol.

We also have attempted to perform the *cis* hydroxylation of *cis*-1,3-diphenylpropene by the action of hydrogen peroxide in formic acid, as described above for the *trans* isomer. In this case the crude product was benzoylated to give a dibenzoate mixture (m.p. 115–130°), from which only a small quantity of the desired *erythro*-dibenzoate (m.p. 152–156°) was recovered by fractional crystallization.

An attempt was also made to perform the *trans* hydroxylation of *cis*-1,3-diphenylpropene, using silver benzoate and iodine in benzene as described above. Here the desired *threo*-dibenzoate also proved rather impure, having m.p. 105–115° after recrystallization from ethanol.

Di-O-benzoyl-*erythro*-1,3-diphenyl-1,2-propanediol by the Catalytic Hydrogenation of Benzalacetophenone Oxide.—Two grams of benzalacetophenone oxide was catalytically reduced using PtO_2 catalyst (50 mg.) in ethanol solvent (30 ml.), after the procedure of Herz.[7] The crude product was benzoylated in the usual way, yielding a crude sample of the *erythro*-dibenzoate having m.p. 150–153.5°. Recrystallization from benzene–hexane gave a product having m.p. 152–155° which, on saponification as described above, yielded a sample of *erythro*-1,3-diphenyl-1,2-propanediol of m.p. 82–84°.

Acknowledgment.—W. A. B. gratefully acknowledges support of a portion of this research by the National Institutes of Health (Grant GM-06232-05).

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(23) N. R. Campbell, *Analyst*, **61**, 392 (1936).