Such impure samples of *cis*-stilbene were dissolved in hexane (20 g. in 200 ml.) and washed with four 50 ml. portions of 10% hydrochloric acid. The solution was then washed with 50 ml. of 10% sodium carbonate, and finally with 50 ml. of water. The resulting solution was dried over anhydrous sodium sulfate, cooled to 0°, and filtered to remove the drying agent and most of the *trans*-stilbene present as a contaminent. Fractional distillation yielded *cis*-stilbene, b.p. 135–136° (10 mm.), which appeared to be relatively pure on the basis of the ultraviolet absorption spectrum[13] and gas chromatography.

A modification of the synthetic procedure[12] for *cis*-stilbene was also carried out. A mixture of 46.0 g. (0.205 mol.) of α-phenylcinnamic acid, 280 ml. of quinoline, and 4 g. of copper chromite was heated at 210–220° for 1.25 hr. The mixture was then filtered and distilled at 10 mm. as quickly as possible. The fraction distilling around 104° was recovered quinoline. The crude product, b.p. 125–140° was dissolved in 200 ml. of hexane and then was washed and purified as described above. A yield of 21 g. (59%) of *cis*-stilbene, b.p. 135–136° (10 mm.), was obtained.

Other materials. The compounds used in the reactions with bromine chloride were either commercially available or were synthesized by methods given in the Organic Syntheses series. Cyclohexene was purified by fractional distillation, b.p. 81.0–81.5° (739 mm.). Chloroform, and methylene chloride were reagent grade. They were used without further purification.

Addition reactions of bromine-chloride. The addition reactions were carried out in a 1 l., three necked flask fitted by means of spherical joints with: (1) a combination dropping funnel, and gas delivery tube extending close to the bottom of the flask, (2) a stirrer, and (3) a cold-finger condenser which was kept filled during the runs with Dry Ice in a mixture of chloroform and carbon tetrachloride. The reaction flask was kept in an ice-water bath. In each run the desired amount of chlorine was liquefied in a trap cooled in a Dry Ice bath and then weighed. The liquid chlorine was then allowed to distill slowly from the trap by way of the delivery tube into the reaction flask containing the solvent (500 ml. per 35.5 g. of chlorine). An amount of liquid bromine equivalent to the amount of chlorine was then added by way of the dropping funnel and washed in with additional solvent. The bromine-chlorine mixture was illuminated with an ultraviolet lamp because the equilibrium between bromine, chlorine, and bromine chloride is set up more rapidly under these conditions.[14] Actually on the basis of the results with ethylene in Table I this illumination may not be strictly necessary. In the dark an equivalent amount of the unsaturated compound was added in solution or as a gas in the case of ethylene. The reaction mixture was allowed to stand in the dark overnight. The solvent was distilled at 30 to 40 mm. pressure. The residue was then crystallized from a suitable solvent or distilled. For most crystallizations ethanol, carbon tetrachloride, or hexane was used. The results of the experiments are summarized in Table I. The identity of most of the bromochlorides was established by comparison with samples obtained in the earlier investigation[1] by the addition of the elements of bromine chloride from *N*-bromoacetamide and hydrochloric acid. The properties of the bromochloride of cyclohexene compared satisfactorily with those reported[15] for *trans*-1-bromo-2-chlorocyclohexane. The bromochloride of diphenylacetylene checked with the α-bromo-α'-chlorostilbene, m.p. 173–174°, reported by Sudborough.[16] The chloride products listed in Table I were identified by comparison with samples obtained in another investigation.[11]

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(13) R. E. Buckles, *J. Am. Chem. Soc.*, **77**, 1040 (1955).

(14) H. G. Vesper and G. K. Rollefson, *J. Am. Chem. Soc.*, **56**, 620 (1934).

(15) M. Mousseron, R. Granger, and J. Valette, *Bull. soc. chim. France*, 244 (1946); M. Mousseron, F. Winternitz, and R. Jacquier, *Bull. soc. chim. France*, 81 (1947).

(16) J. J. Sudborough, *J. Chem. Soc.*, **71**, 218 (1897).

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA AT LOS ANGELES]

Preparation of Homobenzyl and Homoallyl Alcohols by the Hydroboration Method[1,2]

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H. C. Brown's elegant hydroboration-oxidation method for hydrating olefins provides a convenient route to homoallyl and homobenzyl alcohols. Δ^3-Cyclopentenol may be prepared from cyclopentadiene, while the pure diastereomeric *threo*- and *erythro*-3-*p*-anisyl-2-butanols arise in good yield from the *cis*- and *trans*-2-*p*-anisyl-2-butenes, respectively. The stereochemistry of the overall hydration is clearly *cis*.

The elegant method of H. C. Brown and his coworkers[3,4] for accomplishing the hydration of olefins by successive hydroboration and oxidation appeared to offer a convenient route to certain homobenzyl[5] and homoallyl[6] alcohols of interest in various studies in these laboratories. The alcohols desired were Δ^3-cyclopentenol (II) and the diastereomeric *threo*- and *erythro*-3-*p*-anisyl-2-butanols (XII and XIII). Therefore, hydration by means of hy-

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(1) Research supported in part by the National Science Foundation.

(2) This research was supported in part by a grant from The Petroleum Research Fund administered by the American Chemical Society. Grateful acknowledgment is hereby made to the donors of this fund.

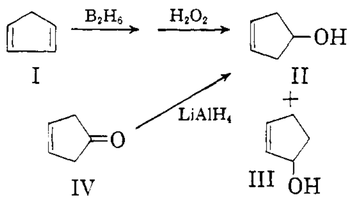
(3) H. C. Brown and G. Zweifel, *J. Am. Chem. Soc.*, **81**, 247 (1959).

(4) H. C. Brown and B. C. Subba Rao, *J. Org. Chem.*, **22**, 1136 (1957).

(5) *e.g.*, (a) S. Winstein, M. Brown, K. C. Schreiber, and A. H. Schlesinger, *J. Am. Chem. Soc.*, **74**, 1140 (1952); (b) S. Winstein and G. C. Robinson, *J. Am. Chem. Soc.*, **80**, 169 (1958).

(6) *e.g.*, (a) S. Winstein, H. M. Walborsky, and K. Schreiber, *J. Am. Chem. Soc.*, **72**, 5795 (1950); (b) S. Winstein, M. Shatavsky, and R. B. Woodward, *J. Am. Chem. Soc.*, **77**, 4183 (1955); (c) S. Winstein and M. Shatavsky, *J. Am. Chem. Soc.*, **78**, 592 (1956).

droboration and subsequent oxidation was studied with cyclopentadiene (I) and the *cis*- and *trans*-2-anisyl-2-butenes (IX and XI). For comparison with the latter, anethole was also investigated. The results are presented in the present manuscript.

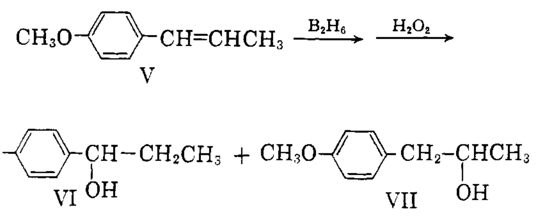


Cyclopentadiene. Hydroboration of cyclopentadiene was carried out by the general method of Brown and Zweifel[3] using a considerable excess of cyclopentadiene in order to minimize glycol formation. Under the conditions employed, the consumption of diborane was incomplete and some dicyclopentadiene was formed from the monomer. A 30% yield of Δ^3-cyclopentenol (II) was obtained, no systematic effort being made to maximize the yield of alcohol. The infrared spectrum of the alcohol product was identical with that of an authentic sample of Δ^3-cyclopentenol (II), and phenylurethane and toluenesulfonate derivatives of the product of hydroboration and oxidation agreed in melting point and mixed melting point with derivatives prepared from authentic alcohol II.

A sample of authentic homoallylic alcohol II was prepared by lithium aluminum hydride reduction of the corresponding ketone IV, which in turn was prepared in poor yield by the method of Alder and Flock[7] from pyrolysis of dicyclopentadienol-3.

The alcohol product from hydroboration of cyclopentadiene proved to be 94% pure by vapor phase chromatographic analysis. While the alcohol contained less than 1% of allylic alcohol III, there was present 3–4% of a material which had the same vapor phase chromatographic behavior as cyclopentanol and which may possibly have arisen from reduction of cyclopentenyl alcohol by diborane. Further, vapor phase chromatographic analysis indicated contamination by 1–2% of an unidentified component. Even on the basis of a 30% yield of 94% pure homoallylic alcohol II, the hydroboration-oxidation method is in our opinion the method of choice for the preparation of a sizable quantity of this homoallylic alcohol.

Anethole. Conversion of anethole (V) to alcohol product by the procedure of Brown and Zweifel[3] gave rise to a 66% yield of alcohol which was separated from unreacted olefin by chromatography. Infrared analysis of the alcohol product indicated it was a mixture of 75% of the benzylic alcohol VI and 25% of the homobenzyl alcohol VII. The low yield of 1-*p*-anisyl-2-propanol(VII) was further substantiated by the low yield (*ca.* 18%) of 1-*p*-anisyl-2-propyl *p*-toluenesulfonate obtained from reaction of the alcohol mixture with *p*-toluenesulfonyl chloride in pyridine under the usual conditions.[5a]



The 2-p-anisylbutenes. A mixture of isomeric 2-*p*-anisylbutenes was produced by the acetic anhydride dehydration of 2-*p*-anisyl-2-butanol (VIII). On the basis of gas phase chromatography, the mixture of olefins contained three components roughly in the ratio 74:14:12. By careful fractionation and refractionation using an efficient fractionating column, it was possible to obtain pure samples of the low and high boiling components and an essentially pure sample of a product with an intermediate boiling point (Table I). With a trace of *p*-toluenesulfonic acid added to the olefin mixture, only the pure low boiling olefin is obtained from slow distillation of the continually equilibrating mixture.

As indicated in Table I, the *trans*-2-anisyl-2-butene designation XI is assigned to the lowest boiling isomer and the *cis*-2-anisyl-2-butene designation IX to the highest boiling isomer. These assignments may be made on the basis of the physical properties of the isomers, especially the ultraviolet absorption spectra, the situation here being completely analogous with that in the case of the isomeric 2-phenyl-2-butenes.[8] The gross structure of the olefins is confirmed by the hydroboration to 3-anisyl-2-butanols described below. Although the structure of the intermediate boiling isomer (Table I) was not proved as definitely as that of the other two, it is almost certainly 2-*p*-anisyl-1-butene (X) on the basis of its ultraviolet absorption spectrum, refractive index and boiling point, and analogy with the isomeric 2-phenylbutenes.[8]

TABLE I

SUMMARY OF THE PROPERTIES OF THE 2-*p*-ANISYLBUTENES

Olefin	B.P., °C. (10 mm.)	n_D^{25}	λ_{max} mμ	ε	Configuration of Derived Alcohol
cis-2-*p*-anisyl-2-butene (IX)	117.5	1.5495	252	14,540	*threo*
2-*p*-anisyl-1-butene (X)	107	1.5400	252	13,780	
trans-2-*p*-anisyl-2-butene (XI)	103	1.5316	243	10,890	*erythro*

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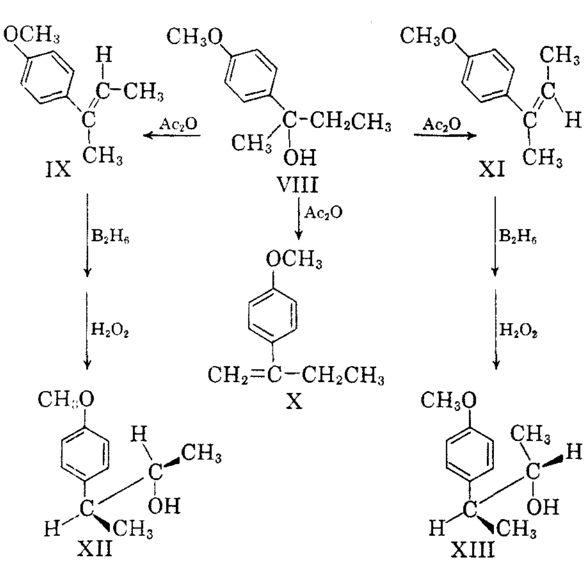
[7] K. Alder and F. H. Flock, *Ber.*, **89**, 1732 (1956).

[8] D. J. Cram, *J. Am. Chem. Soc.*, **71**, 3883 (1949); **74**, 2137 (1952).

Hydration of the *trans*-olefin XI by the hydroboration method of Brown and Zweifel[3] and chromatographic separation of the alcohol products from residual olefin gave a 72% yield of solid alcohol. After one recrystallization this was pure *erythro*-3-*p*-anisyl-2-butanol (XIII). A similar stereospecific result was obtained from the *cis*-olefin IX. Reaction of the crude alcohol product with phthalic anhydride in pyridine resulted in a 77% yield of crude acid phthalate. One recrystallization of the latter gave rise to pure *threo*-3-*p*-anisyl-2-butyl acid phthalate. A comparison of the infrared spectra of the crude alcohol products from the *cis*- and *trans*-2-*p*-anisyl-2-butenes (IX and XI) with the spectrum of authentic 2-*p*-anisyl-2-butanol (VIII) indicated that the formation of the latter is essentially negligible for both olefins.



The present method is certainly convenient for preparation of the pure diastereomeric 3-*p*-anisyl-2-butanols (XII and XIII), especially for the *threo*-isomer XII. This is a distinct improvement over the conventional method formerly employed.[5b]

Stereochemistry and orientation. As regards the stereochemistry of the over-all hydration of the double bond by hydroboration-oxidation, the present results with the *cis*- and *trans*-2-anisyl-2-butenes (IX and XI) illustrate further the stereospecific *cis*-addition already demonstrated with 1-methylcyclohexene, 1-methylcyclopentene, 1,2-dimethylcyclohexene, and 1,2-dimethylcyclopentene,[3] as well as cholesterol.[9]

As regards orientation in the over-all hydration of the double bond, that observed in the case of cyclopentadiene with very predominant formation of the homoallylic alcohol II is what we anticipated on the basis of the reported results with unsymmetrical olefins[3] and styrene.[4] This orientation makes the hydroboration-oxidation method applied to conjugated dienes a promising one for preparation of homoallylic alcohols.[10] The orientation observed in the over-all hydration of the *cis*- and *trans*-2-*p*-anisyl-2-butenes (IX and XI) was also the one anticipated on the basis of the results reported with unsymmetrical olefin[3]. How important the 1-methyl group is in promoting this orientation for the 2-*p*-anisyl-2-butenes (IX and XI) is obvious from the contrasting results obtained with anethole.[5] In this case, the benzylic alcohol VI predominates over the homobenzyl alcohol VII by a factor of *ca.* 3. More extended discussion of this point is probably best deferred until information is available on the orientation of the over-all hydration in more cases.

EXPERIMENTAL

Δ^3-Cyclopentenol. The 0.067 mol. of diborane from 3.8 g. (0.1 mol.) of sodium borohydride in 100 ml. of diglyme (diethyleneglycol dimethyl ether) and 21 g. (0.15 mol.) of boron trifluoride etherate in 35 ml. diglyme was passed into 45 g. (0.68 mol.) of freshly distilled cyclopentadiene in 150 ml. of anhydrous ether at 0° over a period of 30 min., while a slow stream of nitrogen was maintained. A precipitate frequently formed which clogged the fritted glass bubbler and made the complete addition of diborane impossible. Similar results were obtained in tetrahydrofuran.

After the reaction mixture stood at room temperature for 30 min., the excess cyclopentadiene and solvent were removed under vacuum, a viscous oil remaining (65% calculated as tri-Δ^3-cyclopentenyl boron). The oil was hydrolyzed in ether with 3*M* sodium hydroxide, followed by the slow addition of 30% hydrogen peroxide (exothermic reaction). Similar results were obtained by adding small pieces of ice to the original reaction mixture and then hydrolyzing as above.

The organic layer was separated and the aqueous solution continuously extracted with ether. After the ether extract was dried over magnesium sulfate, the ether was removed, and an alcohol fraction boiling in the range 60–70° (36 mm.) was collected. A higher boiling residue remained. The cyclopentenol fraction was freed from dicyclopentadiene, the major impurity, by chromatography on alumina. Distillation through a Podbielniak column gave rise to material, b.p. 67–68° (36 mm.), n_D^{25} 1.4673, in 30% yield. The infrared spectrum of this material was identical with that of an authentic specimen of Δ^3-cyclopentenol described below. The alcohol product also gave a phenylurethan, m.p. 140.4–140.8°, mixed m.p. with authentic material 140.4–141.0°, and a toluenesulfonate (80% yield), m.p. 53.4–54.2°, mixed m.p. with authentic material 53.2–54.2°.

Vapor phase chromatography on carbowax indicated 94% purity of the Δ^3-cyclopentenol, with 3–4% of cyclopentanol and 1–2% of an unidentified component. Vapor phase chromatography on didecyl phthalate indicated a maximum of 1% Δ^2-cyclopentenol and no trace of bis-Δ^2-cyclopentenyl ether.

Authentic Δ^3-cyclopentenol was prepared in 84% yield

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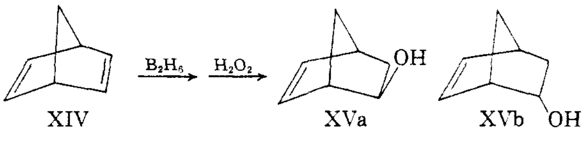
[9] W. J. Wechter, *Chem. & Ind.* (*London*), 294 (1959).

[10] The homoallylic alcohol XV from the unconjugated diene, bicycloheptadiene (XIV), may be prepared by the hydroboration-oxidation method (E. Vogelfanger, unpublished work). The 5-norbornenol XV obtained from the reaction at room temperature is an 85:15 *exo:endo* mixture.



by lithium aluminum hydride reduction of Δ^3-cyclopentenone, the latter ketone being obtained in poor yield by pyrolysis of dicyclopentadienol-3 according to the method of Alder and Flock.[7] The Δ^3-cyclopentenol product displayed b.p. 69–70° (40 mm.) and n_D^{25} 1.4688.

Anal. Calcd. for C_5H_8O: C, 71.39; H, 9.57. Found: C, 71.18; H, 9.58.

The Δ^3-cyclopentenol gave rise to a phenylurethan, m.p. 141.6–142.1°.

Anal. Calcd. for $C_{12}H_{13}NO_2$: C, 70.92; H, 6.45; N, 6.89. Found: C, 70.81; H, 6.41; N, 6.71.

Hydrogenation of the alcohol over platinum oxide in ethanol gave rise to cyclopentanol, whose *N*-phenylcarbamate, m.p. 135.5–136.0°, showed no melting point depression on admixture with authentic material, m.p. 136.4–136.5°.

The Δ^3-cyclopentenol gave rise to a *p*-toluenesulfonate, m.p. 53.4–54.6°, in 90% yield by the usual preparative method from alcohol and toluenesulfonyl chloride in pyridine.

Anal. Calcd. for $C_{12}H_{14}O_3S$: C, 60.48; H, 5.92; S, 13.14. Found: C, 60.63; H, 5.79; S, 12.86.

Δ^2-Cyclopentenol. This material was prepared by addition of hydrogen chloride to cyclopentadiene and hydrolysis of the 1-chloro-2-cyclopentene with aqueous sodium bicarbonate.[7] The alcohol yielded a phenylurethan, m.p. 128.5–129.5°, m.p. depressed by addition of the phenylurethan of Δ^3-cyclopentenol.

1-p-Anisyl-2-propanol. This material was prepared as described previously[5a]; b.p. 119.5° (4 mm.), n_D^{25} 1.5262.

1-p-Anisyl-1-propanol. To the Grignard reagent prepared from 75 g. (0.4 mol.) of *p*-bromoanisole and 9.6 g. (0.4 gram atom) of magnesium in 250 ml. of ether was added, as rapidly as possible, 23.3 g. (0.4 mol.) of freshly distilled propanal in 200 ml. of ether. After an additional stirring time of 30 min., the reaction mixture was decomposed with a saturated ammonium chloride solution. Fractionation of the crude product yielded 52 g. (78%) of 1-*p*-anisyl-1-propanol, b.p. 120.5–121° (4 mm.), n_D^{25} 1.5257.

Anal. Calcd. for $C_{10}H_{14}O_2$: C, 72.26; H, 8.50. Found: C, 72.03; H, 8.44.

Hydroboration of anethole. A 14.8 g. (0.1 mol.) sample of freshly distilled anethole was reacted with diborane and the organoborane converted to alcohol according to the procedure of Brown and Zweifel.[3,11] From this reaction was recovered 16 g. of an alcohol-olefin mixture which on chromatographic separation (alumina) yielded 11.0 g. (66%) of alcohol and 3.4 g. of anethole. The alcohol fraction was distilled; b.p. 119.5–121° (4 mm.), n_D^{25} 1.5262.

Infrared analysis[12] of the alcohol product, using characteristic bands at 900 and 752 cm.$^{-1}$, showed a mixture of 75 ± 2% 1-*p*-anisyl-1-propanol and 25 ± 2% 1-*p*-anisyl-2-propanol, the figures being average values from two separate hydroboration reactions. A known mixture of 78.8% 1-*p*-anisyl-1-propanol and 21.2% 1-*p*-anisyl-2-propanol analyzed 78% and 22%, respectively. This latter spectrum was essentially superimposable with the spectrum of the alcohol product from the above described hydroboration.

A 5.0-g. sample of the hydroboration alcohol product in 10 ml. of pyridine was allowed to react with 5.8 g. of *p*-toluenesulfonyl chloride in 10 ml. of pyridine allowing the reaction mixture to stand overnight at 0°. From this reaction was obtained 1.8 g. (18%) of 1-*p*-anisyl-2-propyl-*p*-toluenesulfonate, m.p. 79–80° after one recrystallization from ether and pentane, mixed m.p. 79–80° with an authentic sample (m.p. 80°). An 80% yield from pure 1-*p*-anisyl-2-propanol has been reported.[5a]

2-p-Anisyl-2-butanol. The Grignard reagent from 364.1 g. (2 mol.) of *p*-bromoanisole and 48.6 g. (2 gram atoms) of magnesium, prepared in *ca.* 800 ml. of ether, was reacted in the usual manner with 144.2 g. (2 mol.) of 2-butanone dissolved in 400 ml. of ether. The reaction complex was decomposed with saturated ammonium chloride solution. On distillation, the crude product yielded 250 g. (70%) of 2-*p*-anisyl-2-butanol, b.p. 98.5–99° (0.5 mm.), n_D^{25} 1.5260.

Anal. Calcd. for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.19; H, 9.07.

Cis and trans-2-p-anisyl-2-butenes. A solution of 187 g. (1.04 mol.) of 2-*p*-anisyl-2-butanol in 450 ml. of freshly distilled acetic anhydride was heated under reflux (b.p. 136–124°) for a period of 4 hr. Following this, the acetic acid and acetic anhydride were removed by distillation and the concentrate distilled through a Vigreux column; weight 150 g. (88%), b.p. 105–118° (10 mm.). Analysis of this material by vapor phase chromatography (Perkin-Elmer Corp. Vapor Fractometer, Model 154-B, fitted with a 2-m. Perkin-Elmer Column C) indicated the presence of three components in the ratio 74:14:12. This material was submitted to fractionation on a center rod column (75 theoretical plates at the highest operating efficiency) at 10 mm. of pressure. Two large fractions were collected: (1) 36.6 g., b.p. 109–116°; (2) 112.3 g., b.p. 116–117.5°. Fraction (2) was refractionated to give 16.9 g. of material, b.p. 108–115° (10 mm.); 23.3 g. of material, b.p. 115–117.5° (10 mm.); and 71.0 g. of pure *cis*-isomer, b.p. 117.5° (10 mm.) (Table I).

Anal. Calcd. for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found for the *cis*-olefin: C, 81.24; H, 8.81.

All of the fractions boiling below 116° were again refractionated to give 6.1 g. of pure *trans*-isomer, b.p. 103° (10 mm.) (Table I), and 8.5 g. of another olefin, b.p. 107° (10 mm.) (Table I), presumably 2-*p*-anisyl-1-butene.

Anal. Calcd. for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found for the *trans*-olefin: C, 81.26; H, 8.62.

A combined fraction (32 g.) with b.p. 116.5–117.5° (10 mm.) was mixed with 0.10 g. of *p*-toluenesulfonic acid and the mixture submitted to slow fractionation at 10 mm. to produce 26.4 g. of pure *trans*-isomer, b.p. 103° (10 mm.), n_D^{25} 1.5325.

Hydroboration of trans-2-p-anisyl-2-butene. A 16.2 g. (0.1 mol.) sample of pure *trans*-2-*p*-anisyl-2-butene was hydroborated and converted to alcohol exactly as described for anethole. From this reaction was recovered 17.5 g. of an alcohol-olefin mixture which on chromatographic separation (alumina) gave 13.0 g. (72%) of a solid alcohol fraction and 4.2 g. of *trans*-olefin (unchanged from starting material according to the infrared spectrum). One recrystallization from pentane gave 10.6 g. of pure *erythro*-3-*p*-anisyl-2-butanol, m.p. 58.5–59.5°, mixed m.p. 59.5–60° with an authentic sample (m.p. 60°).[5b]

A comparison of the infrared spectrum of the crude alcohol product (after chromatography) with the spectrum of pure 2-*p*-anisyl-2-butanol indicated that no more than a very small amount of the latter could have been formed.

Hydroboration of cis-2-p-anisyl-2-butene. A pure sample of *cis*-2-*p*-anisyl-2-butene (28.0 g., 0.17 mol.) was reacted with diborane and converted to the alcohol as described for anethole. A 29.0-g. sample of alcohol (with very little olefin according to the infrared spectrum) was recovered and reacted with 26.2 g. of phthalic anhydride in 85 ml. of dry pyridine for 2 hr. at 75° to yield 40.3 g. (77%) of crude acid phthalate, m.p. 113.5–121.5°. One recrystallization from benzene gave 34.6 g. of pure *threo*-3-*p*-anisyl-2-butyl acid phthalate, m.p. 122–123.5°, mixed m.p. 123–124° with an authentic sample (m.p. 123–124°).[5b] Recrystallization of the residue from the first crystallization gave an additional 5.3 g. of acid phthalate, m.p. 121.5–123.5°.

The infrared spectrum of the crude alcohol product was essentially identical with the spectrum of pure *threo*-alcohol[5b] and showed no detectable amount of 2-*p*-anisyl-2-butanol.

Los Angeles 24, Calif.

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(11) Considerably higher yields were obtained by adding the diborane to the olefin at room temperature and then allowing the reaction mixture to stand several hours before oxidation.

(12) A Perkin-Elmer Model 21 Infrared Spectrophotometer with sodium chloride optics was used for analysis.