The Stereochemistry of the Reduction of 2-Substituted Cyclopentanones with Complex Aluminum Hydrides

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(Received November 13, 1970)

Five different 2-substituted cyclopentanones have been reduced by complex aluminum hydrides in various solvents. In the lithium aluminum hydride reduction, the proportion of *cis* 2-substituted cyclopentanols tends to increase with the steric bulkiness of the substituent. The fact that the composition of the products changes with the sort of solvent is discussed in terms of the difference in the coordinating abilities of the solvents. More *cis* 2-substituted cyclopentanols are obtained by lithium trimethoxyaluminum hydride than by the other complex aluminum hydrides used in the present study. Lithium tri-*t*-butoxyaluminum hydride and lithium aluminum hydride-aluminum chloride reduction are also investigated.

Although the stereochemistry of the reduction of substituted cyclohexanones has been studied in detail,[1] there have been only a few investigations of that of simple cyclopentanone derivatives.[1a,2]

In order to ascertain the effects of the substituents, solvents, and complex metal hydrides on the stereochemistry in the reduction of five-membered ring compounds, the reduction of representative 2-substituted cyclopentanones with lithium aluminum hydride and some of its alkoxy derivatives was undertaken in various solvents. The cyclopentanones (2-methyl-, 2-ethyl-, 2-isopropyl-, 2-cyclopentyl-, and 2-phenylcyclopentanones) were reduced with lithium aluminum hydride in diethyl ether, tetrahydrofuran, dimethoxyethane, triethylamine or pyridine, lithium trimethoxyaluminum hydride in tetrahydrofuran, lithium tri-*t*-butoxyaluminum hydride in tetrahydrofuran, and lithium aluminum hydride-aluminum chloride mixture in diethyl ether. In order to confirm the accuracy of the data, experiments were repeated at least twice in all hydride reductions, and the isomeric ratios of the products were quantitatively analyzed by gas chromatography.

Results

Reduction with Lithium Aluminum Hydride. The reduction of 2-methylcyclopentanone(I) in diethyl ether yielded a mixture of *cis* and *trans* 2-methylcyclopentanol(VI) containing 74—78% of the *trans* isomer. This is in good agreement with the result reported by Umland and Jefraim.[2a] The proportion of *trans*-VI changed with the solvents and increased in the order of diethyl ether, tetrahydrofuran, and dimethoxyethane. A similar isomer distribution was obtained in the reduction of 2-ethylcyclopentanone(II). The reduction of 2-isopropylcyclopentanone (III), which is considered to be relatively hindered ketone, gave slightly more *cis* 2-isopropylcyclopentanol (VIII) than *trans* (55—57% of *cis*-VIII) in diethyl ether. Conversely, the formation of *trans*-VIII was preferred in tetrahydrofuran, and 70% of the *trans* isomer was yielded in dimethoxyethane. On the other hand, the reduction of 2-cyclopentylcyclopentanone (IV) proceeded to give slightly more *trans*-2-cyclopentylcyclopentanol (IX) than *cis* (54% of *trans*-IX) in diethyl ether; a similar solvent dependence in the case of III was observed. The reduction of 2-phenylcyclopentanone (V) afforded 61% of *trans* 2-phenylcyclopentanol (X) in diethyl ether, while no appreciable solvent dependence on the isomer distribution was observed.

When triethylamine was used as the solvent, less of the *trans* isomer was obtained than in the ether-type sovents. Compounds I, II, III, IV, and V gave 68, 67, 37, 45, and 44% of the *trans* isomers respectively.

Lansbury and Peterson[3] reported that the reaction of lithium aluminum hydride with excess pyridine fairly rapidly formed a complex, lithium tetrakis-(1,2-dihydropyridyl)aluminate. They also reported that the complex reacted sluggishly with aliphatic ketones. Actually, the reaction of the cyclopentanones which were used in the present study with the complex gave the corresponding cyclopentanols in a yield of less than 1% even after 24 hr. However, when lithium aluminum hydride was added to the ketone-pyridine mixtures at 0°C, cyclopentanols were obtained in a good yield, so the ketone might be reduced by lithium aluminum hydride itself. In this case, the isomer distribution was similar to that obtained in dimethoxyethane. In all cases, the reduction favored the formation of the *trans* isomer.

*Reduction with Lithium Tri-*t*-butoxyaluminum Hydride.* In this case, inverse addition method was employed

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[1] a) H. C. Brown and H. R. Deck, *J. Amer. Chem. Soc.*, **87**, 5620 (1965) and references cited therein; b) H. C. Brown and J. Muzzio, *ibid.*, **88**, 2811 (1966); c) M. Chèrest and H. Felkin, *Tetrahedron Lett.*, **1968**, 2205; d) J. Klein, E. Dunkelblum, E. L. Eliel, and Y. Senda, *ibid.*, **1968**, 6127; e) D. N. Kirk, *ibid.*, **1969**, 1729; f) E. L. Eliel and Y. Senda, *Tetrahedron*, **26**, 2411 (1970); g) D. C. Ayres, D. N. Kirk, and R. Sawdaye, *J. Chem. Soc., B*, **1970**, 505.

[2] a) J. B. Umland and M. I. Jefraim, *J. Amer. Chem. Soc.*, **78**, 2788 (1956); b) W. Hückel and G. Näher, *Chem. Ber.*, **91**, 792 (1958); c) J-C. Richer and C. Gilardeam, *Can. J. Chem.*, **43**, 3419 (1965); d) R. G. Haber and B. Fuchs, *Tetrahedron Lett.*, **1966**, 1447; e) D. V. Banthrope and H. ff. S. Davies, *J. Chem. Soc., B*, **1968**, 1356; f) B. V. Baddeley and W. L. Shao, *Tetrahedron*, **24**, 6513 (1968).

[3] P. T. Lansbury and J. O. Peterson, *J. Amer. Chem. Soc.*, **85**, 2236 (1963).

TABLE 1. REDUCTION OF 2-SUBSTITUTED CYCLOPENTANONES WITH COMPLEX ALUMINUM HYDRIDES[a)]

		Products (*trans* %)								
Cyclopentanone	Order of addition	$LiAlH_4$					$LiAl(OMe)_3H$	$LiAl(O$-*t*-$Bu)_3H$	$LiAlH_4$-$AlCl_3$	
		(DEE)[b)]	(THF)[c)]	(DME)[d)]	(TEA)[e)]	(PYD)[f)]	(THF)	(THF)	K[g)] (DEE)	T[h)] (DEE)
2-Methyl-	N[i)]	74[k)]	80[l)]	83	68	—	54[m)]	—[n)]	83	100
	I[j)]	78	83	—	—	83	54	72	—	—
2-Ethyl-	N	72	76	75	67	—	40	—	73	100
	I	75	81	—	—	82	38	71	—	—
2-Isopropyl-	N	45[o)]	66	70	37	—	28	—	47	100
	I	43	72	—	—	72	37	53	—	—
2-Cyclopentyl-	N	54	66	79	45	—	33	—	62	100
	I	—	—	—	—	75	26	68	—	—
2-Phenyl-	N	61	59	64	44	—	30	—	58	99+
	I	61	59	—	—	53	24	52	—	—

a) All are simply average percentages from more than two concordant experiments. b) Diethyl ether c) Tetrahydrofuran d) Dimethoxyethane e) Triethylamine f) Pyridine g) Under the condition of kinetic control h) Under the condition of thermodynamic control i) Normal addition j) Inverse addition k) Umland and Jefraim[2a)] reported 75% *trans*, temperature not specified. l) Brown and Deck[1a)] reported 76% *trans* at 0°C. m) Brown and Deck[1a)] reported 56% *trans* at 0°C. n) Brown and Deck[1a)] reported 71% *trans* at 0°C. o) Hückel and Näher[2b)] reported 43% *trans* at 20°C.

for all experiments. Compounds I and II gave results very similar to the isomer distribution realized with lithium aluminum hydride itself. Similar results have already been observed in the reduction of substituted cyclohexanones by Haubenstock and Eliel[4)] and by Brown and Deck.[1a)] In the cases of III and IV, a situation different from that of I and II prevailed and the proportion of the more stable isomer was larger than that realized with lithium aluminum hydride in diethyl ether, the predominant product being the *trans* isomer. The lithium tri-*t*-butoxyaluminum hydride reduction of V gave more of the *cis* isomer than that with the parent hydride, and the ratio of the *cis* to the *trans* isomer became almost unity. In the cases of III, IV, and V, the isomer distribution was not similar to that obtained from lithium aluminum hydride reduction in diethyl ether, but was rather close to that in tetrahydrofuran.

Reduction with Lithium Trimethoxyaluminum Hydride. The replacement of hydrogen in lithium aluminum hydride by methoxyl groups resulted in an isomer distribution of the reduction products different from that resulting from reduction by other reagents. In the case of I, the amount of the more stable isomer decreased as compared to the amounts with other metal hydride reagents, and the isomer ratio became 46% of *cis* and 54% of *trans*-VI. With the increase in the steric requirement of the substituent in the 2 position, in other words, the change in the methyl group into the ethyl group, the predominant product was no longer the *trans* isomer. The *trans* 2-ethylcyclopentanol (VII) thus obtained was 38—40%.

Reduction with Lithium Aluminum Hydride - Aluminum Chloride Mixture. The reduction with a lithium aluminum hydride - aluminum chloride mixture was also performed. Under the conditions of kinetic control (in the presence of excess hydride), the ratio of *trans* 2-substituted cyclopentanols to *cis* isomers was very close to that obtained in the reduction by lithium aluminum hydride alone. However, when acetone was added at the end of the reduction, and when the reaction mixture was then refluxed for 4 hr (in the presence of excess ketone), the products of thermodynamical control, *trans* 2-substituted cyclopentanols, were predominant, probably because equilibriation occurred.

The results of the reduction of 2-substituted cyclopentanones are summarized in Table 1.

Discussion

In 1956 Dauben, Fonken, and Noyce postulated that the steric course of complex metal hydride reduction was controlled by two factors, "product development control" and "steric approach control".[5)] Alternative explanations have also been devised. Richer[6)] seeks it in the steric interference of the approaching nucleophile with the axial hydrogens in the 2 and 6 positions, and Chèrest and Felkin[1c)] ascribe the same interference to bond-eclipsing factors. Eliel and his coworkers deduce, from the kinetic data of the reduction of several cyclohexanones by lithium tri-*t*-butoxyaluminum hydride[1d)] and from a competitive rate study of complex metal hydrides,[1f)] that product development control plays, at best, a minor role especially in the reduction with aluminum hydrides.

Since it has been suggested[7)] that the cyclopentanone exists in the half-chair form with the maximum puckering occurring at the 3 and 4 carbon atoms, the Dreiding model shows the steric environments above and below the plane of the carbonyl group in this molecule

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4) H. Haubenstock and E. L. Eliel, *ibid.*, **84**, 2363 (1962).

5) W. G. Dauben, G. J. Fonken, and D. S. Noyce, *ibid.*, **78**, 2579 (1956).

6) J-C. Richer, *J. Org. Chem.*, **30**, 324 (1965).

7) E. L. Eliel, N. L. Allinger, S. J. Angyl, and G. A. Morison, "Conformational Analysis", Interscience Publishers, Inc., New York, N. Y. (1965), p. 202.

to be identical. Consequently, it seems impossible to introduce the steric factors, for example, Felkin's eclipsing factor, to explain the prior formation of the more stable one of the two possible products. Our results indicate that, in contrast to six-membered cyclic ketones, the steric course in the resuction of relatively unhindered cyclopentanone reflects the relative stability of the two isomeric products.

As was pointed out previously, the rates of reaction of the simple cyclic model systems exhibit this order; cyclohexanone>cyclopentanone.[8] This typical I-strain order can be attributed to the influence of bond-opposition forces on the energetics of the initial and final states, reflected in the transition state.

Since it has been suggested that the complex metal hydride reduction of cyclohexanone has a reactant-like transition state,[1c,1g] the results obtained in the present study and the difference in reactivity between cyclohexanone and cyclopentanone implies that the complex aluminum hydride reduction of the latter has a product-like transition state. In the increase in the steric requirement of the substituent in the 2 position of cyclopentanone, steric approach control is also operative in the course of the reaction.

It is also evident from this study that lithium trimethoxyaluminum hydride reduction gives more of the *cis* isomer which is obtained by the hydride attack from the less-hindered side of the carbonyl group than either lithium aluminum hydride or lithium tri-*t*-butoxyaluminum hydride reduction, as has been observed previously.[1a,4] This may mean, of course, that steric approach control becomes important with the trimethoxyaluminum hydride, even in the reduction of II.

The solvent effect on the isomeric ratio of the products was observed in the lithium aluminum hydride reduction. Our results do not permit a definitive statement of the mechanism, but we have adopted the one presented below as a tentative postulate. The solvent effect on the stereochemistry of the reduction is examined in terms of steric approach control, which is the steric interference between the entering metal hydride and the substituent in the 2 position, and product development control, which is the steric interference between the developing alkoxyaluminum function and the substituent in the 2 position at the transition state. Geneste and Lamaty[9] have postulated a transition state for hydride reduction of the type shown in Fig. 1. For convenience, however, these two factors will be discussed separately.

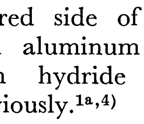

Fig. 1.

Steric Approach Control. Hydride transfer from aluminum to carbonyl carbon is facilitated by the coordination of aluminum with a Lewis base.[10] Since the greater the coordination, the more facile the hydride transfer, the ionic property of hydride may be accentuated and the distance between aluminum and hydride at the transition state seems to be longer in strongly-coordinating solvent than in a weakly-coordinating solvent. Consequently, the steric interaction of the entering hydride reagent with the substituent in the 2 position decreases with an increase in a Lewis base strength; in other words, more of the *trans* isomer will be afforded in a strongly-coordinating solvent than in a weakly-coordinating one.

Product Development Control. When the solvent coordinates on the aluminum atom of the OAl group in the 1 position, the steric strain between this OAl group and the substituent in the 2 position at the transition state may increase with the coordinating ability of the solvents; in other words, more of the *trans* isomer will be obtained in a strongly-coordinating solvent than in a weakly-coordinating one.

Jorgenson and Thacher[11] reported on the basis of a rate study of the reduction of cinnamyl alcohol with lithium aluminum hydride that tetrahydrofuran is more effective than diethyl ether as a solvent medium by a factor of 7.5, and that dimethoxyethane is better than tetrahydrofuran by a factor of 4—6.

Reportedly, the observed Lewis base strengths of the ether-type solvents used in the present study increase in the order: diethyl ether < tetrahydrofuran<dimethoxyethane,[12] an order which is identical with the coordinating ability.

Taking the above discussion of the coordinating ability of the solvents into account, the proportion of the *trans* isomer may increase in the order: diethyl ether<tetrahydrofuran<dimethoxyethane. Actually, the proportion of the *trans* isomers obtained from I, II, III, and IV agrees with the above prediction. Especially in the cases of III and IV, the solvent dependence is very significant.

Pyridine, whose base strength is stronger than that of an ether-type solvent, may behave as a solvent only under the experimental conditions employed. The isomer distributions of the products in pyridine are close to those in dimethoxyethane.

Wiberg and his coworkers[13] found that, when lithium aluminum hydride is treated with an excess of trimethylamine, the molecular compound $LiAlH_4 \cdot 2Me_3N$ is formed in the temperature range between −40° to 0°C. Alternatively, Peters[14] reported that lithium aluminum hydride with one mole of trimethylamine forms $(Me_2N)_2AlH_3$. He observed, however, when an excess of trimethylamine was used, nearly all of the lithium aluminum hydride gave a compound containing lithium and aluminum in an approximately 1 to 1 molar ratio, hydrogen and loosely-bound trimethylamine. He considered that this compound may be the material that Wiberg

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[8] H. C. Brown and K. Ichikawa, *Tetrahedron*, **1**, 221 (1957).

[9] P. Geneste and G. Lamaty, *Bull. Soc. Chim. Fr.*, 669 (1968).

[10] E. I. Synder, *J. Org. Chem.*, **32**, 3531 (1967).

[11] M. J. Jorgenson and A. F. Thacher, *Chem. Commun.*, **1968**, 973.

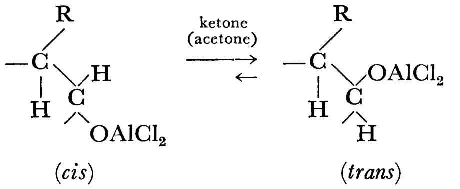
[12] E. M. Annett and Y. Wu, *J. Amer. Chem. Soc.*, **84**, 1680, 1684 (1962).

[13] E. Wibetg, H. Noth, and R. U. Lacal, *Z. Naturforsch.*, *B*, **11**, 486 (1956).

[14] F. M. Peters, *Can. J. Chem.*, **42**, 1755 (1964).

and his coworkers observed. However he did not reach any definite conclusion. The formation of the corresponding compound, $LiAlH_4 \cdot 2Et_3N$ or $(Et_2N)_2AlH_3$, can be expected when triethylamine is used instead of trimethylamine. Either way, although triethylamine is the strongest Lewis base among the solvents used in the present study, the molecular structure of lithium aluminum hydride in triethylamine seems to be a bulkier reagent than that in other solvents and so the steric interaction of the entering hydride reagent with the substituent in the 2 position will increase.

In the reduction of 2-substituted cyclopentanones with the lithium aluminum hydride-aluminum chloride mixture, the isomeric ratios differ according to the reaction conditions. In the presence of an excess of the hydride reagent, the isomer distribution is very close to that obtained in the reduction with lithium aluminum hydride. On the other hand, when excess ketone exists in the reaction system, *trans* isomers are yielded exclusively. Presumably the complex of the *trans* isomer is much more stable than that of the *cis* isomer because of the large bulk of the $-AlCl_2$ group, which is possibly swelled further by the solvation of the aluminum with ether. The equilibrium lies certainly very much more on the side of the *trans* isomer than the corresponding equilibrium of the free 2-substituted cyclopentanols.[15)]



(*cis*) (*trans*)

It is also of interest to relate the conformational equilibrium constant with the steric course of metal hydride reduction. The equilibria of VI, VII, VIII, and IX were studied in dioxane with Raney nickel at 80°C. The composition of the equilibrium mixture is listed in Table 2. The equilibrium compositions of VI and VII are 76 and 75% of the *trans* isomer respectively. However, VIII whose substituent is expected to be of a larger effective size than that of the methyl or the ethyl group gives a proportion of 61% of the *trans* isomer at equilibrium, while IX yields a mixture containing 66% of the *trans* isomer.

Table 2. Equilibrium mixture of 2-substituted cyclopentanols

Cyclopentanol	*Trans* %
2-Methyl-	76[a)]
2-Ethyl-	75
2-Isopropyl-	61[b)]
2-Cyclopentyl-	66

a) Umland and Williams[16)] reported 58% *trans* with aluminum isopropoxide-isopropanol system. b) Hückel and Näher[2a)] reported more than 91% *trans* with aluminum isopropoxide-isopropanol system.

Experimental

Materials. The lithium aluminum hydride and the lithium tri-*t*-butoxyaluminum hydride were purchased from Metal Hydride Inc. The lithium trimethoxyaluminum hydride was prepared *in situ* by adding a calculated amount of anhydrous methanol solution to lithium aluminum hydride in tetrahydrofuran. The hydride solution was titrated with iodine and sodium thiosulfate. The 2-methylcyclopentanone (I) was prepared from 1-methylcyclopentene by the method of Brown and his coworkers.[17),18)] The 2-ethylcyclopentanone (II) was prepared from 1-ethylcyclopentene as the above ketone (I). The 2-isopropylcyclopentanone (III) was prepared by the aldol-type condensation of cyclopentanone with acetone, followed by the hydrogenation over the palladium catalyst. The 2-cyclopentylcyclopentanone (IV) was prepared by the method reported by Hückel and his coworkers.[19)] The 2-phenylcyclopentanone (V) was prepared by the rearrangement of 1-phenylcyclopentene oxide by sulfuric acid. The 1-phenylcyclopentene oxide was dissolved in ether with 5 micro drops of 6 N sulfuric acid, after which the mixture was refluxed for 32 hr.

Reduction Procedures. The gas-chromatographic analysis showed that the yields of isomeric cyclopentanols from the corresponding ketones were in the range between 75—100%.

Reduction with Lithium Aluminum Hydride or Its Alkoxy Derivatives. (*a*) *In the Ether-type Solvent*: Ketone (0.04 mol) which had been dissolved in 15 m*l* of a solvent and an equivalent amount of a hydride solution was mixed at 0°C over a period of 10—15 min. Two orders of addition, normal and inverse addition were employed. After the reaction mixture had been stirred for 30 min, water and crushed ice were added, followed by enough 10% sulfuric acid to dissolve the precipitate. The aqueous layer was extracted with ether which had been washed with saturated sodium hydrogen carbonate, and then by brine, and dried over sodium sulfate. The solution was concentrated, and the residual solution was subjected to gas chromatography.

(*b*) *In Pyridine*: Ketone (0.04 mol) was dissolved in 10 m*l* of pyridine. To this solution, maintained in an ice bath, we then added powdered lithium aluminum hydride (0.01 mol) over a period of 1—2 min. After the reaction mixture had been stirred for 30 min, water and crushed ice were added, followed by enough 10% sulfuric acid to acidify the solution. The solution was extracted with ether which had been washed with saturated sodium hydrogen carbonate, and then by brine, and dried over sodium sulfate. The solution was concentrated, and the residual solution was subjected to gas chromatography.

(*c*) *In Triethylamine*: Lithium aluminum hydride (0.01 mol) was dissolved in 15 m*l* of triethylamine. To this solution, maintained in an ice bath, we then added a ketone (0.04 mol) in 10 m*l* of triethylamine over a period of 10—15 min. After the reaction mixture had been stirred for 30 min, water and crushed ice were added, followed by enough 10% sulfuric acid to acidify the solution. The solution was

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15) E. L. Eliel, *Rec. Chem. Progr.*, **22**, 129 (1961) and references cited therein.

16) J. B. Umland and B. W. Williams, *J. Org. Chem.*, **20**, 2788 (1956).

17) H. C. Brown and C. P. Garg, *J. Amer. Chem. Soc.*, **83**, 2951 (1961).

18) H. C. Brown, K. J. Murray, L. J. Murray, J. A. Sonver, and G. Zweifel, *J. Amer. Chem. Soc.*, **82**, 4233 (1960).

19) W. Hückel, M. Maier, E. Jordan, and W. Seeger, *Ann. Chem.*, **616**, 46 (1958).

20) E. L. Eliel and M. Rerick, *J. Amer. Chem. Soc.*, **82**, 1367 (1960).

extracted with ether which had been washed with saturated sodium hydrogen carbonate, and then brine, and dried over sodium sulfate. The solution was concentrated, and the residual solution was subjected to gas chromatography.

Reduction with Lithium Aluminum Hydride - Aluminum Chloride.[20] (*a*) *Under Conditions of Kinetic Control*: To a solution of 0.050 mol of aluminum chloride in 50 m*l* of ether, we added 0.014 mol of lithium aluminum hydride in ether. After stirring the homogeneous solution for 30 min, a solution of ketone (0.050 mol) in 10 m*l* of ether was added, drop by drop, over a period of 30 min. The mixture was stirred for 30 min at 0°C; then the excess hydride was destroyed with water and crushed ice, followed by enough 10% sulfuric acid to dissolve the precipitate. The aqueous layer was extracted with ether which had been washed with saturated sodium hydrogen carbonate, and then by brine, and dried over sodium sulfate. The solution was concentrated, and the residual solution was subjected to gas chromatography.

(*b*) *Under Conditions of Thermodynamic Control*: The mixed hydride was prepared as in (a) from 0.050 mol of aluminum chloride and 0.014 mol of lithium aluminum hydride. A solution of 0.050 mol of ketone in 10 m*l* of ether was added, after which the mixture was stirred for 30 min at 0°C. To this reaction mixture we then added 0.010 mol of acetone and refluxed the mixture for 4 hr; then water and crushed ice were added, followed by enough 10% sulfuric acid to dissolve the precipitate. The aqueous layer was extracted with ether, which had been washed with saturated sodium hydrogen carbonate, and then by brine, and dried over sodium sulfate. The solution was concentrated, and the residual solution was subjected to gas chromatography.

Equilibrium. Raney nickel which had been prepared from the alloy was thoroughly washed with distilled water, absolute ethanol, and dioxane. For equilibriation 0.3 g of 2-substituted cyclopentanol was dissolved in 10 m*l* of the solvent. Raney nickel (*ca* 2 g) was added in the suspension and the mixture was heated at 80°C. From time to time, samples were withdrawn and analyzed by gas chromatography. Equilibrium was deemed to be reached when several consecutive samples gave identical analyses. In all cases, equilibrium was approached from both sides. Equilibriation was usually complete after 250—300 hr.

Gas Chromatography. Hitachi K-53 or F-6 equipped with 45 m PEG goley column (0.25 or 0.50 mm in diameter) was used along with a flame ionization detector.