

ether was added 20.0 g. of pure cyanuric chloride in several portions. The solution was stirred for another 15 minutes, excess hydride was decomposed with water, 200 ml. of 10% sulfuric acid was added and the mixture was extracted with ether. The ether extract yielded 1.5 g. of colorless needles which after recrystallization from petroleum ether melted at 124–124.5° (cor.).

Anal. Calcd. for $C_5H_6Cl_2N_4$: C, 31.11; H, 3.13; N, 29.02. Found: C, 31.52; H, 3.10; N, 29.13.

A mixture melting point with an authentic sample of 2-dimethylamino-4,6-dichloro-1,3,5-triazine⁸ showed no depression.

Ultraviolet Spectrum.—The ultraviolet absorption spectrum of 2-amino-1,3,5-triazine in a 10^{-5} molar aqueous solution was measured with a Beckman model DU spectrophotometer at 26°. A continuously decreasing absorption was observed from 225 to 240 $m\mu$.

(8) W. M. Pearlman and C. K. Banks, *THIS JOURNAL*, **70**, 3726 (1948).

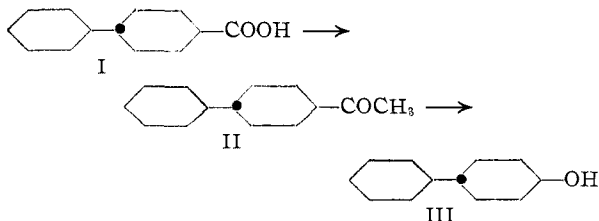
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Determination of the Configuration of 4-Cyclohexylcyclohexanols

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In the course of a study of the hydrogenation of 4-hydroxybiphenyl-4'-carboxylic acid,¹ it became of interest to determine, with certainty, the stereochemistry of the 4-phenyl- and the 4-cyclohexylcyclohexanols. In 1948, Ugnade² prepared the pure *cis* and *trans*-isomers of this series and assigned the configurations on the basis of method of preparation and on thermodynamic stability. Recently, Dauben and Hoerger³ showed that by converting a carboxylic acid to a methyl ketone and cleaving the resulting ketone with perbenzoic acid,⁴ an acid could be related, stereospecifically with retention, to an alcohol. Since Fieser, *et al.*,⁵ have related *trans*-4-cyclohexylcyclohexylcarboxylic acid (I) to *trans*-hexahydroterephthalic acid, a compound whose stereochemistry has been rigorously established by Mills and Keats,⁶ application of the methyl ketone-perbenzoic acid sequence above to *trans*-acid I would determine the stereochemistry of the 4-substituted cyclohexanols in an unequivocal manner.



Such a sequence of reactions was performed and it was found that *trans*-acid I was, indeed, related to the *trans*-alcohol III. Thus, the stereochemical configurations of the 4-phenyl- and 4-cyclohexyl-

cyclohexanols assigned by Ugnade² are correct. It is of passing interest to note that in this series, the thermodynamically stable isomer is the one which has both of its substituents in equatorial conformations.⁷

Experimental

***trans*-4-Cyclohexylcyclohexylcarboxylic Acid (I).**—The acid was prepared following the procedure of Fieser, *et al.*,⁵ m.p. 160.5–161.9° (lit. 161–162°).

***trans*-4-Cyclohexylcyclohexanol (III).**—A solution of 0.9 g. (4.28 millimoles) of *trans*-acid I in 15 ml. of dry ether was added dropwise with stirring to 18 ml. of a 0.6 *M* solution of methylolithium in ether. Following the addition, the mixture was stirred for 12 hours and then poured onto ice. The ethereal layer was removed, washed neutral with water, dried and the solvent removed through a small column. The residue weighs 0.8 g. (89.7%) and was used directly in the oxidation.

Perbenzoic acid (0.638 g.) in 8.8 ml. of chloroform was added to the crude ketone which had been dissolved in 5 ml. of chloroform. The solution was allowed to stand for 8 days at room temperature in the dark. At the end of this time the reaction was diluted with ether and washed thoroughly with dilute sodium hydroxide. The residue remaining after removal of the solvents was heated for 7 hours with 50 ml. of 1 *N* sodium hydroxide in 30 ml. of methanol and the methanol removed under reduced pressure. The aqueous residue was extracted with ether, the ether distilled and the 4-cyclohexylcyclohexanol recrystallized from hexane, yield 0.28 g. (40%), m.p. 99.6–102.4° (lit.² 103–104°). The phenylurethan was prepared and melts 154.9–156.6° (lit.² 156–156.8°).

(7) D. H. R. Barton, *Experientia*, **6**, 316 (1950).

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The Structure of the Solid Product from the Condensation of Methyl γ -Bromocrotonate with Sodium Methoxide¹

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Owen and Sultanbawa² reported the isolation of a small amount of a solid from the reaction of methyl γ -bromocrotonate with dry sodium methoxide in benzene. The product was characterized by its analysis (empirical formula of $C_5H_6O_2$), melting point (168–169°) and ultraviolet absorption spectrum (λ_{max}^{alc} 303 $m\mu$, $E_{1cm}^{1\%}$ 2600). The authors considered that it was possibly methyl cyclopropenecarboxylate.

A re-examination of the reaction confirmed the observations of Owen and Sultanbawa, the solid product being formed in 1.3% yield. However, its high melting point and its spectrum, which appeared uncharacteristic of either of the double bond isomers of methyl cyclopropenecarboxylate, suggested a reconsideration of its structure.

α -Haloesters are known to be attacked by bases with the formation of enolates.³ The vinylogous reaction with methyl γ -bromocrotonate (I) is the abstraction of a proton from the γ -position with the

(1) Supported in part by grants to the Detroit Institute of Cancer Research from the American Cancer Society, Inc., the Michigan Cancer Foundation and The Kresge Foundation.

(2) L. N. Owen and M. U. S. Sultanbawa, *J. Chem. Soc.*, 3098 (1949).

(3) M. S. Newman and B. J. Magerlein, "The Glycidic Ester Condensation" in R. Adams, "Organic Reactions," Vol. 5, John Wiley and Sons, Inc., New York, N. Y., p. 413.

(1) W. G. Dauben and M. Tanabe, in press.

(2) H. E. Ugnade, *J. Org. Chem.*, **13**, 361 (1948).

(3) W. G. Dauben and E. Hoerger, *THIS JOURNAL*, **73**, 1504 (1951).

(4) R. B. Turner, *ibid.*, **72**, 878 (1950); T. F. Gallagher and T. H. Kritchevsky, *ibid.*, **72**, 882 (1950).

(5) L. F. Fieser, *et al.*, *ibid.*, **70**, 3186 (1948).

(6) W. H. Mills and G. H. Keats, *J. Chem. Soc.*, 1373 (1935).