starting material there was obtained 0.5 g. of the acid; colorless plates melting at 197°.

Anal. Calcd. for $C_{12}H_{14}O_2$: C, 75.8; H, 7.37. Found: C, 75.9; H, 7.31.

p-Cyclopentylbenzamide was prepared by the method of reference 1, p. 157. It was recrystallized from dilute alcohol and formed glistening flakes, m. p. 189–190°.

Anal. Calcd. for $C_{12}H_{15}ON$: C, 76.2; H, 7.94; N, 7.41. Found: C, 76.1; H, 8.12; N, 7.30.

The melting points given are uncorrected.

2211 BURLING STREET
CHICAGO 14, ILLINOIS RICHARD D. KLEENE

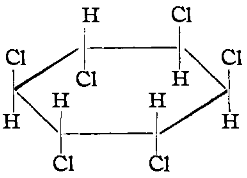
RECEIVED JANUARY 4, 1949

COMMUNICATIONS TO THE EDITOR

THE STRUCTURE OF α-BENZENE HEXACHLORIDE

Sir:

The structures of the isomers of benzene hexachloride (1,2,3,4,5,6-hexachlorocyclohexane) are of considerable interest due to the insecticidal activity of one of them.[1] Of the five known isomers, one, the β-isomer, is of established configuration,[2] being the isomer with all vicinal chlorine (or hydrogen) atoms *trans* to each other. The α-isomer has been assigned the structure



by Burrage and Slade[1] on the basis of a statistical treatment of chlorine addition, whereas Melander[3] has assigned the above structure to the γ-isomer on the basis of dipole moment data.

We wish to report preliminary data at this time regarding the structure of the α-isomer.

The above structure has no element of symmetry and should therefore exist as a racemate capable of resolution.[4] Lucas and Gould[5] have shown that *dl*-dibromoalkanes may be partially resolved by preferred reaction of one modification with optically active bases. With this rate method we have obtained optically active α-benzene hexachloride by the reaction

$$dl\text{-}C_6H_6Cl_6 + 3/2\text{brucine} \longrightarrow 1/2\text{active-}C_6H_6Cl_6 + 3/2\text{brucine}\cdot HCl + 1/2C_6H_3Cl_3$$

The α, γ and δ isomers were treated separately in dioxane with one-half of the amount of brucine required for complete dehydrochlorination. Five grams (0.018 mole) of each isomer, 12.4 g. (0.027 mole) of brucine, and 50 ml. of dioxane were allowed to stand at room temperature for several days. The alkaloid and alkaloid hydrochloride were removed by washing with water and dilute acid, and the residual (ether extraction) oily solids tested for optical activity. (Specific rotations (ethereal solutions): α, −14.6°; γ, −0.1°; δ, −1.3°.)

The optical activity of the α-product was concentrated by distillation, steam distillation, chromatography on alumina, and fractional distillation to give material of m. p. 128–132° (specific rotation over 120° (in acetone)). [*Anal.* calcd. for $C_6H_6Cl_6$: C, 24.78; H, 2.08; Cl, 73.14. Found (Clark Microanalytical Laboratories): C, 24.94; H, 2.32; Cl, 73.04. Hydrolyzable chlorine, calcd., 36.6; found, 36.2.] The activity is unaffected by recrystallization from concd. nitric acid, and is lost in a few minutes upon treatment with 2.5% methanolic sodium hydroxide.[6]

We are presently attempting the isolation of the levorotatory α-isomer with other optically active bases for a study of the phase diagram, as our present preliminary data suggest that the inactive material is a racemic compound.

We wish to acknowledge the assistance of the Office of Naval Research, for partial support of this work, as well as of the Bureau of Entomology and Plant Quarantine, U. S. Department of Agriculture, where initial phases of this research were done.

---

[1] Slade, *Chemistry and Industry*, 314 (1945).

[2] Hendricks and Bilicke, THIS JOURNAL, **48**, 3007 (1926); Dickinson and Bilicke, *ibid.*, **50**, 764 (1928).

[3] Melander, *Svensk Kem. Tid.*, **58**, 231 (1946).

[4] The only structure for a hexachlorocyclohexane capable of resolution. See Shriner, Adams and Marvel in Gilman, "Organic Chemistry," John Wiley and Sons, New York, N. Y., 1943, 2nd ed., pp. 324, 337.

[5] Lucas and Gould, THIS JOURNAL, **64**, 601 (1942).

[6] *Cf.* Cristol, *ibid.*, **69**, 338 (1947).

DEPARTMENT OF CHEMISTRY STANLEY J. CRISTOL
UNIVERSITY OF COLORADO
BOULDER, COLORADO

RECEIVED MARCH 11, 1949

THE EXCHANGE REACTION BETWEEN THE TWO OXIDATION STATES OF CERIUM IN NITRIC ACID SOLUTIONS[1]

Sir:

We have studied the exchange reaction between cerous and ceric ions in nitric acid solutions

---

[1] This work was carried out under the auspices of the Atomic Energy Commission.