

continuous nature of the discharge, may account for the increased rate of production of diboron tetrachloride in a d.c. arc.

Work is in progress to increase the yield still further, and an investigation of electrode materials other than mercury is planned.

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RECEIVED JULY 14, 1958

THE ACTION OF POTASSIUM AMIDE IN AMMONIA
ON *o*-CHLOROPHENYLACETONE: CORRECTION.¹
Sir:

We recently reported a new principle of ring closure, the essence of which is the intramolecular addition of a nucleophilic center to a benzyne structure.²

Amongst examples of the new principle, the conversion of *o*-chlorophenylacetone to indan-2-one, through the action of potassium amide in liquid ammonia, was described. We have now discovered an error: the compound reported as indan-2-one (m.p. 60–61°)³ is actually 2-methylindole (m.p. 62°).⁴ The compound obtained, in 25% yield in recent experiments, strongly depresses the mixed melting point with authentic indan-2-one, but does not alter the mixed melting point with authentic 2-methylindole. Also, the compound gives a positive sodium fusion test for nitrogen. All our efforts to isolate authentic indan-2-one from the reaction in question have been fruitless.

It is of theoretical interest that 2-methylindole is formed in this reaction; the matter will be discussed in a future publication.

(1) Research supported in part by the Office of Ordnance Research, U. S. Army.

(2) B. F. Hrutford and J. F. Bunnett, *THIS JOURNAL*, **80**, 2021 (1958).

(3) H. D. Porter and C. M. Suter, *ibid.*, **57**, 2022 (1935).

(4) L. Marion and C. W. Oldfield, *Can. J. Research*, **25B**, 1 (1947).

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RECEIVED AUGUST 4, 1958

1,2;5,6-DI-*O*-ISOPROPYLIDENE 3-DEOXY-3-AMINO-
 α -D-ALLOSE

Sir:

Hydrolysis of the new antibiotic kanamycin has been found¹ to yield 3-deoxy-3-amino-D-glucose (I).²

The crystalline amine which is formed³ on the aminolysis of 1,2;5,6-di-*O*-isopropylidene α -D-glucopyranose tosylate (II) has been characterized^{2,3} as 1,2;5,6-di-*O*-isopropylidene-3-deoxy-3-amino- α -D-glucose. These reactions therefore appear to provide a convenient route for the preparation of I.

This communication is to report that, as suspected by Cope and Shen,⁴ the aminolysis actually

(1) M. J. Cron, D. L. Evans, F. M. Palermiti, D. F. Whitehead, I. R. Hooper, P. Chu and R. U. Lemieux, *THIS JOURNAL*, **80**, 4741 (1958).

(2) S. Peat and L. F. Wiggins, *J. Chem. Soc.*, 1810 (1938).

(3) K. Freudenberg, O. Burkhart and E. Braun, *Ber.*, **59**, 714 (1926).

(4) A. C. Cope and T. Y. Shen, *THIS JOURNAL*, **78**, 3177 (1956).

proceeds with inversion of carbon-3 to form 1,2,5,6-di-*O*-isopropylidene-3-deoxy-3-amino-D-allose (III).

Compound III can be prepared in 83% yield by hydrogenolysis of the 1,2;5,6-di-*O*-isopropylidene-3-deoxy-3-hydrazino-D-hexose (IV) of Freudenberg and Brauns⁵ in ethanol at 80° using Raney nickel catalyst and 40 p.s.i. of hydrogen. The advantage of this route is that, whereas the aminolysis of II proceeds in 16% yield,³ the hydrazinolysis proceeds in 60% yield.⁵ Acid hydrolysis of III produced an aminosugar of different paper-chromatographic properties than that obtained on the hydrolysis of methyl 4,6-benzylidene-3-deoxy-3-amino- α -D-glucoside diacetate.²

The N-acetyl derivative of III (V), m.p. 127–128°, $[\alpha]_D +71.3^\circ$ (*c*, 2 in chloroform), [calcd. for C₁₄H₂₃O₆N: C, 55.80; H, 7.69; N, 4.65. Found: C, 55.87; H, 7.73; N, 4.85.] was hydrolyzed for two hours at 100° in 0.2 *N* hydrochloric acid. N-Acetylation was then accomplished by the addition of acetic anhydride to the neutralized hydrolyzate. Evaporation of the solvent and extraction of the residue with ethanol gave a 1,2-*O*-isopropylidene-3-deoxy-3-acetamido- α -D-hexose (VI), m.p. 154–156°. Calcd. for C₁₁H₁₉O₆N: C, 50.56; H, 7.33; N, 5.36. Found: C, 50.25; H, 7.44; N, 5.29.

Periodate oxidation of VI, reduction of the product with sodium borohydride and acetylation of the reduced product gave a crystalline substance which was hydrolyzed to aminosugar using *N* hydrochloric acid at 100°. On evaporation, a crystalline product was isolated whose infrared spectrum (KBr disc) and X-ray powder diagram were identical to those obtained with an authentic sample of 3-deoxy-3-amino-D-ribose hydrochloride (VII).⁶ These results establish the *allo*-configuration for III-VI and provide a new synthesis of VII which is a constituent of the antibiotic puromycin.⁷

Acknowledgments.—The authors wish to thank Dr. M. Przybylska for the X-ray analyses.

(5) K. Freudenberg and F. Brauns, *Ber.*, **55**, 3233 (1922).

(6) B. R. Baker and R. E. Schaub, *J. Org. Chem.*, **19**, 646 (1954).

(7) P. W. Fryth, C. W. Waller, B. L. Hutchings and J. H. Williams, *THIS JOURNAL*, **80**, 2736 (1958).

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RECEIVED JULY 21, 1958

STRUCTURE OF THE Ag⁺ (CYCLOÛCTATETRAENE)
COMPLEX

Sir:

Complexes between metal ions and organic π bonding systems have been of interest for some time^{1,2} and are germane to general discussions³ of weak complexes and their reactivities.^{1,2,4} Aside from the Ag⁺ (Benzene) complex,^{5,6} few such com-

(1) S. Winstein and H. J. Lucas, *THIS JOURNAL*, **60**, 836 (1938); **79**, 4339 (1957).

(2) L. J. Andrews, *Chem. Revs.* **54**, 713 (1954).

(3) R. S. Mulliken, *J. Chem. Phys.*, **19**, 514 (1951); *THIS JOURNAL*, **72**, 600 (1950); **74**, 811 (1952).

(4) R. E. Rundle and J. D. Corbett, *ibid.*, **79**, 757 (1957).

(5) R. Rundle and J. Goring, *ibid.*, **72**, 5337 (1950); for a structural study of the styrene-PdCl₂ complex see J. Holden and N. Baenziger, *ibid.*, **77**, 4987 (1955).

(6) H. G. Smith and R. E. Rundle, Abstracts, American Crystallographic Society Meeting, Milwaukee, Wisconsin, June 23–27 (1958).