SHORT COMMUNICATIONS

Reductive Dehalogenation of Aromatic Halides with Fused Zinc Dust

Eiji ŌSAWA[1] and Zen-ichi YOSHIDA

Department of Synthetic Chemistry, Faculty of Engineering, Kyoto University, Yoshida, Kyoto

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Reductive dehalogenation of aromatic halides is hard unless the halogen is activated by electron-attracting groups.[2] Recently it was reported that Raney-alloy and a base in water or toluene is effective at room temperature for a limited number of aromatic halides.[3] We wish to report that fused mixture of zinc dust, zinc chloride, and sodium chloride reduces not only carbonyl groups[4] but also halogen. Both monobromo- and dichloro-isodibenzanthrone gave directly isodibenzanthrene in moderate yield. 9, 10-Dibromoanthracene was reduced to anthracene. This method can be applied to the compounds insoluble in ordinary organic solvents. Attempt to extend this method to replace other substituents with hydrogen is going on. A preliminary test showed that sulfonate group could be removed from sodium anthraquinone-α-sulfonate.

Isodibenzanthrene from Brominated Isodibenzanthrone. Ponsol Brilliant Violet 3B (du Pont, Colour Index No. 60005) was washed with water to remove dispersing agent contaminated, and recrystallized several times from nitrobenzene. Two grams of the purified vat dye, 2 g of sodium

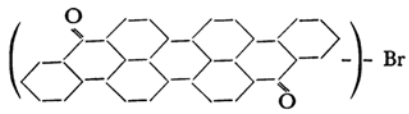

Fig. 1. Ponsol Brilliant Violet 3 B.
(Du Pont, C. I. 60005)

chloride, 1.2 g of zinc chloride, and 2 g of zinc dust are mixed in a glass tube. The tube was immersed in a silicone-oil bath heated at 300°C. The mixture began to melt at 270°C. Temperature was kept at 270—285°C for twenty minutes. The reaction mixture was cooled, poured into 200 m*l* of dilute hydrochloric acid, and triturated well. Collected precipitate was washed with water unitl neutral. To remove the unreacted vat dye, the crude product was treated with warm aqueous solution of sodium hydrosulfite (40 g/*l*), and sodium hydroxide (30 g/*l*) for one hour, and filterated quickly while warm. The hydrosulfite treatment was repeated three times until the vat remained colorless. The insoluble crystal was washed water, boiled in 200 m*l* of dilute hydrochloric acid for thirty minutes, collected, washed with water, and dried. Sublimation at 400°C and 10^{-3} Torr gave 1 g of red-brown crystal. Beilstein test for bromine was negative. Found: C, 95.68; H, 4.23%. Calcd for $C_{34}H_{18}$: C, 95.75; H, 4.25%.

Other reductions mentioned above were conducted in essentially the same way.

1) Present address: Department of Chemistry, the University of Wisconsin, Madison, Wis., U.S.A.
2) R. Stroh in "Houben-Weyl, Methoden der organischen Chemie," **V4**, E. Müller, ed., Georg Thieme Verlag, Stuttgart (1960), p. 769.
3) H. Kämmerer, L. Horner and H. Beck, *Ber.*, **91**, 1376 (1958).
4) E. Clar, *ibid.*, **72**, 1645 (1939).