Fraction	B. p. °C.	Mm.	Grams	n^{25}D	$[\alpha]^{25}$D
1	45–46.5	4	1.3	1.4909	+5.1
2	46.5–80	4	2.9	1.4932	+5.1
3	80–89	4.5	0.7	1.5110	...
4	89–95	5	1.9	1.5095	+2.4

Fraction 1 was concluded to be essentially pure α-phenethyl nitrite. In order to establish the nature of fraction 4, 50 g. of *dl*-α-phenethyl chloride was treated with silver nitrite under the conditions described above, and from the crude reaction product there was obtained by repeated fractionation, in addition to 11.5 g. of α-phenethyl nitrite of b. p. 34–36° at 2 mm. and of n^{25}D 1.4938, 2.5 g. of colorless oil of b. p. 90–95° at 3 mm. and of n^{25}D 1.5210. The latter material, which corresponded roughly to fraction 4 above, was shown to contain largely α-nitroethylbenzene by conversion to the aci-form according to the method of Bamberger and Seligman.[6] The intermediate fractions gave precipitates of silver chloride when warmed with alcoholic silver nitrate solution and were concluded to contain, in addition to the other two components, unreacted α-phenethyl chloride.

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[6] Bamberger and Seligman, *Ber.*, **36**, 707 (1903).

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RECEIVED JULY 19, 1946

Catalytic Decomposition of DDT

BY A. L. FLENNER

From work conducted on the stability of DDT to heating at 115°, Fleck and Haller[1] concluded that impurities present in technical DDT inhibit the action of catalytic substances in eliminating hydrogen chloride, and that removal of these impurities without simultaneous removal of catalytic substances produces an apparent instability of DDT. In the course of work on the manufacture and formulation of DDT, this Laboratory made a study of the stability of samples varying in degrees of purity.

It was found that Technical DDT, having a set point of 88°, and highly purified material, with a melting point of 108.5°, evolved no hydrogen chloride when heated for twenty-four hours at 115°, whereas material which had been once recrystallized from ethanol, and having a melting point of 105–107°, did evolve hydrogen chloride when heated under the same conditions. The material melting at 105 to 107° had not been filtered from a small amount of insoluble impurities during the recrystallization and it was thought that these impurities might be acting as catalytic decomposing substances. A portion of the partially purified material was therefore dissolved in ethanol, filtered, and then the solution evaporated to dryness to recover the DDT. When this filtered DDT, after drying, was subjected to heating at 115° for twenty-four hours, it was found to be stable since no hydrogen chloride was evolved, which indicates that the insoluble impurities acted as catalytic decomposition agents and their removal rendered the DDT stable under the test conditions. To obtain additional evidence, both technical DDT and purified DDT of m. p. 108.5° were heated in the presence of iron oxide at 115°. Hydrogen chloride was evolved almost immediately from different samples of the purified material, while from four to six hours were required to evolve hydrogen chloride from samples of technical DDT.

These results therefore verify those of Fleck and Haller[1] and it is concluded that there is some substance or substances present in technical DDT which act as an inhibitor to the catalytic elimination of HCl. Removal of these inhibiting substances will render the DDT susceptible to catalytic decomposition. It, therefore, seems quite possible that under conditions of use technical DDT may be more stable than purified or partially purified material since catalytic substances present in diluents and natural dusts may act to liberate hydrogen chloride slowly from spray deposits of the purified DDT. Although purified DDT is not being used extensively at the present time any future use under field conditions may require the addition of an inhibitor to protect it from catalytic decomposition.

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[1] Fleck and Haller, THIS JOURNAL, **68**, 142 (1946).

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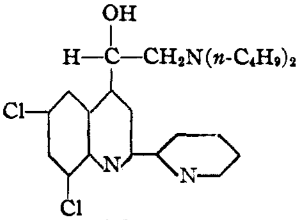
6,8-Dichloro-2-(2′-pyridyl)-α-di-*n*-butylaminomethyl-4-quinolinemethanol[1a]

BY HENRY GILMAN, LEO TOLMAN AND SAMUEL P. MASSIE, JR.

In connection with studies on experimental avian malaria it was desirable to determine the activity of a 6,8-dichloro-4-quinolinemethanol having a nitrogen heterocycle in the 2-position. The compound selected was 6,8-dichloro-2-(2′-pyridyl)-α-di-*n*-butylaminomethyl-4-quinolinemethanol.[1b] This compound [I] was prepared *via* the Pfitzinger reaction by condensation of 5,7-dichloroisatin with methyl α-pyridyl ketone.[2] Ethyl 6,8-dichloro-2-(2′-pyridyl)-cinchoninate was prepared incidental to an examination of its condensation with ethyl acetate to give the corresponding ketoester. The plan was to brominate the keto-ester as a means of preparing the 4-bromoacetyl compound. However, some orienting experiments showed that the condensation of the ethyl ester with ethyl acetate by means of sodium ethoxide was not so satisfactory as the reaction of diazomethane with the acid chloride.



[I]

Experimental

6,8-Dichloro-2-(2′-pyridyl)-cinchonic Acid.—A mixture of 77 g. (0.36 mole) of 5,7-dichloroisatin,[3] 60 g. (1.08

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[1] (a) The work described in this paper was done under a contract recommended by the Committee on Medical Research, between the Office of Scientific Research and Development and Iowa State College; (b) the Survey Number assigned to this drug by The Survey of Antimalarial Drugs is SN-14,143-4. The activities of these compounds will be tabulated in a forthcoming monograph.

[2] See Winstein, Jacobs, *et al.*, THIS JOURNAL, **68**, 1831 (1946). Also, Lindwall, Bandes, and Weinberg *ibid.*, **53**, 317 (1931).

[3] Prepared in accordance with directions kindly provided by Drs. H. Sargent and T. C. Myers of the California Institute of Technology.