A NOTE ON THE CONVERSION OF ω-TRIBROMOQUINALDINE TO ω-DIBROMOQUINALDINE AND THE PRODUCTION OF QUINALDIC ALDEHYDE

By L. K. SHARP.

Received March 3, 1949.

ω-DIBROMOQUINALDINE cannot be prepared by selective bromination of quinaldine, the only product isolated being the ω-tribromo derivative, but the former may be prepared by reduction from the latter. Hammick[1] reduced tribromoquinaldine by means of stannous chloride in acetone solution, obtaining a yield of 60 per cent. of theory. In order to separate the product from tin compounds, steam distillation was employed. As dibromoquinaldine is only slightly volatile in steam the purification process is tedious. The reduction may however be performed by refluxing ω-tribromoquinaldine with a 20 per cent. v/v solution of sulphuric acid in alcohol and pouring the mixture into water. The product separates almost pure in excellent yield, and a single crystallisation from alcohol is all that is necessary. The identity of the product was confirmed by its conversion, by means of alcoholic silver nitrate, to quinaldic aldehyde by the method of Hammick[2].

EXPERIMENTAL.

ω-Tribromoquinaldine (10 g.) prepared by Hammick's[3] method was refluxed for 4 hours with a mixture of alcohol (97 per cent.) (80 ml.) and concentrated sulphuric acid (20 ml.), and the mixture then poured into water. The white precipitate was washed and dried. Yield 7 g. (88 per cent. of theory). M.pt. 119°C. After recrystallisation from alcohol the pure product melted at 120°C. Found: C, 39·95; H, 2·67; N, 4·9; Br, 52·9 per cent.: $C_{10}H_7NBr_2$. requires C, 40·0; H, 2·3; N. 4·7; Br, 53·3 per cent.

PROOF OF CONSTITUTION.

2·5 g. in 15 ml. of boiling alcohol was treated with silver nitrate solution and the product worked up in the manner described by Hammick[2]. After steam distillation 0·9 g. (70 per cent. of theory) of quinaldic aldehyde was obtained: M.pt. 69°C.; mixed melting-point with quinaldic aldehyde, 70°C.

REFERENCES

1. Hammick, *J. chem. Soc.*, 1926, 1302.
2. Hammick, *Ibid.*, 1926, 1303.
3. Hammick, *Ibid.*, 1923, **123**, 2882.