2-CHLORO-3,5-BIS(ACETYLAMINO)TOLUENE*

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Incidental to the identification of o-chlorotoluene in a reaction mixture, 2-chloro-3,5-bis(acetylamino)toluene was prepared. This compound is mentioned in the literature[1,2] but its constants apparently have not been published. It was prepared by nitration of the fraction to be identified, reduction with tin and hydrochloric acid, and acetylation of the amine with acetic anhydride. Mixed melting point determinations showed it to be identical with the product prepared from authentic o-chlorotoluene by the following reactions:

Nitration of o-chlorotoluene with HNO_3-H_2SO_4 yielded a semisolid product from which 2-chloro-3,5-dinitrotoluene, m. 62-3° (cor.) was isolated by repeated recrystallization from carbon tetrachloride. Previously recorded melting points are 63-4°[3], 65°[4] and 45°[1]. The last value is incorrect.

The dinitro compound was reduced with tin and hydrochloric acid to 2-chloro-3,5-diaminotoluene which, after recrystallization from water, melted at 72-3° (cor.). The literature gives 73°[1] or 74°[2].

The diamine was stirred with a slight excess of acetic anhydride until crystals formed; these were washed with cold water and recrystallized from about 400 parts by weight of water. 2-chloro-3,5-bis(acetylamino)toluene forms fine white fibrous needles, m.p. 227-8° (cor.), soluble in acetic acid, acetone and alcohol, difficultly soluble in ether and benzene, almost insoluble in hexane (60-70°) and carbon tetrachloride.

The compound was analyzed by the semi-micro Kjeldahl method by Mr. C. A. Harman. Calcd. for $C_{11}H_{13}ClN_2O_2$: N, 11.64%. Found: 11.80, 11.58.

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[1] R. Nietzke and E. Rehe. Ber. 25, 3005-9 (1892).
[2] G. T. Morgan. J. Chem. Soc. 81, 86-100 (1902).
[3] W. Borsche and A. Fiedler. Ber. *45*, 270-3 (1912).
[4] G. Körner and A. Contardi. Atti. accad. Lincei 24, I, 888-96 (1915).