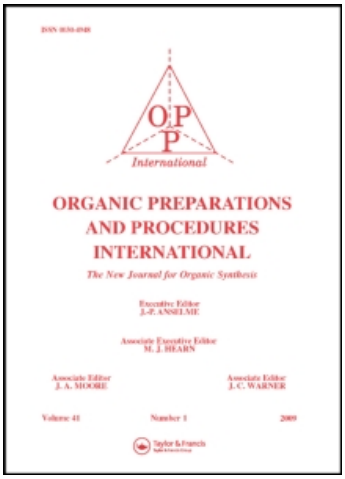

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PREPARATION OF 2-ISOCYANATO-5-CHLOROBENZOYL CHLORIDE

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PREPARATION OF 2-ISOCYANATO-5-CHLOROBENZOYL CHLORIDE

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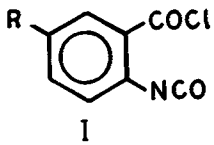
In our studies[1,2] on the reactions of 2-isocyanato benzoyl chlorides(I), we were required to prepare the 5-chloro-derivative(Ib). Its preparation (in 31% yield) from 6-chloro isatoic anhydride(IIb) by the action of thionyl chloride takes

three weeks time.[3] We report here the preparation of 2-isocyanato-5-chlorobenzoyl chloride(Ib) in 75% yield by the chlorination of Ia following a modified procedure for the chlorination of phenyl isocyanate.[4]



I

a) R = H
b) R = Cl

EXPERIMENTAL

Into a stirred solution of 2-isocyanato benzoyl chloride (36.2 g; 0.2 M) in ethylene dichloride (110 g) at 25° was introduced a slow stream of dry chlorine for 5 min. Iodine (0.75 g in 30 g ethylene dichloride was then added and chlorine addition (total 14.9 g; 5% excess) resumed during which period the temperature rose to 50-55°. After stirring for 2 hours at 25°, the solvent was evaporated and the residue treated with dry benzene (75 ml). The separated 6-chloro isatoic anhydride (1.64 g) was filtered out and benzene evaporated from the filtrate. The residue (45 g) on distillation under reduced pressure gave 2-isocyanato-5-chlorobenzoyl chloride at 115-118°/ 1.5 mm Hg, m.p. and m.m.p. 54-57°. Its IR spectrum was superimposable with that of an authentic sample prepared from Ib and PMR spectra of its reaction products[2] confirm the substitution pattern.

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A NEW SYNTHESIS OF 4-BROMOMETHYLBENZAL BROMIDE AND 1,4-bis(DIBROMOMETHYL)-BENZENE

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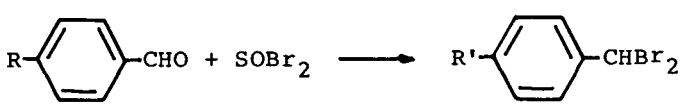
Although two different methods for the preparation of 4-bromomethylbenzal bromide IIa have been studied, a one-step synthesis of this compound in good yield has yet to be achieved. In one such method, p-xylene was brominated at 130° to yield a mixture of brominated products from which IIa was isolated in 23% yield.[1,2] Drefahl and Plotner[3] isolated the same product after treatment of p-tolualdehyde with phosphorous tribromide in carbon disulphide followed by bromination of the intermediate product, 4-methylbenzal bromide, at 140° in the presence of a powerful source of light.



I II

a) R = CH_3 b) R = CHO a) R' = CH_2Br b) R' = $CHBr_2$

The other compound 1,4-bis(dibromomethyl)benzene (IIb) has been synthesized by various research groups, but a high yield synthesis of this compound under ordinary conditions is yet to be realized. In one such method[4,5] dry bromine was