

The Action of the Esters of Chlorosulfonic Acid on Di-*n*-Butylamine

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The reactions between the lower dialkylamines and the lower esters of chlorosulfonic acid at 0°C. were described by Delepine and Demars¹ in 1923. Similar reactions between the lower esters of chlorosulfonic acid and di-*n*-butylamine were conducted by Binkley and Degering in 1939.² The present study was an investigation of the reactions between the lower esters of chlorosulfonic acid and di-*n*-butylamine at the of these experiments are as follows:

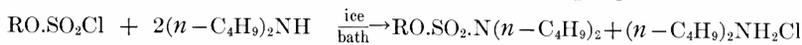
A three-neck-distilling flask was equipped with a mercury-seal mechanical stirrer and a small dropping funnel. The third neck was attached to a gas analysis train containing four traps. The two traps nearest the reaction flask were surrounded by cold water baths while the other two traps were placed in dry ice-acetone baths. The apparatus was constructed in such a manner that a current of dry air could sweep any gases from the reaction flask into the gas analysis train. After one-fifth of a mole of di-*n*-butylamine was placed in the reaction flask, the agitation was begun. The flask was heated with a steam bath during the careful addition of one-tenth of a mole of the alkyl chlorosulfonate from the dropping funnel to the di-*n*-butylamine. The reaction mixture was heated for 30 minutes after the addition of the alkyl chlorosulfonate, while a slow current of dry air was allowed to pass through the apparatus. The cold water baths removed from the two traps nearest the reaction flask, these traps were warmed with steam to distill any volatile substances into the remaining two traps for analysis. The results of these experiments are as follows:

Alkyl chlorosulfonate	Percentage of alkyl chloride formed (based on the alkyl chlorosulfonate used)
methyl	60
ethyl	53
<i>n</i> -propyl	46

The oily residue in the reaction flask was largely di-*n*-butylammonium-*N,N*-di-*n*-butylamidodisulfonate in all of these experiments. This compound was readily converted to di-*n*-butylamine hydrochloride and *N,N*-di-*n*-butylamidodisulfonic acid (m.p. 132-3°C.) with hydrochloric acid.

Conclusions

The reactions between lower esters of chlorosulfonic acid and di-*n*-butylamine at the temperatures of an ice bath and steam bath are markedly different as represented by the following equations:



¹Delepine, M., and R. Demars, 1923. *Bull. Sci. Pharmacol.* 30:577-9.

²A portion of a thesis submitted to the faculty of Purdue University in partial fulfillment of the requirements of the degree of Doctor of Philosophy, June, 1939.