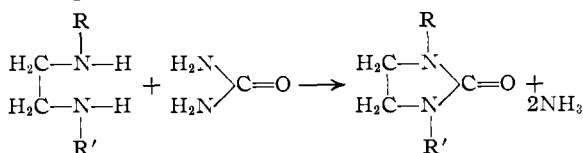


TABLE I

Compound	B. p., °C.	Mm.	M. p., °C.	% yield	N Analyses, %	
					Calcd.	Found
1,3-Dicyclohexyl-2-iminazolidone	165-166	1	92-94	80	11.19	10.91
1,3-Dibutyl-2-iminazolidone	137-140	3	...	81	14.14	14.36
1,3-Dioctyl-2-iminazolidone	195-200	3	5-6	80	9.02	9.30
1,3-Didodecyl-2-iminazolidone	245-252	2	48-49	76	6.63	6.86
1,3-Dibenzyl-2-iminazolidone	183-186	1	93-94	80	10.53	10.43

which ammonia is rapidly evolved, according to the equation



Although the reaction of urea with amines to produce substituted ureas is well known, the only reaction between diamines and urea previously described was reported by Clarke¹ as a method of preparing polymers of interest as synthetic fibers.

A previous synthesis of 2-imidazolidones was described by Boon,² who treated the diamines with phosgene. He reported approximately equal yields of the cyclic urea and of the carbonyl chloride, so that, at best, yields of less than 50% were obtained. In the present investigation, the yield is in general about 80% of the theoretical amount.

Data for the five substances now prepared are shown in Table I. Of these the only one previously reported is the dibenzyl derivative, prepared by Lob³ from phosgene. In this case our data agree very closely to the physical constants which he reported.

The general procedure for the preparation of these substances is given herewith.

One-tenth mole of the diamine is heated in a glass vessel with 0.125 mole of urea with a free flame until vigorous evolution of ammonia takes place. The heating is continued until this slows down to a negligible rate (ten to fifteen minutes). The product is then vacuum distilled and, if crystalline, is recrystallized from absolute alcohol.

Acknowledgment.—The authors express their appreciation to the F. C. Bersworth Laboratories of Framingham, Massachusetts, for financial support for this research.

(1) Clarke, British Patent 530,267, Dec. 9, 1940.

(2) Boon, *J. Chem. Soc.*, 307 (1947).

(3) Lob, *Rec. trav. chim.*, **55**, 866 (1936).

DEPARTMENT OF CHEMISTRY
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The Reduction of Sulfonyl Chlorides to Mercaptans with Lithium Aluminum Hydride

BY C. S. MARVEL AND P. D. CAESAR

The reduction of aliphatic and aromatic sulfonyl chlorides to mercaptans with lithium aluminum hydride has been realized. There is no previous record of the use of this reagent for a reduction of this type.

The reaction may proceed according to the following stoichiometrical equation, although in the preliminary experiments shown here a two- to threefold excess of lithium aluminum hydride was used to assure a positive reaction. Ethyl ether was employed as the solvent.



Initial attempts to reduce arylsulfonamides and diarylsulfones, using ethyl ether as a solvent, proved fruitless. It seems probable that use of a higher boiling solvent such as tetrahydrofuran may improve this reaction.

***p*-Thiocresol and *n*-Butyl Mercaptan.**—These compounds were prepared under the general conditions for lithium aluminum hydride reduction described by Nystrom and Brown.¹ The reductions were carried out at 35° for one and one-half hours.

From 5 g. (0.026 mole) of *p*-toluenesulfonyl chloride and 4 g. (0.1 mole) of lithium aluminum hydride there was obtained 1.6 g. (50% based on *p*-toluenesulfonyl chloride) of *p*-thiocresol, m. p. 42.5-43.5°, alone and when mixed with an authentic sample of *p*-thiocresol.

From 4 g. (0.025 mole) of 1-butanensulfonyl chloride and 3 g. (0.08 mole) of lithium aluminum hydride there was isolated 2.2 g. (45% based on 1-butanensulfonyl chloride) of mercury *n*-butyl mercaptide; m. p. 84-85°, reported m. p. 85-86°. This was obtained by distilling the ether, mercaptan and some of the water from the reaction mixture, after decomposition of the lithium aluminum complex, into a suspension of mercuric oxide in alcohol.

(1) R. F. Nystrom and W. G. Brown, *THIS JOURNAL*, **69**, 1197 (1947).

(2) E. Wertheim, *ibid.*, **51**, 3661 (1929).

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Proton Exchange between Ammonia and Ammonium Ion in Liquid Ammonia¹

BY C. J. NYMAN, SI-CHANG FUNG AND H. W. DODGEN

In the explanation of data observed on the exchange of protons between ND₃ and B₂H₆·2NH₃, it was postulated by Burg² that the exchange of protons between ammonia molecules and ammonium ions was rapid. Several properties of the ammonium ion in liquid ammonia caused the authors to consider the possibility of this reaction being slow. The fact that the conductance³ of the ammonium ion in this solvent is similar to that of lithium indicates that the proton is not nearly so mobile as in aqueous solution. Like-

(1) This project supported in part by the State College of Washington Committee on Research.

(2) A. B. Burg, *THIS JOURNAL*, **69**, 747 (1947).

(3) E. C. Franklin and C. A. Kraus, *ibid.*, **27**, 191 (1905); *Am. Chem. J.*, **23**, 277 (1900).