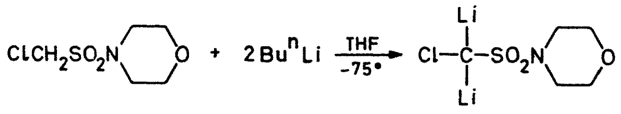
Preparation and Characterisation of an α-Chloro-dicarbanion from 4-(α-Chloromethanesulphonyl)morpholine

By W. E. TRUCE* and L. W. CHRISTENSEN

(*Department of Chemistry, Purdue University, Lafayette, Indiana* 47907)

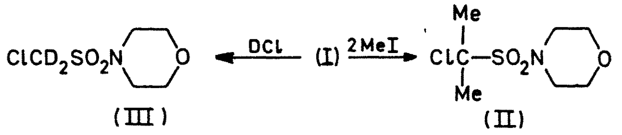
Summary The generation and characterization of the first α-chlorodicarbanion structure is reported.

ALTHOUGH the area of dicarbanion chemistry has received considerable attention, most of the dianions investigated have been 1,3-dianions separated by the stabilizing functional group.[1] So far only three 1,1-dicarbanions have been reported in the literature.[2,3] We are unaware of any reports of the formation of the halogenodilithioalkyl moiety. This would constitute a dianion with a potentially good leaving group, *i.e.*, halogen, attached to the reactive site. We now report the preparation and characterization of such a species. When 4-(α-chloromethanesulphonyl)-morpholine is treated with two equivalents of n-butyllithium in tetrahydrofuran at −75°, it is converted cleanly into the 1,1-dianion (I).



(I)

The α-halogenodilithio-species, (I), was characterized by its ready dialkylation to afford (II) in 83% yield. Significantly, the n.m.r. spectrum of the crude reaction material leading to (II) indicated only the presence of dialkylated product with no detectable monoalkylated product or starting sulphonamide in the reaction mixture. In addition, when a solution of (I) was added to a large excess of deuterium chloride in D_2O, the dideuteriated product was isolated in 94% yield. It was shown by n.m.r. that (III) was at least 97% dideuteriated.



We thank the Public Health Service for financial support of this work under a grant from the National Cancer Institute.

(*Received, March* 15*th*, 1971; *Com.* 249.)

---

[1] K. G. Hampton, T. M. Harris, and C. R. Hauser, *J. Org. Chem.*, 1964, **29**, 3511; S. Boatman and C. R. Hauser, *ibid.*, 1966, **31**, 1785; E. M. Kaiser, D. M. von Schriltz, and C. R. Hauser, *ibid.*, 1968, **33**, 4275.

[2] E. M. Kaiser and C. R. Hauser, *J. Amer. Chem. Soc.*, 1966, **88**, 2348.

[3] E. M. Kaiser and C. R. Hauser, *Tetrahedron Letters*, 1967, 3341.