Synthesis of *N*-Toluene-*p*-sulphonylsulphone Di-imines

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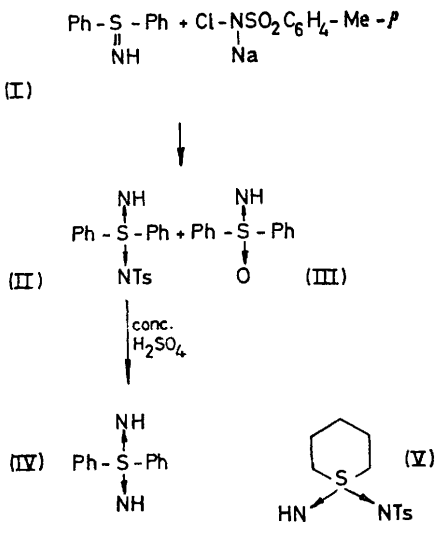
Summary Toluene-*p*-sulphonylsulphone di-imines were synthesized from the corresponding "free" sulphilimines and chloramine-T.

WE describe a convenient synthesis of *N*-toluene-*p*-sulphonylsulphone di-imine[1] from "free" sulphilimine[2] and chloramine-T. In a general reaction diphenylsulphilimine

(I) (200 mg) and chloramine-T (400 mg) were treated in methanol at room temperature, and after evaporation of the solvent, a mixture of the sulphone di-imine (II) and the sulphoximine (III) was obtained in yields of 54 and 39%, respectively. Compound (II), m.p. 151·5—152°, was isolated and recrystallized from benzene. The yield of (II) increased to 92% when a large excess of sodium toluene-*p*-sulphonamide was used. The structure of (II) was determined by means of elemental, i.r., and n.m.r. spectroscopic analyses. The n.m.r. chemical shifts of the phenyl protons of (II) resemble those of *N*-*p*-tosylsulphoximine or the sulphone and not those of *N*-*p*-tosylsulphilimine. Furthermore, (II) is stable under both acidic and alkaline conditions, supporting the quadrivalent S^{IV} structure (II) and not the trivalent S^{III} structure, $Ph_2S{\cdot}N{\cdot}NHTs$. Treatment of (II) with conc. H_2SO_4 afforded diphenylsulphone di-imine (IV) quantitatively, m.p. 88—89°, ν 3170 (NH), 1130, 1095, 1065, 930 (SN), δ($CDCl_3$) 2·2—2·6 (2H, NH), 7·15—7·50 (6H, *m*, *p*-PhH), 7·80—8·20 (4H, *o*-PhH). Compound (V) was obtained similarly in 33% yield, m.p. 145·5—146° and its structure was determined by means of elemental and spectroscopic analyses.



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[1] New dialkylsulphone di-imines have been prepared from the corresponding sulphide and chloramine: R. Appel, H. W. Felhaber, D. Hauugen, and R. Scholhorn, *Chem. Ber.*, 1966, **99**, 3103; M. Haake, *Tetrahedron Letters*, 1970, 4449; J. A. Coglino, and X. Braude, *J. Org. Chem.*, 1964, **29**, 1387. The structure of di-imines is discussed in the following: R. G. Laughlin and W. Yellin, *J. Amer. Chem. Soc.*, 1967, **89**, 2435; N. C. Webb and R. A. Gloss, *Tetrahedron Letters*, 1967, 1043.

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