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SYNTHESIS OF S,S-DIMETHYLSULFILIMINOAZINES

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Nitroso heterocycles are a little known class of compound. One of the few easy (and in this case the only) methods of synthesis is the oxidation of S,S-dimethylsulfilimines [1, 2].

We propose a practical and convenient synthesis of sulfiliminoazines II from the corresponding amino heterocycles I in DMSO using phosphoric anhydride.

Phosphoric anhydride (1 mmole) was added over 30 min at 20-25°C to DMSO (2.5 mole) and stirred for a further 30 min. The amine I was then added at such a rate that the temperature did not exceed 25°C and the product held at this temperature for a further 3 h. The product was poured into water (400 ml), neutralized with NaOH (20%), and extracted with dichloromethane. After evaporation of the extract the residue was precipitated from toluene using hexane to give Ia [78%, mp 94-95°C, PMR spectrum in CDCl3: 2.79 (s, 2CH3), 7.42 (d, J = 2.3 Hz, H4), 7.91 ppm (d, J = 2.3 Hz, 6-H)] or Ib (68%) or Ic (76%). The physicochemical data for Ib and Ic agreed with [2]. The S,S-dimethylsulfilimines Ia-c were readily converted to the corresponding nitroso heterocycles by oxidation with meta-chloroperbenzoic acid.

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