REACTION OF DMSO WITH UREAS — A ONE STEP ROUTE TO 1,3,5-TRIS(METHYLTHIOMETHYL)-1,3,5-TRIAZINE-2,4,6-TRIONE

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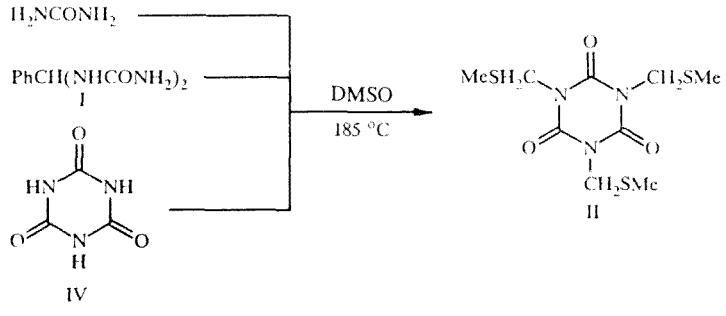
There are a number of literature reports on N-methylthiomethylation of some nitrogen containing compounds with dimethylsulfoxide, e.g., amides [1, 2] and phthalazindiones [3]. However high temperature reactions of urea with DMSO have not yet been studied. We have established that boiling urea or benzylidenediurea (I) in DMSO gave the product (II) in 16 and 20% yield respectively. The product of intramolecular cyclization (III) was also obtained in 33% yield from the diurea (I). This compound has been made previously from benzaldehyde and urea [4].



We have also shown that isocyanuric acid (IV) forms product II in 58% yield on reaction with DMSO. This observation suggests that acid IV is an intermediate in the synthesis of compound II in all the cases mentioned above, and the low yields of II are evidently a result this acid being formed in insufficient amount. The synthesis of compound II most likely occurs by a Pummerer type rearrangement involving dimethylsulfoxide [5] and isocyanuric acid.

The structures of compounds II and III have been confirmed spectroscopically.

1,3,5-Tris(methylthiomethyl)isocyanuric Acid (II, $C_9H_{15}N_3O_3S_3$). m.p. 98-99°C. IR spectrum: 1698 cm^{-1} (C=O). ^1H NMR Spectrum ($CDCl_3$, δ): 2.32 (9H, s, 3 CH_3), 5.01 ppm (6H, s, 3 CH_2). ^{13}C NMR Spectrum (DMSO-D_6, δ): 49.40 (CH_2), 151.21 ppm (C=O).

2-Phenylisocyanuric Acid (III). m.p. 269-270°C (lit. 270°C [4]). IR Spectrum: 1700 (C=O), 3320 (NH), 3390 cm^{-1} (NH). ^1H NMR Spectrum (DMSO-D_6, δ): 5.75 (1H, s, CH), 8.35 (2H, s, 2 NH), 9.60 (1H, s, NH), 7.62 (5H, m, H_{arom}).

Elemental analysis for C,H and N for compounds II and III agreed with the calculated values.

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