LITERATURE CITED

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SYNTHESIS OF 3,4-BIS(3-R-4-SYDNONOYL)FUROXANES

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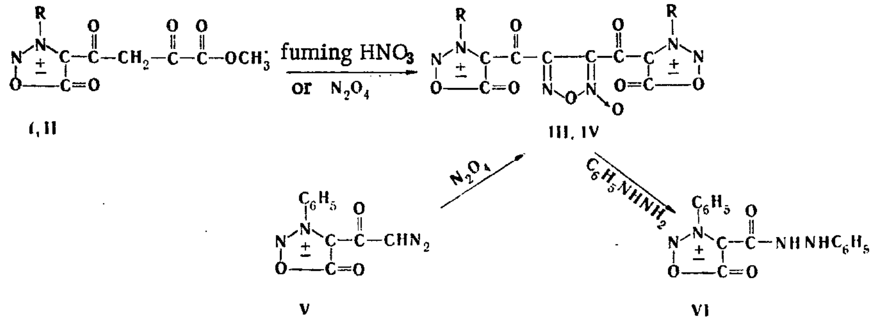
We have observed that an oxalyl residue is split out in the reaction of methyl 4-(3-R-sydnonyl)-2,4-dioxobutanoates (I, II) with fuming nitric acid to give 3,4-bis(3-R-4-sydnonoyl)-furoxanes (III, IV). Thus 2.3 g (0.3 mmole) of fuming nitric acid was added with stirring at 30-35°C to a solution of 5 g (0.15 mmole) of II in 40 ml of chloroform, 2 h after which the solvent was removed, and the residue was washed with ether and recrystallized from chloroform to give 4.53 g (57%) of 3,4-bis(3-phenyl-4-sydnonoyl)furoxane (IV) with mp 198-199°C. Compound III (in 51% yield), with mp 212-214°C (dec.), was similarly obtained. Furoxane IV was also obtained by reaction of II and 3-phenyl-4-diazoacetylsydnone (V) with nitrogen tetroxide.

Compound IV reacts with phenylhydrazine to give N-phenyl-N'-(3-phenyl-4-sydnonoyl)-hydrazine (VI); this is characteristic for furoxanes.



I, III R = CH$_3$; II, IV R = C$_6$H$_5$

A 1-g (0.021 mmole) sample of IV was sprinkled into a solution of 2.3 g (0.21 mmole) of phenylhydrazine in 10 ml of ether, and the mixture was stirred until an exothermic reaction commenced. After 2 h, the solvent was removed, and the residue was washed with ether and recrystallized from ethanol to give 0.3 g (47%) of VI with mp > 230°C (dec.).

The IR spectra of furoxanes III and IV contain absorption bands at 1790-1805 (sydnone ring CO group), 1660-1665 (keto group), 1610-1620 (C=N), 730-915, 790-955, and 1060-1080 cm^{-1} (furoxane ring system). The IR spectrum of VI contains absorption bands at 1720 (sydnone ring CO), 1645 (keto group), and 3319 cm^{-1} (NH). UV spectra of furoxanes in chloroform, λ_{max} (log ε): III, 258 (3.32) and 342 (4.21); IV, 268 (3.96) and 350 nm (4.38).

The results of elementary analysis of III, IV, and VI for C, H, and N are in agreement with the calculated values.

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