

observed. Similarly, $\text{CH}_3\text{CH}_2\text{OC}(=\text{S})\text{N}(\text{CH}_2\text{CH}_3)_2$ and $(\text{CH}_3)_2\text{CHOC}(=\text{S})\text{N}(\text{CH}_2)_2$ were heated together for 5 h at 160°C without the formation of any new products.

Finally, *S*-(methoxythiocarbonyl)thiohydroxylamine was decomposed in the presence of ^{15}N labelled *O*-ethylthiocarbamate (30% ^{15}N). Again, formation of ^{15}N labelled *S*-(methoxythiocarbonyl)thiohydroxylamine or ^{15}N labelled *O*-methylthiocarbamate was not observed.

From these experiments we conclude that, at room temperature, the decomposition of *S*-(alkoxythiocarbonyl)thiohydroxylamines is completely intermolecular.

The mass spectra were obtained on a double focusing mass spectrometer, type AEI-MS 902.

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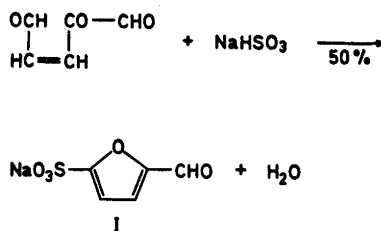
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Synthesis of 5-Formyl-2-furan-sulfonic Acid Sodium Salt from Endialone

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The title compound (I) has previously been prepared by Ivanov and Yankov¹ (cf. also Refs. 2, 3) by sulfonation of furfuraldiacetate with Baumgarten's reagent (sulfur trioxide-pyridine) (yield 37%). It has now been found that I is formed from endialone⁴ (*cis*-oxoglutaconaldehyde) and sodium hydrogen sulfite (cf. Ref. 5). This new synthesis is simpler and cheaper than the sulfonation reaction.



Experimental. Sodium hydrogen sulfite (10.4 g, 0.100 mol) was added to a molar solution of endialone of pH 2.0 and 0°C (100 ml, 0.100 mol), prepared as described previously.⁴ The mixture was stirred at room temperature for 30 min. The resulting clear, slightly yellow solution was heated to 60°C over a period of 30 min and then kept at this temperature for 60 min. About 5 min after the temperature of 60°C had been reached, crystals of a sesquihydrate of I began to separate. The suspension was cooled to 15°C and the crystals isolated by filtration. The wet cake was washed on the filter with ethanol-water (1:1) (20 ml), ethanol (20 ml), and ether (20 ml), and dried (20°C , 15 h). 11.2 g (50%) of I sesquihydrate was obtained, m.p. $> 250^\circ\text{C}$ (decomp.). (Found: C 26.6; H 3.0; S 14.0; ashes from CH-determination 31.9. Calc. for $\text{C}_5\text{H}_4\text{NaO}_5\text{S}$ (225.2): C 26.7; H 2.7; S 14.3; 0.5 Na_2SO_4 31.6). Very rapid Karl Fischer titrations of water gave 12.7% of water (calc. for 1.5 H_2O : 12.0%). Slower titrations showed up to 17.9% of water, indicating formation of water due to acetalization of the aldehyde group during titration.

Drying of the sesquihydrate at 110°C to constant weight gave anhydrous III as a slightly hygroscopic product. (Found: C 30.1; H 1.9; S 15.4; ashes 35.4. Calc. for $\text{C}_5\text{H}_3\text{NaO}_5\text{S}$ (198.1): C 30.3; H 1.5; S 16.2; 0.5 Na_2SO_4 35.9.) The ^1H NMR spectrum of the product agreed with the proposed structure.

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