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A Convenient Method for the Quantitative Determination of Sulfoxides

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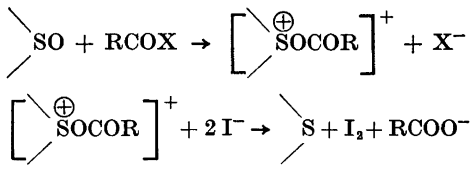
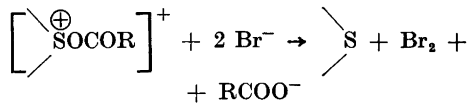
The methods available for quantitative determination of sulfoxides are mainly based on reduction of the sulfoxide group. Thus, the reduction by means of hydrogen bromide in glacial acetic acid [1] has often been used and studied,[2,3] and other reducing agents such as titanium trichloride [4,5] and stannous chloride [6] have been found valuable. Other methods are also known, such as the potentiometric titration of sulfoxides as bases with perchloric acid in acetic anhydride.[7]

During work on the reduction of sulfoxides, the present author found an accurate and rapid method for the quantitative determination of such compounds. The method, which consists in acylation of the sulfoxide in the presence of iodide, involves the intermediate formation of an acyloxysulfonium salt and can be represented by the following scheme:



With bromide, free bromine is formed:



As acylating agent acetyl chloride was used. For analytical purposes acetic acid was found to be a good reaction medium because of the formation of a one-phase system on adding dilute hydrochloric acid. The iodine was titrated with sodium thiosulfate. Details are described in the experimental part.

The method seems to be generally applicable for compounds in which the sulfoxide group is not too sterically hindered and the reaction at room temperature is usually complete within some minutes. In Table 1 the equivalent weights found for some various compounds are represented.

Acyloxysulfonium salts have earlier been proposed by some authors [8-12] as intermediates in the reaction between acid anhydrides and sulfoxides,[13,14] and recently in a reaction consisting in oxidation of alcohols in dimethyl sulfoxide - acid anhydride mixtures.[15]

Experimental. Procedure. The following method was found to be suitable: 0.5—1.0 mmole of the sulfoxide compound is accurately weighed, transferred to a 100 ml Erlenmeyer flask, and dissolved in 20 ml of glacial acetic

Table 1. Equivalent weights found for some different sulfoxides.

Sulfoxide	Reaction time min	Equivalent weights found	calc.
$(CH_3)_2SO$	2	39.4	39.1
$(C_6H_5CH_2)_2SO$	2	114.6	115.2
$CH_3CH_2CH_2SOCH_2CH_2CO_2H$	2	82.4	82.1
$C_6H_5CH_2SOCH_2CH_2CO_2H$	2	106.2	106.1
$CH_3SOC(CH_3)_2CO_2H$	5	75.1	75.1
$CH_3SOC(CH_3)_2CH_2CO_2H$	5	81.8	82.1

acid. When all has passed into solution, 3 g of potassium iodide is added followed by 0.5 ml of acetyl chloride. Iodine is immediately formed. After gentle stirring for 2–5 min, the contents are diluted with 50 ml 1 M hydrochloric acid and titrated at once with 0.1 N sodium thiosulfate. In order to correct for simultaneous oxidation of iodide by oxygen in the air, it is preferable to carry out a blank determination, although the corresponding thiosulfate volume was found to be very small (<0.1 ml).

Materials. Dimethyl sulfoxide (Fisher Certified Reagent) was used without further purification. Dibenzyl sulfoxide (Schuchardt) was recrystallized from ethanol. M.p. 135° (Ref. 16: 135–136°). The other compounds listed in Table 1 were all of the same purities as previously reported.[17-19]

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Significance of Constituents and Processing on the Amino Acid Pattern of Meat Meal

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From a global point of view a marked deficiency, quantitatively as well as qualitatively, exists with regard to protein. This refers both to man and animal. One source of protein in animal feeding has been and still is the considerable amounts of by-products from slaughterhouses, constituting about one fifth of the meat production. Most of the inedible products are worked up to meat meal by sterilizing, dehydration and defattening. The value of the meal thus obtained is dependent on two factors: raw material and processing technics.

The quality of meat meal has been investigated repeatedly. Swedish meat meals as well as other foodstuffs were analysed by Ågren [1-3] about 15 years ago. For the determination of the amino acid composition Ågren applied the microbiological technique after preceding autoclaving in 2 M HCl at 120°C for 10 h. Tryptophan, however, was determined after alkaline hydrolysis, and cystine, too, was analysed after a separate hydrolysis.

Since these investigations were made new meat meal processing technics have been developed and generally introduced. In addition, the ratio between the various by-products may have changed with time. Although the samples analysed by Ågren were not drawn at random, they do not represent an average composition of meat meal for a longer period. This is of importance to consider as the raw material shows qualitative seasonal changes.

Formerly the wet rendering technics was generally employed for the processing of meat meal. It is still in use in smaller plants. Briefly, this method makes use of direct steam for sterilizing and for separating the fat. Simultaneously, however, also some 30 % of the protein will be extracted and get lost as "glue liquor", which is drawn. The meat and bone residue is then dried in steam jacketed drums at atmospheric pressure. The temperature