Table 2. Determination — by reduction with stannous chloride and simultaneous presence of cupric oxide — of sulphate in potassium sulphate pro analysi. 20.00 ml about 0.1 N potassium iodate solution were used in each of the first 6 experiments and 50.00 ml in each of the last 3 experiments. The volumes of potassium iodate solution are recalculated to 0.1000 N.

K_2SO_4 weighed g	KIO_3 ml	K_2SO_4 found g
0.1069	12.19	0.1062
0.1114	12.78	0.1114
0.1126	12.96	0.1129
0.1064	12.11	0.1055
0.1072	12.33	0.1074
0.1115	12.76	0.1112
0.3449	39.59	0.3450
0.3080	35.18	0.3065
0.3062	35.18	0.3065

Presence of other elements which form slightly soluble sulphates. In the same way as mentioned in the previous section, it was investigated if presence of calcium or lead influenced the method. This was not the case. It may be added that the presence of lead never caused formation of visible lead sulphide in the boiling flask.

Presence of elements which form slightly soluble sulphides. An example of such an element is lead which is mentioned above. Further it was tried to add copper salts: To the weighed sample was added about 0.5 g of diverse copper salts. Surprisingly, it now appeared that no hydrogen sulphide distilled over, but sulphur dioxide only, though copper sulphide was formed as an intermediate in the boiling flask.

This fact led up to the possibility of altering the earlier published method so that sulphur dioxide only is produced by the reduction of sulphate. The following method was tried with a favourable result: To a weighed portion of the sample were added 1) 4 g stannous chloride, 2) 1 g large-grained cupric oxide, and 3) 20 ml phosphoric acid (85 %). An ordinary Bunsen burner was used for heating. Further 1) standard potassium iodate solution (about 0.1 N) was pipetted off into the absorption system, and 2) 2 g potassium iodide, and 3) 10 ml 2 M sodium hydroxide were added. According to this method the sulphate content in potassium sulphate pro analysi was determined; the results are given in Table 2.

Introductory experiments indicate that sulphate may be determined also by boiling down with phosphoric acid using cuprous chloride or zinc as reductant. Investigations of these problems will be continued.

1. Rancke-Madsen, E. *Acta Chem. Scand.* **3** (1949) 773.

Received October 12, 1951.

Tellurenyl Sulphur Compounds

OLAV FOSS

Universitetets Kjemiske Institutt,
Blindern — Oslo, Norway

Whereas sulphenyl and selenenyl compounds, with the formulae RSX and RSeX where X is an electronegative atom or group, are well known [1–4], no tellurenyl compounds, RTeX, have been described in literature. We wish to report the syntheses of the first types of tellurenyl compounds, *viz.*, some *p*-methoxybenzenetellurenyl pseudohalides.

p-Anisyl telluritrichloride [5] reacts with sodium methanethiosulphonate to give *p*-methoxybenzenetellurenyl methanethiosulphonate:

$$ArTeCl_3 + 3\ CH_3SO_2SNa = ArTeS_2O_2CH_3 + $$
$$+ S_2(SO_2CH_3)_2 + 3\ NaCl$$

The following procedure was employed: 6.8 g (0.02 mole) of *p*-anisyl telluritrichloride and 10 g (0.075 mole) of finely powdered sodium methanethiosulphonate in 25 ml of ethanol-free chloroform were triturated for a few minutes, first at room temperature and afterwards at boiling temperature of the solvent. The mixture was filtered while hot, and 20 ml of ether were added to the deep red filtrate. On standing, the tellurenyl methanethiosulphonate crystallized as orange red prisms. Yield, 4.7 g (68 %). The compound is stable, readily soluble in chloroform and fairly soluble in warm methanol.

The methanethiosulphonate group possesses pronounced pseudohalogen properties, the nucleophilic reactivity of the anion, in displacements on divalent sulphur, selenium and tellurium, being lower than that of aromatic thiosulphonate, thiosulphate, and thiocarbonyl anions [2,6]. Accordingly, such anions react with the tellurenyl methanethiosulphonate to displace the methanethiosulphonate group. In methanol solutions potassium benzenethiosulphonate reacts as follows:

$$\mathrm{ArTeS_2O_2CH_3 + C_6H_5SO_2S^- = ArTeS_2O_2C_6H_5 + CH_3SO_2S^-}$$

The resulting *p*-methoxybenzenetellurenyl benzenethiosulphonate forms stable, light orange red crystals, readily soluble in warm chloroform, sparingly soluble in ether.

The tellurenyl thiosulphonates react with aqueous sodium thiosulphate to produce aqueous solutions of a compound, presumably sodium *p*-methoxybenzenetellurenyl thiosulphate, having in dilute solutions a greenish yellow colour and in more concentrated solutions a reddish colour (R = methyl or phenyl):

$$\mathrm{ArTeS_2O_2R + S_2O_3^{--} = ArTeS_2O_3^- + RSO_2S}$$

The tellurenyl thiosulphate solutions react with aqueous potassium methylxanthate to give *p*-methoxybenzenetellurenyl methylxanthate:

$$\mathrm{ArTeS_2O_3^- + CH_3OCS_2^- = ArTeS_2COCH_3 + S_2O_3^{--}}$$

The same compound was obtained directly from the tellurenyl thiosulphonates on triturating these with an excess of potassium methylxanthate suspended in warm ether. On filtering, and partial evaporation of the ether, the tellurenyl methylxanthate crystallized as stable, orange red plates or prisms.

Compound (Ar = *p*-anisyl)	M.p. °C	% Te Calc.	% Te Found
$\mathrm{ArTeS_2O_2CH_3}$	107	36.9	37.1
$\mathrm{ArTeS_2O_2C_6H_5}$	110	31.3	31.4
$\mathrm{ArTeS_2COCH_3}$	78	37.3	37.4

Further researches on tellurenyl pseudohalides are being made as part of a program for the study of the relative electrophilic reactivities of divalent sulphur, selenium and tellurium.

The author is indebted to *Styret for Nansenfondet og de dermed forbundne fond* for a grant.

1. Kharasch, N., Potempa, S. J., and Wehrmeister, H. L. *Chem. Reviews* **39** (1946) 269.
2. Foss, O. *Acta Chem. Scand.* **1** (1947) 307.
3. Foss, O. *J. Am. Chem. Soc.* **69** (1947) 2236.
4. Rheinboldt, H., and Perrier, M. *Bull. Soc. Chim. France* **17** (1950) 245.
5. Morgan, G. T., and Kellett, R. E. *J. Chem. Soc.* (1926) 1080.
6. Foss, O. *Sjunde nordiska kemistmötet* (1951) 117, Helsingfors.