

tion of solid particles within microscopic dimensions. But in a study on crystallization of calcium fluoride, Tovborg Jensen⁴ found that a comparatively small number of solid particles (10^8 per cm^3) appear, seemingly formed from an approximately equal number of crystal nuclei by capture of ions from the solution and not by coalescence of a much larger number of nuclei. In the case of calcium fluoride there would not be one solid particle available per cell of radius $R = 10^{-3}$ cm ($10^8 \frac{4}{3} \pi R^3 = 0.4$) and even if in the case of calcium phosphate the number of nuclei per cm^3 exceeded 10^8 by several powers of ten it would be hard to imagine that finer cell structures could come out by well defined local precipitation.

The question therefore arises whether spontaneous precipitation ever occurs in the Gomori system. It seems more likely that precipitation is always induced either by pre-formed crystal nuclei or by other structure elements so that the deposits obtained are determined not only by the localization of enzymic sites but also by the localization of "precipitation centers". Recent experiments⁵ confirm this view.

A more detailed treatment of the problem including aspects such as the distribution of phosphate within the sites themselves and the possibility of wandering of primary deposits of calcium phosphate toward sources of phosphate ions so as to give an increasingly correct picture, will appear in the *Acta Medica Scandinavica*.

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1. Gomori G. *Proc. Soc. Exptl. Biol. Med.* **42** (1939) 23.
2. Takamatsu, H. *Trans. Soc. Path. Japon.* **29** (1939) 492.
3. Reis, J. L. *Biochem. J.* **48** (1951) 548.
4. Tovborg Jensen, A. *Z. physik. Chem.* **A 180** (1937) 93.
5. Novikoff, A. B. *Science* **113** (1951) 320.

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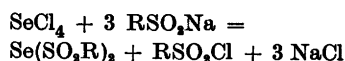
Aromatic Seleno- and Telluropolythionic Compounds

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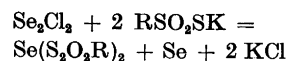
This note gives a preliminary report on three new types of benzene- and *p*-toluenesulphonyl derivatives, *viz.*, selenium disulphates, $\text{Se}(\text{SO}_2\text{R})_2$, and thiosulphonates of divalent selenium and tellurium, $\text{Se}(\text{S}_2\text{O}_2\text{R})_2$ and $\text{Te}(\text{S}_2\text{O}_2\text{R})_2$. The corresponding methanethiosulphonates have been described recently¹, whereas benzene- and *p*-toluenesulphates and -thiosulphonates of divalent sulphur, $\text{S}(\text{SO}_2\text{R})_2$ and $\text{S}(\text{S}_2\text{O}_2\text{R})_2$, are of older date^{2, 3}.

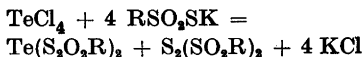
The selenium disulphates were prepared from finely powdered, dry sodium benzene- or *p*-toluenesulphate, suspended in dry benzene or ether, and selenium tetrachloride:



The compounds also result, in equally smooth reactions, if selenium oxychloride, SeOCl_2 , is used instead of selenium tetrachloride. Furthermore, they occur as products, beside triselenium disulphates, when diselenium dichloride reacts with the sodium sulphates suspended in dry ether⁴.

The thiosulphonates of divalent selenium were got from diselenium dichloride and dry, powdered potassium benzene- or *p*-toluenethiosulphonate suspended in dry ether, and the thiosulphonates of divalent tellurium were obtained from the same salts, suspended in dry, ethanol-free chloroform, and tellurium tetrachloride:





The thiosulphonates of divalent selenium are pale greenish, and so are the selenium disulphinates, whereas the thiosulphonates of divalent tellurium are yellow. When kept in a dry, neutral atmosphere, shielded from light, the crystals remain unchanged for months.

In the following, the symbols Bs and Ts denote the benzenesulphonyl and *p*-toluenesulphonyl group, respectively.

Compound	M.p., °C (preheated bath, dec.)	% Se or Te	
		Calc.	Found
SeBs ₂	132–3	21.85	21.94
SeTs ₂	121–2	20.29	20.16
Se(SBs) ₂	152–4	19.00	19.04
Se(STs) ₂	Ca. 200	17.41	17.53
Te(SBs) ₂	Ca. 170	26.91	26.73
Te(STs) ₂	Ca. 215	25.41	25.28

The crystals of the selenium disulphinates, SeBs₂ and SeTs₂, are monoclinic prismatic, and are isomorphous with those of the corresponding sulphur derivatives, the crystal structure of which was described recently by Mathieson and Robertson⁵.

The unit cells and space group of the benzene- and *p*-toluenethiosulphonates of divalent sulphur, S(SBs)₂ and S(STs)₂, have been reported by Dawson, Mathieson and Robertson⁶. The crystals are tetragonal trapezohedral. The crystals of the following four compounds are isomorphous with those of the sulphur thiosulphonates: Selenium di(benzenethiosulphonate), selenium and tellurium di(*p*-toluenethiosulphonate), and triselenium di(*p*-toluenesulphinate), *i.e.*, Se(SBs)₂, Se(STs)₂, Te(STs)₂, and Se₃Ts₂. Tellurium di(benzenethiosulphonate), Te(SBs)₂, is orthorhombic, whereas triselenium di(benzenesulphinate), Se₃Bs₂, is triclinic.

Work on the crystal structure of the *p*-toluene compounds, Se(STs)₂, Te(STs)₂ and Se₃Ts₂, is in progress in this Institute.

1. Foss, O. *Acta Chem. Scand.* **5** (1951) 115.
2. Otto, R., and Troeger, J. *Ber.* **24** (1891) 1125.
3. Troeger, J., and Hornung, V. *J. prakt. Chem.* [2] **60** (1899) 113.
4. Foss, O. *Acta Chem. Scand.* **4** (1950) 1499.
5. Mathieson, A. McL., and Robertson, J. M. *J. Chem. Soc.* (1949) 724.
6. Dawson, I. M., Mathieson, A. McL., and Robertson, J. M. *J. Chem. Soc.* (1948) 322.

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Explosions in the Preparation of Diethylaminoethyl Dinitrate

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In the preparation of diethylaminoethyl dinitrate, O₂NO · CH₂ · CH₂ · N(C₂H₅)₂, HNO³ according to the directions given by Barbière¹, we have experienced several very violent explosions.

Hence we feel compelled to call attention to the potential danger of this compound.

The procedure as described by Barbière (*l.c.*) calls for addition of 23.4 g of diethylaminoethanol to 75.6 g of fuming nitric acid under stirring in an ice bath and evaporating excess nitric acid *in vacuo* being careful not to let the bath temperature exceed 40° C.

When most of the nitric acid had evaporated our mixture invariably exploded. This explosion could not be prevented by nitrating in the presence of urea.

The following alternative procedure is suggested: After addition of the reagents,