

*The selenium and tellurium contents of sulphur from
Krisuvik, Iceland.*¹

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THE deposits of sulphur at Krisuvik, Iceland, have long been known. They were described geologically in detail by Vincent² who also discussed their probable origin, but gave no chemical analyses other than rough determinations of the percentages of sulphur and matrix in various samples. From the flame spectrum Vincent thought that thallium might be present.

Apart from popular descriptions by travellers and casual references in guide-books, little attention appears to have been given in literature to these deposits in more recent years. Sporadic attempts at working the sulphur economically were made by the Danish Government during the eighteenth century and later by a Danish merchant round 1832. These were abandoned, however, owing to cost of transport. There is little likelihood of their renewal in the immediate future unless the deposits are found to contain some other element(s) of sufficient market value.

In view of the increasing commercial applications of selenium and tellurium it seemed of interest to ascertain if either or both of these elements were present in appreciable quantities in the Krisuvik deposits. The results were not encouraging but may be worth placing on record.

About 1 kilogram of the crude mineral was obtained through the good offices of Mr. Stefan Stefánsson of Reykjavik and 50 gram lots were boiled stepwise under reflux with carbon disulphide until all soluble matter had been extracted. The insoluble siliceous residue (3.2 %) and the soluble portion recovered by distillation were examined spectrographically with a Hilger quartz spectrometer.

Both portions showed lines due to selenium and tellurium, whilst the insoluble residue gave also strong lines due to titanium, weaker nickel lines, and lines due to iron, calcium, and barium.

The presence of the nickel was subsequently confirmed by the

¹ A preliminary note appeared in *Nature*, London, 1939, vol. 143, p. 762.

² C. W. Vincent, *Chem. News*, 1873, vol. 27, pp. 111, 126.

dimethylglyoxime test, that of titanium by the peroxide test, the metal being estimated colorimetrically by the same reaction. Tellurium was confirmed qualitatively by precipitation as minute rods with quinol and concentrated sulphuric acid, and examination under the microscope.¹ It was quantitatively determined by digestion of the extracted sulphur and of the insoluble residue respectively with concentrated nitric acid, extraction of the selenium and tellurium dioxides with potash, precipitation of the former from the acidified solution with sodium sulphite, and colorimetric comparison of the filtered solution after reduction with mercurous chloride against a suitable standard.²

Selenium was confirmed qualitatively by the thio-urea test³ and subsequently estimated by oxidation of the extracted sulphur and residue respectively with nitric acid to selenious acid. Excess nitric acid was removed by evaporation with hydrochloric acid to prevent nitration of the pyrrole, which latter was then added, together with orthophosphoric acid and ferric chloride. The blue colour was compared with a standard.⁴ The tellurium was twenty times as abundant as the selenium in both the extracted sulphur and the insoluble residue.

The results expressed in parts per million were as follows:

	Extracted sulphur. p.p.m.	Insoluble residue. p.p.m.	Crude mineral. p.p.m.
Titanium	—	3750	120
Selenium	12.5	200	18.9
Tellurium	250	4000	378

¹ P. C. Putnam, E. J. Roberts, and D. H. Selchow, *Amer. Journ. Sci.*, 1928, ser. 5, vol. 15, p. 253.

² G. G. Pierson, *Ind. Eng. Chem., Anal. Edit.*, 1934, vol. 6, p. 437.

³ M. H. Evans, *Amer. Min.*, 1937, vol. 22, p. 1128.

⁴ R. Berg and M. Teitelbaum, *Mikrochem. Emich Festschr.*, 1930, p. 23.