

Thermolysis of Selenides of Gold, Tin, Iridium, Indium, Molybdenum, Cadmium, Bismuth, Lead and Zinc precipitated by Sodium Selenide

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With 3 Figures

Summary

The thermolysis of precipitates formed by gold, tin, iridium, indium, molybdenum, cadmium, bismuth, lead and zinc with the sodium selenide reagent has been studied.

It has been shown already in two papers¹⁾²⁾ published from this laboratory that sodium selenide can be used for precipitating certain elements as selenides and estimating them in this form. The estimation requires the use of a special apparatus designed to prevent contact of sodium selenide with the oxygen of the atmosphere and to keep the solutions at a low temperature between 5–10 °C. The precipitated selenides behave more or less like sulphides, but can be filtered and weighed more easily.

It was considered desirable to make a thermogravimetric study of those selenides which can be used for the estimation of elements in order to gain definite information regarding the correct conditions for weighing the precipitates. The selenides of arsenic, antimony, rhenium, mercury, silver, platinum, palladium, ruthenium and rhodium have already been studied thermogravimetrically¹⁾. The present paper embodies the results obtained with the selenides of iridium, indium, molybdenum, cadmium, bismuth, lead, zinc, gold and tin.

Experimental

The apparatus used for the purpose was the standard Stanton Thermobalance. It was not possible to attain true programme control below 120 °C. To obtain a linear rate of heating with time automatically the high/low switches were used, high at 2 or 3 and low at 1, according to the maximum temperature 800° or 900 °C used for the observation. The rate of heating was $5.3 \pm 0.2^\circ\text{C}$ per minute and the speed of the chart was 6'' per hour. As the

¹⁾ I. K. TAIMNI and R. RAKSHPAL, *Anal. Chim. Acta* **25**, 438–447 (1961).

²⁾ I. K. TAIMNI and R. RAKSHPAL, *Communicated*.

slope altered abruptly at the change-over point it was necessary to start the cam motor 10–15 minutes after the furnace was switched on. The thermograms shown in these cases were obtained by plotting different points of the original graph on X and Y axes (temperature and weight respectively).

The reagents used and the methods followed in preparing the precipitates were the same as reported earlier. The precipitates taken for thermolysis were also in exactly the same condition as the corresponding precipitates just before they were weighed in estimations of the selenides.

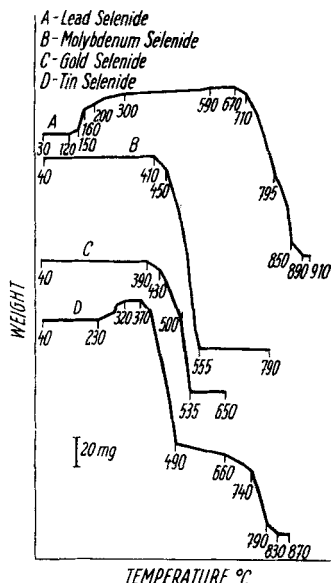


Fig. 1. Thermal decomposition curves of Selenides precipitated by Sodium Selenide reagent

Tin Selenide

The air-dried precipitate of tin selenide¹⁾ gave a horizontal level upto 230°C, gained in weight from 230°C to 320°C and a horizontal was again obtained between 320°C and 370°C. From 370°C there was continuous loss in weight upto 830°C, after which there was a horizontal again corresponding to SnO₂ (Fig. 1).

The results are given in Table 1.

Gold Selenide

The air-dried precipitate of gold selenide¹⁾ gave a horizontal level upto 390°C. There was a rapid loss in weight from 390°C which continued upto 535°C. From 535 to 650°C there was a horizontal which corresponded to the weight of the metal (Fig. 1).

The results are given in Table 2.

Table 1

Weight of SnSe ₂ taken	Weight of SnO ₂ calculated	Weight of SnO ₂ from graph (830–870°C)
310 mg	169 mg	167 mg
577 mg	315 mg	312 mg

Table 2

Weight of gold selenide taken	Weight of gold calculated	Weight of gold from graph (535–650°C)
198 mg	110 mg	110 mg
415 mg	230 mg	229 mg

Iridium Selenide

The air-dried precipitate of iridium selenide gave a horizontal upto 240 °C. A rapid loss in weight was observed from 240 to 540 °C. It gave another horizontal from 540 to 650 °C (Fig. 2).

Indium Selenide

The air-dried precipitate of indium selenide gave a horizontal upto 390 °C. It lost in weight continuously from 390 to 770 °C. A horizontal was obtained between 770 °C and 800 °C (Fig. 3).

Molybdenum Selenide

The air-dried precipitate of molybdenum selenide showed a horizontal level upto 410 °C and from 410 °C there was a rapid loss upto 555 °C, after which there was a horizontal level corresponding to MoO₃, the residue began to volatilize at 800 °C (Fig. 1).

The results are given in Table 3.

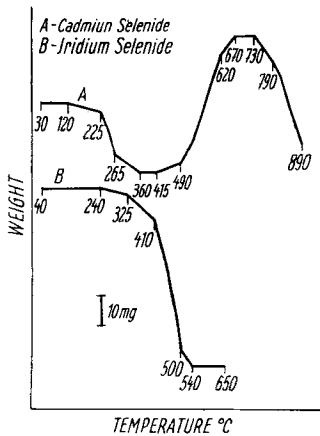


Fig. 2. Thermal decomposition curves of Selenides precipitated by Sodium Selenide reagent

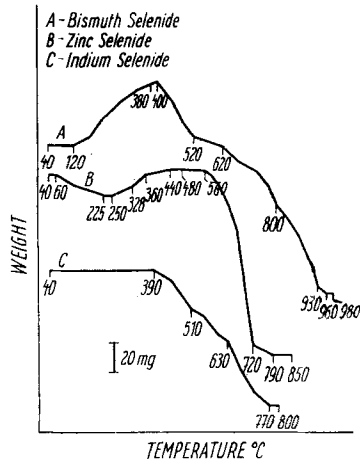


Fig. 3. Thermal decomposition curves of Selenide precipitated by Sodium Selenide reagent

Table 3

Weight of MoSe ₃ taken	Weight of MoO ₃ calculated	Weight of MoO ₃ from graph (555—790 °C)
176 mg	76 mg	67 mg
288 mg	124 mg	115 mg

Cadmium Selenide

The air-dried precipitate of cadmium selenide gave a horizontal upto 120 °C. It lost in weight from 120 to 360 °C and a horizontal was obtained between 360 and 415 °C. There was gain in weight between 415 and 670 °C. A horizontal was obtained from 670 to 730 °C. From 730 to 890 °C there was a continuous loss in weight. No horizontal was obtained even after this temperature (Fig. 2).

Bismuth Selenide

The air-dried precipitate of bismuth selenide gave a horizontal upto 120°C. It gained in weight from 120 to 380°C after which a horizontal was obtained between 380°C and 400°C. The precipitate lost in weight continuously from 400 to 960°C and then there was a horizontal again from 960 to 980°C (Fig. 3).

Lead Selenide

The air-dried precipitate of lead selenide gave a horizontal upto 120°C. There was gain in weight from 120 to 590°C and again a horizontal between 590 and 670°C. It then lost in weight continuously upto 890°C. A horizontal was then obtained from 890 to 910°C (Fig. 1).

Zinc Selenide

The air-dried precipitate of zinc selenide gave a horizontal upto 60°C. From 60 to 225°C there was a loss in weight and then a horizontal was obtained between 225 and 250°C. It gained in weight from 250 to 440°C and then a horizontal was obtained again between 440 and 480°C. There was abrupt loss in weight at 480°C which continued upto 790°C. A horizontal was obtained from 790 to 850°C which corresponded to the weight of ZnO (Fig. 3).

The results are given in Table 4.

Table 4

Weight of ZnSe taken	Weight of ZnO calculated	Weight of ZnO from graph (790–850°C)
260 mg	147 mg	145 mg
278 mg	157 mg	154 mg

These results obtained during the thermogravimetric study of the various selenides indicate a close resemblance with the behaviour of corresponding sulphides i. e. (i) the selenides of tetravalent tin, hexavalent molybdenum and zinc are finally converted into their oxides in a way similar to the corresponding sulphides³⁾, (ii) the selenide of gold leaves a residue of the metal, just like the sulphide and (iii) the selenides of iridium, indium, cadmium, bismuth and lead are finally converted into forms, which do not correspond to any particular chemical formula and in this respect they are similar to the sulphides³⁾.

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³⁾ I. K. TEJANI and S. N. TANDON, *Anal. Chim. Acta* **22** 34–37, 553–557 (1960).

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