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Methods for chemical analysis of ruthenium determination of lead, iron, nickel, aluminum, copper, silver, gold, platinum, palladium, rhodium, iridium and silicon in ruthenium powder by glow discharge mass spectrometry

钌粉化学分析方法 铅、铁、镍、铝、铜、银、金、铂、铱、钯、 铑、硅量的测定 辉光放电质谱法

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Methods for chemical analysis of ruthenium determination of lead, iron, nickel, aluminum, copper, silver, gold, platinum, palladium, rhodium, iridium and silicon in ruthenium powder by glow discharge mass spectrometry

1 Scope

This Standard specifies the method for the determination of lead, iron, nickel, aluminum, copper, silver, gold, platinum, palladium, rhodium, iridium and silicon in ruthenium powder.

This Standard applies to the determination of lead, iron, nickel, aluminum, copper, silver, gold, platinum, palladium, rhodium, iridium and silicon in ruthenium powder, of which the determination ranges are shown in Table 1.

2 Method summary

Prepare the sample by powder-pressing method. Use the sample as the cathode of the glow discharge power supply. The atoms of the sample (cathode) are peeled off from the sample under the impact of argon ions (cathode sputtering), enter the plasma, and become positive ions by electron collision and Penning collision. Positive ions are accelerated through the outlet of the ion source and then enter the mass spectrometer, which analyzes according to the charge-to-mass ratio and ion intensity.

3 Reagents and materials

- **3.1** Absolute ethanol (analytical reagent).
- **3.2** Pure argon.
- **3.3** High-purity argon.

4 Instruments

- **4.1** Pressing machine, whose rated pressure is not less than 15 MPa.
- **4.2** Glow discharge mass spectrometer, whose working parameters are shown in Appendix A

In order to ensure the working state of the instrument, the following conditions must be met:

- -- The high vacuum of the instrument analyzer must be less than 5×10^{-7} mbar, and the fore vacuum must be less than 1×10^{-3} mbar.
- -- The heat transfer between the sample and the sample holder must be good, and the cooling temperature shall be set at 15 °C.
- -- The instrument shall be in a state of good mass calibration and Faraday detector calibration before testing.
- -- Discharge parameters, gas pressure and lens voltage, etc. can be adjusted to obtain good peak shape, resolution and 102 Ru matrix signal of not less than 5×10^7 cps.

5 Sample preparation

Use absolute ethanol (3.1) to wash and air-dry the part in contact with the sample in the steel ring of the tablet-producing mold of the pressing machine in advance; then, weigh $5 \text{ g} \sim 10 \text{ g}$ of the sample and place it in the steel ring of the tablet-producing mold; start the pressing machine, and press at a pressure of 15 MPa for at least 3 min; take out the tablet.

6 Analysis steps

Load the sample into the sample holder on the glow discharge mass spectrometer; adopt an appropriate instrument working condition, to carry out sample pre-sputtering first; perform sample analysis after the signal of the element to be measured is stabilized.

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