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Determination of plutonium in pure plutonium nitrate solutions — Gravimetric method

Détermination du plutonium dans les solutions de nitrate de plutonium pur — Méthode gravimétrique

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Foreword

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Determination of plutonium in pure plutonium nitrate solutions — Gravimetric method

1 Scope and field of application

This International Standard specifies a precise and accurate gravimetric method for determining the concentration of plutonium in pure plutonium nitrate solutions and reference solutions, containing between 100 and 300 g of plutonium per litre, in a nitric acid medium.

2 Principle

Treatment of a weighed portion of the plutonium nitrate solution with sulfuric acid and evaporation to dryness. Decomposition of the plutonium sulfate which is formed to oxide by heating in air. Ignition in air of the oxide at 1 200 to 1 250 °C and weighing as stoichiometric plutonium dioxide, which is stable and non-hygroscopic.

Calculation of the plutonium content using a gravimetric conversion factor which depends slightly on the isotopic composition of the plutonium. If the latter is not known, it shall be measured, usually by mass spectrometry.

3 Interferences

All non-volatile impurities interfere. If the impurity content is greater than 0,05 %, a correction shall be applied. If this correction exceeds about 0,5 %, the accuracy of the impurity measurements may limit the overall performance of the method. There is no interference from up to at least 1 000 ppm of phosphorus (present as phosphate) which is lost during the sulfuric acid treatment. The chloride and fluoride contents of the sample should not exceed 25 ppm.

4 Reagents

4.1 Sulfuric acid, solution at 50 % (V/V).

While stirring, cautiously add 500 ml of analytical reagent quality sulfuric acid (ϱ = 1,84 g/ml) to 500 ml of cold distilled or deionized water. Allow to cool.

5 Apparatus

Normal laboratory equipment for a plutonium laboratory, and

5.1 Platinum crucibles, approximately 8 ml in capacity.

5.2 Polythene weighing burettes.

5.3 Furnace, in an air-atmosphere glove box, with a temperature range from 300 to 1 250 °C.

5.4 Semi-micro balance, in an air-atmosphere glove box, to weigh 25 g with a readability of ± 0,1 mg; the balance and weights should be certified or calibrated to ± 0,05 mg.

5.5 Radiant heater, in a glove box.

6 Procedure

6.1 Ignite a clean crucible (5.1) for 1 h at 1 200 to 1 250 °C. Cool in a desiccator for 20 min and then in the balance (5.4) for 5 min; weigh to within ± 0,1 mg, repeating the ignition until the mass remains constant to within 0,1 mg.

6.2 Weigh out 1 to 2 g of sample solution containing 0,2 to 0,4 g plutonium from a polythene weighing burette (5.2) into the crucible. Record the masses (before sample delivery m_2, after sample delivery m_3) to within ± 0,1 mg.

NOTE — In order to avoid errors due to thermal effects, the weighing burette shall be allowed to adjust to the balance temperature before each weighing.

6.3 Add 1,0 ml of the sulfuric acid solution (4.1) to the crucible and swirl gently to mix.

6.4 Evaporate the solution under a radiant heater (5.5), by heating gently until sulfuric acid fumes are evolved and then more strongly until a dry residue has been obtained and fuming has practically ceased.

NOTE — Plutonium nitrate is converted to plutonium sulfate as the nitrate compound spatters on evaporation to dryness.

6.5 Without delay, transfer the crucible and dried plutonium sulfate to the furnace (5.3) set at about 300 °C. Maintain this temperature for about 15 min. Then raise the temperature by 5 to 10 °C per minute to about 850 °C at which temperature the plutonium sulfate will have decomposed.

6.6 Increase the temperature to 1 200 to 1 250 °C and ignite at this temperature for 1 h.