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Nitric acid feed solutions from reprocessing plants — Spectrophotometric determination of plutonium after oxidation to plutonium(VI)

Solutions nitriques d'entrée des usines de retraitement — Dosage spectrophotométrique du plutonium après oxydation en plutonium(VI)

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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Nitric acid feed solutions from reprocessing plants — Spectrophotometric determination of plutonium after oxidation to plutonium(VI)

1 Scope

This International Standard specifies an analytical method for determining the plutonium concentration of nitric acid feed solutions from reprocessing plants. The method is applicable, without interference, in the presence of numerous cations, it is applicable to test portions containing between 0,5 mg and 2,5 mg of plutonium.

2 Principle

The nitrate concentration is adjusted to 3 mol/l with nitric acid.[1)]

Plutonium is oxidized to the hexavalent state either with cerium(IV) or with argentic oxide, in which case the excess is destroyed by adding sulfamic acid.

The volume is adjusted with 3 mol/l nitric acid.

The optical density of the PuO_2^{2+} absorption peak at 831 nm is measured on a spectrophotometer. The result is based on a calibration established under the same conditions.

The procedure uses 3 mol/l nitric acid as this permits either cerium(IV) or argentic oxide to be used as oxidant and is convenient for most applications. It is acceptable to use cerium(IV) as oxidant at lower acidities and argentic oxide as oxidant at higher acidities provided that the concentration of the nitric acid used for calibration is similarly adjusted.

3 Chemical conditions

3.1 Stability of Pu(VI)

Plutonium(VI) is very stable under the operating conditions of the method over the range 2 mol/l $< c(H^+) <$ 5 mol/l.

3.2 Rate of oxidation of Pu(IV) to Pu(VI)

The rate of oxidation by cerium(IV) decreases as the acidity increases. With the reagent quantities stated in the method, the oxidation is complete in 5 min in 2 mol/l or 3 mol/l nitric acid.

With argentic oxide, the oxidation is very rapid, noticeably faster than with cerium(IV).

3.3 Destruction of the excess oxidant

With cerium(IV) the excess reagent does not interfere and need not be destroyed.

With argentic oxide as oxidant, the excess reagent shall be destroyed by reaction with a small excess of sulfamic acid or by heating (to about 80 °C).

3.4 Molar extinction coefficient of Pu(VI)

The absorbance falls

— when the nitrate ion concentration is increased. The decrease in absorbance becomes more rapid at higher nitrate levels. At about 3 mol/l nitrate, an increase of 0,1 mol/l in the total nitrate content causes a decrease of about 0,7 % in the absorbance;

1) This acidity has been chosen in order to avoid dilution with water due to the fact that the solutions to be measured are wet precipitates with a nitrate ion concentration of 3 mol/l. Dilution with water would risk causing plutonium hydrolysis.