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Nuclear fuel technology — Determination of milligram amounts of plutonium in nitric acid solutions — Potentiometric titration with potassium dichromate after oxidation by Ce(IV) and reduction by Fe(II)

Technologie du combustible nucléaire — Détermination de quelques milligrammes de plutonium dans des solutions d'acide nitrique — Titrage potentiométrique avec le dichromate de potassium, après oxydation par le Ce(IV) et réduction par le Fe(II)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 8298 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

This second edition cancels and replaces the first edition (ISO 8298:1987), which has been technically revised.

Nuclear fuel technology — Determination of milligram amounts of plutonium in nitric acid solutions — Potentiometric titration with potassium dichromate after oxidation by Ce(IV) and reduction by Fe(II)

1 Scope

1.1 This International Standard describes a precise and accurate analytical method for determining 1 mg to 5 mg of plutonium per millilitre in nitric acid solutions.

1.2 The method is very selective for plutonium. It is suitable for the direct determination of plutonium in materials ranging from pure product solutions, to solutions of mixed nuclear materials with a uranium/plutonium ratio up to 20:1. However, potential application to the assay of plutonium in solutions of irradiated nuclear fuels and solutions of mixed nuclear materials with uranium/plutonium ratios of 20:1 to 33:1 has not yet been documented.

1.3 The method recommends that the aliquot be weighed and that the titration burettes be calibrated gravimetrically in order to obtain adequate precision and accuracy. This does not preclude using any alternative technique which can be shown to give an equivalent accuracy.

1.4 As the reproducibility of the reaction conditions is important to maintain good performance, extensive automatization of the procedure is beneficial.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 10980, *Validation of the strength of reference solutions used for measuring concentrations.*

3 Principle

3.1 Plutonium in 1 mol/l nitric acid solution is oxidized to plutonium(VI) using an excess of cerium(IV).

At this acidity 0,2 mmol of cerium(IV) will oxidize more than 5 mg of plutonium(IV) in less than 5 min. Amidosulfonic acid is added to prevent nitrite-induced side-reactions and aluminium(III) to complex the fluoride ions contained in the plutonium sample solution.

The main reactions can be represented as follows:

$$Pu^{4+} + 2Ce^{4+} + 2H_2O \Leftrightarrow PuO_2^{2+} + 2Ce^{3+} + 4H^+ \tag{1}$$

$$Pu^{3+} + 3Ce^{4+} + 2H_2O \Leftrightarrow PuO_2^{2+} + 3Ce^{3+} + 4H^+ \tag{2}$$

The excess of cerium(IV) is reduced with sodium arsenite, in accordance with the following reaction, catalysed by osmium tetroxide with a slight excess of arsenite being added:

$$AsO_2^- + 2Ce^{4+} + H_2O \Leftrightarrow AsO_3^- + 2Ce^{3+} + 2H^+ \qquad (3)$$

The excess of arsenite is oxidized by addition of a slight excess of potassium permanganate solution:

$$5AsO_2^- + 2MnO_4^- + 6H^+ \Leftrightarrow 5AsO_3^- + 2Mn^{2+} + 3H_2O \qquad (4)$$

The excess of permanganate is reduced by addition of oxalic acid:

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \Leftrightarrow 2Mn^{2+} + 10CO_2 + 8H_2O \qquad (5)$$

A small excess of oxalic acid does not interfere in the subsequent plutonium determination.

These reduction and oxidation steps are followed potentiometrically and leave the plutonium in the hexavalent state.

In all these steps, it is necessary to choose carefully the amounts of reagents added, the potentials to be reached and the waiting times in order to minimize possible biases resulting from side-reactions.

3.2 The plutonium(VI) is reduced with a measured volume of standardized iron(II) sulfate solution in 3 mol/l sulfuric acid, the iron(II) sulfate being added in excess of that required for a complete reduction of the plutonium to plutonium(IV).

The excess of iron(II) is back-titrated, together with any plutonium(III) formed, with a standardized potassium dichromate solution.

The reactions involved can be schematized as follows:

$$2Fe^{2+} + PuO_2^{2+} + 4H^+ \Leftrightarrow 2Fe^{3+} + Pu^{4+} + 2H_2O \qquad (6)$$

$$Fe^{2+} + Pu^{4+} \Leftrightarrow Fe^{3+} + Pu^{3+} \qquad (7)$$

$$Cr_2O_7^{2-} + 6Pu^{3+} + 14H^+ \Leftrightarrow 2Cr^{3+} + 6Pu^{4+} + 7H_2O \qquad (8)$$

$$Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \Leftrightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O \qquad (9)$$

3.3 The dichromate solution and the iron(II) sulfate solution are standardized against a standard reference solution of plutonium prepared from a plutonium metal certified to 0,05 % or better.

3.4 The accuracy of the titre of the standard reference solution of plutonium is verified in accordance with ISO 10980 by titration against a standard reference solution of dichromate prepared from NIST SRM 136.

3.5 The plutonium content is calculated from the amount of iron(II) needed to reduce the plutonium(VI) to plutonium(IV).

4 Interference

4.1 1 mg of iron does not interfere. Amounts of Fe(III) greater than 5 mg influence the kinetics of the oxalic acid/permanganate reaction.

4.2 Rubidium, caesium, strontium, barium (80 µg each or 320 µg altogether) or lanthanides (lanthanum, cerium(III), praseodymium, neodymium, samarium, europium, gadolinium (80 µg of each or 560 µg altogether) and 147 µg of yttrium(III) do not interfere with the determination of 1 mg to 5 mg of plutonium.

4.3 No interference has been detected with the following elements up to the amounts given: 41 µg of zirconium, 47 µg of palladium(III), 41 µg of ruthenium(III), 46 µg of tellurium, 40 µg of molybdenum and 41 µg of tin(IV).

4.4 Up to 0,07 mmol of fluorosilicate and 0,7 mmol of fluoride do not interfere if 2,5 mmol to 3 mmol of aluminium(III) are added. 3 mmol of sulfuric acid can be tolerated in the plutonium aliquot without any interference. Up to 0,4 mg of chromium(VI), 0,3 mg of chromium(III), 0,3 mg of manganese(II) and 0,2 mg of gallium(III) do not interfere.

4.5 0,5 mmol of nitrite causes no statistically significant bias when present at the moment of oxidation of the plutonium. Indeed, most of the nitrites should be decomposed by the amidosulfonic acid which is contained in the nitric acid mixture used for the dilution of the plutonium aliquot.

4.6 However, adding more than about 0,5 mmol of amidosulfonic acid with the nitric acid mixture causes a positive bias due to incomplete reduction of the permanganate by the oxalic acid.

4.7 About 40 µg of iodate leads to a significant bias of about + 0,2 %.

4.8 Vanadium(IV) and vanadium(V) interfere almost quantitatively.

4.9 The neptunium present is titrated together with the plutonium, involving the exchange of approximately 1 mole of electrons per mole. For neptunium/plutonium ratios up to 0,01, an empirical formula can be established and used to correct for this interference, provided the neptunium content is known or measured to an accuracy of ± 10 %. The following is an example of such an empirical correction equation:

$$T = F/[1 + k(\mathrm{Np/Pu})]$$

where

F is the "found" plutonium content, expressed in arbitrary units (e.g. mg, mass percent);

T is the "taken" or true plutonium content, expressed in the same units as F;

k is the mass fraction of neptunium, determined by titration with plutonium;

Np/Pu is the mass ratio of neptunium to plutonium in the sample.

The coefficient k is close to 0,57 and has to be measured experimentally. It is sufficiently reproducible provided that the reagents used in clause 7 are added carefully in accordance with the procedure, complying in particular with the potentials and timing specified.

4.10 The effect of americium remains to be studied.

5 Reagents

Use only reagents of recognized analytical grade and distilled or deionized water.

5.1 **0,5 mol/l solution of ammonium hexanitratocerate,** $(NH_4)_2[Ce(NO_3)_6]$, in nitric acid.

Dissolve 27,5 g of ammonium hexanitratocerate in a 1 mol/l nitric acid solution and dilute to 100 ml using the same solution.

5.2 **Nitric acid mixture:** 1 mol/l nitric acid, 0,013 mol/l amidosulfonic acid, 0,002 5 mol/l iron(III) nitrate and 0,05 mol/l aluminium nitrate.

Add 70 ml of concentrated nitric acid (ρ = 1,40 kg/l) to 500 ml of water. Dissolve in this solution successively 1,26 g of amidosulfonic acid, 1,01 g of iron(III) nitrate nonahydrate and 18,75 g of aluminium nitrate nonahydrate and dilute with water to 1 l of solution.

5.3 **0,25 % (*m*/*V*) osmium tetroxide solution.**

WARNING — Osmium tetroxide is a volatile toxic chemical and the vapour attacks the eyes. It should only be handled in a fume cupboard or glove box, wearing safety glasses and surgeon's gloves.

Add progressively and while stirring 4 ml of concentrated sulfuric acid ($\rho = 1{,}84$ kg/l) to 36 ml of water in a glass beaker and allow to cool. Dissolve the content of a 0,1 g ampoule of osmic acid in this solution. Store the solution in a glass bottle with a glass stopper and do not use later than one month after preparation.

5.4 0,05‰ mol/l sodium arsenite solution.

Dissolve 6,5 g of sodium arsenite in 1 l of water.

5.5 0,04 mol/l and 0,004 mol/l potassium permanganate solutions.

5.5.1 Dissolve 0,63 g of potassium permanganate in 100 ml of water to obtain a 0,04 mol/l solution. Boil the solution gently for 15 min. Cool down to room temperature.

5.5.2 Before use, filter the necessary volume of 0,04 mol/l permanganate solution through a Whatman glass-fibre filter or through a plug of glass wool. Dilute 10 ml of the filtrate to 100 ml with water to obtain a 0,004 mol/l solution. Store the diluted solution in a glass bottle and do not use later than one month after preparation.

5.6 0,02 mol/l oxalic acid solution.

Dissolve 0,63 g of oxalic acid dihydrate in water and dilute to 250 ml with water.

5.7 3 mol/l sulfuric acid.

Cautiously, with stirring, add 167 ml of concentrated sulfuric acid ($\rho = 1{,}84$ kg/l) to 750 ml of water in a glass beaker, cool to room temperature, mix and dilute to 1 l with water.

5.8 $6{,}69 \times 10^{-3}$ mol/l iron(II) sulfate solution (^{239}Pu equivalent 0,8 g/kg of solution), in 3 mol/l sulfuric acid and 0,3 mol/l amidosulfonic acid.

Cautiously, with stirring, add 167 ml of concentrated sulfuric acid ($\rho = 1{,}84$ kg/l) to 750 ml of water in a glass beaker, cool to room temperature. Dissolve 29,1 g of amidosulfonic acid and 1,86 g of iron(II) sulfate heptahydrate in this solution and dilute to 1 l with water.

5.9 Potassium dichromate titrant solution (^{239}Pu equivalent 0,5 g/kg of solution).

Introduce 1,03 g of potassium dichromate into a 5 l glass flask, dissolve and dilute to 5 l with water. Standardize the dichromate solution by titrating it with at least five aliquots of the plutonium reference solution (5.10) in accordance with the procedure specified in 5.11 or a more accurate one. Express the titre as the ^{239}Pu equivalent in g/kg of solution.

5.10 Plutonium reference solution (see ISO 10980).

Dissolve an accurately known mass (m_0) of a certified plutonium metal (e.g. NBL CRM 126, CETAMA MP2 or UK/Pu1) in a 10:1 mixture of concentrated nitric acid ($\rho = 1{,}40$ kg/l) and 0,1 mol/l hydrofluoric acid. Heat gently to dissolve without losses of aerosols. After complete dissolution, continue to heat the open vessel for at least 1 h at about 90 °C to destroy and expel nitrous compounds, and then allow to cool to room temperature. Alternatively, the metal may be dissolved in 4 mol/l hydrochloric acid, which can be removed by fuming with nitric acid, if necessary, after the dissolution is completed. Dilute with the required volume of 2 mol/l nitric acid to obtain a solution containing about 2 mg Pu/ml in about 3 mol/l nitric acid. Homogenize, weigh to within 0,000 1 g (m_1) and store tightly stoppered till needed.

The strength of the solution, expressed as the Pu equivalent in g/kg of solution, is calculated using the following equation:

$$T = 0{,}01\, R\, (m_0/m_1)$$

where

R is the decay-corrected concentration of plutonium in the certified metal, as given in the certificate, in mas percent;

m_0 is the certified (or measured) mass of plutonium metal taken, in grams;

m_1 is the mass of the solution, in kilograms.

Measure again the gross mass before use, to verify that no significant evaporation has occurred: take aliquots containing the desired amount (1 mg to 5 mg) of plutonium and weigh them to within 0,000 1 g.

5.11 Potassium dichromate reference solution (^{239}Pu equivalent 0,5 g/kg of solution).

Weigh a clean and dry 5 l flask to within 0,001 g. Weigh about 1,03 g of dried NIST SRM 136 potassium dichromate certified reference material to within 0,000 1 g in a clean and dry beaker and dissolve it in water. Transfer the solution quantitatively into the tared flask and dilute to 5 l with water. Weigh the flask plus contents to within 0,001 g and homogenize well by shaking. Calculate the concentration of potassium dichromate in the solution (T_c) as the ^{239}Pu equivalent in g/kg of solution:

$$T_c = \frac{m_2}{m_3} \times \frac{3A}{M} \times \frac{P}{100}$$

where

m_2 is the mass of dichromate, in grams;

m_3 is the mass of the solution, in kilograms;

A is the atomic mass of ^{239}Pu (= 239,052);

M is the molecular mass of potassium dichromate (= 294,184 4);

P is the chemical purity of the dichromate standard (= 99,984 % for NIST SRM 136E), in mass percent.

Verify the titre of the plutonium reference solution against the potassium dichromate reference solution in accordance with ISO 10980, titrating at least five aliquots with the potassium dichromate reference solution using the procedure specified above or a more accurate one. Repeat this verification on at least one other day. The solution is acceptable if the calculated titre and the mean of the measured titres agree within 0,1 %. If not, repeat the preparation of the reference solutions.

6 Apparatus

Normal laboratory equipment for a laboratory analysing plutonium, plus the following:

6.1 High-impedance millivoltmeter, 100 MΩ input resistance, with a digital read-out capable of discriminating to 1 mV.

6.2 Burettes, capacity 1 ml and 10 ml, capable of delivering increments down to 4 µl, with non-return valves controlling the addition of the reagent into the titration cell through polyethylene tubes, and fitted with a labyrinth-type anti-diffusion tip. The burettes used for the addition of iron(II) sulfate solution and potassium dichromate titrant solution shall be calibrated gravimetrically.

6.3 Magnetic stirrer, with speed control and plastic-coated stirrer bar.

6.4 Temperature sensor, with digital read-out, to measure the temperature of the solutions of iron(II) sulfate (5.8) and potassium dichromate (5.9).

6.5 Balance, in a glove box or a shielded cell, with a readability of 0,000 01 g and an accuracy of 0,000 1 g or better.

6.6 Indicating electrode, Pt/Rh (90:10) wire, 1 mm diameter.

Check the performance of the electrode regularly. If its response begins to deteriorate, clean the electrode by immersing in boiling nitric acid ($\rho = 1{,}42$ kg/l). Rinse the electrode thoroughly with distilled water, and store in concentrated nitric acid between use.

6.7 Hg/HgSO$_4$ reference electrode, in saturated K_2SO_4, placed in a salt bridge filled with saturated K_2SO_4 and dipping in the titration solution.

If the use of mercury is not permitted, an Ag/AgCl reference electrode in 1 mol/l KCl can be used. The latter shall, however, be placed in a salt bridge filled with saturated K_2SO_4 to slow down the diffusion of chloride ions into the titration cell. Subtract 20 mV from all potentials specified in the procedure given in clause 7 if an Ag/AgCl reference electrode in 1 mol/l KCl is used.

7 Procedure

7.1 Transfer a 1 ml to 5 ml aliquot, containing between 1 mg and 5 mg of plutonium, of the sample solution into a clean, dry and tared vial, for instance, and measure the net mass of the aliquot to within 0,000 1 g (m_4).

Any alternative method of measuring the sample aliquot shall be shown to be accurate to better than 0,05 %. Determine whether the weighings need to be corrected for air buoyancy effects.

7.2 Transfer the sample aliquot quantitatively into a 100 ml beaker by rinsing the vial with 25 ml to 30 ml of nitric acid mixture (5.2) and add 0,4 ml of cerium(IV) solution (5.1). Stir gently for 5 min.

7.3 Add 0,1 ml of osmium tetroxide solution (5.3). Plunge the electrodes and the reagent delivery tips into the solution and start gentle stirring.

7.4 After 10 s, add sodium arsenite solution (5.4) until a potential of 310 mV to 340 mV is reached.

7.5 After 20 s, slightly increase the stirring rate and add 0,004 mol/l potassium permanganate solution (5.5.2) to reach a potential of 375 mV.

7.6 Add a preset volume of 0,1 ml of potassium permanganate solution (5.5.2) without delay. Keep stirring the solution for 2 min.

7.7 Add a preset volume of 0,12 ml of oxalic acid solution (5.6). Stir the solution for 3 min.

7.8 Add a preset volume of 3 ml of sulfuric acid (5.7).

7.9 Add iron(II) sulfate solution (5.8) to reach a potential of 115 mV, giving an approximately 1,5-fold excess of iron(II) for the back-titration, and wait for 1 min.

7.10 Back-titrate the excess iron(II) sulfate solution potentiometrically with the dichromate titrant solution (5.9).

7.11 Record the volumes of iron(II) sulfate solution (V_1) and potassium dichromate solution (V_2) added to reach the point of inflection of the titration curve, which is taken as the equivalence point. The potential of the indicating electrode is about 250 mV vs. the Hg/HgSO$_4$ at this point. Also record the temperatures of the iron(II) and dichromate burettes, t and t' respectively, during the titration.

8 Standardization of the iron(II) sulfate solution

8.1 Standardize the iron(II) sulfate solution every day using the following procedure, and record its titre T_{Fe} on a control chart.

8.2 Use the titrated solution of a plutonium aliquot and record the volume V_{Cr} of excess dichromate titrant solution added beyond the last equivalence point.

8.3 Add 3 ml (V_3) of iron(II) sulfate solution (5.8) to the analysed solution, record the temperature t of the iron(II) burette and titrate with dichromate titrant solution (5.9).

8.4 Record the volume of dichromate solution (V_4) added up to the equivalence point, together with the temperature t' of the dichromate burette at this point.

9 Expression of results

9.1 Whenever appropriate, correct the weighings used in the calculation of the result of the analysis for the effects of air buoyancy.

9.2 Calculate the titre T_{Fe}, expressed as the ^{239}Pu equivalent in grams per kilogram of solution, for the iron(II) sulfate solution from the equation:

$$T_{\mathrm{Fe}} = \frac{(V_4 + V_{\mathrm{Cr}}) \times K_{\mathrm{Cr}} \times \rho_{\mathrm{Cr}} \times T_{\mathrm{Cr}}}{V_3 \times K_{\mathrm{Fe}} \times \rho_{\mathrm{Fe}}}$$

where

V_4 is the volume of dichromate titrant solution added to complete the titration of Fe(II), in millilitres;

V_{Cr} is the excess of dichromate titrant solution from the previous titration, in millilitres;

K_{Cr} is the calibration factor for the dichromate titrant burette;

ρ_{Cr} is the density of the dichromate titrant solution at temperature t' (°C), in kilograms per litre, calculated from the expression $\rho_{\mathrm{Cr}} = 0{,}997\,1 - 0{,}000\,215\,(t' - 20)$;

T_{Cr} is the titre, expressed as the ^{239}Pu equivalent in grams per kilogram of solution, of dichromate titrant solution (5.9);

V_3 is the volume of iron(II) solution used, in millilitres;

K_{Fe} is the calibration factor for the iron(II) burette;

ρ_{Fe} is the density of the iron(II) solution at temperature t (°C), in kilograms per litre, calculated from the expression $\rho_{\mathrm{Fe}} = 1{,}202\,3 - 0{,}000\,71\,(t - 20)$.

9.3 Calculate the relative atomic mass of the plutonium A_{r} in the sample material from the equation:

$$A_{\mathrm{r}} = 238{,}050a_8 + 239{,}052a_9 + 240{,}054a_0 + 241{,}057a_1 + 242{,}059a_2 + 244{,}064a_4$$

where a_8, a_9, … are the atom fractions of the ^{238}Pu, ^{239}Pu, ... isotopes in the sample, as determined by mass spectrometry.

9.4 Calculate the plutonium content of the sample solution $w(\mathrm{Pu})$, expressed in grams of plutonium per kg of sample solution, from the equation:

$$w(\mathrm{Pu}) = \frac{(V_1 \times K_{\mathrm{Fe}} \times T_{\mathrm{Fe}} \times \rho_{\mathrm{Fe}}) - (V_2 \times K_{\mathrm{Cr}} \times T_{\mathrm{Cr}} \times \rho_{\mathrm{Cr}})}{m} \times \frac{A_{\mathrm{r}}}{A}$$

where

V_1 is the volume of iron(II) solution used, in millilitres;

K_{Fe} is the calibration factor for the iron(II) burette;

T_{Fe} is the titre of the iron(II) solution, expressed as the $^{239}\mathrm{Pu}$ equivalent in grams per kilogram of solution;

ρ_{Fe} is the density of the iron(II) solution at temperature t (°C), in kilograms per litre, calculated from the expression $\rho_{\mathrm{Fe}} = 1{,}202\,3 - 0{,}000\,71(t - 20)$;

V_2 is the volume of the dichromate titrant solution needed to titrate the excess of Fe(II), in millilitres;

K_{Cr} is the calibration factor for the dichromate titrant solution burette;

T_{Cr} is the titre of the dichromate titrant solution, expressed as a $^{239}\mathrm{Pu}$ equivalent in grams per kilogram of solution;

ρ_{Cr} is the density of the dichromate titrant solution at temperature t' (°C), in kilograms per litre, calculated from the expression $\rho_{\mathrm{Cr}} = 0{,}997\,1 - 0{,}000\,215\,(t' - 20)$;

m_4 is the mass of the aliquot taken, in g;

A_{r} is the relative atomic mass of the plutonium in the sample, calculated in accordance with (9.3);

A is the atomic mass of $^{239}\mathrm{Pu}$ (= 239,052).

10 Repeatability and accuracy

10.1 Repeatability

The coefficient of variation of the repeatability for a determination of 4 mg of plutonium is lower than 0,1 %.

10.2 Accuracy

The accuracy of the method depends mainly on the accuracy of the reference solution and is expected to be better than 0,1 % when no interfering substances are present. The results of the titration of 71 aliquots taken from five different dissolutions from an NBL CRM 122 reference material over a period of 19 months had a mean bias of – 0,021 % with a relative standard deviation of 0,070 %.