

INTERNATIONAL STANDARD

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Determination of uranium in uranium dioxide powder and pellets — Iron(II) sulfate reduction/potassium dichromate oxidation titrimetric method

*Dosage de l'uranium dans la poudre et les pastilles de dioxyde
d'uranium — Méthode titrimétrique par réduction par le sulfate de fer(II) et
oxydation par le dichromate de potassium*



Reference number
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Foreword

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Determination of uranium in uranium dioxide powder and pellets — Iron(II) sulfate reduction/potassium dichromate oxidation titrimetric method

1 Scope

1.1 This International Standard specifies an analytical method for the determination of uranium in uranium dioxide powder and pellets.

1.2 The method recommends that the aliquot of sample is weighed and that a mass titration is used, in order to obtain adequate precision and accuracy. This does not preclude the use of any alternative technique which can be shown to give an equivalent performance.

As the performance of some steps of the method is critical, the use of certain automatic devices has some advantages, mainly in the case of routine analyses.

2 Principle

2.1 Uranium(VI) is reduced to uranium(IV) in concentrated phosphoric acid solution by reaction with iron(II) sulfate. The excess iron(II) sulfate is subsequently oxidized by nitric acid in the presence of molybdenum, and the uranium(IV) is determined by mass titration with standardized potassium dichromate solution to a potentiometric end-point.

2.2 A portion of sample containing about 0,2 g of uranium in nitric acid solution is diluted with orthophosphoric acid containing a little dichromate to oxidize any reducing species which may be present. An excess of iron(II) sulfate solution is then added to reduce all the uranium to the quadrivalent state. Amidosulfuric acid is added to remove oxides of nitrogen formed at this stage.

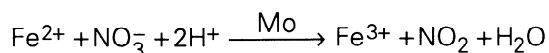
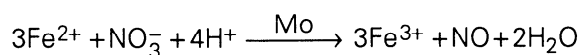
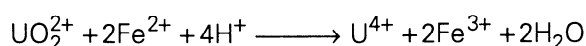
The excess of iron(II) sulfate is destroyed by oxidation with nitric acid, catalysed by molybdenum, in a time and temperature controlled operation. After dilution, the uranium is determined by mass titration with standardized potassium dichromate solution to a potentiometric end-point. To improve precision, the end-point is approached using dilute potassium dichromate solution and the titration is performed in the presence of vanadium(IV), which increases the rate of equilibrium attainment and enhances the potential step at the equivalence point.

2.3 The standard potassium dichromate solution is checked either against an internationally recognized uranium standard using the same potassium dichromate titration procedure.

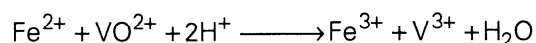
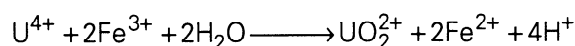
3 Reactions

Under the given experimental conditions, the principal reactions are as follows.

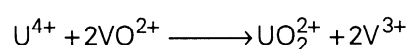
3.1 In concentrated phosphoric acid solution



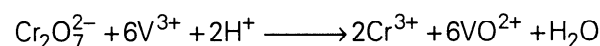
3.2 In diluted phosphoric acid solution



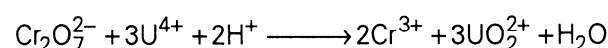
The overall reaction may be represented:



3.3 On titration with potassium dichromate solution



which is equivalent to the titration of U^{4+} with dichromate:



4 Interferences

This procedure is less subject to interference from foreign ions than most other methods of determining uranium. Ions and metals which do not interfere in any reasonable concentration include fluoride, perchlorate, sulfate, nitrate, peroxide, Al, Be, Si, Zr, Nb, Ti, Cr, Fe, Co, Ni, W, Cu, Pb, Pu, Th, the rare earths, the alkali metals and the alkaline earth metals. Molybdenum interferes only if large amounts of nitrate are also present and vice versa. Bromide, oxalate, Au, Sn, Mn and some platinum group metals interfere slightly. Interference from iodide, iodate, Ag, V and Tc is more severe. There is inadequate information on the effect of chloride, Hg, As, Sb, Np and Am and further studies are required.

5 Reagents

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

5.1 Hydrofluoric acid, ρ 1,13 g/ml.

5.2 Nitric acid, ρ 1,42 g/ml.

5.3 Orthophosphoric acid, ρ 1,75 g/ml.

5.4 Sulfuric acid, 50 % (V/V) solution.

Add 100 ml of sulfuric acid (ρ 1,84 g/ml) slowly and carefully to 100 ml of water, whilst stirring.

5.5 Iron(II) sulfate, 280 g/l solution

Add 50 ml of sulfuric acid (ρ 1,84 g/ml) slowly and carefully to 300 ml of water in a 2 litre beaker with constant stirring. Add $140 \text{ g} \pm 1 \text{ g}$ of iron(II) sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and stir until it is dissolved. Dilute to 500 ml and mix. This solution should not be used more than one month after preparation.