

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*



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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.

International Standard ISO 9989 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

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.