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Uranium metal and uranium dioxide powder and pellets — Determination of nitrogen content — Method using ammonia-sensing electrode

Uranium métal, et poudre et pastilles de dioxyde d'uranium — Dosage de l'azote — Méthode utilisant l'électrode sensible à l'ammoniac

Reference number
ISO 9006:1994(E)

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 9006 was prepared by Technical Committee ISO/TC 85, *Nuclear energy,* Subcommittee SC 5, *Nuclear fuel technology.*

Annex A forms an integral part of this International Standard.

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Uranium metal and uranium dioxide powder and pellets — Determination of nitrogen content — Method using ammonia-sensing electrode

1 Scope

This International Standard specifies an analytical method for determining the nitrogen content in uranium metal and uranium dioxide powder and pellets.

It is applicable to the determination of nitrogen, present as nitride, in uranium metal and uranium dioxide powder and pellets. The concentration range within which the method can be used is between 9 µg and 600 µg of nitrogen per gram. Interference can occur from metals which form complex ammines, but these are not normally present in significant amounts.

2 Principle

2.1 The sample is dissolved in a mixture of hydrochloric acid and hydrogen peroxide, producing a solution of uranium(VI) and converting any nitrogen, present as nitride, to the ammonium ion. Potassium carbonate is added to convert the ammonium ion to ammonia and uranium(VI) is retained in solution as the carbonate complex anion. Ethylene diaminetetraacetic acid (EDTA) is present to complex metals which form ammines. The ammonia content is measured using an ammonia-sensing electrode and a standard addition procedure.

2.2 A portion of sample containing about 0,5 g of uranium is dissolved in hydrochloric acid and hydrogen peroxide. The quantity of hydrochloric acid is kept to a minimum to ensure a low acidity after dissolution and to minimize the reagent blank. Nitrogen, present as nitride, is converted to ammonium ion by the acid, and hydrogen peroxide converts the uranium to the hexavalent state. Initially, a solid pale-yellow peroxy-uranium complex is formed; further heating decomposes this complex resulting in a clear yellow solution of uranium(VI).

The acid solution is made alkaline by the addition of excess potassium carbonate solution, the low free acid after dissolution ensuring that the evolution of carbon dioxide is minimal. The excess carbonate converts ammonium ion to ammonia gas and maintains uranium(VI) in solution as a complex anion, $UO_2(CO_3)_3^{4-}$.

The quantity of ammonia present in the alkaline solution is determined by recording the potential indicated by the ammonia-sensing electrode when it is inserted in the solution, adding a known amount of ammonia and again recording the potential indicated by the electrode.

The difference between the two values enables the ammonia content of the solution, and hence the nitrogen content of the sample, to be determined. The ammonia is added in the form of a standard solution of ammonium chloride, the ammonium ion being instantly converted to ammonia when it is added to the alkaline solution.

A blank test is carried out using the amounts of hydrochloric acid, hydrogen peroxide and water that are used for dissolving the test portion, and following the same procedure for measuring the ammonia content.

3 Reactions

$U^{4+} + (OO)^{2-} \rightarrow UO_2^{2+}$

$UO_2^{2+} + 3CO_3^{2-} \rightarrow UO_2(CO_3)_3^{4-}$

$NH_4^+ + OH^- \rightarrow NH_3 + H_2O$

4 Reagents

Use only reagents of recognized analytical grade and distilled water.

4.1 Distilled water.

The ammonia concentration shall be less than 0,1 µg/ml; laboratory grade water usually conforms to this requirement.