

INSTITUTO DE ENERGIA ATÔMICA

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CIDADE UNIVERSITÁRIA

SIE.0549/07/79.

FEG/MCB.

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SÃO PAULO

São Paulo, July 05, 1979.

UNITED STATES NUCLEAR REGULATORY COMMISSION
Gerald G. Oplinger, Assistant Director.
Export/Import and International Safeguards
Office of International Programs.
Washington, D.C. 20555

XSNM01475
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Dear Mr. Oplinger

We were very much surprised with the terms of your letter of May 22, addressed to Globe Shipping Co- N.Y. New York.

Your point of view regarding our order to import some NBS standards is for us too difficult to understand.

Our purpose is to get uranium NBS standards of varying isotopic concentrations and not enriched uranium in the form of NBS standards. There is quite a big difference in these two statements.

Anyone familiar with uranium thermionic -/ mass spectrometric analysis knows about the need of reference samples. Just for your knowledge we are sending, in annexe, a couple of papers (all from U.S.A) dealing with this matter. In these papers you could realise the -/ importance of these materials including uranium over 20% of enrichment.

The NBS standards, as specified in our -/ application are for checking discriminator factors, like is made in any / laboratory over the world. We do not need this material for manufacture / nuclear fuels. In this field our program is quite modest, all of the -/ activities are of laboratory scale and for do that we have imported the / necessary material inclusive from USA. Always under safeguards. It looks / for us rather impossible to make fuels with five gramms of uranium. As / regard an other point of ^{your} letter you should know our country signed an / agreement with Germany which cover all for our activities in the field of uranium enrichment.

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(cont.)

We hope to have clarified your questions and doubts but if you still have further questions regarding this matter / do not hesitate to write me.

Sincerely

HERNANI AUGUSTO LOPES DE AMORIM

Superintendent

CC. Mrs. Alvine R. Spies.
Globe Shipping Co.

PROC. 1589/77.

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LOS ALAMOS SCIENTIFIC LABORATORY
of the
University of California
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Methods of Chemical Analysis for
FBR Uranium-Plutonium Oxide
Fuel and Source Materials

Edited by

- James E. Rein
- George M. Matlack
- Glenn R. Waterbury
- Robert T. Phelps
- Charles F. Metz

This work was sponsored by the Fuels and Materials Branch of the Division of Reactor Development and Technology of the U. S. Atomic Energy Commission.

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2. Set the amplifier on the 10-mV scale and apply 10 mV from the potentiometer. Record the deflection.

3. Repeat step 2 for each amplifier sensitivity setting and the corresponding potentiometer voltage.

4. Determine exact scale factors from the ratio of applied voltage/recorder deflection for each sensitivity setting. Use these factors in subsequent determinations.

b. Recorder linearity

1. Connect a precision potentiometer as in step a-1 above.

2. Set the amplifier on the 1-V scale and apply voltage in increments of 0.1 V up to 1 V. Record the recorder deflection for each increment.

3. Find the cause for and correct any non-linearity greater than 0.25% of full scale.

c. Grid resistor linearity and ratio determination: If a standard current source is available, the test described in TID-7029 is advisable.

Calibration of Complete System

Calibrate the complete mass spectrometer at startup, at least every three months, and following any alteration, as follows:

a. Determine the mass discrimination bias both for the electron multiplier and the Faraday cup by measuring the $^{235}\text{U}/^{238}\text{U}$ ratio on at least three separate filament loadings of the NBS U-500 standard for each detector. Average the results and calculate the respective bias correction factors by:

$$\bar{R}_o = \frac{R_o - 1}{B}$$

where B = mass discrimination bias factor per amu in the U-Pu mass range.

\bar{R}_o = average of observed atom ratio $^{235}\text{U}/^{238}\text{U}$.

R_s = NBS stated atom ratio $^{235}\text{U}/^{238}\text{U} = 1.0003$.

Apply these bias corrections to subsequent determinations.

b. Establish the linear range of both detection systems by measuring the $^{234}\text{U}/^{235}\text{U}$ ratio on separate filament loadings of the NBS U-510 standard for each system over a wide range of ion currents at increments corresponding to the amplifier scale factors. To aid interpretation, plot the observed ratio vs the ^{235}U ion current on semilog paper. Analyze samples only within the linear range.

Daily Verification of Mass Spectrometer Stability

Verify the stability of each mass spectrometer used in any 8-hr working shift with the NBS U-010 uranium isotopic standard when uranium samples are analyzed during that shift. Maintain a control chart for each instrument for the ratio of $^{235}\text{U}/^{238}\text{U}$. When the value of this ratio changes at the .05 significance level, do not analyze samples until the cause is corrected. Possible sources of instability are a deterioration of the electron multiplier, low gain in the electrometer amplifier, electrical or mechanical malfunctions in the recorder, and inadequate regulation in the filament power supply.

Sample Analysis

1. Dissolve 1 mg of the uranium dioxide sample in 0.5 ml of 5M HNO_3 in a 10-ml beaker with the aid of moderate heating, dilute to 5 ml with water, and mix.

The sample weight and reagent volumes need not be quantitative because only isotopic abundances are to be measured. If a whole insulator pellet has been dissolved, dilute an aliquot of the solution to a uranium concentration of 0.2 $\mu\text{g}/\text{ml}$ with 1M HNO_3 .

[4] R. J. Jones, Ed., Method 2-500, Selected Measurement Methods for Uranium and Plutonium in the Nuclear Fuel Cycle, USAEC Report TID-7029 (1963).

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TID 7029(2nd Ed.) MASTER

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**Selected Measurement Methods
for Plutonium and Uranium
in the Nuclear Fuel Cycle**
Second Edition

Edited by
CLEMENT J. RODDEN

Office of Information Services
U.S. Atomic Energy Commission
1972

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used. Also, for instruments with expanded-scale strip-chart recording, see Ref. 3.

D. Calibration

1. *Mass Discrimination* The mass-discrimination bias factor for both uranium and plutonium is determined by analyzing a suitable NBS isotopic reference standard. The general procedure is to obtain several sets of isotopic ratios from several separate loadings of the reference sample and to calculate an average mass-discrimination bias factor, B ,

$$B = \frac{1}{C} \left[\left(\frac{\bar{R}}{R_s} \right) - 1 \right]$$

where B = mass-discrimination bias factor

$C = \Delta M/M = (238 - 235)/238$ for U

R_s = known value of $^{235}U/^{238}U$ for the reference sample

\bar{R} = grand-average measured value of $^{235}U/^{238}U$ for all sets of data

2. *Linearity* The linearity of the ion-current measuring system, including electron multiplier, amplifier, and data-recording system, can be checked using the uranium isotopic reference standard NBS U-930 and measuring the $^{235}U/^{238}U$ ratio over the range of ion intensities normally used.

E. Sample Preparation

1. *Uranium* Prepare uranium samples as a solution of uranyl nitrate of approximately 0.1 to 1 mg of uranium per milliliter in 0.5N to 2N HNO_3 . The sample may contain other elements as impurities up to 10 times the amount of uranium, except for alkali elements, which should be not greater than 10% of the uranium. For purification of samples see Methods 1.1.2.1 and 1.2.2.1.

2. *Plutonium* Prepare plutonium samples as a solution of approximately 0.01 to 0.1 mg of plutonium per milliliter in 0.5N HNO_3 . The sample may contain other elements, including uranium, as impurities up to 100 to 1000 times the plutonium concentration, except for alkali elements, which should be not greater than 100% of the plutonium. For purification of samples, see Method 2.1.2.1.

F. Procedures

1. *Filament Preparation* In preparing the filaments of a multiple-filament assembly, make certain that the filaments are not con-

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