WESTINGHOUSE MATERIALS
COMPANY OF OHIO

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Method No: 4017

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ANALYTICAL LABORATORIES

THE RADIOMETRIC NONDESTRUCTIVE ANALYSIS OF URANIUM-235 IN URANIUM TETRAFLUORIDE

Revision: 1

Date: 1/27/87

1.0 SCOPE

This method permits the nondestructive measurement of U-235 in uranium tetrafluoride (UF_4) using a Ge(Li) gamma spectrometer. The method was evaluated for UF_4 in the 0.20 to 1.25 wt% U-235 concentration range, but it could be easily extended to higher U-235 concentrations by using the appropriate standard. A nondestructive U-235 measurement can be performed in about 20 minutes. No measurement interferences have been observed.

2.0 PRINCIPLE

An unweighed subsample of UF_4 is transferred to a plastic vial and its 185.7 keV U-235 gamma activity is measured on a Ge(Li) gamma spectrometer. The U-235 concentration of an unknown sample is determined by ratio with the measured activity of a known UF_4 standard.

3.0 SPECIAL APPARATUS

3.1 Ge(Li) Detector System, Nuclear Data model ND-66. See Attachment B for a listing of the individual components and control settings for the system. This is the manually loaded Ge(Li) detector system.

3.2 Polystyrene Vials (with caps), 88 cm³ capacity, 4 cm I.D. x 7 cm H.

3.3 Exhaust Fume Hood.

4.0 REAGENTS

Not Applicable.

5.0 SAFETY PRECAUTIONS

5.1 WEAR SAFETY GLASSES IN THE LABORATORY AT ALL TIMES.

5.2 Perform all sample preparations in an exhaust hood. Wear rubber gloves.

6.0 SAMPLE PREPARATION

COMMENTS

6.1 Standard selection and determination of sample volume.

MASTER

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SAMPLE PREPARATION	COMMENTS
6.1.1 Select any UF ₄ sample which has been previously analyzed by mass spectrometry, and is close to the U-235 level to be measured, as the standard.	
6.1.2 Weigh 50 $\pm$ 0.1 g of the selected UF ₄ sample and transfer into a vial.	The standard is the only material actually weighed.
6.1.3 Label the vial with the wt% of U-235 and the designation "Standard".	
6.1.4 Place a mark on a vial for each sample at the same height as that of the standard.	The mark corresponds to a weight of about 50 g of UF ₄ . Vials and caps are used once and discarded.
6.2 Carefully transfer enough of the unknown UF ₄ into the vial to just exceed the mark (Step 6.1.4).	Work in an exhaust hood.
6.2.1 Place a clean sheet of paper under the vial to catch any spills.	
6.3 Cap the vial and wipe off the exterior surface to minimize detector contamination.	
6.4 Write the lab number on the cap.	
6.5 Seal the vial with plastic tape.	
6.6 Tamp each vial on a hard surface several times to compact the sample.	

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7.0 PROCEDURE**COMMENTS**

7.1 Increase (clockwise rotation) the intensity on the Nuclear Data model ND-66 MCA cathode ray tube (CRT) using the intensity control.

The intensity control is located on the left back side of the MCA.

7.2 Press the **ON LINE** button on the Centronics model 122-1 system printer.

Located on the front of the printer.

7.3 Place the appropriate standard in the detector shield.

Refer to Step 6.1.1.

The standard U-235 concentration should be as close as possible to the claimed U-235 value (viz, ± 0.25 wt. % U-235).

At least six measurements will be made on the same vial to obtain a good average count rate.

See Attachment B for instrument settings.

7.3.1 Open the clasp on the detector shield.

7.3.2 Open the shield by moving the tops in opposite (horizontal) directions.

7.3.3 Remove the previously counted vial (if one is present).

7.3.4 Insert the vial to be counted.

PROCEDURE	COMMENTS
7.3.5 Close the top sections and lock the clasp.	
7.4 Type the following command using the MCA keyboard: AU^1,X where "X" is the number of standard measurements to be performed (minimum is X=6).	The "^" symbol means press the space bar. Each measurement is 15 minutes in duration. See Attachment C for a description of the tasks included in the command.
7.4.1 Press the RETURN key.	
7.5 While the first count of the standard is in process, type the following into the MCA keyboard: ID^"Standard Identification".	The "^" symbol means press the space bar. The standard identification is the word "standard" and the wt% U-235 value of the appropriate standard. For example, type: Standard 0.2004 wt % U-235, and then press the RETURN key.
7.5.1 Press the RETURN key.	The new I.D. will be displayed on bottom of the CRT.
7.6 After the last count of the standard has concluded, insert the first unknown sample into the detector shield using Steps 7.3.1 - 7.3.5.	The red light on the analog-to-digital converter (ADC) will go out when counting is completed.
7.7. Press the TASK key and then the INIT key on the MCA keyboard.	Each unknown sample is counted once. See Attachment C for a description of this command.

PROCEDURE	COMMENTS
7.8 Type the following using the MCA keyboard: ID^ Lab. No.	The "^" symbol means press the space bar.
7.8.1 Enter the actual laboratory number after "ID" and not the words, in this command (e.g., 4-5761).	
7.8.2 Press the RETURN key.	
7.9 Repeat steps 7.7 and 7.8 until all samples have been counted.	
7.9.1 When the printer starts to print out data, then insert the next sample into the shield.	
7.10 Decrease (counterclockwise) the intensity on the CRT using the intensity control knob before leaving the instrument for extended periods of time.	The intensity control knob is on the back left side of the MCA.
8.0 STANDARDIZATION	
The U-235 concentration of an unknown sample is based on UF ₄ standards which have been destructively analyzed by gas mass spectrometry. Three standards are presently available: 0.2002, 0.8511, and 1.248 wt% U-235.	
9.0 CALCULATIONS	
9.1 Average all net (gross counts/15 minutes-background counts/15 minutes) standard counts (C _S) obtained in Steps 7.3 - 7.5.	
9.2 Calculate an unknown enrichment (I _X) using the relationship:	
$I_X = \frac{C_X I_S}{C_S} \text{ wt\% U-235}$	
Where,	
I _X = U-235 concentration of an unknown sample (in wt% U-235)	
I _S = U-235 concentration of the standard (in wt% U-235)	
C _X = Net counts/15 minutes for the unknown sample	
C _S = Net counts/15 minutes for the standard	

9.3 Example calculation:

$$C_X = 47,739$$

$$C_S = 47,308$$

$$I_S = 0.2002$$

$$I_X = \frac{(47,739)(0.2002)}{(47,308)} = 0.2020 \text{ wt\% U-235}$$

9.4 If the above example was to be reported the calculated value would be rounded to two significant decimal places and reported as 0.20 wt% U-235.

9.5 If the above example was for an ALQA sample the result would be rounded to three significant decimals and reported as 0.202 wt% U-235.

9.6 See Attachment A for the derivation of the above equation.

10.0 PRECISION AND ACCURACY

10.1 The analysis precision is dependent on the U-235 level. The precisions for the three standard levels (95% C.L.) are:

<u>Level (wt% U-235)</u>	<u>Precision (wt% U-235)</u>
0.20	± 0.017
0.85	± 0.026
1.25	± 0.033

10.2 Measurements at all three U-235 concentrations were determined to be unbiased, when compared to mass spectrometric results.

11.0 QUALITY ASSURANCE

11.1 Cross check all calculations before reporting results.

11.2 At least 10% of all analyses shall be quality control samples.

12.0 REFERENCES

12.1 Previous Method No. G-04-1283, dated 12/5/83.

12.2 H.W. Humphrey, Analytical Project No. ANAL-422, Gamma Analysis. "The Nondestructive Determination of U-235 in UF₄", (1/6/84).

13.0 ATTACHMENTS

13.1 Attachment A - Equation Derivation.

13.2 Attachment B - Ge(Li) Detector System Components and Settings.

13.3 Attachment C - Autosequence Command Tasks.

Revision 1 - prepared by E.L. Adkins and H.W. Humphrey, 1/27/87.
Prepared on HP-Touchscreen, HP-Word Wordprocessor.

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ATTACHMENT A
EQUATION DERIVATION

The measured 185.7 keV gamma ray count rate of a UF_4 sample is directly proportional to the U-235 concentration. Mathematically,

$$C_X = KI_X \quad (1)$$

Where,

C_X = Measured net counts/15 minutes for the sample

I_X = Weight percent U-235 concentration of the sample

K = Proportionality constant

If the standard is: (1) of the same matrix as the sample, (2) counted identically to the sample, and (3) it is of the same weight as the sample, then the proportionality constant "K" is the same as for the sample. Or,

$$C_S = KI_S \quad (2)$$

Where,

C_S = Measured net counts/15 minutes for the standard

I_S = Weight percent U-235 concentration of the standard

and "K" is the same proportionality constant as for Equation (1).

If Equations (1) and (2) are rearranged we have the following:

$$K = \frac{C_X}{I_X} = \frac{C_S}{I_S} \quad (3)$$

or,

$$\frac{C_X}{I_X} = \frac{C_S}{I_S} \quad (4)$$

Solving for the unknown U-235 concentration " I_X ", we have

$$I_X = \frac{C_X I_S}{C_S} \quad \text{wt\% U-235} \quad (5)$$

It should be noted that 50 g of sample (and standard) is counted. This weight is approximately 40% more than an "infinite thickness" for the 185.7 keV U-235 gamma ray. The weight of material to be counted therefore only needs to be close to 50 grams.

ATTACHMENT B

GE(LI) DETECTOR SYSTEM COMPONENTS AND SETTINGS

SYSTEM COMPONENTS

<u>Item</u>	<u>Manufacturer</u>	<u>Model No.</u>	<u>Serial No.</u>
Graded Pb Shield	Nuclear Data	SPG-16 (K-8)	783/269
Ge(Li) Detector	Princeton Gamma-Tech	LGTC35 SD	1825
Preamplifier	Princeton Gamma-Tech	RG-11 A/C	5046
Spectroscopy Amplifier	EG&G ORTEC	572	2735
Bias Voltage Supply	Princeton Gamma-Tech	314	165
Analog-to-Digital Converter	Nuclear Data	575	761489
Multichannel Analyzer	Nuclear Data	ND66	79-511
Electronics Enclosure	Nuclear Data	ND-11	80101
Winchester/Floppy Disk	Data Systems Design	880-D8	03495
Printer/Plotter	Centronics	122-1	A22700894

ATTACHMENT B (Continued)

GE(LI) DETECTOR SYSTEM COMPONENTS AND SETTINGS

INSTRUMENT SETTINGSNUCLEAR DATA MODEL ND-66 MULTICHANNEL ANALYZER (MCA)

ADC - #1
Group Size - 4096 Channels
Integration Area - Channels 268-293 (179.5 - 192.4 keV)
Zero (y-intercept) - 42.109 keV
Slope - 0.512822 keV/Channel
Energy Range - 42.109 - 2142.63 keV

NUCLEAR DATA MODEL ND-575 ANALOG TO DIGITAL CONVERTER (ADC)

Conversion Gain - 4K
Coin./Anticoin. - OFF
ACQ/STRG - ACQ

ORTEC MODEL 572 AMPLIFIER

Coarse gain - 50
Fine Gain (0.5 - 1.5) - 0.9015
Shaping time - 3 us
BLR Auto/PZADJ/THRES - PZ ADJ
DELAY/OUT - OUT
POS/NEG - NEG

PRINCETON GAMMA TECH MODEL PGT 314 Bias Supply

Polarity - Pos
ON/OFF - ON
Output Potentiometer - 4.8 (+4800V)

CENTRONICS MODEL 122 PRINTER

Select Light - On
Power Light - On

ATTACHMENT C

AUTOSEQUENCE COMMAND TASKS

<u>Step</u>	<u>Task</u>	<u>Task Description</u>
1	ER	Erase memory
2	LI 900	Sets 900 sec (15 min.) live time counter
3	ON	Starts acquisition
4	DE LP	Defines the Line Printer as the system output device
5	WA 1	Wait until ADC #1 times out
6	ST	Print out the MCA status (includes the sample I.D.)
7	PT	Print total, net, and background counts for the U-235 peak.
8	PR	Print the individual data contents for each channel in the U-235 peak.
9	WA IO	Wait until print out is completed (I/O)

Notes: (1) Task steps 1-9 may be executed in two ways:

- (a) Press **TASK** and **INIT** - performs Steps 1-9 one time and the MCA stops.
- (b) Type: AU 1, "X" and then press the **RETURN** key - performs Steps 1-9 "X" times and the MCA stops. "AU" is the MCA pneumatic for autosequence.