

TABLE 5b – ^{235}U Enrichment (NDA)

Method/measurement system (including software): [TO](#) / [XMCN](#)

Application: [Measurements of Uranium Enrichment in \$\text{UF}_6\$ and Pure \$\text{UO}_x\$](#)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
XMCN is a portable gamma-spectrometry system, which is based on the $\varnothing 2'' \times 0.5''$ NaI(Tl) scintillation detector (typical FWHM $\approx 11\%$ @ 185.7 keV) coupled to an InSpector-2000 Multichannel Analyzer (IMCN) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-166/527 (MMCN) from GBS Elektronik GmbH. The MCA is typically set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. The NaI(Tl) crystal is usually seeded by ^{241}Am- isotope for temperature stabilization purposes. The detector's field of view is shaped by a 44 mm diameter by 20 mm length collimator made of lead. The ^{235}U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ^{235}U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness > 7 mean-free-paths of the 185.7 keV gammas, e.g. > 1.5 cm for UF_6 or > 2.7 cm for UO_2 powder with $\rho = 2 \text{ g/cm}^3$). Typically, the spectrum is measured for 300 s live time. The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using the two-regions-of-interest (2ROI) approach [2], e.g., as implemented in WinU235 [3] and IMCA [4] codes. In this approach, two non-overlapping ROIs are defined within and above the 185.7 keV peak and intended for net peak area determination using a linear combination of the gross numbers of counts registered within their limits. The method requires at least two "infinitely" thick standards for calibration. More standards can be employed to achieve better calibration accuracy. Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term. The term includes the container wall thickness, which is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.
Factors affecting the method performance
Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ^{235}U enrichment will impact the results. Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with poorly known uranium content. A negative bias in the measured enrichment should be expected for materials containing burnable poison additives, such as Gd, Er etc. Due to the uncontrolled contribution from the forward scattering into the lower ROI, the 2ROI approach usually performs better in narrow wall thickness intervals. Multiple calibrations are normally prepared to cover a wider wall thickness interval. Additionally, a significant bias should be expected for materials containing reprocessed uranium due to the uncontrolled contribution of ^{232}U decay products to the upper ROI.

Measurements in industrial environments require observation of background conditions for avoiding biasing to higher enrichment values. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment. Temperature instabilities of system's parameters may shift energy calibration, thereby leading to an improper definition of the ROIs and biasing the enrichment result.

Source of top-down value and ITV applicability

The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.

Source of bottom-up value (only for prospective ITV not based on historical data)

n/a

Method references

- [1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.
- [2] D. Reilly, N. Ensslin, H. Smith Jr., S. Kreiner, Passive Nondestructive Assay of Nuclear Materials, United States Nuclear Regulatory Commission, Washington, D.C, NUREG/CR-5550, LA-UR90-732 (1991) 211–214.
- [3] WinU235 - Program for ^{235}U enrichment measurements with NaI-detectors, Ver. 2.02, GBS Elektronik GmbH, <https://www.gbs-elektronik.de/en/downloads/downloads-nuclear-measurements.php>
- [4] G.H. Gardner, M. Koskelo, M. Moeslinger, R.L. Mayer, B.R. McGinnis, The IMCA: A Field Instrument for Uranium Enrichment Measurements, Proceedings of the 37th Annual Meeting of the Institute of Nuclear Materials Management, Naples, FL, USA, 28 July - 1 August 1996, p. 680-685.

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / ECGS

Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

ECGS stands for Electrically Cooled Gamma Spectrometer, which is a portable high-energy resolution HPGe-detector-based system with an integrated MCA, a microcomputer and an electrical cooler (typical FWHM ≈ 1.1 keV @ 122 keV). The IAEA's ECGS is represented by the ORTEC Micro-UF₆TM system, a variant of the standard ORTEC Micro-trans-SPEC portable gamma-spectrometer. The system includes a coaxial HPGe crystal with $\varnothing 50$ mm $\times$ 30 mm nominal dimensions, furnished with a $\varnothing 50$ mm $\times$ 8 mm front-end tungsten collimator.

The spectrometer is normally set up to acquire a 4k-channel gamma-spectrum with the 0.075 keV/chn gain. The ²³⁵U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness > 7 mean-free-paths of the 185.7 keV gammas, e.g. > 1.5 cm for UF₆ or > 2.7 cm for UO₂ powder with $\rho = 2$ g/cm³). The gamma-spectrum is typically measured for 300 s live time.

The measurement geometry ensures that the examined material completely (i.e. without voids) fills the space within the detector's field of view. The on-board *UF₆ Enrichment* program [2] determines the 185.7 keV peak area via a linear background subtraction using two non-symmetrical continuum regions-of-interest (ROIs) around the peak ROI.

Other spectrum evaluation algorithms, e.g. smoothed step background subtraction or peak fitting, can be utilized as well. The method relies on one-point enrichment calibration. Owing to the high-energy resolution of the employed detector, the method is barely affected by the effects of the low-angle photon scattering and isotopic interference (e.g. from ²³²U decay daughters in the case of reprocessed uranium).

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction factor with an empirical quadratic term in the exponent that adequately addresses the ECGS's wide solid angle measurement geometry [2]. The container wall thickness is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results. Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities.

Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content or for materials containing burnable poison additives, such as Gd, Er etc. Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments.

Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment. The ORTEC Micro-trans-SPEC experiences high deadtimes and prone to overloading when measuring

materials with high contents of reprocessed uranium. Therefore, lower performance is to be expected in this case due to the need to use a narrower collimator and significantly deteriorated peak-to-continuum ratio.

Source of top-down value and ITV applicability

The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.

Source of bottom-up value (only for prospective ITV not based on historical data)

n/a

Method references

- [1] [1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.
- [2] [2] ORTEC Micro-UF6™ Portable UF6 Cylinder Verification System, Software v.2.3 — User's Manual, ORTEC Part No. 932500, 177 p.

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / HM-5

Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

HM-5 is a handheld gamma-spectrometric instrument [1], which is based on the Ø23 mm x 21 mm NaI(Tl) scintillation detector (typical FWHM $\approx$ 8.5% @ 662 keV). The detector has an internal 5 mm thick tungsten side shielding and a not-thallium-activated rear NaI crystal, serving as a back shielding. The main outward-facing NaI(Tl) crystal is recessed into the internal collimator by 5 mm, thereby determining a wide-solid-angle field of view of the detector.

The spectrometer has a LED-based temperature stabilization of the photomultiplier tube and a built-in $\sim$ 500 Bq Cs-137 check source for instrument's gain initialization on startup. The HM-5 is set up to acquire a 1k-channel gamma-spectrum with the 3 keV/chn gain. The ^{235}U enrichment is determined using the "enrichment meter" principle [2], i.e. on the absolute intensity of counts in the 185.7 keV peak of ^{235}U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF₆ or >2.7 cm for UO₂ powder with $\rho = 2$ g/cm³). Typically, a spectrum is measured for 120 s (LEU) or 180 s (NU) live time. The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using the peak fitting approach implemented in the NaIGEM code [3].

The method relies on one-point enrichment calibration. A wide range of container wall thicknesses is covered owing to the application of the effective wall thickness concept and accurate background delineation under the 185.7 keV peak. The method accounts for spectrum distortion effects due to decay products of ^{232}U in the case of reprocessed uranium.

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term with the collimator dependent term for calculating effective wall thickness. The container wall thickness is determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ^{235}U enrichment will impact the results.

Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content or materials containing burnable poison additives, such as Gd, Er, etc.

Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment.

Source of top-down value and ITV applicability
The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties. The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
n/a
Method references
[1] Safeguards Techniques and Equipment: 2011 Edition, International Nuclear Verification Series No.1 (Rev. 2), International Atomic Energy Agency, Vienna, 2011, 162 p. [2] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19. [3] Gunnink, R. Arlt, R. Bernt, New Ge and NaI analysis methods for measuring ^{235}U enrichments, Proceeding of the 19th Annual ESARDA Symposium on European Safeguards Research and Development Association, Luxembourg, ESARDA Joint Research Centre, Ispra, Italy, May 13-15, 1997.

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / XMCC

Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

XMCC is a portable gamma-spectrometry system, which is based on a $\geq 1500 \text{ mm}^3$ (for NU/LEU) or $\geq 500 \text{ mm}^3$ (for HEU) cadmium zinc telluride (CZT) semiconductor room temperature detector with typical FWHM $\approx 2.5\%$ @ 122 keV. The detector can be coupled to an InSpector-2000 Multichannel Analyzer (IMCC) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-527 (MMCC) from GBS Elektronik GmbH, or another MCA. The MMCC system operated via the MCA Touch (MCAT) v.2.X software from GBS Elektronik GmbH [1] represents a standard IAEA setup.

In this setup, a CZT detector is arranged inside a tungsten shielding with the collimator opening matching dimensions of the detector's active area, e.g., 15 mm in diameter in the case of a $15 \times 15 \times 7.5 \text{ mm}^3$ hemispherical CZT/1500 from Ritec Ltd. [2]. A typical collimator length is 5 mm. The system is set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. The ^{235}U enrichment is determined using the "enrichment meter" principle [3], i.e. on the absolute intensity of counts in the 185.7 keV peak of ^{235}U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness > 7 mean-free-paths of the 185.7 keV gammas, e.g. $> 1.5 \text{ cm}$ for UF₆ or $> 2.7 \text{ cm}$ for UO₂ powder with $\rho = 2 \text{ g/cm}^3$).

Typical spectrum measurement live time is 300 s. The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using a peak fitting approach implemented in the GEM software [4]. The method relies on one-point enrichment calibration. It covers a wide range of container wall thicknesses owing to the application of the effective wall thickness concept and accurate background delineation under the 185.7 keV peak. The latter removes a considerable systematic bias (inherent to the older two-region method) due to the contribution of the low-angle Compton scattering under the full energy peaks. The method accounts for the spectrum distortion effects due to decay products of ^{232}U in the case of reprocessed uranium.

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term with the collimator dependent term for calculating effective wall thickness. The container wall thickness is determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured. For an unknown compound, the matrix correction can be calculated using the declared or independently measured uranium content. Additional correction is implemented to account for the presence of burnable poison additives, such as Gd, Er etc.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ^{235}U enrichment will impact the results. Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content.

Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. The container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment.

Source of bottom-up value (only for prospective ITV not based on historical data)

The prospective ITVs were derived considering different factors contributing to both systematic and random uncertainty components. Various parameters in the applied analytical expressions were determined based on the carried-out laboratory and limited field tests. Details of the approach are presented in the Annex.

Method references

- [1] MCA Touch user manual, GBS Elektronik GmbH, JOPAG/05.12-DOC-99 Joint Program on the technical development and further improvement of IAEA safeguards between the government of the Federal Republic of Germany and the IAEA, Task C.40/A7191, February 2016, ver. 1.8, 61 p.
- [2] ZRF Ritec SIA, CZT/1500, <http://www.ritec.lv/html/czt500.html>.
- [3] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.
- [4] A. Berlizov, GEM: A Next-Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, Vienna, IAEA, Feb 2021 (to be published in JNMM)

Prepared by: Andriy Berlizov

ANNEX: Descriptions of the bottom-up evaluations of the measurement uncertainties for XMCC

A. Berlizov (IAEA, SGTVT)

1. Random uncertainty

The random component of the measurement uncertainty was calculated using the following formula:

$$u^2(r) = u_{PEAK}^2 + u_{WALL}^2$$

Here, u_{PEAK} is the random component of the relative uncertainty (%) of the 186 keV peak area determination from the measured gamma spectrum; u_{WALL} is the random component of the relative uncertainty (%) of the wall thickness determination using ultrasonic thickness gauge.

Based on the practically achievable accuracy of the wall thickness determination using IAEA's standard equipment, the value $u_{WALL} = 1.5\%$ was assumed in the current evaluations.

The peak area uncertainty was calculated using the following formula:

$$u_{PEAK}^2 = \frac{10^4}{N} \times \left(1 + 2 \frac{B}{N}\right)$$

Here, N is the net number of counts in the 186 keV peak (net peak area), and B is the number of counts in the continuum background under the peak. The intensity of the 186 keV peak in the ambient background was assumed to be zero.

The net peak area was estimated using the following formula:

$$N = Enr \times K^{-1} \times F(x) \times F_M \times LT$$

Where, Enr is the material ^{235}U enrichment (wt %), K is the enrichment calibration constant (= 0.0310 wt %/cps), $F(x)$ is the attenuation correction factor for container wall thickness x (cm); F_M is the matrix correction factor (= 1.0153 for U_3O_8); and LT is the spectrum acquisition live time (s).

The attenuation correction factor was calculated using the following formula:

$$F(x) = \exp(-\mu a(1 - bx)x)$$

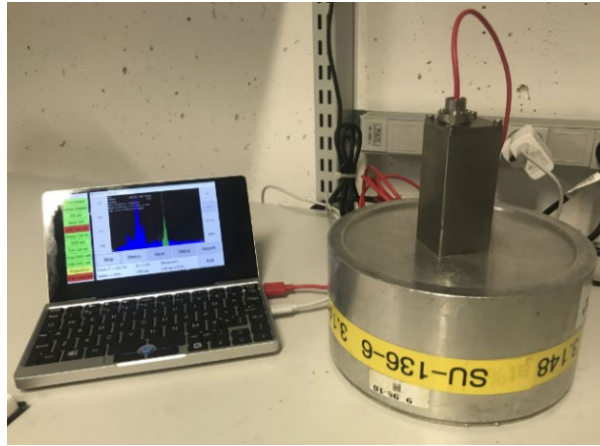
Where, $\mu = 1.21 \text{ cm}^{-1}$ is the linear attenuation coefficient for the 186 keV photons in steel; $a = 1.1136$ and $b = 0.0143 \text{ cm}^{-1}$ are the collimator specific correction constants.

The background-to-peak count ratio B/N was parameterized as function of material enrichment. The parameterization was based on the set of experimental spectra measured with the 16 mm (the thickest) steel absorber:

$$\frac{B}{N} = 0.958 + \frac{4.3}{Enr}$$

Experimental measurements were performed using the following equipment and experimental setup:

- Detector: CZT 1500 mm³ (RITEC), crystal dimensions = 15 × 15 × 7.5 mm³, collimator dimensions = Ø15 mm × 5 mm (tungsten).
- Multiple channel analyzer: MCA-527, GBS Elektronik
- Software: MCAT 2.0, GEM v.2.2.5
- Working standard materials: SU-135 (19.82 wt %), SU-136 (3.105 wt %), SU-137 (0.708 wt %), SU-138 (0.217 wt %), U3O8 powder, quasi-infinitely thick layer = 20 mm, 163 mm diameter, aluminum can with 2 mm thick working window.
- Shielding: 0 mm, 4 mm, 8 mm, 12 mm, 16 mm
- Measurement geometry:



2. Systematic uncertainty

The systematic uncertainty was calculated taking into account the uncertainty of the system enrichment calibration (typical value is $u_{CAL} = 0.6 \%$) and the systematic uncertainty of the spectrum fitting. The latter was approximated by the equation: $u_{FIT} = 1.243 + 0.0782 \cdot (B/N)$. The total systematic uncertainty was thus calculated as follows:

$$u^2(s) = u_{CAL}^2 + u_{FIT}^2$$

Method/measurement system (including software): T1 / XMCG

Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

XMCG is a portable high-resolution gamma-spectrometry system, which is based on a liquid nitrogen (LN₂) cooled high-purity germanium (HPGe) detector coupled to an InSpector-2000 Multichannel Analyzer (IMCG) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-166/527 (MMCG) from GBS Elektronik GmbH. A typical IAEA XMCG is based on the low-energy GL1015 or broad-energy BE1015 planar detector from Mirion Technologies.

The detector has a 1000 mm² x 15 mm HPGe crystal and typical FWHM ≤ 600 eV @ 122 keV. The detector is positioned inside a lead collimator-shielding, recessed by 25 mm with respect to its $\varnothing 35$ mm x 15 mm opening. The ²³⁵U enrichment is determined using the “enrichment meter” principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an “infinitely” thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF₆ or >2.7 cm for UO₂ powder with $\rho = 2$ g/cm³).

The gamma-spectrum is typically measured for 300 s live time. The measurement geometry ensures that the examined material completely (i.e. without voids) fills the space within the detector’s field of view. A modified version of the IMCA program [2] determines the 185.7 keV peak area via a linear background subtraction using two continuum regions-of-interest (ROIs) around the peak ROI. The method relies on one-point enrichment calibration. Owing to the high-energy resolution of the detector, the method is barely affected by the effects of the low-angle photon scattering and isotopic interference (e.g. from ²³²U decay daughters in the case of reprocessed uranium).

Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction factor with an empirical quadratic term in the exponent [2]. The container wall thickness is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured.

Factors affecting the method performance

Incompliance with the basic “infinite” thickness geometry assumptions regarding material layer thickness and coverage of the detector’s field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results.

Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content or for materials containing burnable poison additives, such as Gd, Er etc.

Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment. In the case of reprocessed uranium, the measurement performance is expected to deteriorate due to the lower peak-to-continuum ratio.

Source of top-down value and ITV applicability
The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties. The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
n/a
Method references
[1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19. [2] G.H. Gardner, M. Koskelo, M. Moeslinger, R.L. Mayer, B.R. McGinnis, The IMCA: A Field Instrument for Uranium Enrichment Measurements, Proceedings of the 37th Annual Meeting of the Institute of Nuclear Materials Management, Naples, FL, USA, 28 July - 1 August 1996, p. 680-685. [3] ORTEC Micro-UF6™ Portable UF6 Cylinder Verification System, Software v.2.3 — User's Manual, ORTEC Part No. 932500, 177 p.

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T1 / XMCL

Application: Measurements of Uranium Enrichment in UF₆ and Pure UO_x

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

XMCL is a portable gamma-spectrometry system, which is based on a Ø2" x 0.5" LaBr₃(Ce) scintillation detector (typical FWHM $\approx$ 5% @ 185.7 keV) coupled to an InSpector-2000 Multichannel Analyzer (IMCL) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-527 (MMCL) from GBS Elektronik GmbH. The LaBr₃(Ce) crystal is not normally temperature stabilized, thus, in this case, the temperature instabilities are compensated by spectrum processing algorithms. The detector's field of view is shaped by a 44 mm diameter by 20 mm length collimator made of lead or tungsten. The ²³⁵U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum measured from an "infinitely" thick layer of a uranium bearing material (thickness >7 mean-free-paths of the 185.7 keV gammas, e.g. >1.5 cm for UF₆ or >2.7 cm for UO₂ powder with $\rho = 2 \text{ g/cm}^3$). The XMCL system is set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. Typically, a spectrum is measured for 300 s live time.

The measurement geometry ensures that the material completely (i.e. without voids) fills the space within the detector's field of view. The 185.7 keV peak intensity is determined using the peak fitting approach, such as NaIGEM [2], GEM [3] or similar. The method relies on one-point enrichment calibration, which covers a wide range of container wall thicknesses owing to the application of the effective wall thickness concept and accurate background delineation under the 185.7 keV peak. The latter removes a considerable systematic bias (inherent to the older two-region method) due to the contribution of the low-angle Compton scattering under the full energy peaks.

The method accounts for the spectrum distortion effects due to decay products of ²³²U in the case of reprocessed uranium. Data evaluation includes application of the container wall and material matrix corrections. The attenuation by the container wall is considered using an exponential correction term with the collimator dependent term for calculating effective wall thickness. The container wall thickness is normally determined on the spot using an ultrasonic gauge. The matrix correction is applied based on the information about the compound being measured. For an unknown compound, the matrix correction can be calculated using the declared or independently measured uranium content. Additional correction is implemented to account for the presence of burnable poison additives, such as Gd, Er etc.

Factors affecting the method performance

Incompliance with the basic "infinite" thickness geometry assumptions regarding material layer thickness and coverage of the detector's field of view will bias the measured enrichment to a lower value. The heterogeneity and non-uniformity of material with respect to the uranium content and ²³⁵U enrichment will impact the results.

Performing multiple measurements at different spots of a given item mitigates the potential bias due to the non-uniformities. Non-stoichiometric mixtures may bias measured enrichment, e.g. in the case of low-concentrated scraps with unknown uranium content. Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak. The wall thickness determination by ultrasonic gauge may be affected by non-uniformity of the container wall, multiple layers of paint, rust and extra packing layers. In the case of steel, a 0.1 mm inaccuracy in the measured wall thickness leads to about 1% relative bias in the measured enrichment. Moreover, the container surface curvature may lead to up to a several percent bias to lower enrichments. Assuming incorrect material (e.g., steel instead of Monel) may significantly bias the measured enrichment.

Source of top-down value and ITV applicability
The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties. The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in "Short description", on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the 'Notes' column in the table.
Source of bottom-up value (<u>only for prospective ITV not based on historical data</u>)
n/a
Method references
[1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19. [2] Gunnink, R. Arlt, R. Bernt, New Ge and NaI analysis methods for measuring ^{235}U enrichments, Proceeding of the 19th Annual ESARDA Symposium on European Safeguards Research and Development Association, Luxembourg, ESARDA Joint Research Centre, Ispra, Italy, May 13-15, 1997. [3] A. Berlizov, GEM: A Next-Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, Vienna, IAEA, Feb 2021 (to be published in JNMM)

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): T2 / XMCN/XMCL**Application:** Measurements of Uranium Enrichment in UO_x Pellets and Fuel Rods

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

IAEA inspectors perform in-field measurements of ²³⁵U enrichment of UO_x pellets and fuel rods using a portable low- or medium-resolution gamma-spectrometer. The spectrometer is based on an IAEA standard NaI(Tl) (XMCN) or LaBr₃(Ce) (XMCL) Ø2" x 0.5" scintillation detector with typical energy resolution (FWHM at 185.7 keV) of ≈11% (XMCN) or ≈5% (XMCL). The detector is arranged inside a 10 mm thick lead shielding that is opened from the detector's front-end. The detector is coupled to an InSpector-2000 Multichannel Analyzer (IMCN/IMCL) from Mirion Technologies (former Canberra Industries) or a Miniature Multichannel Analyzer MCA-166/527 (MMCN/MMCL) from GBS Elektronik GmbH.

The measurement system is set up to acquire a 512-channel gamma-spectrum with the 185.7 keV peak positioned in channel 300. The ²³⁵U enrichment is determined using the "enrichment meter" principle [1], i.e. on the absolute intensity of counts in the 185.7 keV peak of ²³⁵U in a gamma-ray spectrum. Unlike conventional "enrichment meter" applications, requiring that the measured material completely fills the space within the detector's field of view, the pellet/rod measurements rely on a controlled, reproducible positioning of measured items (normally five pellets or a fuel rod) at a fixed distance from the detector. The high accuracy and reproducibility of positioning are achieved by the use of special pellet/rod holders. The method is calibrated using a standard set of reference pellets or a standard fuel rod measured using the same holder. Field measurements are typically done for 500 s (LEU) or 1000 s (NU/DU) live time. The 185.7 keV peak intensity is determined using a peak fitting approach, such as NaIGEM [2] or GEM [3]. The spectrum fitting accounts for possible presence of decay products of ²³²U in the case of reprocessed uranium.

Data evaluations include application of a matrix correction (including effects of burnable poison additives, such as Gd, Er etc.) and a geometry correction that accounts for possible deviations between the calibration and measured pellets/rods properties (external diameter, inner hole diameter, pellet's height, rod's cladding material and cladding thickness). The external dimensions of pellets/rod are measured/verified in the field using an electronic caliper.

Factors affecting the method performance

Inaccurate measurement geometry setup (e.g., a gap between detector endcap and pellets/rod holder) or inaccurate pellets/rod positioning will bias the measured enrichment. Unknown or inaccurate information about the content of the burnable poison additive will result in a systematic bias. Inaccuracies in the pellets/rods dimension measurements as well as selection of wrong cladding material for a fuel rod (e.g., zircaloy instead of stainless steel) will bias the measured enrichment. Measurements in industrial environments may be affected by the background counts in the 185.7 keV peak.

Source of top-down value and ITV applicability

The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random u(r) and short-term systematic u(s) uncertainties.

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in “Short description”, on material type, grade and containment specified in relevant columns in the Table 5b. The recommended measurement times are provided in the ‘Notes’ column in the table.

Source of bottom-up value (only for prospective ITV not based on historical data)

n/a

Method references

- [1] T.D. Reilly, R.B. Walton, J.L. Parker, Progress report LA-4605-MS, Los Alamos National Laboratory, NNM, 1970, p. 19.
- [2] Gunnink, R. Arlt, R. Bernt, New Ge and NaI analysis methods for measuring ²³⁵U enrichments, Proceeding of the 19th Annual ESARDA Symposium on European Safeguards Research and Development Association, Luxembourg, ESARDA Joint Research Centre, Ispra, Italy, May 13-15, 1997.
- [3] A. Berlizov, GEM: A Next-Generation Gamma Enrichment Measurements Code, Proceedings of the International Workshop on U/Pu Isotopic Analysis by Nondestructive Assay Techniques for Nuclear Safeguards, Vienna, IAEA, Feb 2021 (to be published in JNMM)

Prepared by: Andriy Berlizov and Kamil Krzysztozek

Method/measurement system (including software): HRGS/XMCG Peak Ratio/FRAM-MGAU**Application:** ^{235}U enrichment measurements employing the low energy (< 250 keV) or high energy (> 120 keV) region of the spectrum

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

The method derives the ^{235}U enrichment from ratios of specific nuclear and detection parameters related to two different isotopes, including the peak area of selected gamma-rays in a high-resolution gamma-spectrum. The method can be used to measure items of arbitrary size, shape and composition. It is applicable to any enrichment level and is useful for in-situ measurements of aged bulk materials with net weight of a few grams and up. A typical measurement system includes a collimated HPGe detector, an integrated or stand-alone digital MCA, and an instrumentation laptop.

For the low-energy (< 250 keV) region uranium analysis, the technique uses the pair of ^{238}U 's daughter ^{234}Th peaks at 92.4 and 92.8 keV to measure the activity of ^{238}U . This requires ^{234}Th to be in secular equilibrium with its parent ^{238}U or the chemical separation date is known so correction can be applied. For the measurement of ^{235}U activity, the peaks at 89 and 93 keV, as well as some other peaks at higher energy, may be used. In order to avoid fitting problems due to poor quality (resolution) spectra, this low-energy region analysis requires detector with resolution < 600 eV at 122 keV Co-57, such as the planar HPGe detector. Spectra with 4K channels are typically acquired, with energy calibration of 0.075 keV/ch. Typical measurement time is 300-1800 s, depending on material amount in the item and container wall thickness (< 1.5 mm Fe). The two software codes employing this technique are FRAM [1, 2] and MGAU [3].

For the high-energy (> 120 keV) region uranium analysis, the technique uses the gamma rays of ^{238}U 's granddaughter $^{234}\text{Pa}/^{234\text{m}}\text{Pa}$ peaks to measure the activity of ^{238}U . This requires ^{234}Pa to be in secular equilibrium with its grandparent ^{238}U or the chemical separation date is known so correction can be applied. The method uses the gamma ray peaks from 120 keV up to 1001 keV. The high energy peaks mean that the measurements are more efficient with high efficiency detector such as a large coaxial or semi-planar HPGe detector. Spectra with 8K channels are typically acquired, with energy calibration of 0.125 keV/ch. Typical measurement time is 300-1800 s, depending on material amount in the item and container wall thickness (< 2 cm Fe). The software code employing this technique is FRAM [1, 2].

Factors affecting the method performance

The present ITV values are for homogeneous materials only. The method based on low energy works well with bare or lightly shielded samples (< 1.5 mm Fe).

The method based on high energy works well with bare samples up to moderately shielded (< 2 cm steel) samples. It can also analyze samples that are heavily shielded up to 5 cm steel, but the biases may be significantly larger. For HEU, the method depends somewhat on the intense gamma ray peaks of $^{232}\text{U} \rightarrow ^{228}\text{Th}$ decay chain to bridge the large gap between the low energy peaks of ^{235}U and the high energy peaks of ^{238}U . The method therefore is less accurate and precise when analysing HEU that does not include any reprocessed uranium. For LEU, the method does not depend on the peaks of $^{232}\text{U} \rightarrow ^{228}\text{Th}$ decay chain and can analyze uranium items that do or do not contain reprocessed uranium equally well.

Both the low-energy and high-energy methods require the uranium to be aged (> 6 months after chemical separation). For fresh uranium, both the MGAU and the FRAM codes allow the user to enter the separation date, if well-known, and the codes will correct the results for the freshness of the material. However, enrichment results with higher uncertainty levels can be observed due to this additional correction. Using HPGe planar detectors with resolution poorer than the recommended level may also compromise the measurement uncertainty. Finally, measurements at too high counting rates

may lead to degraded detector resolution, tailing caused by pulse pileup, and random and coincidence sum peaks, all of which can lead to significant measurement biases.

Source of bottom-up value (only for prospective ITV not based on historical data)

To date, ITV values are from the analysis of lab data using FRAM in the high energy mode and counting time of 600 s. Depending on the source sizes and the measurement distances, the counting rates for the data varied from few kHz up to 50 kHz. For the low energy range, ITV values for DU, NU, and LEU are typically based on field data using MGAU and 300 s as counting time. In any case, only spectra with low to medium counting rates (< 10 kHz) were considered. The ITV values for HEU are from lab data at 300 s counting time.

Method references

- [1] T.E. Sampson, T.A. Kelley, PC/FRAM: a code for the non-destructive measurement of the isotopic composition of actinides for safeguards applications, Appl. Radiat. Isot. Vol. 48 (1997) 1543-1548.
- [2] D.T. Vo, "Improvements of FRAM version 6.1," LANL Report LA-UR-19-28449 (2019).
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