



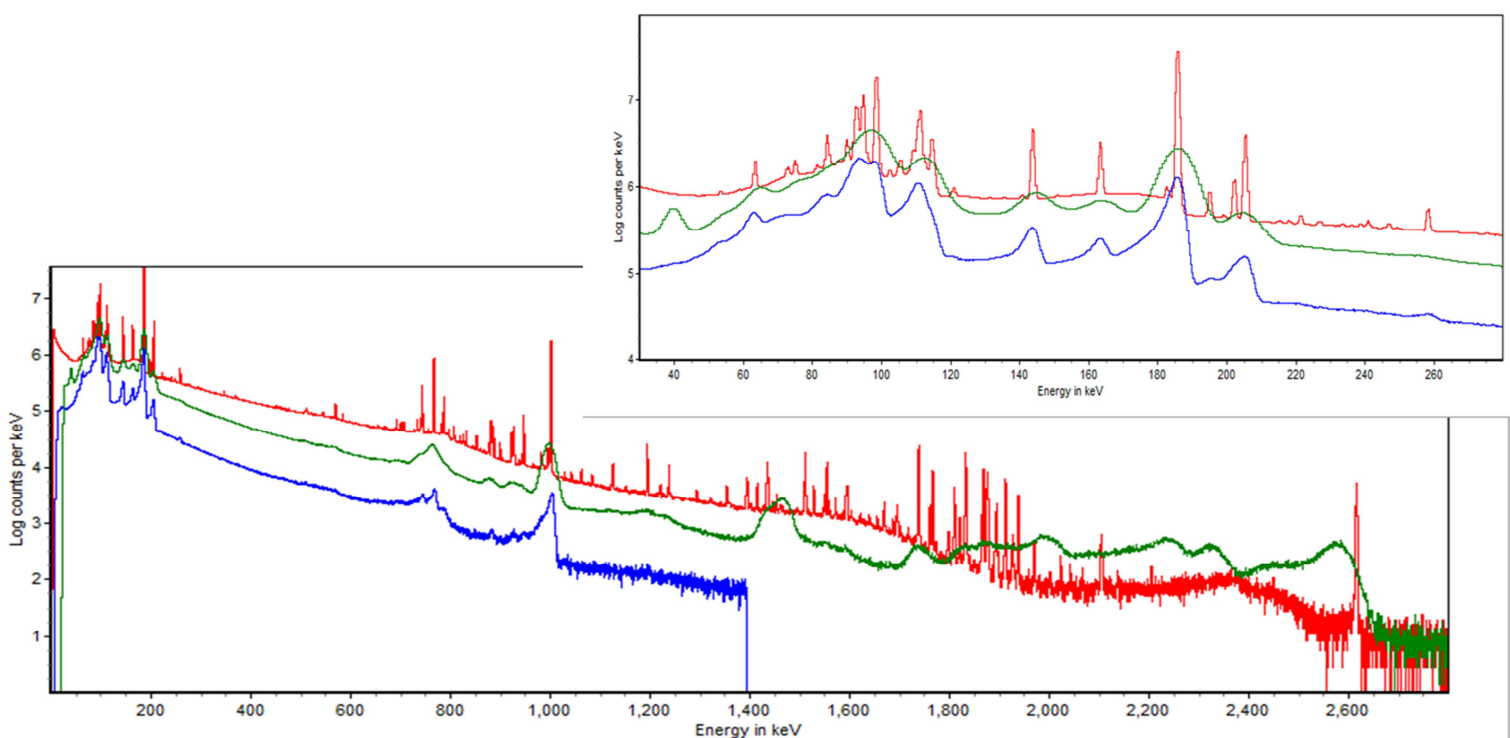
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Collection of medium-resolution gamma spectra of certified U reference materials

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Miguel Ramos Pascual

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Abstract

A collection of medium resolution gamma-ray spectra from well-characterized U certified reference materials has been recorded using LaBr₃(Ce) (2.0" x 0.5") and CZT (500 mm³) detectors. Aiming to acquire the highest quality reference data, the spectra were measured for long acquisition times, ensuring very good counting statistics across potentially useful spectral intervals - up to 1.4 MeV for the CZT and up to 2.8 MeV for the LaBr₃(Ce) detectors. The experimental setup assures that the measurement geometry is stable and reproducible, and that the spectra have minimum influence from background radiation and pile-up effects. The spectra are available at the data library of The international working group on gamma spectroscopy techniques for U and Pu isotopics. The spectra feed phase I of the U isotopic inter-comparison exercise jointly organized by the ESARDA NDA Working Group and IAEA.

1. Introduction

Medium resolution gamma detectors are becoming widely used for (in-field) nuclear safeguards-related measurements. Such detectors do not have to be cooled, so they have obvious benefits for in-field applications, like portability and easy maintenance. There still seems to be room for (further) development of medium resolution spectra analysis software and of applications for such detectors. High quality spectra are needed for testing/development.

This report describes collecting a set of high-quality reference medium-resolution gamma-spectra of U certified reference materials with CZT and LaBr₃ detectors in well-controlled conditions. The collection of analogous spectra of Pu reference materials is described in JRC technical report 98337 (Ref. 1). This work has been performed as a part of IAEA support project JNT A 01684 EC on Sustainability and Maintenance of Software for Pu-isotopics and U-enrichment. The spectra will be used within the international exercise on medium-resolution gamma spectrometry as reference spectra for gamma-ray analysis code developers, for testing their codes.

2. Experimental setup for U measurements

The spectra have been measured at the Institute of Transuranium Elements (ITU) using equipment (detectors, electronics and power supplies) provided by the IAEA. This equipment consisted of Ritec CZT 500S large volume hemispherical detector (500 mm³, no. 427), Saint Gobain Crystals LaBr₃:Ce detector (2"x0.5", mod. 51sea13, S/N 2301 with PM R6231-100-01), absorbers (3mm steel and 1mm Cd), Canberra InSpector 2000 electronics and netbooks with installed corresponding GENIE2000 software. The recorded spectra had 4096 channels with energy range up to 2.8 MeV for LaBr₃ and 1.4 MeV for CZT detector. The electronics has been set up accordingly.

A shielded experimental setup with well-defined geometry has been set for the gamma-spectrometric measurements in ITU hot lab. The shield consists of 2 mm of Cu and 5 cm Pb (with painted sheet metal on the outside). Mechanical setups for holding the Uranium reference samples at a fixed distance above the LaBr₃ and CZT detectors have been manufactured in the ITU workshop. The distances between elements of the experimental setup and the thicknesses of materials are presented in Table 1. PMMA there stands for poly(methyl methacrylate) ("plexiglass") with chemical composition (C₅O₂H₈)_n and density of ca. 1.18g/cm³. The sample-to-detector distances are distances between the top of the detector and the bottom of the sample's encapsulation (samples have been always placed on a flat PMMA surface, distance is always from the top of that surface to the detector's top). The samples have been positioned on the detector's axis.

Table 1: Setup distances and the thicknesses of materials

| Detector | Distance (mm) to the sample | Absolute uncertainty (mm) | Total thickness of material (mm) | |
|-------------------------|-----------------------------|---------------------------|----------------------------------|-------|
| | | | PMMA | Air |
| CZT | 19.63 | 0.05 | 5 | 14.63 |
| LaBr₃ | 120.0 | 0.1 | 10 | 110 |

Figures 1 and 2 show the dimensions relevant to the gamma spectrometric measurement. Figure 3 presents the photo of the experimental setup for CZT detector and U reference samples. Figure 4 presents the U LaBr₃ setup. Here the detector that is outside the experimental box is inside a cylinder with 1 cm Pb and 3 mm Cu shield.

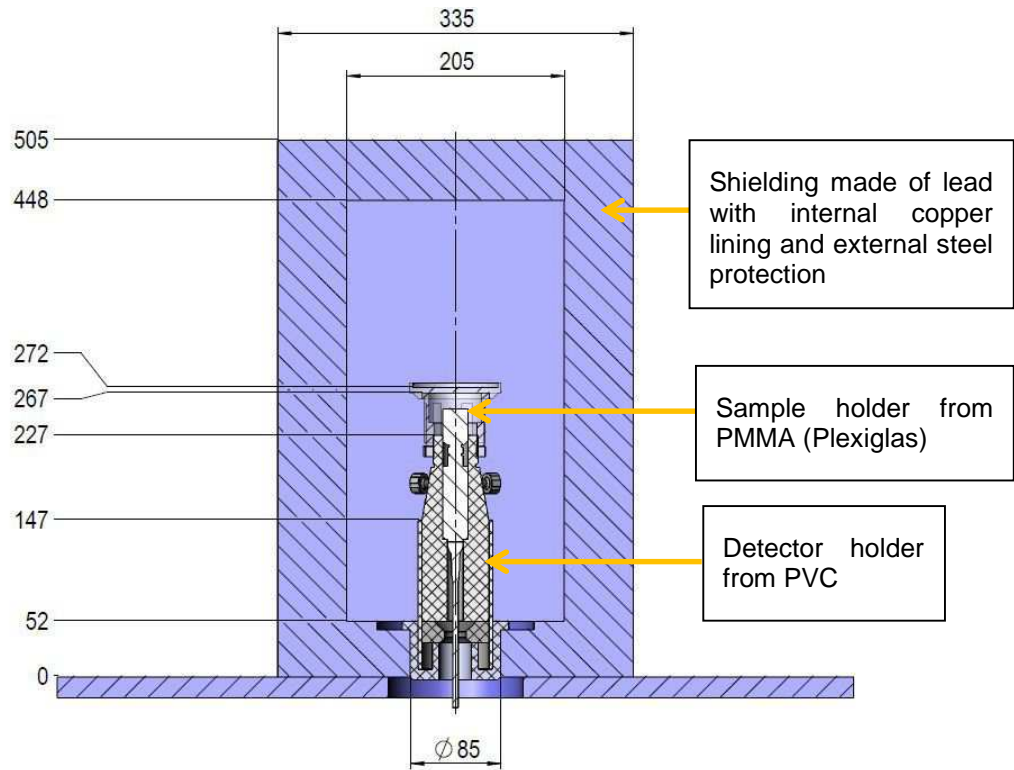


Figure 1: Experimental setup for the CZT detector inside lead shielding (detector to sample distance can be varied). The top of the detector is at 252.37 mm.

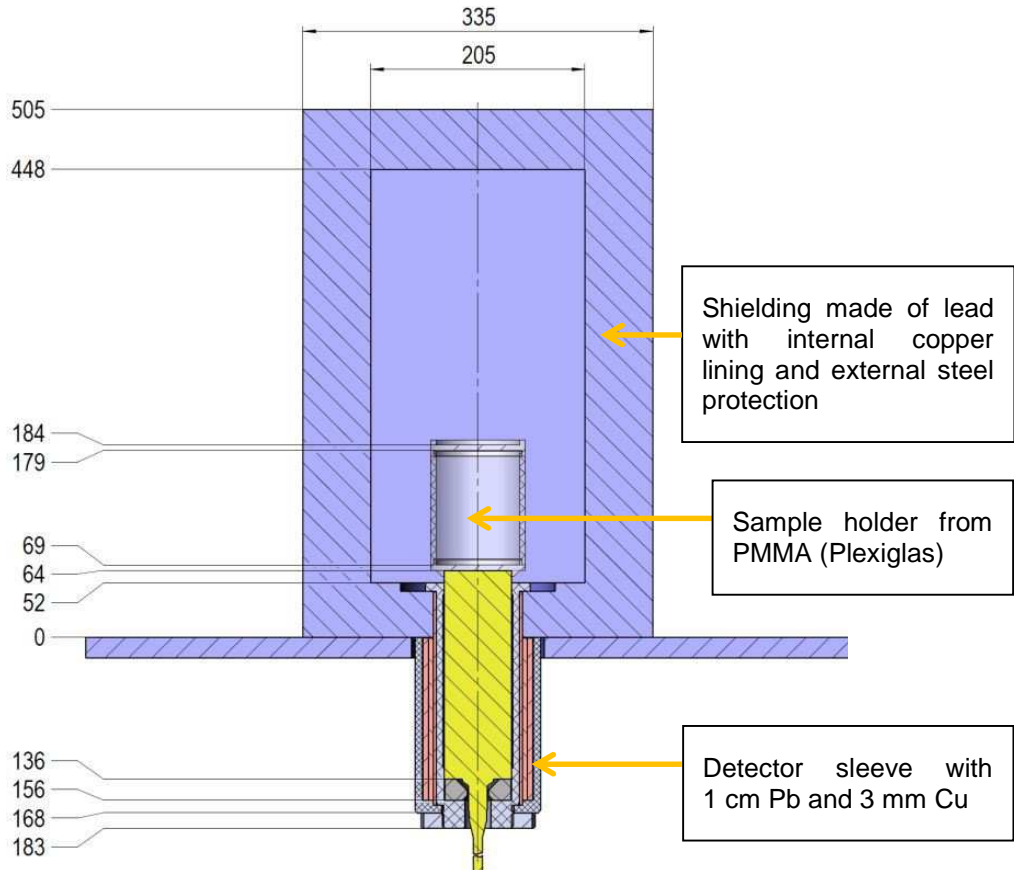


Figure 2: Experimental setup for the LaBr₃ detector inside lead shielding. The top of the detector is at 64 mm.

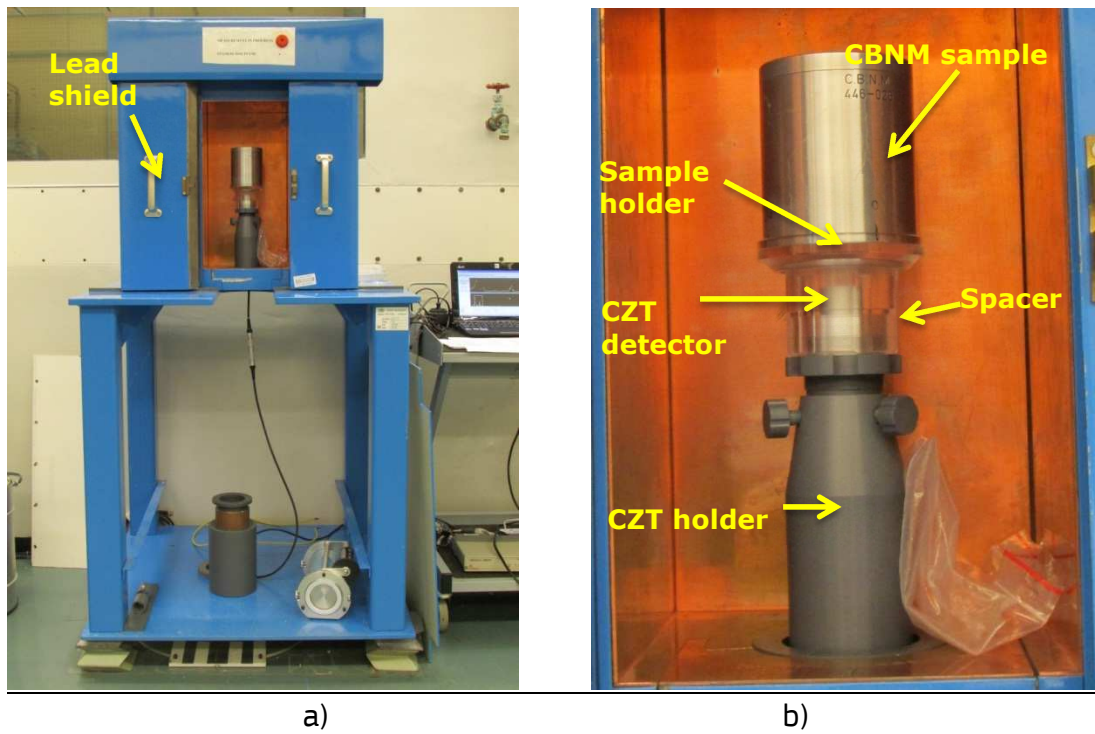


Figure 3: a) Open shield with CZT detector inside. b) Interior of the shield with the CZT detector and a CBNM sample above it.

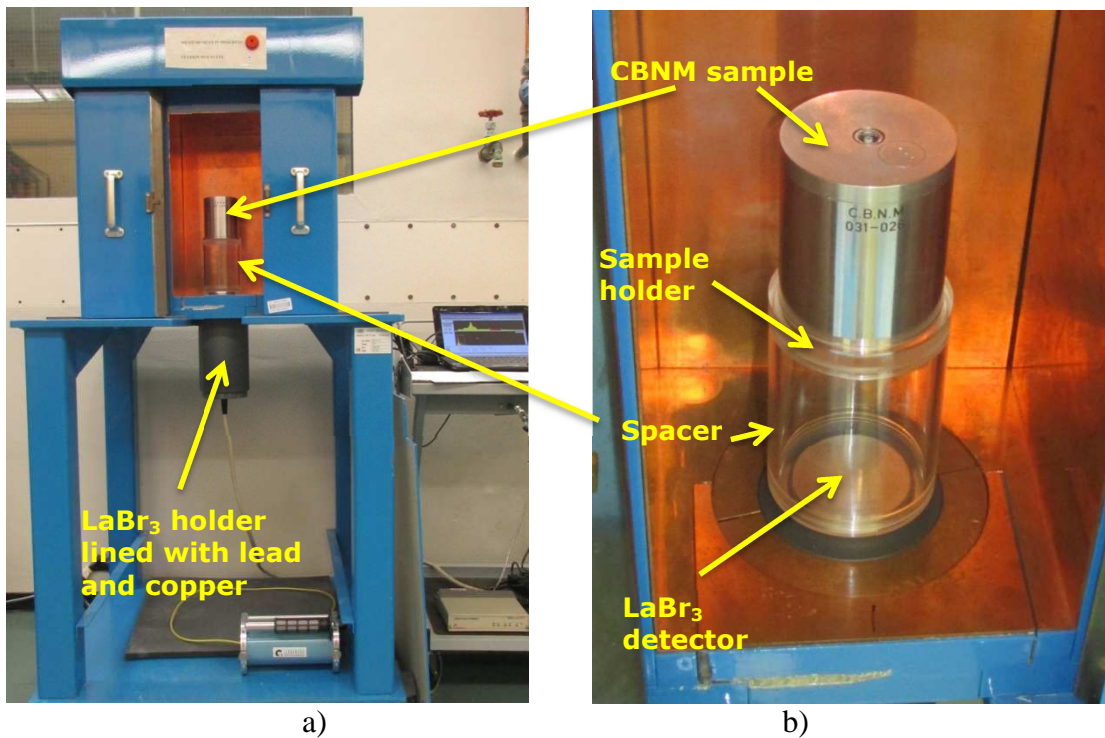


Figure 4: a) Open shield with LaBr₃ detector. b) Interior of the shield with CBNM sample above the LaBr₃ detector.

3. U reference samples

The set of reference samples measured has been set number 026 from the "EC Certified Nuclear Reference Material 171" (EC NRM 171). It consists of five items as described in Refs. 2 and 3. EC NRM 171 corresponds to NBS standard reference material 969 (Ref. 4).

Table 2: Isotopic composition of Uranium of EC NRM 171 reference samples in mass % (with 2s absolute uncertainty in parenthesis).

| Reference material | Mass % | | | | |
|--------------------|------------------|---------------------------|------------------|------------------|------------------|
| | ^{235}U | ^{232}U [Ref. 5] | ^{234}U | ^{236}U | ^{238}U |
| CBNM 031 | 0.3166(2) | $2.1(5)\times 10^{-10}$ | 0.0020(2) | 0.0146(3) | 99.6669(4) |
| CBNM 071 | 0.7119(5) | $<1\times 10^{-11}$ | 0.0052(2) | <0.00002 | 99.2828(4) |
| CBNM 194 | 1.9420(14) | $3(3)\times 10^{-11}$ | 0.0171(2) | 0.0003(1) | 98.0406(18) |
| CBNM 295 | 2.9492(21) | $6(2)\times 10^{-11}$ | 0.0279(4) | 0.0033(2) | 97.0196(29) |
| CBNM 446 | 4.4623(32) | $3.7(3)\times 10^{-10}$ | 0.0359(3) | 0.0068(2) | 95.4950(32) |

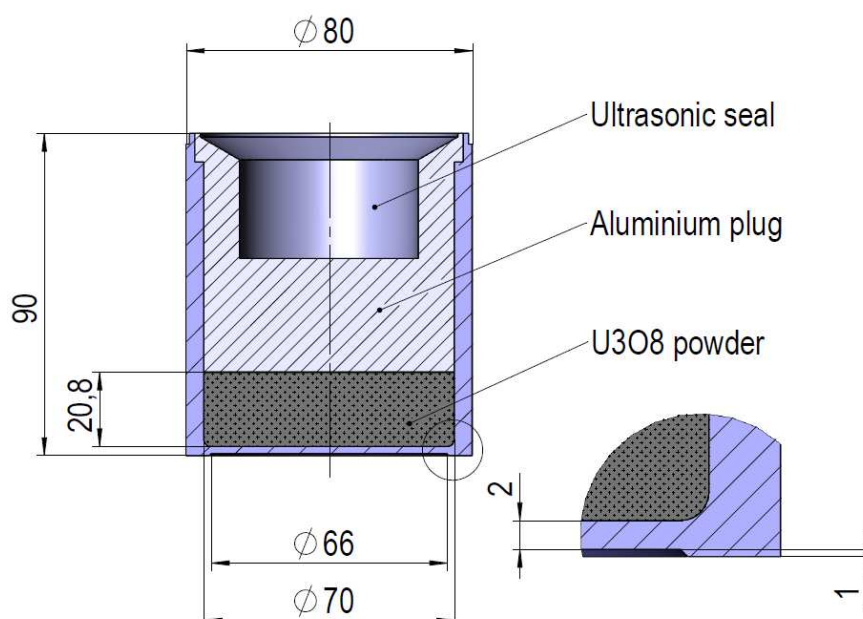


Figure 5: The uranium standard's drawing with nominal dimensions. (Reproduced based on Ref. 3) Filling height/material thickness is 20.8 mm for all samples except CBNM446. For CBNM446 it is 15.8 mm. The can material is Al-alloy ASTM 6061-T6.

EC NRM 171 has been produced by Central Bureau for Nuclear Measurements (CBNM) of the European Commission. Its reference samples consist of 200.1 ± 0.2 g U_3O_8 with surface density 5.2 ± 0.3 g $\times\text{cm}^{-2}$ in a sealed aluminum can. The isotopic composition of the samples is presented in Table 2, the sample's drawing is presented in Fig. 5 while the nominal dimensions and tolerances are given in Table 3. More details and samples' drawings and description are presented in Refs. 2-4. Only ^{235}U abundance is certified. Minor U isotopes are present in very small quantities (^{232}U less than 10^{-9} , ^{233}U less than 5×10^{-5} and $^{237}\text{U}+^{237}\text{Np}$ less than 3×10^{-6} of the mass of ^{235}U) as given in Refs. 2 and 5.

Table 3: Reference samples' nominal dimensions with tolerances (Ref. 3) (all data in mm). Specific dimensions for the particular set "026" are not available.

| | | | | |
|---------------------------------------|----------------------------|------|--------|-----------|
| Can | Height | 90 | ± | 0.2 |
| | Outer diameter | 80 | ± | (no data) |
| | Edge below window | 1 | ± | 0.05 |
| Can window | Thickness | 2 | ± | 0.02 |
| | Deviation from flatness | | ± | 0.1 |
| | Diameter | 66 | ± | 0.05 |
| U ₃ O ₈ filling | Height for material "446" | 15.8 | ± | 0.5 |
| | Height for other materials | 20.8 | ± | 0.5 |
| | Diameter | 70 | + - | 0.05 0 |

4. Measurements and experimental conditions

Five spectra of each reference sample with 100000s live time per spectrum have been recorded with each detector in experimental setup as described in chapter 2. Measurements in some series of measurements (but never a whole series of measurements) have been done by automatic repetition of the previous measurement (without the sample or anything else being moved in-between). Such measurements are marked in List of spectra.

The lab in which the measurements were performed had a slightly elevated temperature (up to ca. 28 degrees C) that was not very stable. This might have affected stability of the measurements (energy scale) and resolution of the spectra (especially spectra measured with LaBr₃ detector). Fine gain of LaBr₃ detector had to be corrected sometimes to maintain the same energy scale. Automatic energy scale stabilization was not used.

The power supply of the Inspector2000 has developed a fault during the measurements and became unable to provide stable power for the LaBr₃ detector and its electronics. The power supply has been replaced with a commercial one.

The logbook in List of spectra provides names of the recorded spectra, dates of the measurements as well as data on detector-to-sample distances used. The spectra, converted to Ortec CHN format, are available via the data library of The international working group on gamma spectroscopy techniques for U and Pu isotopics (Ref. 6).

5. Conclusion

High quality medium-resolution gamma spectra of U reference samples have been measured by CZT and LaBr₃ detectors. They will be used as reference in phase I of the international exercise on medium resolution gamma spectrometry. All spectra are available at no charge via the data library of The international working group on gamma spectroscopy techniques for U and Pu isotopics (Ref. 6).

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List of spectra

| Spectra of U standards recorded by CZT detector | | | | | | | | | |
|--|----------|-------------|--------------|--------|---------|-------|------------|------------------------------|------------------|
| Meas # | Sample | Replicate # | Distance, mm | Shield | LT, sec | DT, % | Meas. date | Spectrum name | Comment |
| 1 | None | 1 | N/A | None | 100000 | 0.00 | 2014-05-20 | CZT500_Ubackground_01.cnf | |
| 2 | CBNM-031 | 1 | 20 | None | 100000 | 0.55 | 2014-05-21 | CZT500_CBNM-031_@20mm_01.cnf | |
| 3 | CBNM-031 | 2 | 20 | None | 100000 | 0.56 | 2014-05-26 | CZT500_CBNM-031_@20mm_02.cnf | |
| 4 | CBNM-031 | 3 | 20 | None | 100000 | 0.56 | 2014-05-27 | CZT500_CBNM-031_@20mm_03.cnf | 3x auto sequence |
| 5 | CBNM-031 | 4 | 20 | None | 100000 | 0.56 | 2014-05-28 | CZT500_CBNM-031_@20mm_04.cnf | 3x auto sequence |
| 6 | CBNM-031 | 5 | 20 | None | 100000 | 0.56 | 2014-05-29 | CZT500_CBNM-031_@20mm_05.cnf | 3x auto sequence |
| 7 | None | 2 | N/A | None | 20000 | 0.00 | 2014-06-02 | CZT500_Ubackground_02.cnf | |
| 8 | CBNM-071 | 1 | 20 | None | 100000 | 0.61 | 2014-06-02 | CZT500_CBNM-071_@20mm_01.cnf | |
| 9 | CBNM-071 | 2 | 20 | None | 100000 | 0.61 | 2014-06-04 | CZT500_CBNM-071_@20mm_02.cnf | |
| 10 | CBNM-071 | 3 | 20 | None | 100000 | 0.61 | 2014-06-05 | CZT500_CBNM-071_@20mm_03.cnf | 3x auto sequence |
| 11 | CBNM-071 | 4 | 20 | None | 100000 | 0.60 | 2014-06-06 | CZT500_CBNM-071_@20mm_04.cnf | 3x auto sequence |
| 12 | CBNM-071 | 5 | 20 | None | 100000 | 0.60 | 2014-06-07 | CZT500_CBNM-071_@20mm_05.cnf | 3x auto sequence |
| 13 | None | 3 | N/A | None | 20000 | 0.00 | 2014-06-10 | CZT500_Ubackground_03.cnf | |
| 14 | CBNM-194 | 1 | 20 | None | 100000 | 0.75 | 2014-06-11 | CZT500_CBNM-194_@20mm_01.cnf | |
| 15 | CBNM-194 | 2 | 20 | None | 100000 | 0.75 | 2014-06-12 | CZT500_CBNM-194_@20mm_02.cnf | |
| 16 | CBNM-194 | 3 | 20 | None | 100000 | 0.75 | 2014-06-23 | CZT500_CBNM-194_@20mm_03.cnf | |
| 17 | CBNM-194 | 4 | 20 | None | 100000 | 0.75 | 2014-06-24 | CZT500_CBNM-194_@20mm_04.cnf | |
| 18 | CBNM-194 | 5 | 20 | None | 100000 | 0.76 | 2014-06-26 | CZT500_CBNM-194_@20mm_05.cnf | |

| | | | | | | | | | |
|----|----------|---|-----|------|--------|------|------------|------------------------------|------------------|
| 19 | None | 4 | N/A | None | 20000 | 0.00 | 2014-06-27 | CZT500_Ubackground_04.cnf | |
| 20 | CBNM-295 | 1 | 20 | None | 100000 | 0.88 | 2014-06-30 | CZT500_CBNM-295_@20mm_01.cnf | |
| 21 | CBNM-295 | 2 | 20 | None | 100000 | 0.88 | 2014-07-01 | CZT500_CBNM-295_@20mm_02.cnf | |
| 22 | CBNM-295 | 3 | 20 | None | 100000 | 0.87 | 2014-07-03 | CZT500_CBNM-295_@20mm_03.cnf | |
| 23 | CBNM-295 | 4 | 20 | None | 100000 | 0.87 | 2014-07-04 | CZT500_CBNM-295_@20mm_04.cnf | 2x auto sequence |
| 24 | CBNM-295 | 5 | 20 | None | 100000 | 0.87 | 2014-07-05 | CZT500_CBNM-295_@20mm_05.cnf | 2x auto sequence |
| 25 | None | 5 | N/A | None | 20000 | 0.00 | 2014-07-07 | CZT500_Ubackground_05.cnf | |
| 26 | CBNM-446 | 1 | 20 | None | 100000 | 1.08 | 2014-07-07 | CZT500_CBNM-446_@20mm_01.cnf | |
| 27 | CBNM-446 | 2 | 20 | None | 100000 | 1.09 | 2014-07-09 | CZT500_CBNM-446_@20mm_02.cnf | |
| 28 | CBNM-446 | 3 | 20 | None | 100000 | 1.09 | 2014-07-10 | CZT500_CBNM-446_@20mm_03.cnf | 3x auto sequence |
| 29 | CBNM-446 | 4 | 20 | None | 100000 | 1.09 | 2014-07-11 | CZT500_CBNM-446_@20mm_04.cnf | 3x auto sequence |
| 30 | CBNM-446 | 5 | 20 | None | 100000 | 1.09 | 2014-07-12 | CZT500_CBNM-446_@20mm_05.cnf | 3x auto sequence |
| 31 | None | 6 | N/A | None | 20000 | 0.00 | 2014-07-14 | CZT500_Ubackground_06.cnf | |

Notes:

Requirements/setup: DT <= 3%
RT = 5.6 us
FT = 0.8 us
4096 channels
0.34 keV/chn
keep 185.7keV peak in approx. 546.5 channel.

Exact distance detector-standard is 19.63 mm +- 0.05 mm (measured with vernier caliper).
Auto sequence means automatic remeasurement by software.

Spectra of U standards recorded by LaBr₃ detector

| Meas # | Sample | Replicate # | Distance, mm | Shield | LT, sec | DT, % | Meas. date | Spectrum name | Comment |
|--------|----------|-------------|--------------|--------|---------|-------|------------|------------------------------|--|
| 1 | None | 1 | None | None | 100000 | 0.01 | 2014-04-07 | LaBr3_Ubackground_01.cnf | |
| 2 | CBNM-031 | 1 | 120 | None | 100000 | 0.27 | 2014-04-09 | LaBr3_CBNM-031_@120mm_01.cnf | |
| 3 | CBNM-031 | 2 | 120 | None | 100000 | 0.27 | 2014-04-11 | LaBr3_CBNM-031_@120mm_02.cnf | |
| 4 | CBNM-031 | 3 | 120 | None | 100000 | 0.27 | 2014-04-14 | LaBr3_CBNM-031_@120mm_03.cnf | |
| 5 | CBNM-031 | 4 | 120 | None | 100000 | 0.27 | 2014-04-15 | LaBr3_CBNM-031_@120mm_04.cnf | |
| 6 | CBNM-031 | 5 | 120 | None | 100000 | 0.27 | 2014-04-17 | LaBr3_CBNM-031_@120mm_05.cnf | |
| 7 | None | 2 | None | None | 20000 | 0.01 | 2014-04-22 | LaBr3_Ubackground_02.cnf | |
| 8 | CBNM-071 | 1 | 120 | None | 100000 | 0.29 | 2014-04-22 | LaBr3_CBNM-071_@120mm_01.cnf | |
| 9 | CBNM-071 | 2 | 120 | None | 100000 | 0.29 | 2014-04-24 | LaBr3_CBNM-071_@120mm_02.cnf | |
| 10 | CBNM-071 | 3 | 120 | None | 100000 | 0.29 | 2014-04-25 | LaBr3_CBNM-071_@120mm_03.cnf | |
| 11 | CBNM-071 | 4 | 120 | None | 100000 | 0.29 | 2014-04-28 | LaBr3_CBNM-071_@120mm_04.cnf | |
| 12 | CBNM-071 | 5 | 120 | None | 100000 | 0.29 | 2014-04-29 | LaBr3_CBNM-071_@120mm_05.cnf | |
| 13 | None | 3 | None | None | 20000 | 0.01 | 2014-05-05 | LaBr3_Ubackground_03.cnf | thereafter new power supply |
| | | | | | | | | | fine gain changed from 1.5327 to 1.500 |
| 14 | None | 4 | None | None | 20000 | 0.01 | 2014-07-18 | LaBr3_Ubackground_04.cnf | |
| 15 | CBNM-295 | 1 | 120 | None | 100000 | 0.42 | 2014-07-18 | LaBr3_CBNM-295_@120mm_01.cnf | 3X automatic sequence measurement |
| 16 | CBNM-295 | 2 | 120 | None | 100000 | 0.42 | 2014-07-19 | LaBr3_CBNM-295_@120mm_02.cnf | 3X automatic sequence measurement |
| 17 | CBNM-295 | 3 | 120 | None | 100000 | 0.42 | 2014-07-20 | LaBr3_CBNM-295_@120mm_03.cnf | 3X automatic sequence measurement |
| 18 | CBNM-295 | 4 | 120 | None | 100000 | 0.42 | 2014-07-22 | LaBr3_CBNM-295_@120mm_04.cnf | |
| 19 | CBNM-295 | 5 | 120 | None | 100000 | 0.42 | 2014-07-23 | LaBr3_CBNM-295_@120mm_05.cnf | |

| | | | | | | | | | |
|----|----------|---|------|------|--------|------|------------|------------------------------|--|
| 20 | None | 5 | None | None | 20000 | 0.01 | 2014-07-24 | LaBr3_Ubackground_05.cnf | |
| | | | | | | | | | Restart of measurements |
| | | | | | | | | | Std settings, fine gain of 1.5327 |
| 21 | None | 6 | None | None | 20000 | 0.01 | 2014-08-25 | LaBr3_Ubackground_06.cnf | |
| | | | | | | | | | fine gain changed from 1.5327 to 1.500 |
| 21 | CBNM-194 | 1 | 120 | None | 100000 | 0.37 | 2014-08-26 | LaBr3_CBNM-194_@120mm_01.cnf | |
| 22 | CBNM-194 | 2 | 120 | None | 100000 | 0.37 | 2014-08-27 | LaBr3_CBNM-194_@120mm_02.cnf | |
| 23 | CBNM-194 | 3 | 120 | None | 100000 | 0.37 | 2014-08-29 | LaBr3_CBNM-194_@120mm_03.cnf | |
| 24 | CBNM-194 | 4 | 120 | None | 100000 | 0.37 | 2014-09-01 | LaBr3_CBNM-194_@120mm_04.cnf | |
| 25 | CBNM-194 | 5 | 120 | None | 100000 | 0.37 | 2014-09-02 | LaBr3_CBNM-194_@120mm_05.cnf | |
| 26 | None | 7 | None | None | 20000 | 0.01 | 2014-09-04 | LaBr3_Ubackground_07.cnf | |
| 27 | CBNM-446 | 1 | 120 | None | 100000 | 0.51 | 2014-09-05 | LaBr3_CBNM-446_@120mm_01.cnf | 3X automatic sequence measurement |
| 28 | CBNM-446 | 2 | 120 | None | 100000 | 0.51 | 2014-09-06 | LaBr3_CBNM-446_@120mm_02.cnf | 3X automatic sequence measurement |
| 29 | CBNM-446 | 3 | 120 | None | 100000 | 0.51 | 2014-09-07 | LaBr3_CBNM-446_@120mm_03.cnf | 3X automatic sequence measurement |
| 30 | CBNM-446 | 4 | 120 | None | 100000 | 0.51 | 2014-09-09 | LaBr3_CBNM-446_@120mm_04.cnf | 2X automatic sequence measurement |
| 31 | CBNM-446 | 5 | 120 | None | 100000 | 0.51 | 2014-09-10 | LaBr3_CBNM-446_@120mm_05.cnf | 2X automatic sequence measurement |
| 32 | None | 8 | None | None | 100000 | 0.01 | 2014-09-12 | LaBr3_Ubackground_08.cnf | |

Notes:

Requirements/setup: DT <= 3%
RT = 5.6 us
FT = 0.8 us
4096 channels
0.68 keV/chn

Automatic sequence measurement means the measurements have been automatically repeated by software.
Fine gain changed during measurements to keep energy scale close to 0.68 keV/ch.

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