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Certification of Activity Concentration of Radionuclides in IAEA-479 Milk Powder

IAEA
International Atomic Energy Agency

CERTIFICATION OF ACTIVITY CONCENTRATION OF RADIONUCLIDES IN IAEA-479 MILK POWDER

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CERTIFICATION OF ACTIVITY CONCENTRATION OF RADIONUCLIDES IN IAEA-479 MILK POWDER

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FOREWORD

To ensure the reliable evaluation of potential radiological hazards and proper decision making on radiation protection measures, the IAEA's Terrestrial Environmental Radiochemistry Laboratory supports Member State laboratories in maintaining their emergency response preparedness and improving the quality of analytical results. It does so by producing certified reference materials, conducting interlaboratory comparisons and proficiency tests, and developing standardized methods for sample collection and analysis that can be used as tools for external quality control of analytical results.

Since the accident at the Fukushima Daiichi nuclear power plant in 2011 and the subsequent release of radionuclides to the surrounding environment, the demand for contaminated food-based certified reference materials has remained high. Additionally, laboratories will ideally maintain their radioanalytical analysis capabilities during normal conditions to ensure that reliable analytical results are available for any emergency response purposes. Following the sale of all units of the certified reference material IAEA-473 (short-lived radionuclides in milk powder), a new certified reference material, IAEA-479 (radionuclides in milk powder), was prepared with known activity concentrations of ^{90}Sr, ^{133}Ba, ^{134}Cs and ^{137}Cs. This publication presents the methodologies used for the production and certification of IAEA-479.

The IAEA officers responsible for this publication were S. Tarjan and S. Patterson of the Division of Physical and Chemical Sciences.

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1. INTRODUCTION

To meet the IAEA Member States' needs for a certified reference material (CRM) of short-lived radionuclides in milk powder following the Fukushima Daiichi nuclear accident, the Terrestrial Environmental Radiochemistry Laboratory (TERC) prepared IAEA-479 CRM as a replacement of the previous out-of-stock material IAEA-473.

Its matrix is a full fat milk powder from which milk with a fat content 3.5% can be reconstituted. This CRM contains one radio-strontium isotope ^{90}Sr as a typical contaminant in milk during and after a nuclear emergency. To simulate real accidental circumstances, both radio-caesium ^{134}Cs and ^{137}Cs are added to the CRM. ^{131}I is also an important radionuclide in nuclear accidents, however considering its short half-life it is not possible to include it in a CRM. Therefore ^{133}Ba was added which has similar characteristics in terms of radiation energy, to simulate the presence of ^{131}I in the CRM.

This publication presents the preparation methodology, materials, and assignment of property values and their associated uncertainties for ^{90}Sr, ^{133}Ba, ^{134}Cs, and ^{137}Cs. The milk powder also contains readily detectable ^{40}K due to its high natural potassium content, and its activity concentration has been determined by measurement of four samples. The activity concentration of ^{40}K is included as an information value of the CRM. Similarly to IAEA-473, IAEA-479 CRM can be used for the verification of performance indicators (precision, repeatability, reproducibility) of ^{90}Sr in presence of radio-caesium and ^{133}Ba isotopes.

2. METHODOLOGY

2.1. PREPARATION OF THE MATERIAL

The raw material used for production of IAEA-479 was high quality milk powder (26% fat content) manufactured in Austria by the ALPI Milchverarbeitungs und Handels GMBH. The raw milk powder was analysed for both anthropogenic gamma-ray emitting radionuclides and radio-strontium isotopes.

For gamma emitting radionuclides, a 60% relative efficiency P-type extended range HPGe detector was used, in 10 cm multi-layer shielding made of lead, cadmium and high purity electrolyte copper. The conditions of the gamma-ray spectrum analysis are summarized in Table 1.

TABLE 1. CONDITIONS OF THE GAMMA-RAY SPECTROMETRY ANALYSIS OF THE RAW MATERIAL

Detector	Broad Energy HPGe in U-style cryostat BEGe5030
Energy range	20-2000 keV
Resolution at 122 keV	0.72 keV
Resolution at 1332.5 keV	2.0 keV
Size of the spectrum	8192 channels
Shielding	CANBERRA
Sample geometry	100 cm^3 cylindrical sample container
Sample mass	72.0 g
Counting time	320000 s
Spectrum evaluation	Genie 2000
Efficiency calibration	LABSOCS (using detector characterization) and isotope calibration with efficiency transfer by EFFTRAN

The results for ^{133}Ba, ^{134}Cs and ^{137}Cs are less than their detection limits. 0.37, 0.41 and 0.26 Bq·kg^{-1} respectively, with a confidence level of approx. 95 % for a one-sided normal distribution, calculated according to ISO11929:2019 [1]. The ^{90}Sr activity concentration was determined by traditional radiochemical separation and beta counting from solid preparatum measured by a low background gas proportional counter (LB5). The ^{90}Sr activity concentration was below the 0.3 Bq·kg^{-1} detection limit.

2.1.1. Spiking

The CRM was prepared by an in-house spiking technique, using dilutions of high precision radioactive reference solutions (RSSs) from metrological institutes.

The main steps of the applied spiking technique are listed below:

1. Planning the composition of the master spike solution (MSS):
2. The required aliquots of the diluted RSSs are added to the MSS using plastic ampoules, the material balances were measured by weighing on a five-digit balance:
3. The isotope mixture was diluted with analytical purity 2-propanol (iso-propanol) up to 50 cm^3 volume:
4. The prepared MSS was dispersed quantitatively in 500 g of raw milk powder material and dried under ambient laboratory conditions.

2.1.2. Homogenization

The homogenization was carried out in two steps:

1. The spiked concentrate (500 g) was homogenized in a TURBULA T2G powder blender (see Fig.1) with ceramic balls (added to remove potential clumps):
2. The homogenized spiked concentrate was diluted to 25 kg with additional raw milk powder and further homogenized in the TURBULA 51 power blender.

FIG. 1. Turbula T2G powder blender.

2.1.3. Bulk homogeneity test

The bulk homogeneity test was necessary to ascertain the suitability of the material prior to bottling. The target uncertainty of the assigned values was planned to be less than 5%, when analysing a 15 g sample size.

After 100 hours of homogenization, five samples (~ 12 g) were taken for the bulk homogeneity test. Two samples were taken immediately after the homogenizer had stopped, then the homogenizer was restarted for additional 5 minutes. After this short remixing, three more samples were taken.

2.2.3.1. Measurements

No chemical or physical reasons for radionuclide separation during the spiking process of the raw material were identified, as one MSS containing all the radionuclides was prepared and added. Therefore gross beta counting was a suitable method to check the bulk homogeneity of the material.

The bulk homogeneity of the material was tested two ways; by gross beta measurement using a liquid scintillation counter (LSC) Quantulus 1220, and by a low background alpha-beta counter (LB5 ULB) equipped with gas-flow proportional detector. The LSC conditions are summarized in Table 2.

TABLE 2. CONDITIONS OF THE BULK HOMOGENEITY TEST BY LSC

Counting technique	Liquid scintillation
Equipment	Quantulus 1220
Counting mode	High energy beta
Scintillator	Insta-Gel Plus
Type of the vial	Low diffusion, plastic
Sample	1 g milk powder[1]
High purity water	5 cm^3
Scintillator	15 cm^3
Counting time	3600 s
Number of Cycles	4
Number of samples to be analysed	25 (5 subsamples from 5 samples)

[1]The sample was first dissolved in water (shaking), then the scintillator was added.

The stability of the sample was demonstrated for the applied measurement period. The counting time was 1 hour per sample and counting was repeated four times (4 cycles).

For gas-flow proportional beta counting, 1 g of sample was distributed evenly on the surface of planchet using a few drops of iso-propanol and allowed to air-dry. Once completely dried, all samples were measured on the LB5 gas-flow counter for 1 hour, repeated for four cycles. Both the LSC and gas-flow measurements were performed using the same sample sets and cycles, so they were directly comparable.

Using the average value of the four runs, the analysis of variance statistical model (ANOVA), using a single factor analysis shows that the sample is homogenous using this sampling pattern and sample size (1g). These results are shown in Table 3.

TABLE 3. ANOVA RESULTS ON SAMPLE AVERAGE VALUES (LSC)

Source of Variation	Sum of Squares *(SS)*	Degrees of Freedom *(df)*	Mean of Squares *(MS)*	ANOVA test statistic *(F)*	Probability *(P-value)*	F_{crit}
Between Groups	33298.5	4	8324.6	1.3691	0.279997	2.8661
Within Groups	121603.6	20	6080.2			
Total	154902.1	24				

The calculation of the arithmetical mean of the four repeated cycles decreases the variation from the full data set. Therefore, to eliminate this smoothing effect the ANOVA was repeated on the full dataset (see results in Table 4).

TABLE 4. ANOVA RESULTS ON FULL DATASET (LSC)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	F_{crit}
Between Groups	172180.7	19	9062.1	**1.0560**	0,410871	**1.7180**
Within Groups	686517.2	80	8581.5			
Total	858697.9	99				

In both cases (Table 3 and 4), the experimental F value is less than the F_{crit}, therefore the sample is considered to be homogeneous from a statistical point of view.
The estimated average variance is 8625 from the repeated measurement cycles, and this the typical standard deviation (square root of the average variance) due to the repeatability of the counting process is 92.9 (3.2%) with a repeatability limit of 9.3%.

The average variance within samples (subsamples to sample) is very low (2.9%) and the estimated repeatability limit covers the range of the measurement results from -4.7% up to 9.0%. The detailed statistical parameters regarding the LSC measurements are in Table 5.

TABLE 5. DETAILED STATISTICAL PARAMETERS (LSC)

Parameters	Samples	All repeated measurements
Number of available results	25	100
Minimum, counts/h (%)	2746 (-4.7)	2669 (-7.4)
Maximum, counts/h (%)	3141 (9.0)	3192 (10.7)
Median, counts/hr	2891	2876
Arithmetical mean, counts/h	2883	2883
Standard deviation (SD), counts/h	78.7	92.7
Relative (rel) SD, %	2.7	3.2
Detection uncertainty (det unc), counts/h	53.7	53.7
Rel det unc, %	1.9	1.9
Estimated measurement repeatability, %	3.2	
Repeatability limit, %	9.3	

Measurement results by gas proportional counter (LB5) using the same sampling pattern and evaluation are presented in Table 6.

TABLE 6. ANOVA RESULTS ON SAMPLE AVERAGE VALUES (LB5)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	2483.8	4	620.95	1.5878	0.216314	2.8661
Within Groups	7821.7	20	391.1			
Total	10305.5	24				

The results of the ANOVA on the full dataset also confirms the homogeneity of the material in Table 7, and the detailed basic statistical summary is presented in Table 8.

TABLE 7. ANOVA RESULTS ON FULL DATASET (LB5)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	23574.1	19	1240.7	1.1555	0.3159	1.7180
Within Groups	85872.8	80	1073.4			
Total	109446.9	99				

TABLE 8. DETAILED STATISTICAL PARAMETERS (LB5)

Parameters (gas proportional counter)	Samples	All repeated measurements
Number of available results	25	100
Minimum, counts/h (%)[1]	817 (-3.4)	772 (-8.9)
Maximum, counts/h (%)[1]	893 (5.7)	928 (9.5)
Median, counts/h (%)[1]	842 (-0.3)	850 (0.35)
Arithmetical mean, counts/h	845	847
SD, counts/h	20.1	33.1
Rel SD, %	2.4	3.9
Det unc, counts/h	29.1	29.1
Rel det unc, %	3.4	3.4
Estimated measurement repeatability, %	3.9	
Repeatability limit, %	11.2	

[1] The distance from the arithmetical mean value expressed in % is in brackets

The trend is the same as for the LSC measurements, however, the estimated repeatability and repeatability limits are larger due to counting statistics. The symmetry of the distribution is slightly better, which is confirmed by the range and the distance between the median and arithmetical mean.

Using the average values of the four cycles, the ANOVA single factor analysis shows that the material is homogeneous for this sampling pattern and sample size. Both measurement series demonstrate that the material is homogeneous, is suitable for the planned purpose, and is ready for bottling.

2.1.4. Particle size distribution

The particle size distribution was determined by a laser scattering particle size analyser (Horiba LA 950) using dry conditions. The 10% and 90% limits of the grain size distribution are 50 and 280 microns respectively.

2.1.5. Bottling

The IAEA-479 CRM was bottled in normal laboratory conditions during one working day. The bottles were labelled with pre-printed stickers bearing a serial number according to the production order, arranged into plastic boxes and sterilized using gamma-ray irradiation with a total dose of 25 kGy using a ^{60}Co source.

The unit size was determined to be 100 g; the exact weight of the material in each bottle was recorded and used for establishing the final material balance of the entire production process. The bottles have a wide secure-sealed lid to preserve the integrity of the CRM in the bottle.

The average moisture content of the material after bottling was determined by drying 2 g overnight at 80 °C and was 3.94%.

2.2. FINAL HOMOGENEITY STUDY

Immediately after bottling, seven out of the 200 bottles were randomly selected for the final homogeneity study. During the planning phase the following parameters were taken into consideration;

- The method repeatability:
- The minimum sample size equal to the homogeneity test portion (15 g):
- Less than 1% detection uncertainty (at minimum 10000 detected counts).

The measurements were carried out by gamma-ray spectrometry on pellets prepared using ~15 g of material with a high-pressure pelletizer under 35 tons pressure (see Fig. 2). Three subsamples were taken from each bottle (without mixing). The exact weight of the pellets was determined and considered during the evaluation of the results. The thickness of the pellets was controlled by precision callipers for ensuring the uniformity of the sample geometry.

FIG. 2. Pellets from milk powder produced by high pressure pelletizer

The conditions of the sample preparation and gamma-ray spectrum analysis are summarized in Table 9.

TABLE 9. CONDITIONS OF THE HOMOGENEITY STUDY

Sampling and Sample Preparation	
Number of subsamples from each bottle	3
Sample mass	15.0 g
Pelletizer	Retsch PP35
Applied pressure	35 tons
Dwell time	20 s
Release time	Automatic
Diameter of the pellet	40 mm
Thickness of the pellet	9.7±0.05 mm
Measurement by the following gamma-ray spectrometer system	
Detector	N-type HPGe
Relative efficiency	45%
Resolution	0.85 keV (at 122 keV) 1.95 keV (at 1332.5 keV)
Cryostat	Low background, Slim line
Energy range	20-2000 keV
Spectrum size	8196 channels
Shielding	10 cm lead and 1 mm copper
Counting time (live time)	160000 s
Software	Genie 2K

The spectra were evaluated by mass and decay corrected net peak areas (counts detected during the measurement time) of the 604.72 keV energy peak of ^{134}Cs and the 661.67 keV energy peak of ^{137}Cs. Decay correction was necessary for both radionuclides due to the required counting time (2 days), and the duration of the homogeneity and short-term stability study period (118 days). Caesium-134 and ^{137}Cs were selected as homogeneity markers, and the results are summarized in Table 10.

TABLE 10. STATISTICAL EVALUATION OF THE FINAL HOMOGENEITY STUDY

Parameter	^{134}Cs (604.72 keV)	^{137}Cs (661.67 keV)
Number of samples	21	
Number of considered measurements	21	
Sample mass	15.04-16.78 g	
Average count number (mass and decay corrected)	16399	20315
SD	300	385
Rel SD, %	1.8	1.9
Median count number	16382	20435
Minimum count number	15945	19584
Maximum count number	16957	20932
Rel unc from radioactive decay[1], %	0.8	0.7
Estimated unc contribution heterogeneity, %	**1.7**	**1.8**
F (crit, α=0.05)	2.85	2.85
F (experimental)	2.74	2.72
s^2_{wb} (within bottle)	61871	102568
s^2_{bb} (between bottle)	35970	58940
u_{wb}	249	320
u_{bb}	190	243
Estimated rel unc contribution ANOVA [2], %	1.61	1.35

[1] Calculated from the square-root of the detected count numbers

The measurement results (including stability measurements) are shown in Fig. 3, where ^{134}Cs and ^{137}Cs measurement results are shown in blue and red respectively.

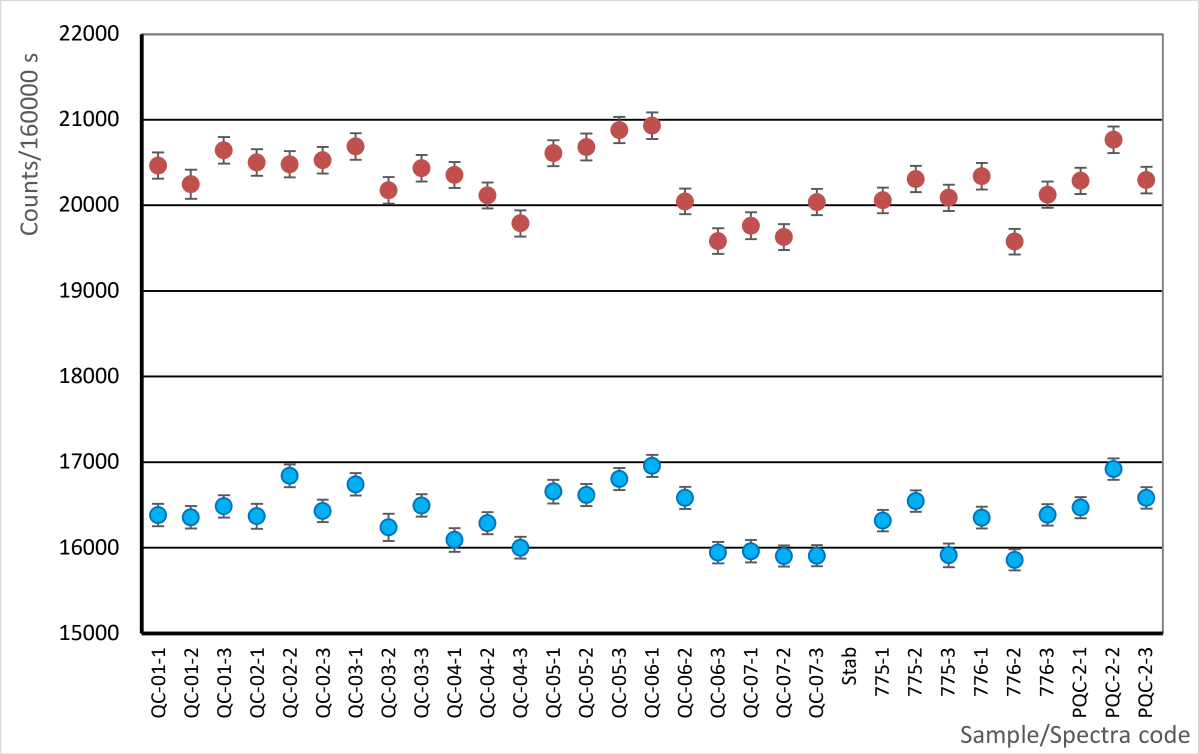

FIG. 3. Results of the homogeneity test for ^{134}Cs and ^{137}Cs.

A comprehensive evaluation of measurement results was carried out on this CRM to estimate the uncertainty from its heterogeneity. Between-bottle homogeneity was calculated from the net counts to eliminate other uncertainty components, increasing the visibility the contribution of heterogeneity. The sampling pattern was three uniform subsamples from each of the randomly selected seven bottles of IAEA-479. The measurements have been carried out on the gamma-ray spectrometer system detailed in Table 9. The uncertainty contribution of the material heterogeneity was estimated two different ways, and the conservative values 1.7 and 1.8 % have been applied in the final uncertainty budget.

At first the known uncertainty components were subtracted from the standard deviation of measurement results and it was assumed that the remaining uncertainty represents the sample heterogeneity.

Secondly, the dataset was evaluated by ANOVA, according to the recommendation of ISO Guide 35:2017 [2]. From regular ANOVA parameters, the within and between bottle homogeneity was determined using the recommended formula from ISO Guide 35:2017:

$$s_{wb}^2 = MS_{within} \tag{1}$$

$$s_{bb}^2 = \frac{MS_{among} - MS_{within}}{n_0} \tag{2}$$

$$u_{hom} = \sqrt{u_{bb}^2 + u_{wb}^2} \tag{3}$$

Where:

MS_{among}	Mean square (ANOVA) between bottles
MS_{within}	Mean square (ANOVA) within bottles
n	Number of observations
n_{bot}	Number of bottles
n_0	Effective number of group members (number of observations)
s_{bb}^2	Variance between bottles
s_{wb}^2	Variance within bottles
u_{bb}, u_{wb}	Uncertainty associated with the between-bottle and within bottle heterogeneity, respectively.

The s_{wb}^2 were less than the s_{bb}^2 for both isotopes, therefore equation (4) [2] was used for the calculation of u_{bb}:

$$u_{bb} = \sqrt{\frac{MS_{within}}{n_0}} \cdot \sqrt[4]{\frac{2}{Df_{within}}} \tag{4}$$

Where:

Df_{within}	Degree of freedom (within bottles).

No data was excluded from the ANOVA pattern, thus the $n_0 = n$ (number of observations) is used for the estimation of the s_{bb} value. The results for ^{134}Cs and ^{137}Cs were 1.61% and 1.35% respectively, however they were considered as unrealistically low values.

2.3. SHORT-TERM STABILITY ANALYSIS

The short-term stability test is intended to simulate possible harsh transport conditions and give indications about the behaviour of the material under extreme conditions. Storing milk powder at high temperatures may change its colour and re-solubility (in water), which can limit its utilization as a CRM. Whilst it is unlikely that these physical property changes would affect the assigned values or uncertainties, it is recommended that the milk powder CRM should not be used if changes to these physical properties are observed, especially if the laboratory reconstitutes it to milk prior to analysis.

The initial test (two weeks stored at 70 °C) resulted in the milk powder turning brown. Therefore, the stability test was performed at -25 °C and +40 °C for 2 weeks, after which time, the observable physical properties remained the same.

To increase sensitivity for stability issues [3], the same sample size, geometry and other counting conditions were the same as detailed in Table 9, enabling a comparative measurement based on the detected count numbers after correction for radioactive decay and mass. The arithmetical mean and its standard deviation derived from the homogeneity study has been used as the reference value. In this way any stability issues should be more specific to the material properties, without contributions from the sample preparation and counting uncertainty. The short-term stability results, for ^{134}Cs and ^{137}Cs are summarized in Table 11 and 12.

TABLE 11. SHORT-TERM STABILITY RESULTS FOR ^{134}Cs, 604.72 keV

		Detected counts (160000 s) [1]		
Sample ID	Temperature, °C	Counts	Count unc	Zeta-score
775-1	-25	16071.0	132.8	-0.95
775-2	-25	16546.1	135.2	0.49
775-3	-25	15913.8	150.1	-1.40
776-1	-25	16353.9	135.2	-0.09
776-2	-25	15860.3	133.2	-1.60
776-3	-25	16385.2	136.0	0.00
PQC-2-1	+40	16438.2	136.6	0.16
PQC-2-2	+40	16931.6	139.8	1.65
PQC-2-3	+40	16474.8	137.1	0.28
PQC-1-1	+40	16199.8	137.0	-0.56
PQC-3-1	+40	16728.5	152.5	1.02
PQC-4-1	+40	16540.0	138.8	0.47
Reference value		16384	300	

[1] Mass and decay corrected

TABLE 12. SHORT-TERM STABILITY RESULTS FOR ^{137}Cs, 661.67 keV

		Detected counts (160000 s) [1]		
Sample ID	Temperature, °C	Counts	Count unc	Zeta-score
775-1	-25	19756.6	148.0	-1.32
775-2	-25	20310.2	152.3	0.02
775-3	-25	20090.6	153.9	-0.51
776-1	-25	20340.7	153.7	0.10
776-2	-25	19577.5	150.1	-1.75
776-3	-25	20126.5	151.3	-0.42
PQC-2-1	+40	20248.0	151.4	-0.13
PQC-2-2	+40	20781.2	155.2	1.16

TABLE 12. SHORT-TERM STABILITY RESULTS FOR ^{137}Cs, 661.67 keV

		Detected counts (160000 s)[1]		
Sample ID	Temperature, °C	Counts	Count unc	Zeta-score
PQC-2-3	+40	20163.9	152.4	-0.33
PQC-1-1	+40	19770.0	150.9	-1.28
PQC-3-1	+40	20670.0	154.6	0.89
PQC-4-1	+40	20038.8	150.1	-0.63
Reference value		20301	385	▨

[1] Mass and decay corrected.

The Zeta-score indicates there is no significant difference in the results and therefore the short-term stability is acceptable for these conditions.

2.4. LONG-TERM STABILITY

Based on the experience of IAEA-473 Milk Powder CRM, the expiry date is set for four years from the release date, assuming the material is stored as described in the handling and storage section.

3. ASSIGNMENT OF PROPERTY VALUES AND ASSOCIATED UNCERTAINTIES

3.1. CALCULATION OF CERTIFIED PROPERTY VALUES

The assigned values were determined by "formulation" based on the specified activity concentration on the certificates of the high precision RSSs and the applied dilutions. The raw milk powder does not contain any detectable radio-caesium and radio-strontium isotopes. Therefore, the absolute activities of the MSS and the identified losses during the preparation steps were used for calculation of the property values.

As few tools as reasonably possible were used in the spiking process to minimize losses due to contact with the spike solution and the spiked material. Any losses were determined by measurement of the remaining activity on the surface of the tools by gamma-ray spectrometry.

The assigned certified values and estimated uncertainties, and the half-life used for decay correction of each radionuclide [5] are presented in Table 13.

TABLE 13. CERTIFIED VALUES FOR ACTIVITY CONCENTRATIONS
(based on dry mass at reference date 01 January 2020)

Radionuclide	**Certified value** [Bq kg^{-1}]	**Uncertainty[1]** [Bq kg^{-1}]	**Half-life[5]**
^{90}Sr	41.2	1.0	28.80 (7) years
^{133}Ba	30.3	0.7	10.539 (6) years
^{134}Cs	213.0	4.7	2.0644 (14) years
^{137}Cs	228.6	5.0	30.05 (8) years

[1] The uncertainty is expressed as a combined standard uncertainty using a coverage factor $k = 1$ estimated in accordance with the JCGM 100:2008 [4] and ISO Guide 35 [2].

For the uncertainty estimation the following contributors were included in the calculation budget:

- Uncertainty of the high precision RSSs (from the certificates):
- Uncertainty of the dilution steps (gravimetric, negligible component):
- Losses of the spiked activity (negligible component):
- Uncertainty of the milk powder mass determination (gravimetric, negligible component):
- Uncertainty of the dry content determination (negligible):
- Heterogeneity component.

The uncertainty components for each radionuclide are summarized in Table 14. Any revision to the certificate will include uncertainty from decay corrections.

TABLE 14. UNCERTAINTIES BY RADIONUCLIDE IN % (at k=1)

Radionuclide	Spike solution	From preparation[1]	From heterogeneity[2]	Combined standard uncertainty
^{90}Sr	0.93	0.52	1.8	2.10
^{133}Ba	0.62	0.52	1.8	1.98
^{134}Cs	0.63	0.52	1.8	1.98
^{137}Cs	0.62	0.52	1.8	1.97

[1] Includes the weighing components and uncertainty due to the dry content determination

[2] From the most conservative estimation

The property values were verified by high precision control measurements by gamma-ray spectrometry. Both radio-caesium isotopes and the ^{133}Ba were considered as marker isotopes for this purpose. The results are summarized in Table 15.

TABLE 15. RESULTS OF THE CONFIRMATION MEASUREMENTS

(based on dry mass)

Sample ID	Info	Spectrum file	Radionuclide	Activity Bq kg^{-1}		Uncertainty[1]
810/1	Raw material (unspiked milk powder)	BBE20122	K-40	376	±	15
			Ba-133	*MDA*	<	*0.37*
			Cs-134	*MDA*	<	*0.41*
			Cs-137	*MDA*	<	0.26
811/1	Spiked milk powder #1	BBE20123	K-40	382	±	15
			Ba-133	28.3	±	1.5
			Cs-134	201	±	8
			Cs-137	219	±	7
812/1	Spiked milk powder #2	BBE20135	K-40	377	±	15
			Ba-133	30.4	±	1.7
			Cs-134	214	±	8
			Cs-137	233	±	7
813/1	Spiked milk powder #3	BBE20134	K-40	376	±	15
			Ba-133	30.1	±	1.5
			Cs-134	204	±	8
			Cs-137	221	±	7

[1] The uncertainty is expressed as a combined standard uncertainty using a coverage factor k = 1 estimated in accordance with the JCGM 100:2008 [4] and ISO Guide 35 [2].

3.2. DRY CONTENT DETERMINATION

The reference value for calculations was 96.06 ± 0.13 g/100 g at 80 °C determined from several test portions by overnight drying in a laboratory drying oven without air circulation. The uncertainty is reported as an expanded uncertainty, using coverage factor k =2 for confidence level of approximately 95%.

Proposed method for dry content determination for general use: the dry content of the material should be determined in a laboratory drying oven without air circulation using the conditions listed below:

- Mass of the test portion 2-5 g:
- Oven temperature 80 ± 0.5 °C:
- Drying period: minimum 8 hours.

3.3. METROLOGICAL TRACEABILITY

Evidence of metrological traceability to the SI units for the preparation process and value assignment included:

- The traceability chain for the RSS solutions used in the preparation of the MSS can be established to the SI unit Bq via certificates from metrological institutes:
- The traceability to the SI unit kg can be established via use of calibrated balances and calibrated control weights:
- The drying temperature during dry mass determination was measured via a calibrated thermometer, traceable to the calibration certificate.

The details of metrological traceability are presented in Table 16.

TABLE 16. METROLOGICALTRACEABILITY DETAILS

Item	Serial number of the certificate	Certificate issued by
^{90}Sr solution	NIST SRM 4234 A	NIST
^{133}Ba solution	PTB-6.11-2007-1525	PTB
^{134}Cs solution	PTB-6.11-2008-1119	PTB
^{137}Cs solution	PTB-6.11-2008-1367	PTB
Control weight (analytical series)	G1-545 D-K-19408-01-00 2020-03	KERN and Sohn GmbH
Control weight (1 kg)	G1-548 D-K-19408-01-00 2020-03	KERN and Sohn GmbH
Thermometer	4056-20/OAKTON/Marz 2020	TUV Austria Services

For the preparation of the MSS, the Mettler XP205 SNR: B342901875 analytical balance was used. For the weight determination of bulk material, the Ohaus Adventurer ARC-120 SNR: 1120173131 balance was used. Both balances are maintained regularly by an authorized service company and checked before use using certified control weights.

3.4. INTENDED USE

This CRM is intended to be used for the verification of performance indicators (precision, repeatability, reproducibility) of measurement of ^{90}Sr in presence of barium and radio-caesium isotopes. The isotope composition of the CRM is intended to simulate the expected radionuclides and activities from a real nuclear emergency. The relatively low uncertainty of the assigned values minimizes the contribution of the CRM to the combined standard uncertainty derived from measurements.

The IAEA-479 CRM is intended to be used for quality assurance and quality control purposes and is suitable for method development and validation of analytical procedures. IAEA-479 is not to be used for calibration.

3.5. INSTRUCTIONS FOR USE

The IAEA-479 certified reference material is supplied in 100 g units. The material homogeneity is guaranteed if a minimum test portion of 15 g is used. To reconstitute the milk powder to 3.5% fat content milk: mix 33.5 g of milk powder in 500 cm^3 warm water (~60 °C) for 10 minutes or until any clumps dissolve completely.

To overcome any segregation effects due to storage or transportation, the material should be mixed before opening the bottle. All necessary precautions should be taken when opening the bottle to prevent any spread of the powder in the laboratory.

Since the moisture content can vary with ambient humidity and temperature, it is recommended to check it prior to analysis and to calculate all results on a dry mass basis.

The original unopened bottle should be stored securely at ambient temperature in a dark and dry place. It is recommended to avoid direct exposure to sunlight or to a source of heat. The material should be handled by experienced persons and is for laboratory purposes only. Any remaining material in the opened bottle should be stored in the same conditions, however the stability of the material cannot be guaranteed after initial use.

A visual inspection of the material is recommended and if discolouration is observed, please advise by contacting the laboratory using the details listed under further information.

3.6. MATERIAL SAFETY

The radioactive content of the material is below the exemption level; thus, it does not require special handling and storage [6].

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