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Evaluation of the 2017 EC Proficiency Test on ^{131}I, ^{134}Cs and ^{137}Cs in maize powder

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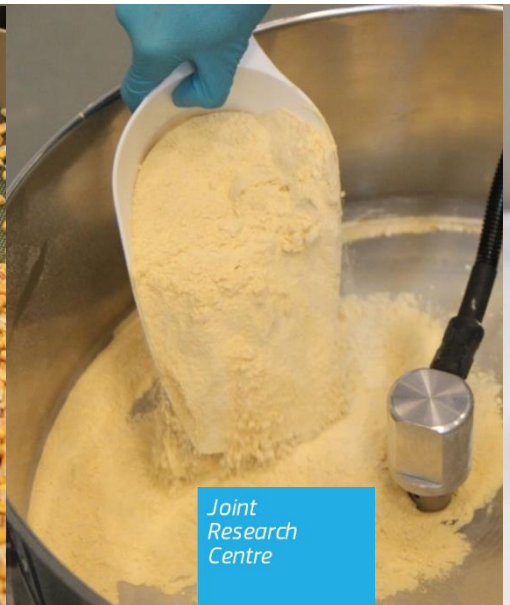

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Authors

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Abstract

A proficiency test (PT) on the measurements of ^{131}I, ^{134}Cs and ^{137}Cs in maize powder was organised by European Commission Directorate-General (EC DG) Joint Research Centre in Geel on request of the EC DG for Energy. This PT was an integral part of the EC's work of realising verification of Member State's obligations towards Article 35 of the Euratom Treaty. It is part of the quality control programme, which JRC Geel is coordinating in order to assess the quality of the results collected in the Radioactivity Environmental Monitoring (REM) database.

The PT reference material was prepared by spiking blank maize powder. The samples were sent to 123 laboratories of which 120 laboratories submitted their results. The results of the PT were evaluated according to ISO 13528:2015. The zed (z) and zeta (ζ) and E_n scores were calculated.

The z scores of the reported results for ^{131}I were acceptable for 92% of laboratories. For both ^{134}Cs and ^{137}Cs they were acceptable for 94% laboratories. The ζ scores were satisfactory for 66%, 56% and 68% of results for ^{131}I, ^{134}Cs and ^{137}Cs respectively.

1 Introduction

Within the framework of the European Atomic Energy Community (Euratom) Treaty and derived European legislation, Member States (MS) of the European Union are obliged to perform measurements of the radioactivity levels in their environment. The results shall be reported to the European Commission (EC). The Radioactivity Environmental Monitoring (REM) group of the EC DG JRC collects, validates and publishes the reported data. In order to verify the performance of the monitoring laboratories and to ensure comparability of reported results, regular proficiency tests (PTs) are organised by the EC. Since 2003, the EC DG Joint Research Centre Geel (JRC Geel) is organising the PTs. The past ten PTs are summarised in a recent report by Hult, Jobággy and Sobiech-Matura (2019). The full reports are also available from the REMON website: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests

The physical properties as well as the radioactivity levels of JRC Geel PT reference material are generally closer to the real samples measured in monitoring laboratories than calibration standards. Therefore, they give a realistic estimate of the performance of these laboratories in their routine monitoring tasks.

This report describes in detail the PT organised in 2017. It covers the production of the reference material, the analytical methods used to establish the reference value, the treatment of the reported data and provided details regarding the procedures used to the evaluation and comparison of the individual results with the reference values. The performance of individual laboratories was evaluated according to ISO 13528:2015 (2015).

2 The EC Proficiency Test 2017

The aim of this PT was to verify the performance of EU Member States (MS) monitoring laboratories for the determination of the massic activity of ^{131}I, ^{134}Cs and ^{137}Cs in maize powder.

2.1 Reference material

Maize powder was produced using commercially available maize grains. It was spiked with radioactive solutions of ^{131}I, ^{134}Cs and ^{137}Cs. A bottle containing no less than 100 g of spiked powder was sent to each participating laboratory. Details on the reference material production and reference values determination can be found in Chapter 3.

2.2 Participating laboratories

In total, 123 laboratories registered to participate in this PT. The laboratories were mainly national research institutes, authorities and radioactivity monitoring laboratories. From EU MS, 112 laboratories were nominated by the national representatives in the Euratom Treaty Art. 35/36 Expert Group. Ten laboratories from accession and pre-accession countries (AC) and one laboratory from Switzerland also registered in this PT. These laboratories were invited as they report their monitoring results to EURDEP.

In total 120 laboratories (109 from EU MS, 10 from AC and 1 from Switzerland) reported results. The list of all participating laboratories is shown in Annex 7. Since anonymity is a requirement in the PTs according to ISO 17043 (2010) the identity of the laboratories is not shown in this compilation of the results. The laboratory numbers used throughout the data evaluation in this report are not related to the order of listing the participants in Annex 7.

Laboratories that submitted the results in this PT have different functions – 48 laboratories are only monitoring radioactivity in the environment, whereas 61 combine this function with others, like research and development (31 laboratories), monitoring of nuclear facilities (7 laboratories) or both (11 laboratories). Four laboratories carry out only research and development activities and 7 laboratories have different functions, like for example disaster management, food monitoring or civil protection.

Table 0-1. The origin of participating laboratories.

	Nominated laboratories from EU Member States	**EU Pre-accession Countries**	**Other**	**Total**
Sample sent	112	10	1	123
Results submitted	109	10	1	120

Source: JRC Geel

2.3 Reporting of results

Participants were instructed to report the results and the associated uncertainty as massic activity normalised to dry mass (Bq/kg d.m.). The organiser recommended performing the moisture content determination on small subsamples that shall not be used for the radionuclide analysis to avoid the loss of the ^{131}I in the radionuclide analysis sample. The participants were instructed to take these subsamples from the bottles at the same time as the samples for radionuclide analysis. The recommended method for moisture content determination was the oven-drying procedure (Annex 5). The detailed description of the method was sent together with the sample accompanying letter (Annex 4).

The reference date for all results was 1 June 2017 00:00:00 UTC. The Monographie BIPM-5 vol. 3, 7 and 8 (2006, 2013 and 2016) was recommended as source of nuclear decay data to be used in the analysis. This data arise from the Decay Data Evaluation Project (DDEP), which is recommended by the ICRM (International Committee for Radionuclide Metrology) as the first choice of decay data to be used in radiometric analyses.

The results were reported via a web-based tool called MILC (developed by JRC Geel), which served also as the tool for a questionnaire. Participants were asked to answer all relevant questions regarding the measurement procedures used. Information given in the questionnaire enables a more detailed evaluation of the PT results. It helps also to discover sources of possible discrepancies and gives an overview of the methods used among the laboratories.

For the first time in a REM PT supporting Article 35, the possibility to perform so-called "emergency reporting" was introduced. The participants were asked on a voluntary basis to report their results within 48 hours after receipt of the PT reference material. These results were not formally reported to national authorities or DG ENER. They will be discussed in a separate publication. For the routine reporting the laboratories had two months to report after receiving the material. These results are reported to the laboratories national authority and DG ENER.

2.4 Timetable of the PT

29/03/2017	Invitation letter (Annex 1) sent to the national representatives to nominate laboratories in their countries
28/04/2017	Nominations from the national representatives collected
11/05/2017	Invitation letter sent to the non-EU laboratories from countries reporting to EURDEP
10-31/05/2017	Registration of laboratories
01-09/06/2017	Material and additional information on the PT sent to the participants via express mail
02-14/06/2017	Laboratories submit their emergency results to the JRC Geel
02/06-28/07/2017	Laboratories submit their routine results and questionnaire to the JRC Geel
25/10/2017	Preliminary evaluation of reported results sent to participants (Annex 6)
31/1-2/2/2018	Workshop and follow-up training
05/03/2018	Distribution of laboratory's final PT results via a new MS Excel-based tool (REMPES)

3 Material preparation and reference values

3.1 Reference material

The reference material used as a test item in this PT was blank maize powder spiked with radioactive solutions of ^{131}I, ^{134}Cs and ^{137}Cs. Due to the short half-life of ^{131}I (8 days), spiking was the only available method for producing this PT material. The Reference Materials Unit of the JRC Directorate F located at JRC Geel performed the processing of the material.

The bulk raw material consisted of 75 kg of maize grains available on the local market as animal feed. It was purchased from a commercial supplier in Belgium. The maize grains were placed in metallic drums and cooled down in liquid nitrogen (-196 °C). Once cold, they were milled using a vibrating cryogenic mill (KHD Humboldt Wedag, Köln, Germany). After the processing a total amount of 64.7 kg of powder was collected. The powder was sieved at room temperature over a 250 µm stainless steel mesh (Russel Finex Industrial sieve Model 17300, London, United Kingdom) connected to an ultrasonication probe (Russel Finex Vibrasonic 2000, London, United Kingdom). The fine fraction represented 57.10 kg and the coarse fraction 7.15 kg. The coarse fraction was re-milled and re-sieved as previously described. The fine fraction of the re-milled coarse fraction (7.13 kg) was added to the 57.10 kg of the fine fraction obtained from the first milling/sieving sequence. The total fine fraction (64.3 kg) was then placed in a 200 L metallic drum. It was mixed for two hours with a DynaMIX-CM200 mixer (WAB, Basel, Switzerland). The mixed powder was then split in 7 different plastic drums containing ca. 10 kg of powder each. The material was stored at +4 °C.

Figure 1 Maize grains processing (cooling, milling and sieving).

Source: JRC Geel

For the spiking, 503.26 g of blank maize powder was placed in a rotary evaporator powder flask of 2 L total volume. Then 600 mL of ethanol was added to obtain a slurry in the flask. The radioactive solutions of ^{131}I, ^{134}Cs and ^{137}Cs were gravimetrically added to the slurry using pycnometers (Canis, Canada) and an analytical balance (Mettler Tolledo type AT21). The activity of the solutions used for spiking was determined by measuring gravimetrically made point and volume sources. These sources were prepared at the same time as the spiked maize powder.

The flask containing the slurry was placed in a rotary evaporator (Hei-VAP, Heidolph, Germany) and rotated for 10 hours at room temperature. Mixing continued for 20 more hours during evaporation of ethanol. In order to retrieve the powder form of the material the water bath of the device was heated up to 65 °C and the pressure in the system was lowered to 500 mBar. In the vertical condenser water at 3 °C was circulated. When the powder was dried, it was transferred from the rotary evaporator powder flask to a 2 L polypropylene container with a screw cap. The powder was mixed together with 6 porcelain balls in a Turbula shaker-mixer (T2F, Glen Mills, USA) for 4 hours.

The spiked powder was transferred to a polypropylene drum containing 19.79 kg of blank maize powder. The content of the drum was mixed in a DynaMIX-CM200 mixer (WAB, Basel, Switzerland) for 4 hours. After mixing, the powder was bottled in 250 mL amber glass bottles containing at least 100 g each. The bottles were closed with a screw cap with a break-ring.

Since the homogeneity of so-prepared material was not satisfactory an additional step was introduced. All available material was collected again in a plastic drum. It was cooled down in liquid nitrogen (-196 ºC) and milled in Cryogenic Mill (CryoMill, Retsch, Germany). Additional mixing in DynaMIX-CM200 mixer (WAB, Basel, Switzerland) for 4 h was applied. After this additional homogenisation, the powder was as previously described bottled into 161 new amber 250 mL glass bottles.

The moisture content of the material after bottling was determined according to procedure sent to the participants together with the PT material. The moisture content of the final product was (12.85 ± 0.02)%.

3.2 Reference material characterisation

The characterisation of the PT reference material was performed by four laboratories (IAEA Monaco, IAEA Seibersdorf, CEA Saclay and JRC Geel). All these laboratories used 2-3 bottles of the PT reference material. They reported one mean value obtained by measuring different samples covering all received bottles. The reported values for each laboratory used for calculations can be found in Table 2.

Table 0-2. Measurement results and uncertainty reported by the laboratories used for the reference value calculations (k=1).

Laboratory name	^{131}I		^{134}Cs		^{137}Cs		^{40}K	
	Activity (Bq/kg d.m.)	unc. (Bq/kg d.m.)	Activity (Bq/kg d.m.)	unc. (Bq/kg d.m.)	Activity (Bq/kg d.m.)	unc. (Bq/kg d.m.)	Activity (Bq/kg d.m.)	unc. (Bq/kg d.m.)
IAEA Seibersdorf	184	7.0	913	23	550	14	88	7.0
IAEA Monaco	190	10	885	14	534	9.0	107	7.0
CEA Saclay	190	11	911	50	561	30	103	5.5
JRC Geel	197	6.0	921	28	563	17	105	7.0

Source: JRC Geel

The homogeneity and short-term stability (under the transport conditions) of the analytes in the reference material used were assessed at JRC Geel. The study was executed in accordance with the ISO Guide 35 (2015).

3.2.1 Homogeneity

Ten bottles of the PT reference material for the homogeneity study were selected in a random stratified manner covering the whole batch. Three samples of 20 g were prepared from each bottle. All samples were measured on the same high-purity Germanium (HPGe) detector under the same measurement conditions. The relative standard deviation of all measurements was 1.8%, 0.8% and 1.0% for ^{131}I, ^{134}Cs and ^{137}Cs respectively. One-way ANOVA calculations were performed using SoftCRM software (Bonas, et al., 2003). The relative uncertainty contribution due to inhomogeneity (u_{bb}) was estimated by calculation of u_{bb*} (Linsinger, et al., 2001). The results of the measurements and the calculations are shown in Annex 10 and the relative components of uncertainty resulting from inhomogeneity equal to 0.6%, 0.3% and 0.3% for ^{131}I, ^{134}Cs and ^{137}Cs respectively. These values were used to calculate the total uncertainty on the reference values.

3.2.2 Short-term stability

The short-term stability of the PT reference material was assessed at two temperatures (40 and 60 °C). Samples stored at room temperature were used as reference. Four bottles were selected in a randomly stratified manner to study the stability at each temperature. All bottles were weighted on one balance at the beginning of the study and at the time of the second measurement. They were all measured on the same HPGe detector at the beginning of the study. Four bottles per temperature were stored in two ovens set at 40 °C and 60 °C respectively. Two bottles were stored at the reference temperature. One bottle was taken out of each oven after 1, 2, 3 and 4 weeks. It was cooled down to room temperature and measured on the same detector under the same measurement conditions as at the beginning of the study. The net count rate was calculated for both measurements. A linear regression was fitted to the net count rates of the measurement results for each test temperatures. A Student t-test described in the ISO Guide 35 (2015) was used to evaluate whether the slope of the linear regression curve is significantly different from zero. For all tree radionuclides and for both temperatures there was no significant statistical trend. Therefore, in accordance with ISO

13528 (2015), the component of uncertainty due to instability (u_{sts}) was not taken into account for the calculation of uncertainty of the reference value.

3.2.3 Reference values

The reference values, given in Table 0-3, are composed of the reference values (x_{pt}) and an expanded uncertainty ($U(x_{pt})$). The reference value (x_{pt}) for each radionuclide was calculated based on power-moderated mean (PMM) approach (Pommé & Keightley, 2015). The standard uncertainty of the reference value ($u(x_{pt})$) was calculated according to the following equation:

$$u(x_{pt}) = \sqrt{u_{char}^2 + u_{hom}^2}$$

where

u_{char} is the uncertainty from the power-moderated mean calculated for the results of the characterisation study;

u_{hom} is the uncertainty component associated with reference material heterogeneity.

The expanded uncertainty ($U(x_{pt})$) was calculated according to the following equation:

$$U(x_{pt}) = k \cdot u(x_{pt}) \quad (1)$$

where

k = 2 (corresponding to a level of confidence of about 95%).

The reference values for ^{131}I, ^{134}Cs and ^{137}Cs are further on used for the evaluation of the results reported by the PT participants. The reference value for ^{40}K is only informative.

Table 0-3. Reference values of massic activity x_{pt} with expanded uncertainties $U(x_{pt})$ (k = 2) for the PT reference material at the reference date (1 June 2017 00:00:00 UTC).

Radionuclide	$x_{pt} \pm U(x_{pt})$ (Bq/kg d.m.)
^{131}I	191 ± 8
^{134}Cs	901 ± 23
^{137}Cs	547 ± 14
^{40}K (*)	101 ± 9

(*) The value for ^{40}K is only informative.

Source: JRC Geel

4 Methods used by the participating laboratories

Participants in this PT could freely choose the measurement method, although it should preferably be their routine procedure for measuring ^{131}I, ^{134}Cs and ^{137}Cs in environmental matrices. They were asked to report for each radionuclide the specific activity per dry mass of the spiked maize powder. Participants could also, on a voluntary basis, report specific activity results per dry mass for ^{40}K in the PT reference material.

The reporting of the results was realised via a web-based platform called MILC, where participants were also asked to fill in a questionnaire. The information in this chapter is extracted from the files obtained from MILC.

4.1 Moisture content

All results of massic activity had to be normalized to dry mass. It was recommended to determine the moisture content in separate small samples not used for other analysis. The description of the recommended oven drying method for the moisture content determination was sent to participants together with the PT material (Annex 5). In this way, the influence of differences resulting from moisture content determination procedures were minimised. The mean value of moisture content reported by the participants equalled to 12.14%, which was close to the reference value of (12.85 ± 0.02)%. The reported results varied from 0.123% to 89.48%. In one third of the cases, the relative difference between the reference value and reported value for the moisture content varied more than 20%. It has to be noted however that the discrepancies may be resulting from misunderstanding the question, i.e. the reported value 0.123% may mean 12.3%, and the value 89.48% may refer to the dry mass content (wet mass being 10.52%). For 21 participating laboratories moisture content determination in food samples is not routinely performed. Those laboratories had problems with proper determination of the moisture content. One laboratory (8) informed about difficulties due to lack of experience and adequate equipment for the drying procedure.

Seven laboratories reported that they did not follow the provided oven drying method. Laboratory 36 dried for a longer time at lower temperature. Their result (10.8 ± 0.4)%, was lower than the reference value.

Laboratories 21 and 54 dried the samples for 15 and 18 hours respectively (instead of recommended 1 hour intervals), reporting results of (12.5671 ± 0.0035)% and (12.32 ± 0.12)% respectively.

Laboratory 43 used different aliquots (5 g) and the moisture content reported by this laboratory was also underestimated (8.6 ± 0.18)%.

Laboratories 64 and 92 used their routine methods (drying overnight at 105 °C) instead of the recommended method. The moisture content reported by laboratory 64 was underestimated (9.1 ± 1)%, but the result reported by laboratory 92 was correct (12 ± 1)%.

Laboratory 114 could not perform the sample preparation for moisture content determination at the same time as the measurement of the PT reference material. The reported result was correct (12.14%), but an uncertainty component for moisture content determination was not reported by this laboratory.

Laboratory 115 did not report the moisture content.

The applied correction factor for dry mass as reported by the participating laboratories varied from 0 to 92.8. Laboratories applied different approaches to calculating this factor. Most of the laboratories divided the dry mass of the sample by the wet mass of the sample, resulting in correction factor ranging from 0.85 to 0.97. Others calculated it as wet mass divided by dry mass, with results ranging from 1.08 to 1.15. Some laboratories

simply used the result of moisture content determination. Eleven laboratories did not provide a value for the dry mass correction factor.

4.2 Measurements

4.2.1 Techniques, instrumentation and quality system

All participants used gamma-ray spectrometry for determining the massic activities of ^{131}I, ^{134}Cs and ^{137}Cs in the samples. One laboratory (number 66) used a NaI(Tl)-detector, one laboratory (number 98) used a Ge(Li)-detector, whilst the remaining 118 laboratories all used HPGe detectors. The relative efficiency of HPGe the detectors used ranged from 5 to 150%.

The participants used in this study mostly methods routinely applied in their laboratories (91 out of 120). Most of the participants (104 out of 120) routinely measure food or feed samples. Out of these 104, 31% measure routinely less than 50 food or feed samples per year, 15% measure 50 to 100 samples per year, 33% measure 100 to 500 samples of this type per year, 12% measure 500 to 1000, and more than 1000 samples are analysed by 10% of laboratories (Table 0-4).

Table 0-4 Number of samples per year analysed by laboratories routinely measuring food or feed samples

Number of analysed samples per year	**Number of laboratories**
< 50	32 (31%)
50 to 100	16 (15%)
100 to 500	33 (32%)
500 to 1000	12 (12%)
> 1000	11 (10%)

Source: JRC Geel

Out of the 120 participating laboratories, 47 are accredited according to ISO 17025 for gamma-ray spectrometry and 28 laboratories are authorised. The ISO 9001 management system is applied in 13 laboratories.

4.2.2 Sample preparation

The typical masses of the samples routinely analysed in the participating laboratories using gamma-ray spectrometry varies from 0.01 g to 15 kg. On average it is 750 g. In most of the laboratories (104 out of 120) pre-treatment of samples is routinely performed (e.g. grinding, mixing, ashing, drying, cutting, calcination).

The PT material was treated according to the same procedure as a routine sample by 91 laboratories. Five laboratories applied a procedure to compact the material and 25 laboratories homogenised the sample. For 8 laboratories the detector was calibrated to a geometry where more material than provided is required. Therefore, to respect the geometry, they prepared a mixture of the PT material with another type of blank material (e.g. maize powder, silicon). Special equipment for preparation of the PT material was used by 13 laboratories, mostly for compacting or homogenising the sample.

The smallest sample mass used for a single measurement was (1.9781 ± 0.0003) g by laboratory 120. One fourth of the laboratories used all of the provided PT material

(~90-100 g) remaining after preparation of the moisture content determination samples. Four laboratories (2, 21, 60, 120) used for a single measurement less material than the minimum prescribed sample intake of 20 g. This could have influenced their results as homogeneity of the PT reference material was only guaranteed down to the sample intake of 20 g.

The majority of laboratories used cylindrical containers, 16 laboratories used a Marinelli beaker and 6 a Petri dish. The sample containers were mostly made of plastic; laboratory 49 used an aluminium container, laboratories 50, 67 and 75 containers made out of other metals and laboratories 9 and 109 a glass container. Approximately half of the laboratories completely filled the measurement container with the sample.

4.2.3 Sample positioning

The sample was centred on the detector by eye in 75 laboratories and centred using sample holders in 36 laboratories. In 9 other laboratories either a Marinelli beaker was used or other methods were applied. Laboratory 42 covered the detector with parafilm, which was marked with the diameter of the cylindrical measurement containers. The sample was placed directly on the detector end-cap by 41 laboratories. The rest of participants reported a different source-detector distance from 0.1 to 50 mm, with a mean of 3 mm and a median of 1 mm.

4.2.4 Efficiency calibration

The full energy peak efficiency calibration of the detector was mostly performed using a calibration source of a geometry similar to the PT reference material measured. Sixteen laboratories used LabSOCS™ (Mirion) and 6 laboratories Monte Carlo simulations. Laboratory number 3 indicated that no calibration was performed.

From the answers it was not possible to understand in detail how every laboratories performed efficiency calibration but Table 5 gives an indication.

Five laboratories used one or more certified reference material (CRM). It should be noted that the approach of using CRMs for either obtaining a calibration curve or directly the calibration factors for the radionuclides of interest is may result in enlarging the measurement uncertainties. It is therefore in many cases not recommended to use CRMs in this way. The uncertainties of the radionuclide activity in CRMs are generally higher than what can be obtained in standard calibration sources of liquids and resins. It is recommended to use CRMs for method validation and testing.

Table 0-5 Efficiency calibration methods used by the participating laboratories

Method	Number of laboratories	Comment
Set up calibration curves using volume sources of relatively similar geometry from a liquid solution or resin	50	
Set up calibration curves using a CRM	5	
One of the two above and additionally using geometry transfer method	38	
LabSOCS™	16	Software from manufacturer with a factory-developed computer model of the detector
ISOCS™	3	Software from manufacturer with a factory-developed computer model of the detector (main intended use is for large sources like drums)
Monte Carlo simulation	6	Codes: GEANT 3.0, MCNP, Gespecor, Angle 3, EFFTRAN, RadiationHelper coupled with DetectorCalibration (based on GEANT4)
Deriving calibration factors from another PT material with radiocaesium and producing an own spiked calibration standard for ^{131}I	1	Laboratory using this method overestimated results for ^{131}I
Calibration by external expert	1	

Source: JRC Geel

4.2.5 Analysis and calculation software

The software used for spectrum analysis was mostly Mirion's Genie™ 2000 (74 laboratories). Ortec's GammaVision® was used by 34 laboratories. Fourteen laboratories used other, sometimes home-made, software.

Activity calculation was performed using Genie™ 2000 software by more than half of participating laboratories. Thirty-one used GammaVision® for this purpose and 14 used spreadsheets for the calculations. Twelve laboratories used other, sometimes home-made, software.

4.2.6 Corrections

True coincidence summing corrections were applied by 80 laboratories. Ten laboratories reported applying self-attenuation correction and 37 geometry transfer correction. Correction for the decay during the measurement was applied by 84 laboratories.

4.2.7 Nuclear data

The recommended source of nuclear data was used by 42 laboratories, out of which 11 used the Nucléide-Lara library, a website using data from DDEP. Twenty-four participants used the data available in the library of the software used (Genie™ 2000 or GammaVision®). Five laboratories used the Lund/LBNL Nuclear Data Search (http://nucleardata.nuclear.lu.se/nucleardata/toi/). The reference data tables of Physikalisch-Technische Bundesanstalt (PTB) were used by 4 laboratories. The rest of the responding participants used various other sources. Four laboratories (46, 72, 79 and 93) provided an answer not adequate to the question asked and 5 laboratories did not answer this question.

4.2.8 Background measurements

A background measurement was performed by 115 laboratories. Details on the duration of the background measurement were not provided by 15 laboratories. Forty-five laboratories measured the background with an empty sample container. 44 with an empty shield, 18 with a sample container filled with (usually distilled) water in the same type of container as used for the measurement of the PT reference material. Eight laboratories measured background with a container filled with a blank organic substance - 4 used blank corn flour and 2 cellulose. One laboratory used a solution of agar (known to have about 0.8 Bq/g of ^{40}K), which might be the reason for a too high result for ^{40}K. One laboratory reported using a blank sample without specifying its type.

The length of the background measurements varied from 989 to 2,000,000 seconds (equal to 16 min to 23 days).

4.2.9 Detection limit calculation

The laboratories were asked which method they used to calculate the minimum detectable activity. Only 30 laboratories replied. ISO 11929:2000 was indicated by 4 laboratories, ISO 11929:2010 by 15 laboratories, and the "Currie method" by 6. The rest of laboratories reported other methods like calculation built in to Genie™ 2000 or GammaVision®.

5 Reported results

As explained in Chapter 2.3, for the first time in this series of proficiency tests two types of reporting were used – emergency and routine reporting. The emergency reporting will be described in detail in a separate report. For the routine reporting, participants were given two months after the sample receipt. The massic activities of ^{131}I, ^{134}Cs and ^{137}Cs with their corresponding expanded uncertainties and the coverage factor used (k) reported in the routine mode are presented in Annex 10. The results for ^{40}K are also provided for information purposes only.

One laboratory (37) noticed after the deadline of submission that by mistake their reported results were filled in in the wrong order into the reporting form. The results reported before the deadline were retained, which resulted in very big deviations from the reference values. Another laboratory (3) reported "0" as a value for the massic activity of ^{131}I. This laboratory has lost their first analysis results, and the second measurement was performed too late to report ^{131}I.

6 Evaluation of the results

6.1 Scores

The evaluation of the reported results was conducted according to ISO 13528:2015 (ISO 13528:2015, 2015). The results were compared to the reference values presented in

The reference values for ^{131}I, ^{134}Cs and ^{137}Cs are further on used for the evaluation of the results reported by the PT participants. The reference value for ^{40}K is only informative.

Table 0-3. Three different scores were calculated z, ζ and E_n score. The results can be found in Annex 10.

6.1.1 z score

In the calculation of the z score the standard deviation for proficiency assessment (σ_{pt}) is used. This parameter was set at 20% of the assigned reference value (x_{pt}), based on the experience from previous PTs and on what should be expected that a modern laboratory today should be able to produce under routine conditions.

The z score was calculated according to the following equation:

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}}$$

where

x_i is the participant's results;

x_{pt} is the assigned reference value.

The interpretation of the z score is as follows:

- If the participant's result gives $|z| \leq 2.0$, it is acceptable;
- If the participant's result gives $2.0 < |z| < 3.0$, it gives a warning signal;
- If the participant's result gives $|z| \geq 3.0$, it is unacceptable and gives an action signal.

6.1.2 Zeta (ζ) score

In order to assess the agreement of the participants' estimations of uncertainty with that of the reference value two additional scores were calculated namely ζ and E_n score.

The ζ score is taking into account the standard uncertainty (k=1) of both the participant's result and the reference value and is calculated according to the following equation:

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}}$$

where

$u(x_i)$ is the standard uncertainty of the participant's result;

$u(x_{pt})$ is the standard uncertainty of the assigned reference value.

When uncertainties are correctly estimated according to the Guide to the Expression of Uncertainty Measurement (GUM) (ISO/IEC GUIDE 98-3:2008, 2008), a measurement result with its uncertainty interval should overlap with the reference value and its uncertainty.

The ζ score indicates whether a participant's result agree with the reference value within the provided uncertainty value. An unsatisfactory ζ score can be related to incorrect measurement result, poor uncertainty estimation, or both of these. It is interpreted as follows:

- If $|\zeta| \leq 2.0$, the participant's result is acceptable;
- If $2.0 < |\zeta| < 3.0$, the participant's result gives a warning signal;
- If $|\zeta| \geq 3.0$, the participant's result gives an action signal.

6.1.3 E_n score

To take the expanded uncertainty (k=2) of the participant's result and that of the reference values into account in the analysis of the reported results, a performance test using E_n score was applied (ISO, 2015). The calculation of the E_n score was carried out according to the following formula:

$$E_n = \frac{x_i - x_{pt}}{\sqrt{U^2(x_i) + U^2(x_{pt})}}$$

where

$U(x_i)$ is the expanded uncertainty of the participant's result;

$U(x_{pt})$ is the expanded uncertainty of the assigned reference value.

E_n scores are interpreted as following:

- If $|E_n| < 1$, the uncertainty of the participant's result is consistent with the uncertainty of the reference value;
- If $|E_n| \geq 1$, the uncertainty of the participant's result are inconsistent with the uncertainty of the reference value and the sources of deviation should be investigated and corrected.

6.2 Evaluation

Overview of the participants performance is presented in Table 0-6. In addition, the results of the z and ζ scores are presented in Figs. 2 to 9. Out of the 120 participants according to the z score the results were acceptable in 92% for ^{131}I and in 94% for both ^{134}Cs and ^{137}Cs. For ^{131}I, only 1 laboratory received a warning signal, 8 laboratories an action signal and 1 laboratory reported a value below the detection limit. In case of ^{134}Cs, also only 1 laboratory received a warning signal and 6 an action signal, and for ^{137}Cs there were 2 laboratories with a warning signal and 5 with an action signal. For the optional ^{40}K, 81% of reported results were acceptable, whereas 7 laboratories received a warning signal and 13 an action signal.

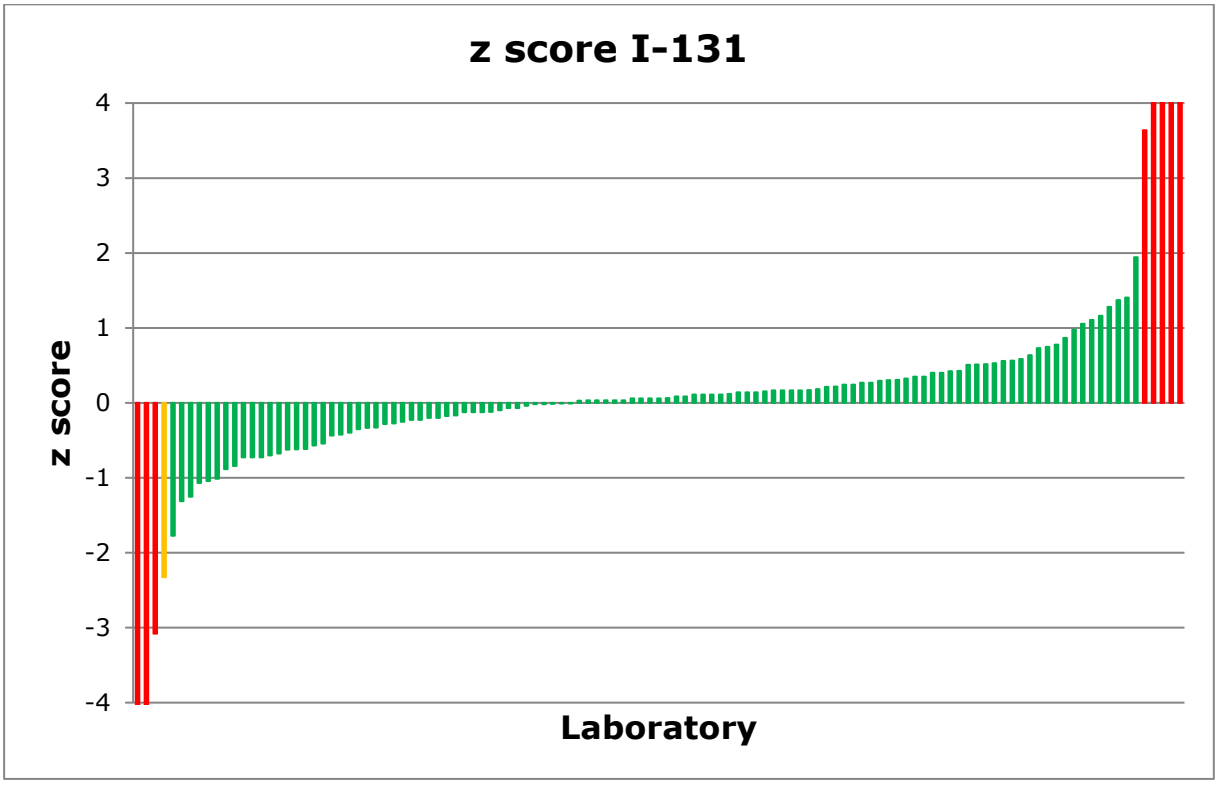
The absolute value of ζ score was found to be lower than 2 for 66% of participants for ^{131}I, 56% for ^{134}Cs and 68% for both ^{137}Cs and ^{40}K. These laboratories reported values having uncertainties consistent with the uncertainty of the reference value. In case of E_n score, the number of consistent results was the same as for ζ score.

Table 0-6 Overview of the laboratories performance. The numbers within brackets are the number of reported results.

Radionuclide	z score			ζ score			E_n score	
	acceptable	warning signal	action signal	acceptable	warning signal	action signal	consistent	inconsistent
^{131}I	92% (110)	1% (1)	7% (8)	66% (79)	11% (13)	23% (27)	66% (79)	34% (40)
^{134}Cs	94% (113)	1% (1)	5% (6)	56% (67)	7% (9)	37% (44)	56% (67)	44% (53)
^{137}Cs	94% (113)	2% (2)	4% (5)	68% (81)	5% (6)	27% (33)	68% (81)	32% (39)
^{40}K*	81% (85)	7% (7)	12% (13)	68% (71)	12% (14)	17% (20)	68% (71)	29% (34)

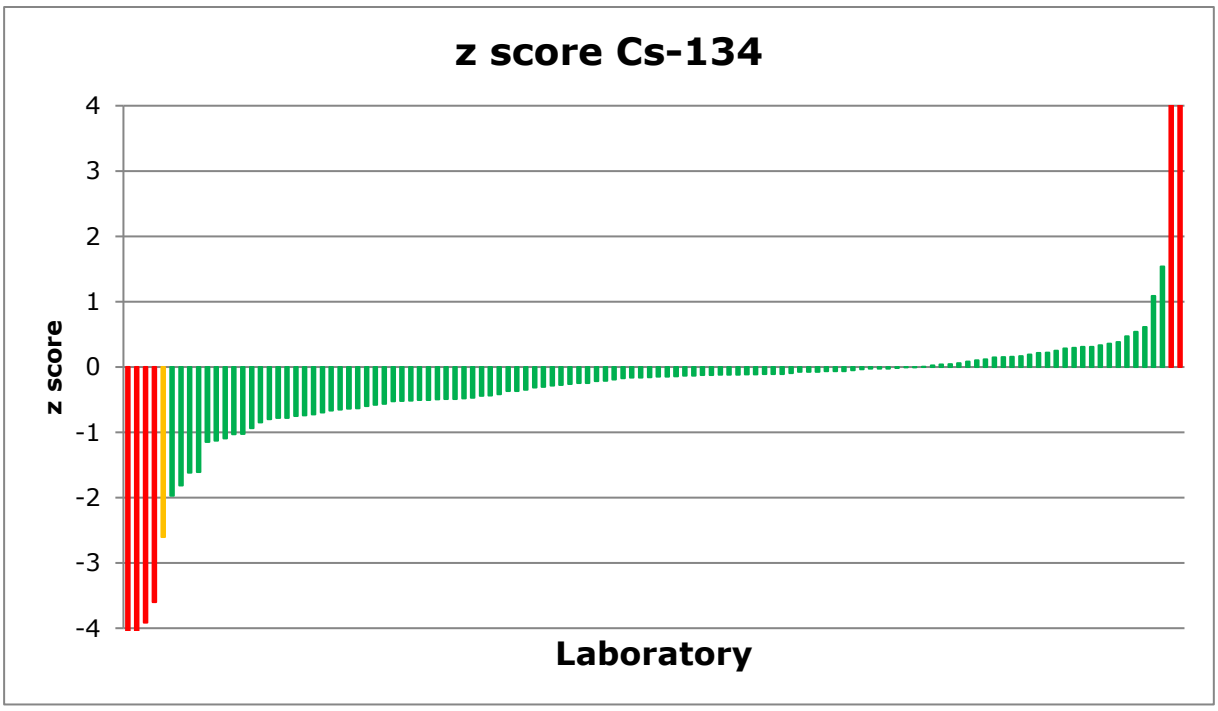
Source: JRC Geel

Figure 2 Results of z score calculations for ^{131}I



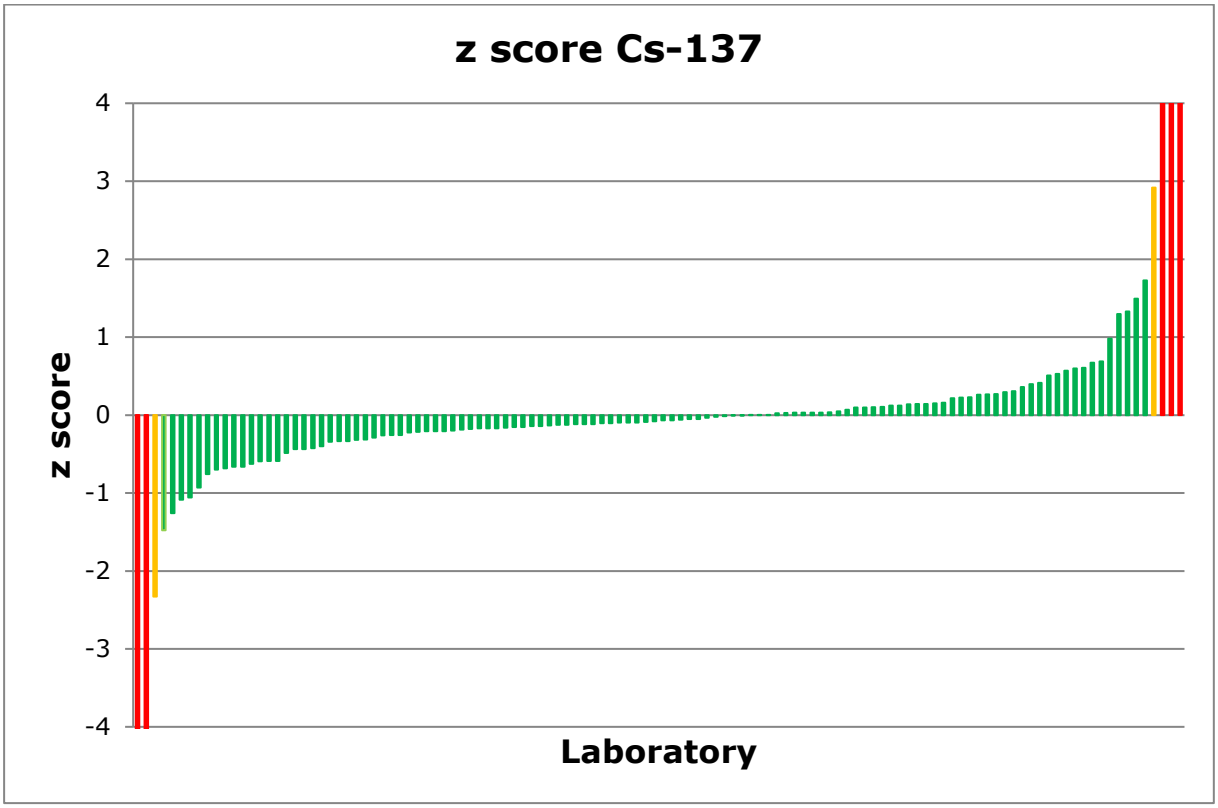
Source: JRC Geel

Figure 3 Results of z score calculations for ^{134}Cs



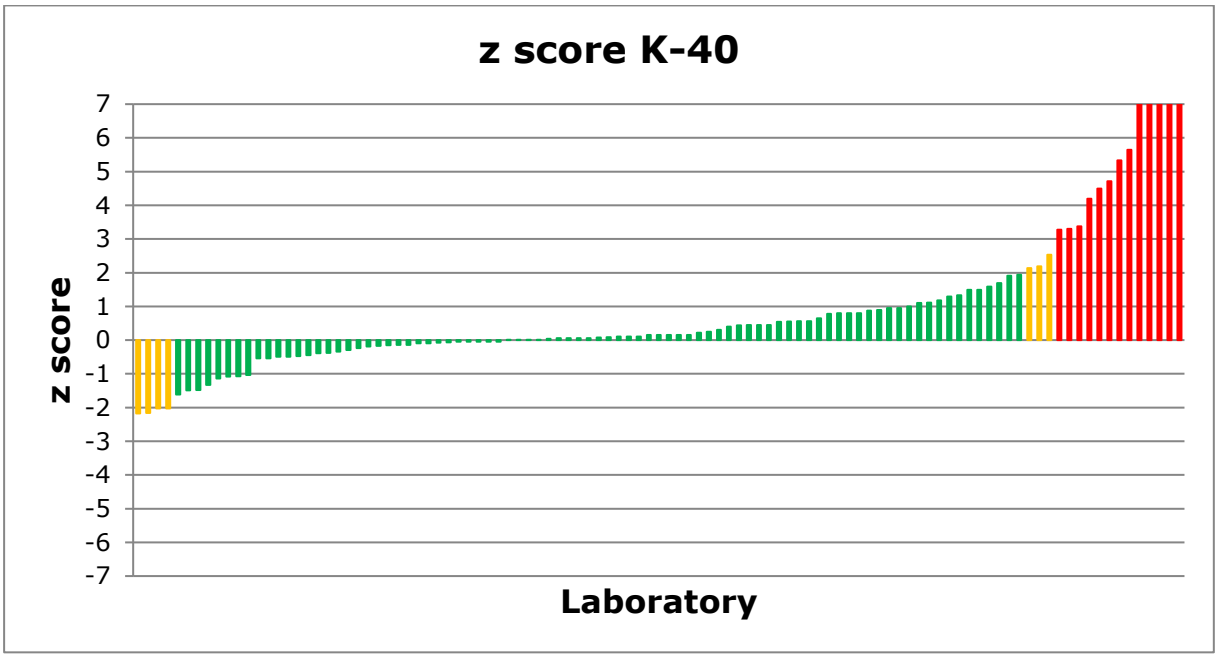
Source: JRC Geel

Figure 4 Results of z score calculations for ^{137}Cs



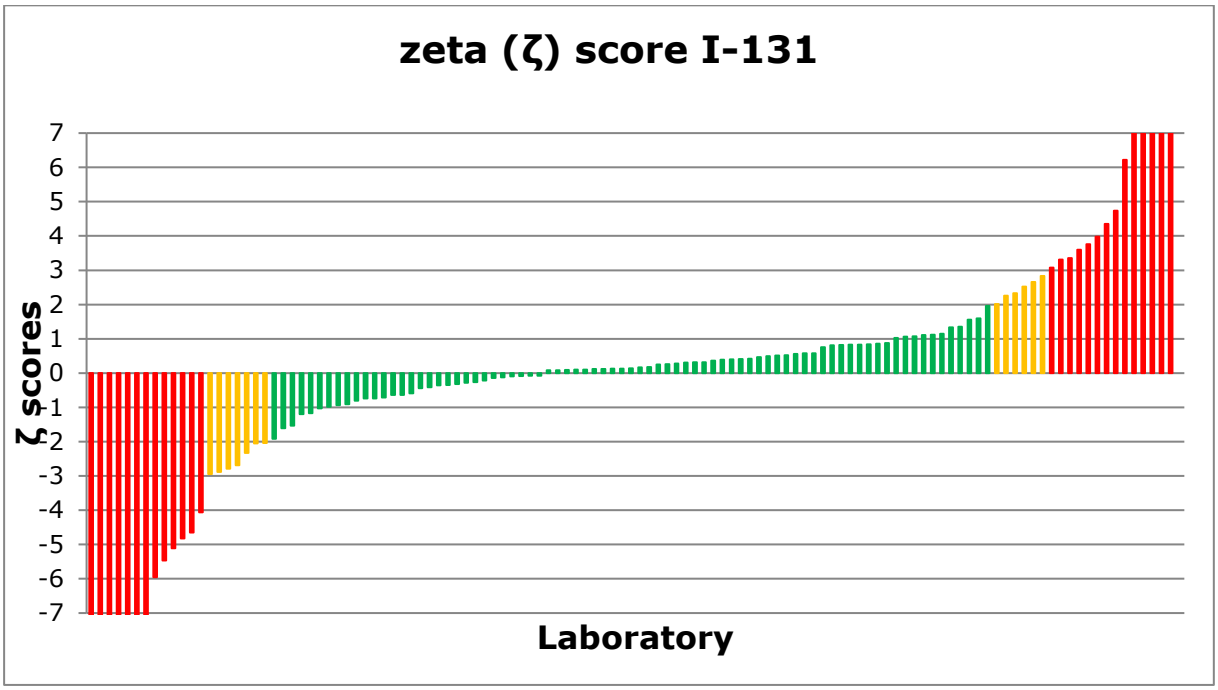
Source: JRC Geel

Figure 5 Results of z score calculations for ^{40}K



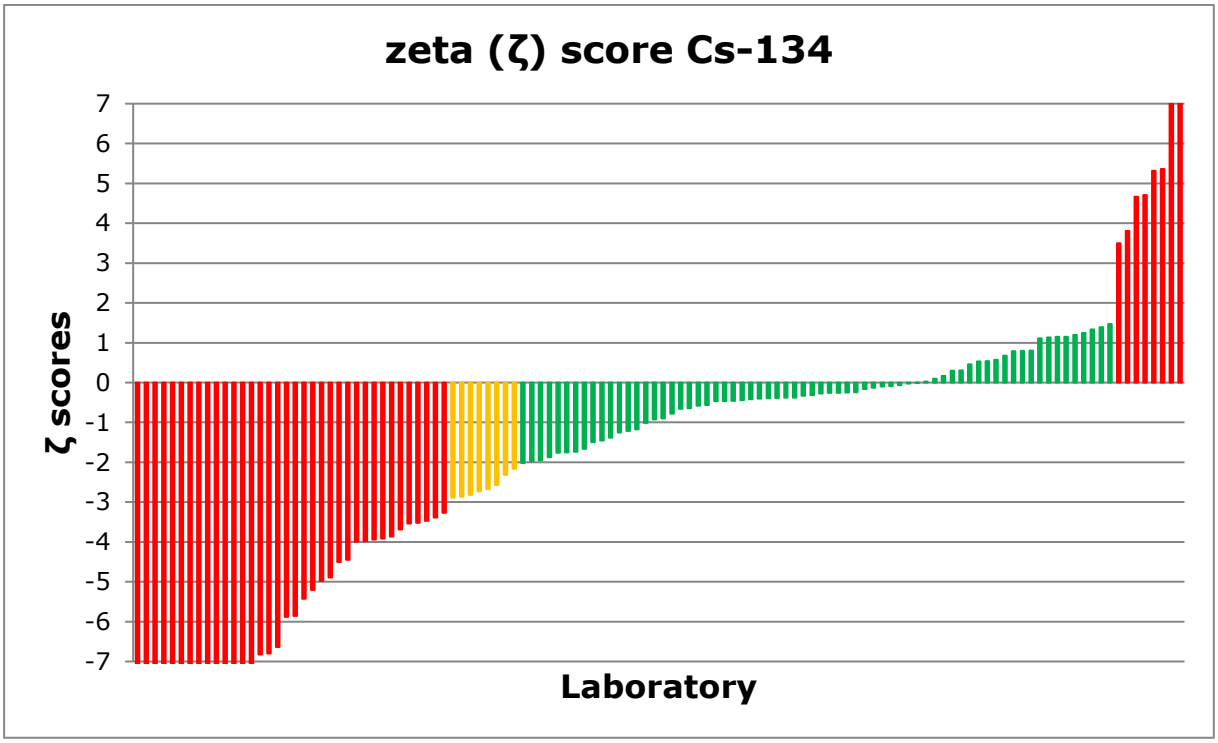
Source: JRC Geel

Figure 6 Results of ζ score calculations for ^{131}I



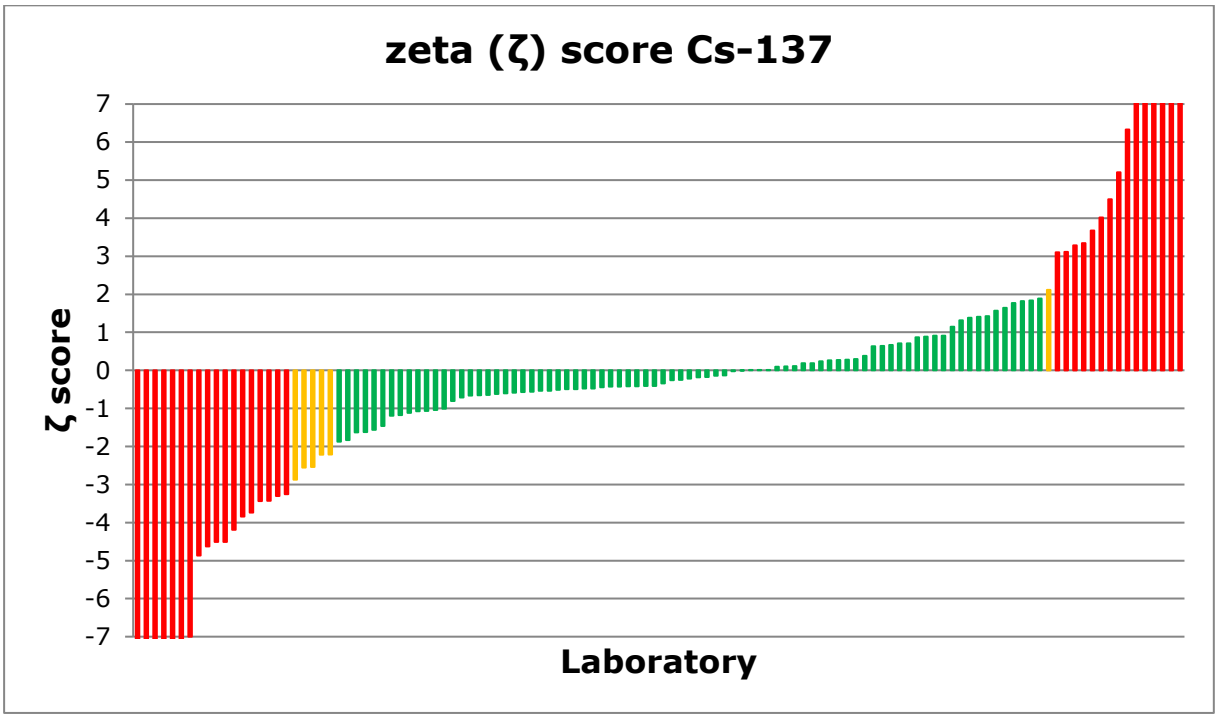
Source: JRC Geel

Figure 7 Results of ζ score calculations for ^{134}Cs



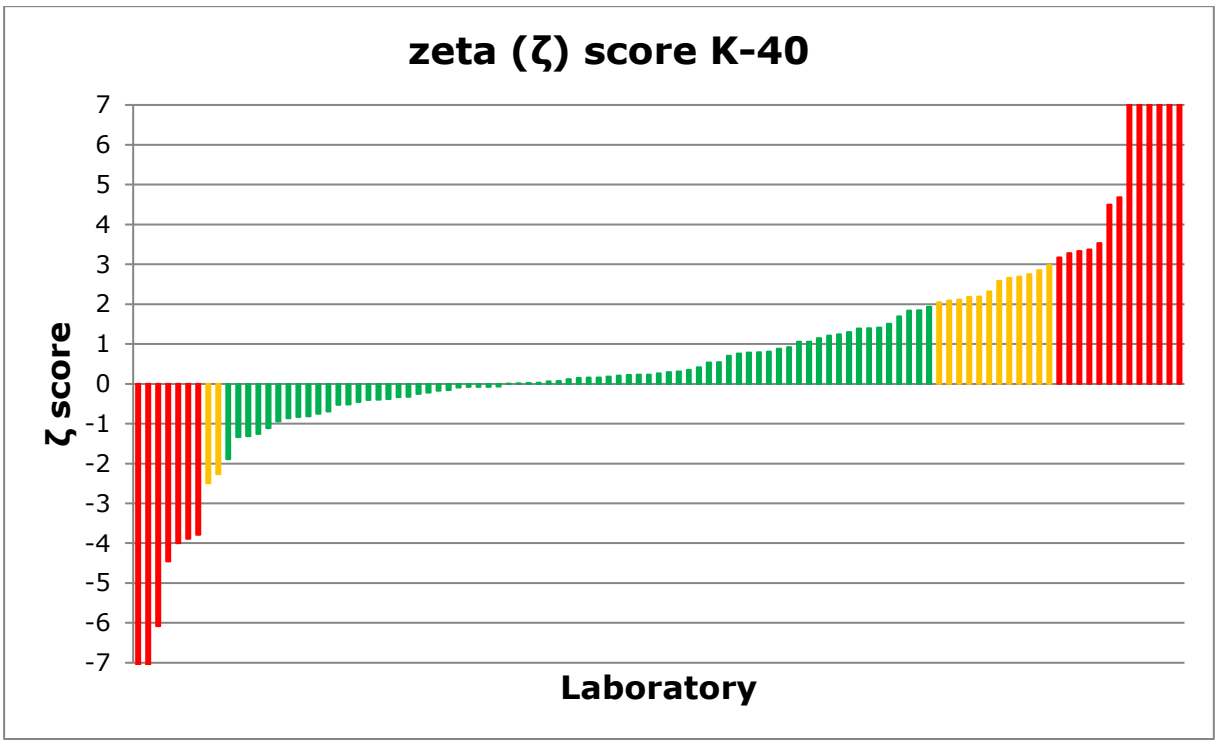
Source: JRC Geel

Figure 8 Results of ζ score calculations for ^{137}Cs



Source: JRC Geel

Figure 9 Results of ζ score calculations for ^{40}K



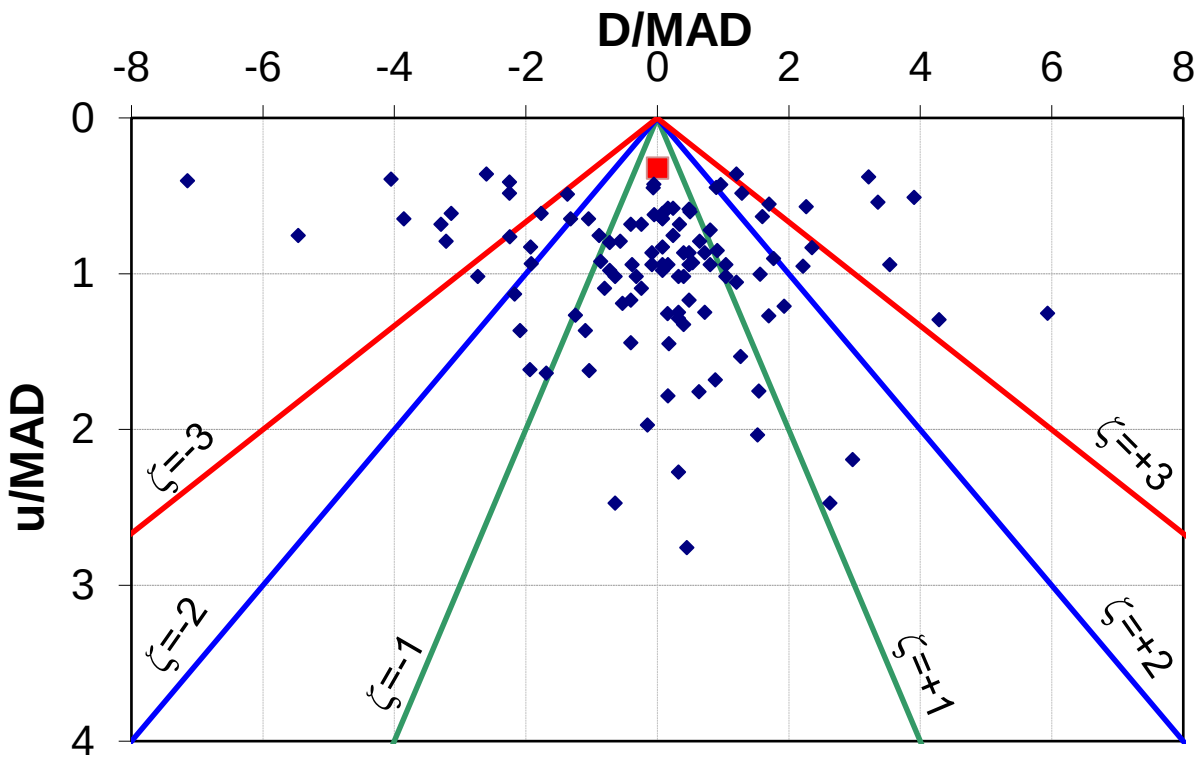
Source: JRC Geel

6.3 PomPlots

Another tool to display the results is a PomPlot. Detailed information regarding this graph can be found in (Spasova, et al., 2007). If the reported result and its uncertainty are correctly assessed by the laboratory, the result on the graph should be found in between the green lines (ζ=1). The laboratories with results close to the top of the pyramid, between the green lines (ζ=1), reported the most accurate results with small uncertainties (see Annex 12). The MAD is the Median of the Absolute Deviation and is used as it is a robust measure of the typical statistical spread of the data set, which is useful for normalising the axes in the PomPlot. D is the deviation from the reference value and u is the standard uncertainty of the laboratory and the reference value.

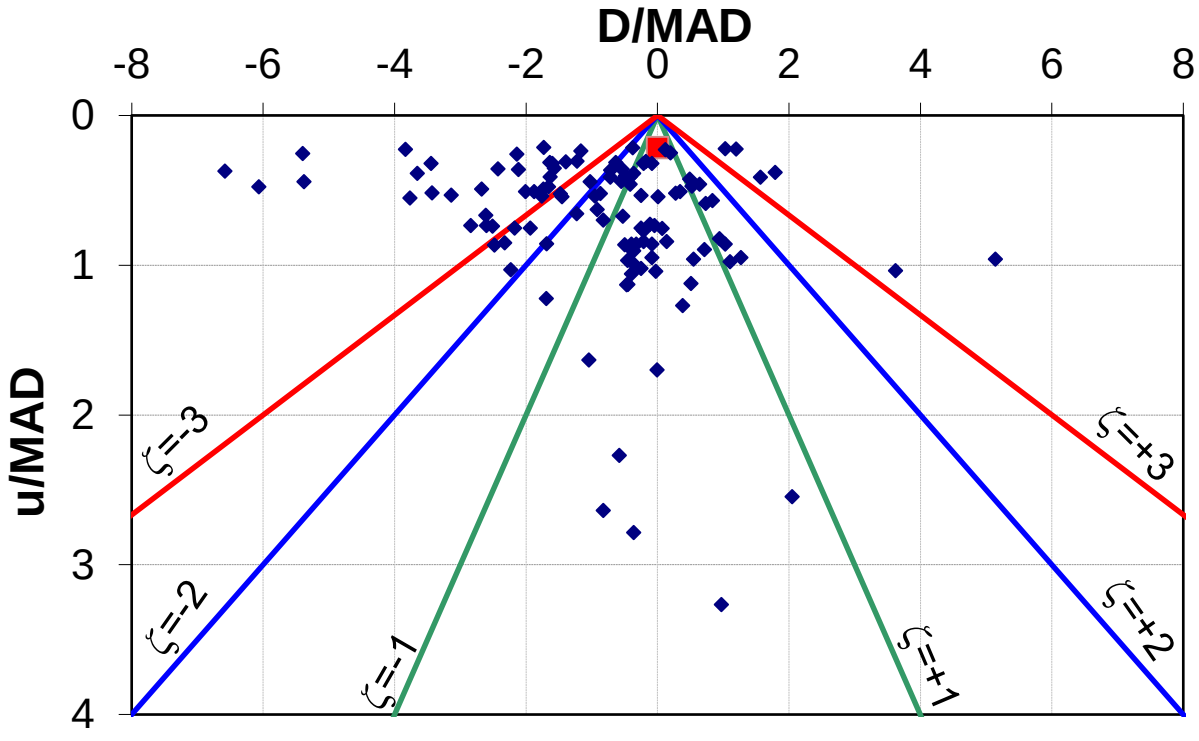
The PomPlots based on the results of this PT are presented in Figures 10 to 13. For ^{131}I most of the results are gathered in the centre. There are however some results falling outside the red lines (ζ=3). There is also a group of laboratories with high uncertainties (u/MAD>1.5). For ^{134}Cs there are many laboratories with too low results (left part of the graph, outside the green, blue and red lines). The same group of laboratories also seem to underestimate their uncertainties (u/MAD<0.7). The results are similar for ^{137}Cs except that there is not a distinct group on the left hand side of the plot as in the case of ^{134}Cs. Also for ^{40}K it can be noted that some laboratories underestimate the uncertainties and that more laboratories have too high results than too low.

Figure 10 PomPlot of the ^{131}I results. Green, blue and red solid lines indicate ζ scores. The reference value is presented as a red square, the participants' results are presented as blue diamonds



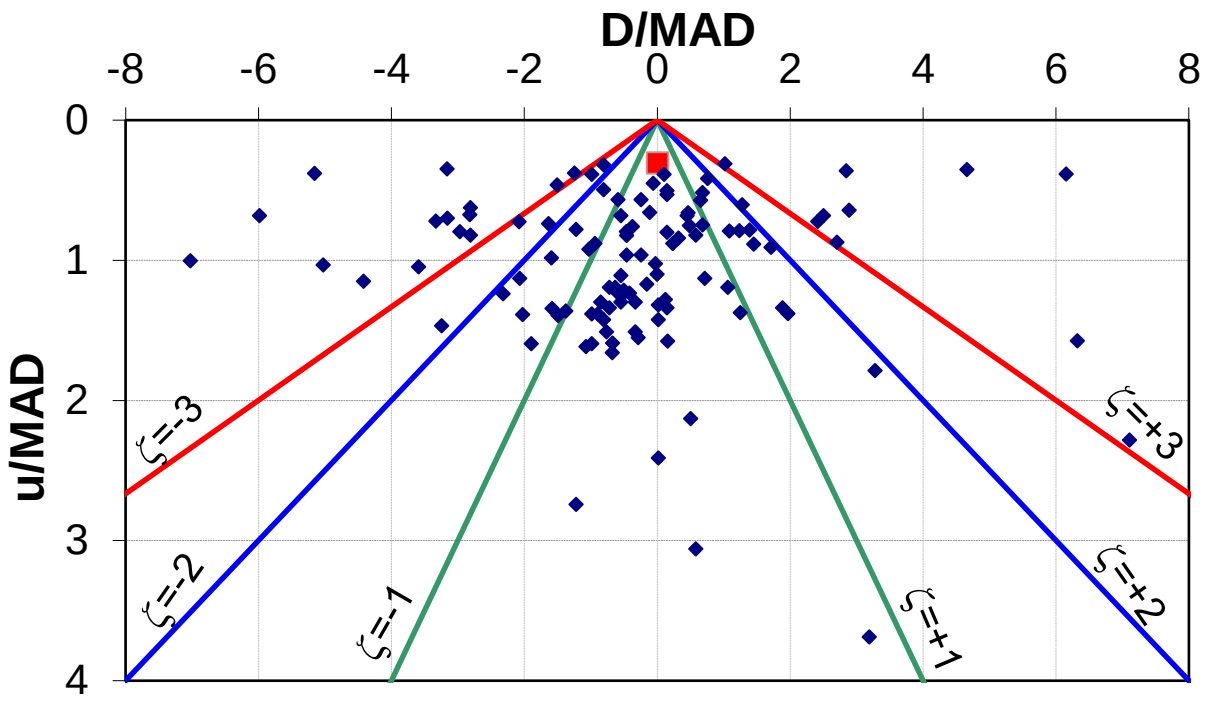
Source: JRC Geel

Figure 11 PomPlot of the ^{134}Cs results. Green, blue and red solid lines indicate ζ scores. The reference value is presented as a red square, the participants' results are presented as blue diamonds



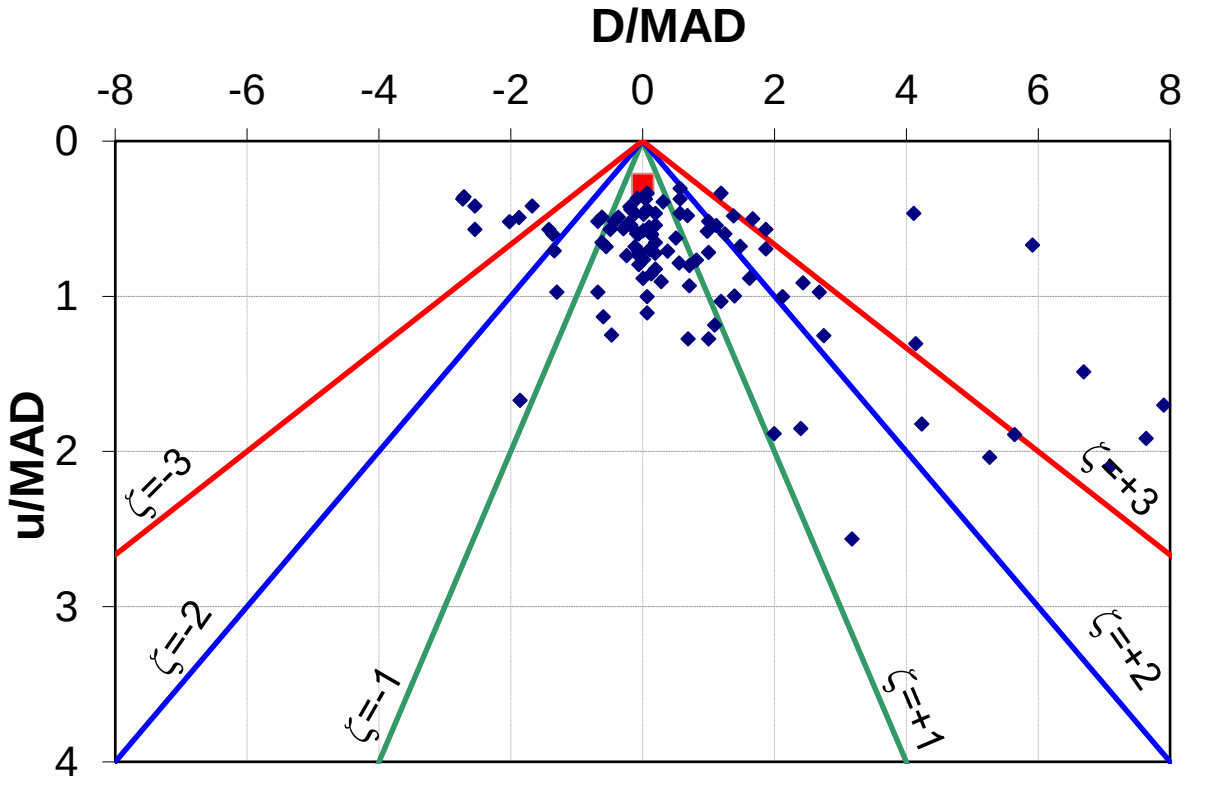
Source: JRC Geel

Figure 12 PomPlot of the ^{137}Cs results. Green, blue and red solid lines indicate ζ scores. The reference value is presented as a red square, the participants' results are presented as blue diamonds



Source: JRC Geel

Figure 13 PomPlot of the ^{40}K results. Green, blue and red solid lines indicate ζ scores. The reference value is presented as a red square, the participants' results are presented as blue diamonds



Source: JRC Geel

6.4 Possible influencing parameters

Based on the reported results and answers to the questionnaire an analysis of possible reasons for errors and areas of improvement are identified. This includes looking at parameters such as application of true coincidence summing corrections, accreditation and experience of laboratories in measurement of food or feed samples.

For ^{131}I, there was no relation between the z scores below -3 and lack of true coincidence summing correction. For ^{134}Cs, only 1 laboratory with z score below -3 and 1 with z score above 3 used more than one γ-ray line and did not apply true coincidence summing correction. The remaining laboratories with unacceptable results according to z score used only one γ-ray line, used more than one γ-ray line and applied true coincidence summing correction or did not provide any information regarding the number of γ-ray lines used. This indicates that the procedures applied for the efficiency calibration calculation and/or true coincidence summing corrections should be revised.

Eleven out of 16 laboratories with z score above 2 for ^{40}K had also z score for ^{134}Cs lower than the reference value (z score -0.14 to -3.60). For ^{134}Cs, 2 out of 5 laboratories with results lower than the reference value reported for ^{40}K results higher than the reference value (z score 1.11 and 4.49) and 2 did not report a result for ^{40}K. This may indicate that the efficiency calibration is not accurate enough for ^{40}K. If the efficiency calibration source contains ^{60}Co and/or ^{88}Y the true coincidence summing corrections must be correctly applied in the calculations of efficiencies.

7 Conclusions

Overall, the results of the participating laboratories are good and no major problems have been discerned. Some more detailed observations are:

- Although many laboratories (80) reported applying true coincidence summing corrections, the negative bias on the results for ^{134}Cs indicate that many laboratories still fail to properly apply these corrections and/or include the associated uncertainty components in the overall uncertainty budget. The same seems to be true for the efficiency calibration sources (especially those containing ^{60}Co). This is further made clear by the ^{40}K results that have a positive bias, which is likely to be caused by either failing to perform proper true coincidence summing correction on certain radionuclides in the calibration source or improper background subtraction.
- Some laboratories use CRMs for setting up calibration curves. This is not always suitable. A calibration source must have low uncertainty, which is not always the case with CRMs as some are aimed for method validation and testing.
- Some laboratories use Monte Carlo calculations for absolute efficiency calculation, which can be treacherous, as a computer models tends to be accurate only for very specific samples. Even a small error in a model can result in incorrect results. Measuring the sample at a distance of 2-8 cm (depending on a count rate) from the detector could reduce the effect of small errors in Monte Carlo calculations.
- A recurrent problem is the underestimation of uncertainties. This can be seen by the slightly worse results of ζ score compared to the z scores.
- Awareness of correct decay data is increasing but some laboratories still use data from sources that are not updated with the latest high quality evaluations. In this case the effect is however of minor importance.
- The too low amount of sample material that was used by some laboratories could possibly have influenced some results. The prescribed minimum sample intake shall be respected.

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List of abbreviations and definitions

AC	pre-accession countries
ANOVA	analysis of variance
BIPM	Bureau International des Poids et Mesures
Bq	Becquerel, SI derived unit of radioactivity
CEA	Commissariat à l'énergie atomique et aux énergies alternatives
CRM	Certified Reference Material
DDEP	Decay Data Evaluation Project
d.f.	degrees of freedom
DG	Directorate General
d.m.	dry mass
EC	European Commission
EU	European Union
EURATOM	European Atomic Energy Community
EURDEP	European Radiological Data Exchange Platform
F	test statistic used in one way ANOVA analysis
F_{crit}	value of the F statistic at the threshold probability α of mistakenly rejecting a true null hypothesis
GUM	Guide to the Expression of Uncertainty in Measurement
HPGe	high-purity germanium
IAEA	International Atomic Energy Agency
ICRM	International Committee for Radionuclide Metrology
ISO	International Organization for Standardization
JRC	Joint Research Centre
k	coverage factor
kg	kilogram
MAD	median of the absolute deviation
MILC	Management of Interlaboratory Comparisons software
MS	member states (of the European Union)

MSB	between-bottle variance
MSW	within-bottle variance
PMM	power-moderated mean
PT	proficiency testing
PTB	Physikalisch-Technische Bundesanstalt
REM	Radioactivity Environmental Monitoring
SS	sum of squares
S_{bb}	between bottles standard deviation
S_{wb}	within bottles standard deviation
StDev	standard deviation
u_{bb}	uncertainty contribution due to inhomogeneity
u_{bb}*	estimation of uncertainty contribution due to inhomogeneity
u_{char}	uncertainty from the power-moderated mean calculated for the results of the characterisation study
u_{hom}	uncertainty component associated with reference material heterogeneity (this report equal to u_{bb}*)
U_{pt}	expanded uncertainty of the reference value
u_{pt}	uncertainty of the reference value
x_{pt}	reference value

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Annexes

Annex 1. Letter of invitation for laboratory nomination

Ref. Ares(2017)1671938 - 29/03/2017

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate G - Nuclear Safety and Security
Standards for Nuclear Safety, Security and Safeguards

Geel, 29 March 2017

Subject: Article 35-36 of the Euratom Treaty
EC Proficiency Testing on I-131, Cs-134, and Cs-137 in maize powder running under the ICS-REM* programme

Ms	M. J. Bação Madruga (PT)	Mr	A. Abramenkovs (LV)	P. Lipinski (PL)
	H. Halachliyska (BG)		K. G. Andersson (DK)	A. Maltezos (EL)
	R. Kamenova-Totzeva (BG)		P. Andersson (SE)	G. Manificat (FR)
	S. Krca (HR)		P. Brejza (MT)	G. Menna (IT)
	M. Lecomte (LU)		O. Chochola (CZ)	S. P. Nielsen (DK)
	M. Lepasson (EE)		M. Cindro (SI)	J. Peter (DE)
	C. McGuire (UK)		J. Claes (BE)	A. Polt (EE)
	L. Peake (UK)		P. Demetriades (CY)	S. Runacres (UK)
	S. Quell (LU)		J. J. Diana (FR)	L. Sombré (BE)
	M. R. Salas (ES)		V. Jurina (SK)	R. Stapel (DE)
	E. Simion (RO)		C. Katzlberger (AT)	G. Torri (IT)
	P. Vesterbacka (FI)		K. Kelleher (IE)	M. Tzortzis (CY)
	B. Vilimaite Silobritiene (LT)		P. Kwakman (NL)	A. Vincze (HU)
			F. Leprieur (FR)	

Dear colleague,

As you know, EU Member States are obliged under Art. 35 and 36 of the EURATOM Treaty to inform the European Commission (EC) on a regular basis of the radioactivity levels in their environment. In order to obtain more information on the measurement methods and on the quality of the values reported by the Member States, the EC Directorate-General Joint Research Centre (DG JRC) is organising Proficiency Testing (PT) exercises for the MS laboratories. These PTs are organised under the ICS-REM* programme in which the EC is testing measurement capabilities as well as providing technical support to the participating laboratories.

After discussions with DG ENER.D.3 and during the Euratom Treaty Art. 35-36 Experts meetings, it was agreed that next PT exercise will be on **I-131, Cs-134, and Cs-137 measurements in maize powder**.

The schedule for the PT is as follows:

- early June 2017 – PT material (maize powder) dispatched
- end of July 2017 – participating laboratories requested to send their results to the JRC
- September 2017 – preliminary report available
- t.b.d. – workshop and short training course for participating laboratory practitioners

I would like to encourage you to investigate which laboratories in your country would be interested in participating in this exercise or which laboratories you would like to see

*ICS-REM – International Comparison Scheme for Radioactivity Environmental Monitoring

participating and provide us with the contact data of the nominated laboratories (responsible person, complete postal address, telephone, and e-mail). To proceed according to the plan, we require your (nationally coordinated) response by **28 April 2017**.

Please, send your replies to the functional e-mail box
JRC-GEE-REM-COMPARISONS@ec.europa.eu

Looking forward to hearing from you with the laboratory nominations,

Yours sincerely,

Katarzyna Sobiech-Matura

Project Coordinator

Joint Research Centre
Nuclear Safety and Security
Standards for Nuclear Safety, Security & Safeguards
Retieseweg 111
B-2440 Geel
Belgium

cc: Messrs. Michael Hübel, Vesa Tanner, Alan Ryan (DG ENER.D3)
Mr. Marc De Cort (JRC Ispra)
Messrs. Willy Mondelaers, Mikael Hult, Viktor Jobbagy (JRC Geel)

Annex 2. Invitation to laboratories for registration

Ref. Ares(2017)2398937 - 11/05/2017

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate G - Nuclear Safety and Security (Karlsruhe)
Standards for Nuclear Safety, Security and Safeguards

Geel, 10/05/2017

EC Proficiency Test on ^{131}I, ^{134}Cs, and ^{137}Cs activity measurements in maize powder

Dear colleague,

EU Member States are obliged under Art. 35 and 36 of the Euratom Treaty (and as further specified in Commission Recommendation 2000/473/Euratom) to inform the European Commission (EC) on a regular basis on the radioactivity levels in their environment, in some food products and in drinking water. In order to obtain more information on the quality of the values reported by the Member States, the EC (through its Directorate General JRC) organises regularly proficiency testing exercises for monitoring laboratories.

Your laboratory has been nominated by your national representative(s) or authority to participate in EC Proficiency Test (PT) on ^{131}I, ^{134}Cs, and ^{137}Cs measurements in maize powder organised by DG JRC Geel.

Material information

The material consists of maize powder containing elevated levels of ^{131}I, ^{134}Cs, and ^{137}Cs. The levels of activity for all of these radionuclides are below the exemption levels such that the material can be transported freely and handled in the laboratory without any radiological restrictions. In order to obtain material containing elevated levels of short-lived radionuclides spiking with radioactive solutions was applied in case of all three radionuclides.

The material was homogenized and bottled in units of approximately 100 g. The water content of the material after bottling was determined, but it needs to be re-measured in each laboratory.

Protocol for the PT

1. Each participant shall receive one bottle of about 100 g maize powder. The activity concentration (massic activity, in **Bq/kg**) of each of the radionuclides **^{131}I, ^{134}Cs, and ^{137}Cs shall be determined**. The specific activity of ^{40}K can also be reported, but it will not be used for the performance evaluation and will be treated only as an information value.
2. The laboratory may use a measurement procedure of its choice, which preferably is consistent with routine procedures used in the laboratory.
3. All results are to be reported **normalized to dry mass**. Water/moisture content is to be determined by the participant on small aliquots that will NOT be used for the radionuclide determination. Water/moisture content should be determined by Karl-Fischer-titration or oven-drying (the detailed procedure will be send together with the PT material).
4. The minimum sample intake for radionuclide analysis is 50 g. It can be reduced down to 20 g if a correspondingly higher number of samples is analysed.

Retieseweg 111, B-2440 Geel - Belgium.

5. The reference date to be used is **1 June 2017 00:00 UTC**. As source of nuclear decay data, the use of Monographie BIPM-5 *, available also under the following link http://www.nucleide.org/DDEP_WG/DDEPdata.htm, is recommended.
6. Web-based registration is obligatory for all laboratories participating in this PT. The deadline for the registration is **31 May 2017**. Please read carefully the attached instructions for registration. Note that the registration in only definitive when we receive a **signed registration form** from you. The signed form can be send by e-mail, fax or post. We kindly ask you to register your laboratory via the following link: https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1742
7. The reporting of laboratory results will be done in two ways:
    - **Emergency reporting (voluntary)**, the results of which will remain anonymous and are collected as a part of a scientific study. This reporting will be collected via e-mail up to 48 h from the moment of sample receipt.
    - **Routine reporting (mandatory)** the results of which will be revealed to relevant authorities and used for performance evaluation. It will be conducted via the JRC online reporting tool. Relevant link will be provided during sample dispatch.
8. Timing and deadlines:
    - Registration deadline: **31 May 2017**
    - Sample dispatch: early June 2017
    - Emergency reporting (anonymous): **48 h** from the moment of sample receipt
    - Routine reporting deadline: **28 July 2017**

Preliminary information on the individual laboratory's performance will be sent by e-mail in September 2017. The final report of this comparison exercise is foreseen to be available beginning of 2018. Also for the beginning of 2018 a workshop for the participants to discuss the results of the performance evaluation is planned.

The results and performance on the basis of the routine reporting of each laboratory will be made available to its national representative(s) (the nominating authority) and to the relevant services of the European Commission at Directorate General for Energy. Apart from informing these authorities, each laboratory's results will be treated anonymously.

Should you have any question, please feel free to contact us at:
JRC-GEE-REM-COMPARISONS@ec.europa.eu

Looking forward to hearing from you,
Yours sincerely,

Katarzyna Sobiech-Matura
Project Coordinator

European Commission
DG Joint Research Centre
Directorate G - Nuclear Safety & Security
Unit G2 - Standards for Nuclear Safety, Security and Safeguards
Retieseweg 111
B-2440 Geel, Belgium
+32 14 571290
JRC-GEE-REM-COMPARISONS@ec.europa.eu
http://ec.europa.eu/dgs/jrc

*Monographie BIPM-5, Table of Radionuclides, 2006, 2013 and 2016. Bé, M.-M., Christé, V., Dulieu, C., Browne, E., Chechev, V., Kuzmenko, N., Helmer, R., Nichols, A., Schönfeld, E., Dersch, R. Bureau International des Poids et Mesures, Pavillon de Breteuil, F-92310, Sèvres, France.

Annex 3. Sample dispatch information

Subject: EC PT on I-131, Cs-134 and Cs-137 in maize powder - samples dispatch

Thu 01/06/2017 15:23

Dear colleague,

Your laboratory is registered for the EC PT on I-131, Cs-134 and Cs-137 in maize powder. We have started dispatching the samples today and due to a large number of participants we will continue with the shipment during the following days.

The parcel will contain:

- a bottle with spiked maize powder,
- instruction for moisture content determination,
- a letter with **your password key** required for the routine reporting of results.

The reporting of laboratory results will be done in two ways:

- **Emergency reporting (voluntary)**, the results of which will remain anonymous and are collected as a part of a scientific study. This reporting will be collected up to **48 h** from the moment of sample receipt by filling in the attached excel file and sending it back to the e-mail address: JRC-GEE-REM-COMPARISONS@ec.europa.eu. The most important is the **Tab nr. 2 (results)**, but we will be grateful if you could fill in also the other information.
- **Routine reporting (mandatory)** the results of which will be revealed to relevant authorities and used for performance evaluation. The reporting of the results is done via the following URL: https://web.jrc.ec.europa.eu/ilcReportingWeb and will be opened from **5 June 2017** to **28 July 2017**. To report your results, you will need your password key which is unique to this proficiency test and your laboratory. Please find the attached pdf file with instructions for the routine reporting.

Should you have any question, please feel free to contact us at:

Email: JRC-GEE-REM-COMPARISONS@ec.europa.eu

Looking forward to hearing from you,

Yours sincerely,

Katarzyna Sobiech-Matura
Project Coordinator

Petya Malo
Logistic Assistant

European Commission
DG Joint Research Centre
Directorate G - Nuclear Safety & Security

Unit G2 - Standards for Nuclear Safety, Security and Safeguards

Retieseweg 111
B-2440 Geel, Belgium
+32 14 571290
JRC-GEE-REM-COMPARISONS@ec.europa.eu

http://ec.europa.eu/dgs/jrc

Annex 4. Sample accompanying letter

Ref. Ares(2017)2739755 - 31/05/2017

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate G - Nuclear Safety and Security (Karlsruhe)
Standards for Nuclear Safety, Security and Safeguards

Geel, 01 June 2017

«Title» «First_Name» «Surname»
«Company»
«Department»
«Street_Name»
«Zip_Code» «City»
«Country»

Participation in the EC Proficiency Test on ^{131}I, ^{134}Cs and ^{137}Cs activity measurements in maize powder

Dear «Title» «Surname»,

Thank you for participating in the EC proficiency test (PT) on ^{131}I, ^{134}Cs and ^{137}Cs measurements in maize powder.

This parcel contains:

a) Test item: glass bottle of maize powder spiked with radioactive solutions of ^{131}I, ^{134}Cs and ^{137}Cs (100 g).
b) Instruction for moisture content determination.
c) This accompanying letter.

Important notes

1) Please confirm the receipt of the sample by e-mail to JRC-GEE-REM-COMPARISONS@ec.europa.eu.

2) Please check the bottle carefully and in case of any damage report to the above e-mail address.

3) **Please keep this letter** as you will need it for the routine reporting of your results.

4) For the measurement and analysis, you should follow the procedure you routinely use.

5) The minimum sample intake for radionuclide analysis is 50 g. It can be reduced down to 20 g if a correspondingly higher number of samples is analysed.

6) The results must be reported for the reference date of **1 June 2017 00:00 UTC**.

7) For your calculations, we recommend to use the data provided by the Decay Data Evaluation Project (DDEP) at http://www.nucleide.org/DDEP_WG/DDEPdata.htm

JRC Geel
Retieseweg 111, B-2440 Geel - Belgium. Telephone: +32 (0)14 571 290

E-mail: JRC-GEE-REM-COMPARISONS@ec.europa.eu

8) Measure the spiked maize powder as soon as possible after reception, as the activity of the relatively short-lived ^{131}I will be significantly reduced by time.

9) Remember to determine the moisture content of the material according to the enclosed instruction. All results are to be reported **normalized to dry mass**.

Reporting of the results

Please report massic activities of ^{131}I, ^{134}Cs and ^{137}Cs. The specific activity of ^{40}K can also be reported, but only as an information value, which will not be used later on for performance evaluation. The reporting of the results, as it was announced before, will be done in two ways:

- **Emergency reporting (voluntary)**, the results of which will remain anonymous and are collected as a part of a scientific study. This reporting will be collected up to **48 h** from the moment of sample receipt to the e-mail address: to JRC-GEE-REM-COMPARISONS@ec.europa.eu.
- **Routine reporting (mandatory)** the results of which will be revealed to relevant authorities and used for performance evaluation. The reporting of the results is done via the login page using the following URL: https://web.jrc.ec.europa.eu/ilcReportingWeb and will be opened from **5 June 2017** to **28 July 2017**. To report your results you need a password key which is unique to this proficiency test and your laboratory.

Your password key is: **«Password_key»**

Please note that only **submitted** results will be taken into account, therefore, do not only ***Save*** your results but also click on the ***Submit*** button. Once you have submitted your results and questionnaire, please remember to send us a signed copy by e-mail to JRC-GEE-REM-COMPARISONS@ec.europa.eu

The uncertainty of the reported results must be in the same units as the massic activity (i.e. **Bq/kg d.w.**) with the coverage factor **k=2**.

The description of your analytical and measurement procedures will be collected via questionnaire using the same URL link as for reporting the results. We kindly ask you to answer all relevant questions regarding the procedures you employed for the measurement of the test sample. Disregard questions which are not relevant to the methods used in your laboratory.

Please, notice that during the reporting of your results the *Cancel* button serves as an exit or return button.

Should you have any question, please do not hesitate to contact us.

We wish you success with your measurements.

Kind regards,

Katarzyna SOBIECH-MATURA
Project Coordinator

Annex 5. Instruction for moisture content determination

Ref. Ares(2017)2739755 - 31/05/2017

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate G - Nuclear Safety and Security (Karlsruhe)
Standards for Nuclear Safety, Security and Safeguards

Moisture content determination

The moisture content is to be determined in one or two small aliquots (sub-samples) that will NOT be used for the radionuclide determination. Aliquots of 1 g to 2 g spread out evenly in the drying/weighing container should be used. Each sub-sample should be placed in a container where it will remain throughout this test.

The sub-sample(s) of the maize powder material to be tested for moisture content should be prepared at the same time and from the same bottle as the PT sample(s) for radionuclides determination.

EQUIPMENT

- Weighing device: A balance or scale sensitive to 0.1% of the mass of the sub-sample.
- Drying device: An oven or another suitable thermostatically controlled heating chamber calibrated and capable of maintaining a temperature up to (105 ± 2) °C.

MATERIALS

- Closed container withstanding the drying temperature and suitable to contain the test sample without loss while permitting the water to evaporate.

PROCEDURE

1) Label each container with a sub-sample ID.

2) Determine the mass of the container.

3) Determine the mass of the sub-sample and the container, subtract the mass of the container and record the remaining value mass as the "Wet mass" (m_{wet}).
   The mass of the sub-sample should be determined immediately after preparation, as a cover on the container does not completely prevent evaporation or absorption of water.

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E-mail: JRC-GEE-REM-COMPARISONS@ec.europa.eu

4) Dry in the drying device at 105 °C for one hour.

5) Remove the sub-sample from the drying device and cool to room temperature in a desiccator.

6) Determine the mass of the sub-sample.

   The mass of the sub-sample should be determined immediately after cooling, each time using the same time interval, e.g. 20 minutes.

7) Repeat steps 6 to 7 until constant mass is attained (separate weighings should not differ by more than 5 mg). Record the last weighing as the "Dry mass" (m_{dry}).

CALCULATION

Determine the moisture content (w) as follows:

$$w = \frac{m_{wet} - m_{dry}}{m_{wet}} * 100\ (\%)$$

PRECAUTIONS

The drying rate of sub-samples will be affected by the moisture conditions and number of samples in the drying device. Avoid placing wet samples in the drying device together with nearly dry samples, to avoid possible absorption of moisture into the dry samples.

Annex 6. Communication of preliminary results

Ref. Ares(2017)5212011 - 25/10/2017

24 October 2017

Subject: Preliminary results of the EC PT 2017 on I-131, Cs-134 and Cs-137 measurements in maize powder

Dear «Firstname» «Surname»,

First of all, thank you for your participation in the EC PT 2017 on ^{131}I, ^{134}Cs and ^{137}Cs measurements in maize powder and your timely provision of results. Currently, we are evaluating the results for the final report. However, for your information we are sending you a preliminary evaluation of the results of this comparison in the form of graphs attached to this email. As anonymity is a requirement, each laboratory was assigned a code number.

The code number for your laboratory is «Lab_Code».

With this number you can identify your laboratory in each of the graphs and find out how your reported result compares to the reference value.

In Figures 1, 2 and 3 the reported massic activities of ^{131}I, ^{134}Cs and ^{137}Cs respectively, with their corresponding expanded uncertainties (k = 2), are plotted in ascending order. The solid red line, present in each graph, indicates the reference value of massic activity for the examined radionuclide. The black dashed lines represent the expanded uncertainty of the reference value. The red dashed lines are representing the ± 20 % deviation from the reference value, as the σ_{PT} (following the definition in ISO13528:2015) was set to 0.2 for each radionuclide in this PT. The values of massic activities for all examined radionuclides are presented in Table 1. The reference date is 01 June 2017 0:00 UTC. The reported values of ^{40}K were also evaluated but as they were not formally part of the proficiency test we will not report them at this stage.

Table 1. The reference massic activities with their expanded uncertainties (k = 2).

Radionuclide used for spiking	Massic activity (Bq·kg^{-1} d.m.)
^{131}I	191 ± 16
^{134}Cs	901 ± 45
^{137}Cs	547 ± 28

The final report of this PT exercise is foreseen to be available by the end of January 2018. Please let us know if you wish to receive a printed version. We will distribute it then during the workshop, which will take place on 31 January 2018. The report will also be published online as a pdf file.

We would like to invite you on this occasion to participate in the workshop as well as in the associated gamma-ray spectrometry training, which will take place on 30 January and 1 February 2018. A lab tour is foreseen for 2 February. More details can be found in the leaflet attached to this email. You can register online for all these events using the following link:

https://web.jrc.ec.europa.eu/rem/app.html?+-+%2Fsubscription-form-screen%2FmeetingId=94151#/subscription-form-screen/meetingId=96151

The deadline for registration is 15 December 2017.

If you have any further questions with respect to this comparison, please feel free to contact us at JRC-GEE-REM-COMPARISONS@ec.europa.eu.

Yours sincerely,

Katarzyna Sobiech-Matura	Mikael Hult	Petya Malo
Project Coordinator	*Team Leader*	*Logistics Assistant*

European Commission
DG Joint Research Centre
Directorate G - Nuclear Safety & Security
Unit G2 - Standards for Nuclear Safety, Security and Safeguards
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B-2440 Geel, Belgium

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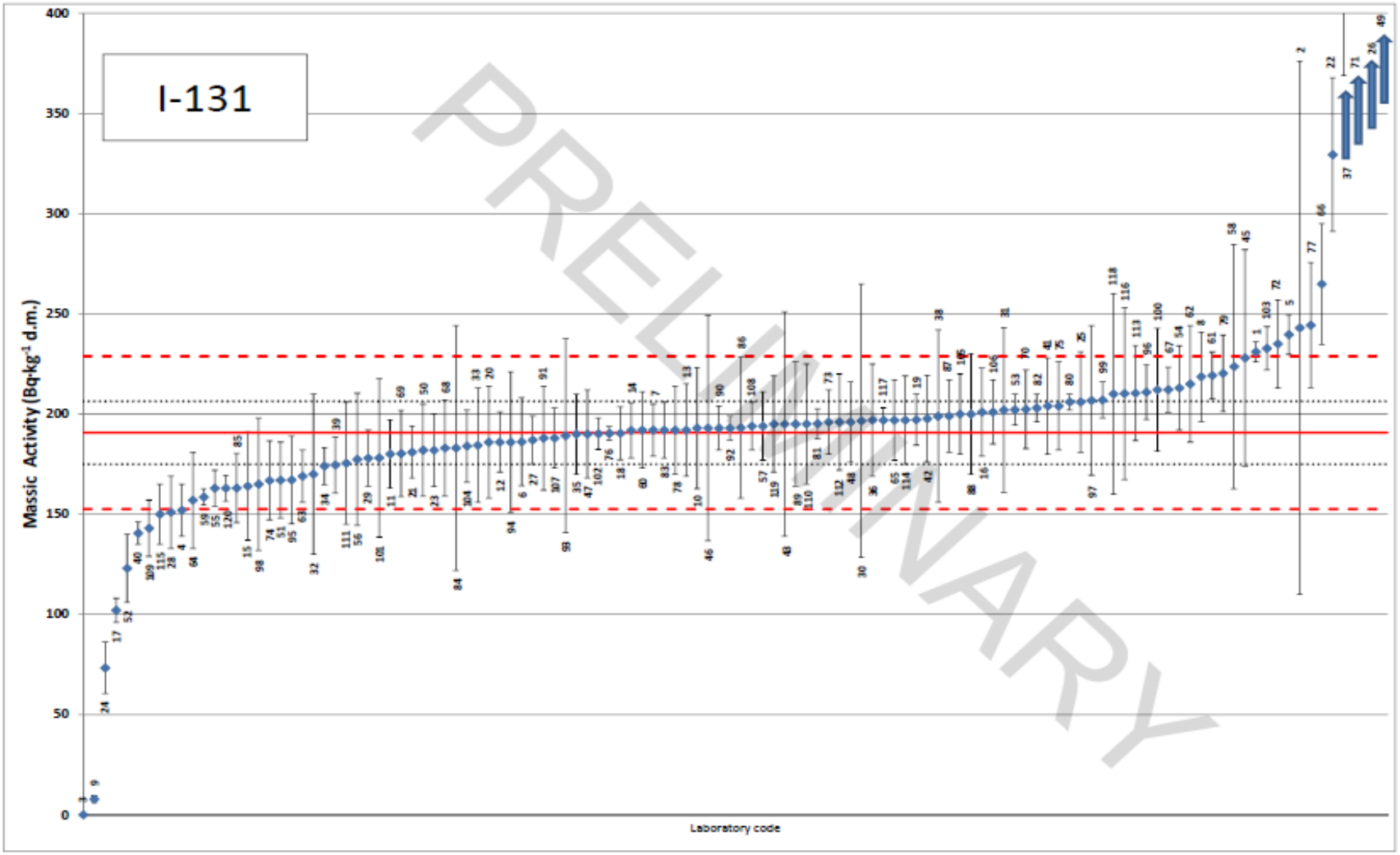
http://ec.europa.eu/dgs/jrc

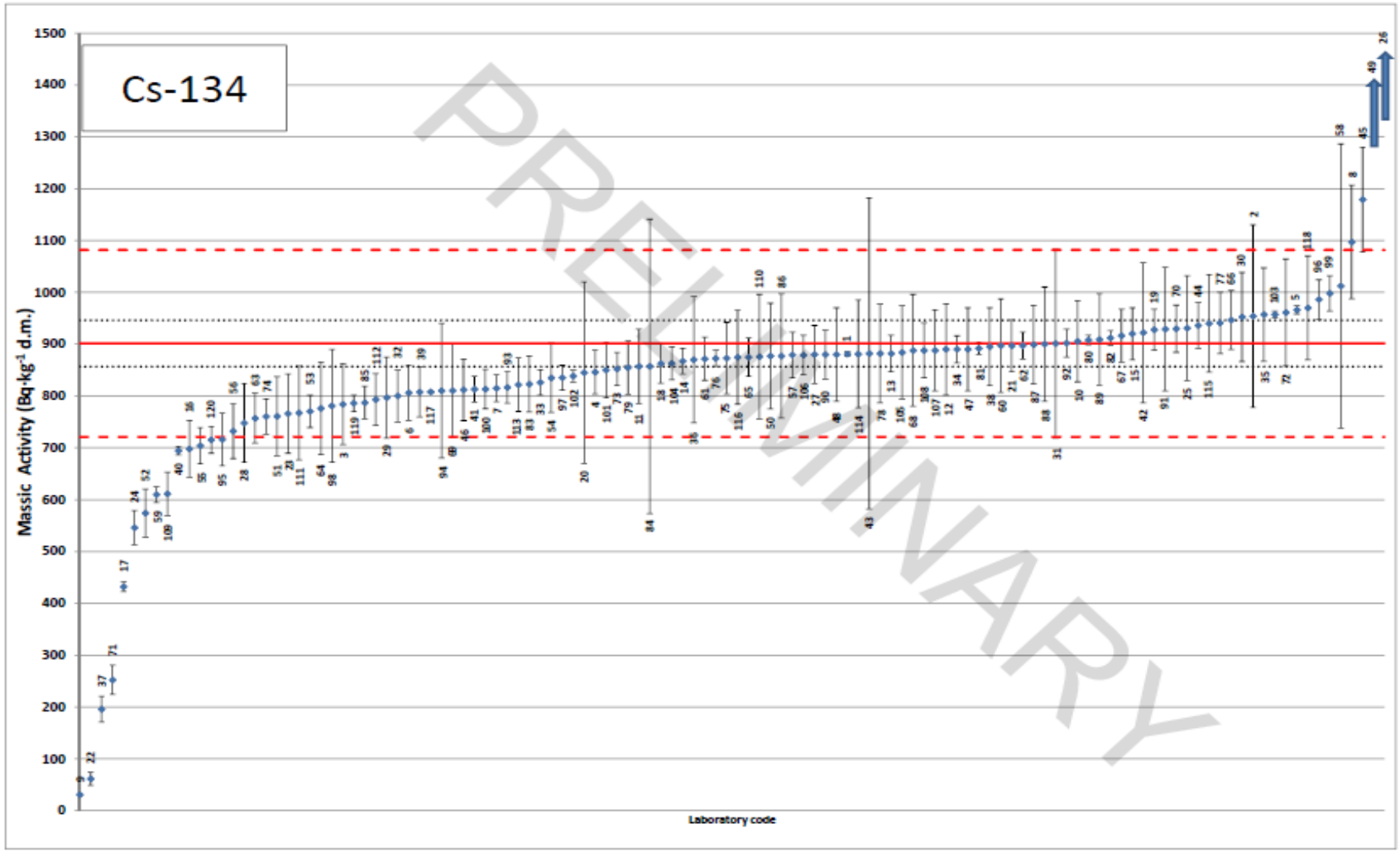
Preliminary results of the 2017 EC Proficiency Test on I-131, Cs-134 and Cs-137 measurements in maize powder



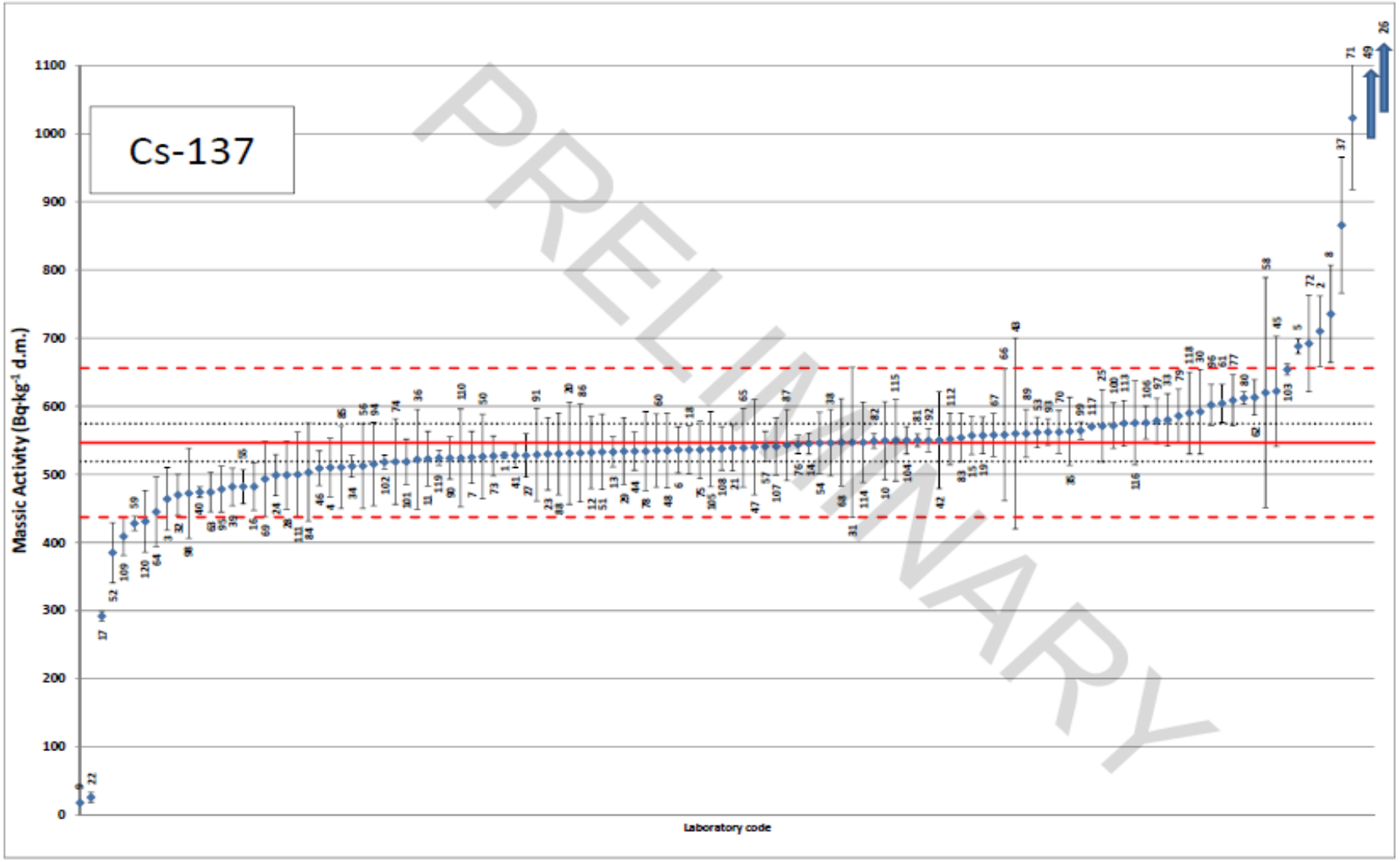
24/10/2017

European Commission

Preliminary results of the 2017 EC Proficiency Test on I-131, Cs-134 and Cs-137 measurements in maize powder



European Commission


Cs-137
Massic Activity (Bq·kg⁻¹ d.m.)
Laboratory code
PRELIMINARY

Annex 7. List of participating laboratories (countries in alphabetical order)

AUSTRIA

Austrian Agency for Health and Food Safety (AGES)
STRA
Spargelfeldstraße 191
1220 Vienna

Austrian Agency for Health and Food Safety (AGES)
Radon and Radioecology
Wieningerstrasse 8
4020 Linz

BELGIUM

IRE-ELIT
LMR
Avenue de l'Espérance 1
6220 Fleurus

SCK•CEN
EHS-LRM
Boeretang 200
2400 Mol

BOSNIA-HERZEGOVINA

Institute for Public Health of Federation of Bosnia and Herzegovina
Radiation Protection Centre
Marsala Tita 9
71000 Sarajevo

BULGARIA

Executive Environment Agency
Regional Laboratory - Pleven
Storgoziya District PBox:525
"Measures and Measuring Devices", RO Building, floor 4
5800 Pleven

Executive Environment Agency
Regional Laboratory - Vratza
Exarh Josif 81
3000 Vratza

Executive Environment Agency
Regional Laboratory – Montana
"Jlius Irasek" Str. 4
3400 Montana

Executive Environment Agency
Blvd. Tzar Boris III, 136
1618 Sofia

National Center of Radiobiology and Radiation Protection (NCRRP)
Radiation Protection
Georgi Sofiiski Blvd. 3
1606 Sofia

Regional Health Inspectorate of Burgas
Control of radiation
Alexandrovska str.120
8000 Burgas

CROATIA

Institute for Medical Research and Occupational Health
Radiation Protection Unit
Ksaverska cesta 2
10000 Zagreb

Ruđrer Bošković Institute
Laboratory for radioecology
Bijenička cesta 54
10000 Zagreb

CYPRUS

State General Laboratory of Cyprus
Kimonos Str. 44
1451 Nicosia

CZECH REPUBLIC

Statni ustav radiacni ochrany, v.v.i.
Hradec Kralove
Pileticka 57
500 03 Hradec Kralove

National Radiation Protection Institute (SÚRO)
Monitoring
Bartoškova 28
140 00 Prague

DENMARK

Technical University of Denmark
Center for Nuclear Technologies
Frederiksborgvej 399, Building 201
4000 Roskilde

ESTONIA

Environmental Board Republic of Estonia
Radiation Safety Department
Kopli 76
10416 Tallinn

University of Tartu
Institute of Physics
W. Ostwaldi Str 1
50411 Tartu

FINLAND

STUK - Radiation and Nuclear Safety Authority
VALO
Laippaite 4
00880 Helsinki

FRANCE

Institut de Radioprotection et de Sureté Nucléaire (IRSN)
STEME
31 rue de l'Ecluse,
BP 40035
78116 Le Vésinet

GERMANY

Max Rubner-Institute (MRI)
Safety & Quality of Milk &Fish
Hermann-Weigmann-Strasse 1
24103 Kiel

GREECE

Aristotle University of Thessaloniki
Nuclear Technology Laboratory
Egnatia street
54124 Thessaloniki

HUNGARY

National Food Chain Safety Office
Food and Feed Safety Directorate
Fogoly utca 13-15
1182 Budapest

University of Pannonia
Institute of R & R
Egyetem str. 10
8200 Veszprém

National Public Health Institute
Anna street 5
1221 Budapest

Government Office for Gyor-Moson-Sopron County
National Public Health
Josika Street 16
9024 Gyor

Hungarian Academy of Sciences Centre for Energy Research
29-33 Konkoly T M
1121 Budapest

Government Office of the Capital City Budapest
Nemetvölgyi 37-39
1124 Budapest

Hungarian Defence Forces CBRN Area Control Centre (MH GAVIK)
Radiological Laboratory
Jaszberenyi ut 39-45
1106 Budapest

IRELAND

Environmental Protection Agency
Radiation Monitoring
3 Clonskeagh Square, Clonskeagh Road

Dublin 14 Dublin

ITALY

ARPA Lombardia
CRR Milano
Via Filippo Juvara 22
20129 Milan

ARPA Lombardia
CRR sede di Bergamo
via Clara Maffei 4
24121 Bergamo

ARPACAL
RC - Physical Laboratory
Via Troncovito SNS
89135 Reggio Calabria

ARPAS - Agenzia Regionale per la Protezione dell'Ambiente della Sardegna
DTS-CMVA Agenti Fisici
Viale F. Ciusa 6
09131 Cagliari

ARPA Sicilia
S.T. Catania
Via Carlo Ardizzone, 35
95124 Catania

Arpa Sicilia
Struttura Territoriale Palermo
via Nairobi 4
90129 Palermo

Istituto Zooprofilattico Sperimentale Lazio e Toscana "M.Aleandri"
Produzioni Zootecniche
Via Appia Nuova, 1411
00178 Rome

Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata
CRN Radioattività, S.C.Chimica
Via Manfredonia, 20
71121 Foggia

ARPA Molise
CRR Molise
Contrada Selva Piana snc
86100 Campobasso

ARPA Lazio
Sezione Provinciale di Latina
Via G. Carducci, 7
04100 Latina

ARPA Lazio
Sezione provinciale di Viterbo
Via Montezebio 17
01100 Viterbo

A.R.P.A.Cal - Agenzia Regionale per la protezione dell'Ambiente della Calabria
Cosenza - Lab. Fisico
L. da Vinci n° 49/51
87040 Castrolibero

ARPA Valle d'Aosta
Environmental Radioactivity
Loc Grande Charrière 44
11020 Saint-Christophe

A.R.P.A.B. - Centro Regionale Radioattività
Dipartim. Provinc. di Matera
via dell'Industria snc
75100 Matera

ARPA FVG (Environmental Protection Agency of Friuli Venezia Giulia - North-East Italy)
Radiation Protection Center
Via Colugna, 42
33100 Udine

ARPA Piemonte
Dipartimento Radiazioni
Via Jervis 30
10015 Ivrea (TO)

I.S.P.R.A. - Italian National Institute for the Environmental Protection and Research
Radiometric Measurement Lab
via di Castel Romano, 100
00128 Rome

ARPAV
DRL - UO CRA-CRR
Via Alberto Dominutti 8
37135 Verona

ARPA Marche
U.O. Radioattività Ambientale
via Colombo, 106
60127 Ancona

APPA TN - Local Environmental Protection Agency
Settore Laboratorio
via Lidorno, 1
38123 Trento

Environmental Protection Agency - Tuscany Region
Radioattività e Amianto
via Ponte alle Mosse 211
50144 Florence

Arpa Piemonte
Struttura Semplice Siti Nucleari
Via Trino 89

13100 Vercelli

ARPA Umbria
Servizio Radiazioni Ionizzanti
Via Pievaiola 207 B-3
06132 Perugia

ARPAE Emilia-Romagna
CTR Radioattività ambientale
via XXI Aprile 48
29121 Piacenza

Agenzia Regionale per la Tutela dell'Ambiente - ARTA Abruzzo
Distretto Prov. di Pescara
Via Guglielmo Marconi, 51
65126 Pescara

ARPA Puglia
BARI
Via Oberdan 18/E
70126 Bari

LATVIA

Institute of Food Safety, Animal Health and Environment - "BIOR"
Lejupes Street 3
1076 Riga

LITHUANIA

National Food and Veterinary Risk Assessment Institute
Radiology
J.Kairiukscio st.10
08409 Vilnius

Radiation Protection Centre
Expertise and Exposure Monitor
Kalvariju 153
08221 Vilnius

LUXEMBOURG

Ministère de la Santé - Direction de la Santé
Division de la Radioprotection
Villa Louvigny, Allée Marconi
2120 Luxembourg

MALTA

Environmental Health Directorate
Public Health Laboratory
Merchant street
VLT1179 Valletta

MONTENEGRO

LLC Center for Ecotoxicological Research Podgorica
Radionuclide analytics Unit
Put Sarla de Gola 2
81000 Podgorica

NETHERLANDS

RIKILT Wageningen University & Research
Contaminants
Akkermaalsbos 2
6708 WB Wageningen

POLAND

Central Mining Institute
Centre for Environmental Radio
Plac Gwarkow 1
40-166 Katowice

Central Laboratory for Radiological Protection
Dosimetry Department
Konwaliowa St. 7
03-194 Warszawa

AGH University of Science and Technology
Faculty of Physics and ACS
Al. Mickiewicza 30
30-059 Krakow

Institute of Nuclear Physics (IFJ PAN)
Radzikowskiego 152
31-342 Krakow

National Centre for Nuclear Research
LPD
A. Soltana 7
05-400 Otwock

PORTUGAL

Instituto Superior Técnico
Laboratório de Proteção e Segurança Radiológica
Estrada Nacional 10 (km 139,7)
2695-066 Bobadela LRS

ROMANIA

Environmental Protection Agency Dolj
SSRM Craiova
Calea Bucuresti 150
200349 Craiova

Environmental Protection Agency
Splaiul Muresului FN
310132 Arad

Environmental Protection Agency Constanta
300 Mamaia B-dul, Room nr.19, (C.M.R. "Dobrogea" Building)
900581 Constanta

Environmental Protection Agency Iasi
Calea Chisnaului nr.43
700179 Iasi

Environmental Protection Agency Maramures
Radioactivity Station
Iza street nr 1°
430073 Baia Mare

National Environmental Protection Agency
Radioactivity Laboratory
294 Splaiul Independentei
060031 Bucharest

National Institute of Public Health - Regional Center of Public Health Cluj
Radiation Hygiene
Pasteur 6
400349 Cluj Napoca

National Institute of Public Health-Regional Center Iasi
Radiation Laboratory
Victor Babes 14
700465 Iasi

Institute for Hygiene and Veterinary Public Health
Chemistry and Radioactivity
Campul Mosilor no. 5, sector 2
021201 Bucharest

SERBIA

Vinča Institute of Nuclear Science
Chemical Dynamics Department
Mike Petrovića Alasa 12-14
11351 Belgrade

Vinča Institute of Nuclear Science
Laboratory 011
Mike Petrovića Alasa 12-14
11351 Belgrade

Vinča Institute of Nuclear Science
Radiation and Envir.Protection
Mike Petrovića Alasa 12-14
11351 Belgrade

Institute for the Application of Nuclear Energy-INEP
Banatska 31b
11080 Belgrade

ANAHEM d.o.o
Mocartova 10
11160 Belgrade

SLOVAKIA

Slovenské elektrárne, a.s.
Závod Atómové elektrárne Mochovce
935 39 Mochovce

Atómové elektrárne Bohunice
Off-site radiation monitoring
Okružná 14
91701 Trnava

VUJE, Inc.
Okružná 5
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Annex 8. Questionnaire

Milc questionnaire

Comparison for EC Proficiency Test 2017

The description of your measurement(s) with detailed information about the measurement method(s) will be collected based on your answers to this questionnaire. We kindly ask you to answer all relevant questions regarding the procedure(s) that you have used. The questions mandatory to answer are marked with an asterisk (*). If it might be useful, you can send separate files by e-mail to Katarzyna.SOBIECH-MATURA@ec.europa.eu Reporting of results for K-40 is not mandatory. Uncertainties should be reported with the coverage factor k=2. Please respect the deadline for the submission of the results and the questionnaire: 28/07/2017. Thank you very much for your cooperation.

Submission Form

1. **General**

1.1. Which are the main activities carried out in your laboratory? (More than one option possible) *

- ☐ a) Research and development
- ☐ b) Monitoring of radioactivity in the environment
- ☐ c) Monitoring of nuclear facilities
- ☐ d) Measurements for fissile material control or safeguards
- ☐ e) Other

1.1.1. If other, please specify

1.2. Your laboratory is: (More than one option possible) *

- ☐ a) Certified (ISO 9000)
- ☐ b) Accredited (ISO 17025)
- ☐ c) Authorised
- ☐ d) Other

1.2.1. If accredited, which method(s) is/are accredited? (e.g. liquid scintillation counting: tritium in water)

2. **Samples**

2.1. On which kind of food/feed samples does your laboratory routinely perform gamma-ray spectrometric analysis? *

2.2. How many food/feed samples are analysed by gamma-ray spectrometry in your laboratory per year? *

2.3. Which radionuclides at which range of activity (in Bq/kg dry weight (d.w.)) are present in the food/feed samples your laboratory routinely measures by gamma-ray spectrometry? *

2.4. Describe the form in which your laboratory typically receives the food/feed samples for gamma-ray spectrometric analysis (e.g. powder, whole plants, grains, cooked meal). *

2.5. Does your laboratory perform sample preparation/homogenisation (e.g. grinding or mixing) of the food/feed samples that are routinely measured? *

- a) Yes
- b) No

2.5.1. If yes, please describe the procedure used. *

2.6. Describe the method of the moisture content determination used routinely for food/feed samples in your laboratory. *

2.7. Provide the range of masses of the food/feed samples routinely analysed in your laboratory using gamma-ray spectrometric measurements. *

3. **Proficiency test (PT) sample treatment**

3.1. Describe the method(s) of PT sample(s) preparation. *

3.2. Was the PT sample treated according to the same analytical procedure as routinely used in your laboratory for this type of samples (powdered food/feed)? *

- a) Yes
- b) No

3.2.1. If not, please specify the deviations from the routine procedure. *

3.3. Was drying applied to the PT sample? *

- a) Yes
- b) No

3.3.1. If yes, how long and at what temperature? *

3.4. Did you use any equipment for the sample preparation (e.g. mixer, vibrating table, tapper)? *

- ◯ a) Yes
- ◯ b) No

3.4.1. If yes, please provide more information (e.g. type, manufacturer, model). If it might be useful, send separate files (e.g. a drawing) by e-mail to Katarzyna.SOBIECH-MATURA@ec.europa.eu. *

3.5. Describe the container(s) used to contain the PT sample(s) for measurement(s) (shape, material, etc.). *

3.6. Provide the dimensions of the container(s) used for measurements (in mm). *

3.7. Was the container completely filled with the PT sample material? *

- ◯ a) Yes
- ◯ b) No

3.7.1. If not, provide the PT sample height (in mm). *

(number)

3.8. Specify mass of sample (in grams) for each PT sample measured (up to 3 samples). Please remember to report uncertainty with k=2. *

See table **Mass(es) of the PT sample(s)** at bottom

4. **Moisture content determination**

4.1. Did you follow closely the provided instruction for moisture content determination? *

- ○ a) Yes
- ○ b) No

4.1.1. If not, please describe the differences. *

4.2. What was/were the mass(es) of the sample used for determination of moisture content (in grams)? *

See table **Mass(es) of the sampe(s) for the moisture content determination** at bottom

4.3. What was the moisture content in % of the PT material? *

(number)

4.4. What was the standard uncertainty on the moisture content of the PT material? *

(number)

4.5. What was the applied correction factor for dry mass? *

5. **Measurements. If more than one detector or more than one sample was used to establish the reported massic activity value, please fill in the suitable fields in sections 5.2 (Measurement 2) and 5.3 (Measurement 3).**

5.1. Measurement 1

5.1.1. Which type of detector was used in Measurement 1 for the determination of I-131, Cs-134, and Cs-137 in maize powder? *

- ◯ a) Ge(Li) detector
- ◯ b) HPGe detector
- ◯ c) NaI(Tl) detector
- ◯ d) Other

5.1.1.1. If other, please specify. *

5.1.2. Provide the manufacturer, the type (e.g. BEGe, SAGe, LEGe, planar, coaxial, well), the nominal relative efficiency of the detector and the acquisition software used. *

See table **Details on the detector used for Measurement 1** at bottom

5.1.3. How was the Full Energy Peak (FEP) efficiency calibration of the detector system determined? *

- ◯ a) Monte Carlo
- ◯ b) Calibration source(s)
- ◯ c) LabSOCS
- ◯ d) Other

5.1.3.1. If other, please specify. *

5.1.3.2. If Monte Carlo code was used, with which software was it used? *

5.1.3.3. If calibration source(s) was/were used, please provide the description, reference ID, material, geometry, radionuclides etc. *

5.1.4. Describe the data acquisition system used. *

5.1.5. Specify the software used for peak analysis. *

- a) Genie 2000 family
- b) GammaVision
- c) InterWinner
- d) Gamma-W
- d) Gamma-track
- e) EMCA+
- e) SAMPO
- f) HYPERMET
- g) Other

5.1.5.1. If other, please specify. *

5.1.6. Specify the software used for the activity calculation. *

- a) Genie 2000 family
- b) GammaVision
- c) InterWinner
- d) Gamma-W
- e) EMCA+
- f) Gamma-track

- ◯ g) SAMPO
- ◯ h) HYPERMET
- ◯ i) MS Excel / spreadsheet
- ◯ j) ORIGIN
- ◯ k) Other

5.1.6.1. If other, please specify. *

5.1.7. How was the sample positioned on the detector? *

- ◯ a) Centred by eye
- ◯ b) Centred using rings or holders
- ◯ c) Other

5.1.7.1. If other, please specify. *

5.1.8. What was the distance between the sample and the endcap of the detector (in mm)? *

(number)

5.1.9. Provide the following data on Measurement 1 (sample number should correspond to the number in question 3.8 and remember to report uncertainty with the coverage factor k=2). *

See table **Measurement 1 details** at bottom

5.1.10. Provide further data on Measurement 1. *

See table **Measurement 1 details cont.** at bottom

5.1.11. If reported, please describe the correction factor used, its components and how the associated uncertainties were estimated.

5.1.12. If the detection limit(s) is/are reported, please provide detailed information on calculation method (e.g. ISO 11929:2000, ISO 11929:2010, etc.).

5.1.13. Comments on details provided in the questions 5.1.9 and 5.1.10.

5.1.14. Provide information on the background/blank measurement(s) for Measurement 1. *

See table **Background/blank measurments for Measurement 1 (max. 3 sets of data)** at bottom

5.1.15. Provide information on setup of the blank/background measurement(s) (e.g. type of blank sample, with holder, empty container). *

5.1.16. Provide the source of nuclear data used for the calculations. *

5.1.17. Which method was used for true coincidence summing corrections? *

- ○ a) No correction
- ○ b) Monte Carlo
- ○ c) Other

5.1.17.1. If other, please specify. *

5.1.18. Which method was used for geometry transfer corrections? *

- a) No correction
- b) Monte Carlo
- c) Other

5.1.18.1. If other, please specify. *

5.1.19. Was the correction for decay during the measurement applied in the calculations?

- a) Yes
- b) No

5.1.19.1. If yes, please specify for which radionuclides. *

5.1.20. Fill in the following table (remember to report uncertainty with the coverage factor k=2).

See table **Massic activities of radionuclides from Measurement 1** at bottom

5.1.21. Describe the method of massic activity calculation. Provide information on the method of calculation, the gamma-lines used, etc.

5.1.21.1. For I-131 *

5.1.21.2. For Cs-134 *

5.1.21.3. For Cs-137 *

5.1.21.4. For K-40

5.1.22. Comments on Measurement 1.

5.2. Measurement 2

5.2.1. Which type of detector was used in Measurement 2 for the determination of I-131, Cs-134, and Cs-137 in maize powder?

- a) Ge(Li) detector
- b) HPGe detector
- c) NaI(Tl) detector
- d) Other

5.2.1.1. If other, please specify. *

5.2.2. Provide the manufacturer, the type (e.g. BEGe, SAGe, LEGe, planar, coaxial, well), the nominal relative efficiency of the detector and the acquisition software.

See table **Details on the detector used for Measurement 2** at bottom

5.2.3. How was the Full Energy Peak (FEP) efficiency calibration of the detector system determined?

- ◯ a) Monte Carlo
- ◯ b) Calibration source(s)
- ◯ c) LabSOCS
- ◯ d) Other

5.2.3.1. If Monte Carlo code was used, with which software was it used? *

5.2.3.2. If calibration source(s) was/were used, please provide the description, reference ID, material, geometry, radionuclides etc. *

5.2.3.3. If other, please specify. *

5.2.4. Describe the data acquisition system used.

5.2.5. Specify the software used for peak analysis.

- ◯ a) Genie 2000 family
- ◯ b) GammaVision
- ◯ c) InterWinner
- ◯ d) Gamma-W
- ◯ e) EMCA+
- ◯ f) Gamma-track

- g) SAMPO
- h) HYPERMET
- i) Other

5.2.5.1. If other, please specify. *

5.2.6. Specify the software used for the activity calculation.

- a) Genie 2000 family
- b) GammaVision
- c) InterWinner
- d) Gamma-W
- e) EMCA+
- f) Gamma-track
- g) SAMPO
- h) HYPERMET
- i) MS Excel / spreadsheet
- j) ORIGIN
- k) Other

5.2.6.1. If other, please specify. *

5.2.7. How was the sample positioned on the detector?

- a) Centred by eye
- b) Centred using rings or holders
- b) Other

5.2.7.1. If other, please specify. *

5.2.8. What was the distance between the sample and the endcap of the detector (in mm)?

(number)

5.2.9. Provide the following data on Measurement 2 (sample number should correspond to the number in question 3.8 and remember to report uncertainty with the coverage factor k=2).

See table **Measurement 2 details** at bottom

5.2.10. Provide further data on Measurement 2.

See table **Measurement 2 details cont.** at bottom

5.2.11. If reported, please describe the correction factor used and how the associated uncertainties were estimated.

5.2.12. If the detection limit(s) is/are reported, please provide detailed information on calculation method (e.g. ISO11929-2000, ISO11929-2010, etc.).

5.2.13. Comments on details provided in the questions 5.2.9 and 5.2.10.

5.2.14. Provide information on the background/blank measurement(s) for Measurement 2.

See table **Background/blank measurments for Measurement 2 (max. 3 sets of data)** at bottom

5.2.15. Provide information on setup of the blank/background measurement(s) (e.g. type of blank sample, with holder, empty container).

5.2.16. Provide the source of nuclear data used for the calculations.

5.2.17. Which method was used for true coincidence summing corrections?

- a) No correction
- b) Monte Carlo
- c) Other

5.2.17.1. If other, please specify. *

5.2.18. Which method was used for geometry transfer corrections?

- a) No correction
- b) Monte Carlo
- c) Other

5.2.18.1. If other, please specify. *

5.2.19. Was the correction for decay during the measurement applied in the calculations?

- a) Yes
- b) No

5.2.19.1. If yes, please specify for which radionuclides. *

5.2.20. Fill in the following table (remember to report uncertainty with the coverage factor k=2).

See table **Massic activities of radionuclides from Measurement 2** at bottom

5.2.21. Describe the method of massic activity calculation. Provide information on the method of calculation, the gamma-lines used, etc.

5.2.21.1. For I-131

5.2.21.2. For Cs-134

5.2.21.3. For Cs-137

5.2.21.4. For K-40

5.2.22. Comments on Measurement 2.

5.3. Measurement 3

5.3.1. Which type of detector was used in Measurement 3 for the determination of I-131, Cs-134, and Cs-137 in maize powder?

- ◯ a) Ge(Li) detector
- ◯ b) HPGe detector
- ◯ c) NaI(Tl) detector
- ◯ d) Other

5.3.1.1. If other, please specify. *

5.3.2. Provide the manufacturer, the type (e.g. BEGe, SAGe, LEGe, planar, coaxial, well), and the nominal relative efficiency of the detector.

See table **Details on the detector used for Measurement 3** at bottom

5.3.3. How was the Full Energy Peak (FEP) efficiency calibration of the detector system determined?

- ◯ a) Monte Carlo
- ◯ b) Calibration source(s)
- ◯ c) LabSOCS
- ◯ d) Other

5.3.3.1. If Monte Carlo code was used with which software it was used? *

5.3.3.2. If calibration source(s) was/were used please provide the description, reference ID, material, geometry, radionuclides etc. *

5.3.3.3. If other, please specify. *

5.3.4. Describe the data acquisition system used.

5.3.5. Specify the software used for peak analysis.

- a) Genie 2000 family
- b) GammaVision
- c) InterWinner
- d) Gamma-W
- e) EMCA+
- f) Gamma-track
- g) SAMPO
- h) HYPERMET
- i) Other

5.3.5.1. If other, please specify. *

5.3.6. Specify the software used for the activity calculation.

- a) Genie 2000 family
- b) GammaVision
- c) InterWinner
- d) Gamma-W
- e) EMCA+
- f) Gamma-track
- g) SAMPO

- ◯ h) HYPERMET
- ◯ i) MS Excel / spreadsheet
- ◯ j) ORIGIN
- ◯ k) Other

5.3.6.1. If other, please specify. *

5.3.7. How was the sample positioned on the detector?

- ◯ a) Centred by eye
- ◯ b) Centred using rings or holders
- ◯ c) Other

5.3.7.1. If other, please specify. *

5.3.8. What was the distance between the sample and the endcap of the detector (in mm)?

(number)

5.3.9. Provide the following data on Measurement 3 (sample number should correspond to the number in question 3.8 and remember to report uncertainty with the coverage factor k=2).

See table **Measurement 3 details** at bottom

5.3.10. Provide further data on Measurement 3.

See table **Measurement 3 details cont.** at bottom

5.3.11. If reported, please describe the correction factor used and how the associated uncertainties were estimated.

5.3.12. If the detection limit(s) is/are reported; please provide detailed information on calculation method (e.g. ISO11929-2000, ISO11929-2010, etc.).

5.3.13. Comments on details provided in the questions 5.3.9 and 5.3.10.

5.3.14. Provide information on the background/blank measurement(s) for Measurement 3.

See table **Background/blank measurments for Measurement 3 (max. 3 sets of data)** at bottom

5.3.15. Provide information on setup of the blank/background measurement(s) (e.g. type of blank sample, with holder, empty container).

5.3.16. Provide the source of nuclear data used for analysis.

5.3.17. Which method was used for true coincidence summing corrections?

- ◯ a) No correction
- ◯ b) Monte Carlo
- ◯ c) Other

5.3.17.1. If other, please specify. *

5.3.18. Which method was used for geometry transfer corrections?

- a) No correction
- b) Monte Carlo
- c) Other

5.3.18.1. If other, please specify. *

5.3.19. Was the correction for decay during the measurement applied in the calculations?

- a) Yes
- b) No

5.3.19.1. If yes, please specify for which radionuclides. *

5.3.20. Fill in the following table (remember to report uncertainty with the coverage factor k=2).

See table **Massic activities of radionuclides from Measurement 3** at bottom

5.3.21. Describe the method of massic activity calculation. Provide information on the method of calculation, the gamma-lines used, etc.

5.3.21.1. For I-131

5.3.21.2. For Cs-134

5.3.21.3. For Cs-137

5.3.21.4. For K-40

5.3.22. Comments on Measurement 3.

6. **Reported massic activity values**

6.1. Emergency reporting

6.1.1. What was the mass of the sample (in grams) measured in the emergency mode?

(number)

6.1.2. What was the live time (in seconds) of the emergency measurement?

(number)

6.1.3. Comments on the emergency reporting.

6.2. Routine reporting

6.2.1. Was the calculation of the reported value of I-131 based on one measurement result? *

- a) Yes
- b) No

6.2.1.1. If not, then what was the method of calculation (e.g. mean, weighted mean)? *

6.2.2. Was the calculation of the reported value of Cs-134 based on one measurement result? *

- a) Yes
- b) No

6.2.2.1. If not, then what was the method of calculation (e.g. mean, weighted mean)? *

6.2.3. Was the calculation of the reported value of Cs-137 based on one measurement result? *

- a) Yes
- b) No

6.2.3.1. If not, then what was the method of calculation (e.g. mean, weighted mean)? *

6.2.4. Was the calculation of the reported value of K-40 based on one measurement result?

- a) Yes
- b) No

6.2.4.1. If not, then what was the method of calculation (e.g. mean, weighted mean)? *

6.3. Comments on the reported values.

7. **Additional information**

7.1. Difficulties encountered.

7.2. Further comments on this proficiency testing exercise.

7.3. Further comments on this questionnaire.

Background/blank measurments for Measurement 1 (max. 3 sets of data)

Questions/Response table	*Live time (s)*	*Dead time (%)*
Background/blank 1		
Background/blank 2		
Background/blank 3		

Background/blank measurments for Measurement 2 (max. 3 sets of data)

Questions/Response table	*Live time (s)*	*Dead time (%)*
Background/blank 1		
Background/blank 2		
Background/blank 3		

Background/blank measurments for Measurement 3 (max. 3 sets of data)

Questions/Response table	*Live time (s)*	*Dead time (%)*
Background/blank 1		

Questions/Response table	*Live time (s)*	*Dead time (%)*
Background/blank 2		
Background/blank 3		

Details on the detector used for Measurement 1

Questions/Response table	*Manufacturer*	*Type*	*Relative efficiency*	*Acquisition software*
Detector				

Details on the detector used for Measurement 2

Questions/Response table	*Manufacturer*	*Type*	*Relative efficiency*	*Acquisition software*
Detector				

Details on the detector used for Measurement 3

Questions/Response table	*Manufacturer*	*Type*	*Relative efficiency*	*Acquisition software*
Detector				

Mass(es) of the PT sample(s)

Questions/Response table	Mass (g)	Uncertainty Mass (g)
Sample 1		
Sample 2		
Sample 3		

Mass(es) of the sampe(s) for the moisture content determination

Questions/Response table	Mass (g)	Uncertainty Mass (g)
Sample 1		
Sample 2		
Sample 3		

Massic activities of radionuclides from Measurement 1

Questions/Response table	Massic activity (Bq/kg d.w.)	Uncertainty (Bq/kg d.w.)
I-131		
Cs-134		

Questions/Response table	Massic activity (Bq/kg d.w.)	Uncertainty (Bq/kg d.w.)
Cs-137		
K-40		

Massic activities of radionuclides from Measurement 2

Questions/Response table	Massic activity (Bq/kg d.w.)	Uncertainty (Bq/kg d.w.)
I-131		
Cs-134		
Cs-137		
K-40		

Massic activities of radionuclides from Measurement 3

Questions/Response table	Massic activity (Bq/kg d.w.)	Uncertainty (Bq/kg d.w.)
I-131		
Cs-134		
Cs-137		
K-40		

Measurement 1 details

Questions/Response table	Sample nr	Starting date (dd/mm/yyyy)	Live time (s)	Dead time (%)
Measurement 1				

Measurement 1 details cont.

Questions/Response table	Net counts	U net counts	Continuum (under peak) counts	Net counts (background)	U net counts (background)	FEP eff	U FEP eff	Correction factor	U correction factor
I-131 [80,2 keV]									
I-131 [284,3 keV]									
I-131 [364,5 keV]									
I-131 [637 keV]									
Cs-134 [604,7 keV]									
Cs-134 [795,9 keV]									
Cs-137 [661,7 keV]									
K-40 [1460,8 keV]									

Measurement 2 details

Questions/Response table	Sample nr	Starting date (dd/mm/yyyy)	Live time (s)	Dead time (%)
Measurement 2				

Measurement 2 details cont.

Questions/Response table	Net counts	U net counts	Continuum (under peak) counts	Net background counts	U net background counts	FEP eff	U FEP eff	Correction factor	U correction factor
I-131 [80,2 keV]									
I-131 [284,3 keV]									
I-131 [364,5 keV]									
I-131 [637 keV]									
Cs-134 [604,7 keV]									
Cs-134 [795,9 keV]									
Cs-137 [661,7 keV]									
K-40 [1460,8 keV]									

Measurement 3 details

Questions/Response table	Sample nr	Starting date (dd/mm/yyyy)	Live time (s)	Dead time (%)
Measurement 3				

Measurement 3 details cont.

Questions/Response table	Net counts	U net counts	Continuum (under peak) counts	Net background counts	U net background counts	FEP eff	U FEP eff	Correction factor	U correction factor
I-131 [80,2 keV]									
I-131 [284,3 keV]									
I-131 [364,5 keV]									
I-131 [637 keV]									
Cs-134 [604,7 keV]									
Cs-134 [795,9 keV]									
Cs-137 [661,7 keV]									
K-40 [1460,8 keV]									

Annex 9. Results of the laboratories (routine reporting). For each laboratory reported massic activity and expanded uncertainty (U) is provided, as well as results of z and ζ scores calculations

Lab code	Massic activity of ^{131}I (Bq/kg d.m.)	U	z score	ζ score	Massic activity of ^{134}Cs (Bq/kg d.m.)	U	z score	ζ score	Massic activity of ^{137}Cs (Bq/kg d.m.)	U	z score	ζ score	Massic activity of ^{40}K (Bq/kg d.m.)	U
1	231	5	1.05	8.48	881	4	-0.11	-1.71	528	4	-0.17	-2.61	110	4
2	243	266	1.36	0.39	954	352	0.29	0.30	710	104	1.49	3.11	-	-
3	0	0	-5.00	-47.75	784	78	-0.65	-2.88	464	46	-0.76	-3.45	-	-
4	152	13	-1.02	-5.11	846	42	-0.31	-2.30	510	43	-0.34	-1.64	-	-
5	239.63	9.85	1.29	7.66	966.07	7.93	0.36	5.35	688.08	10.78	1.29	15.97	2863.52	81.62
6	186.2	22	-0.13	-0.41	806.4	53.4	-0.52	-3.25	535.9	33.8	-0.10	-0.61	96.2	15.8
7	192	13	0.03	0.13	815	26	-0.48	-4.95	525	38	-0.20	-1.09	339	42
8	218.6	22.3	0.72	2.33	1097.0	109.5	1.09	3.50	735.5	71.2	1.72	5.20	131.0	20.5
9	7.8	1.7	-4.80	-44.80	31.0	2.5	-4.83	-75.21	18.0	2.1	-4.84	-74.74	57.3	7.3
10	192.98	30.24	0.05	0.13	905.42	78.11	0.02	0.11	549.45	57.15	0.02	0.08	101.12	22.88
11	180	17	-0.29	-1.17	857	72	-0.24	-1.16	523	40	-0.22	-1.13	100	22
12	186	15	-0.13	-0.59	890	88	-0.06	-0.24	532	53	-0.14	-0.55	-	-
13	192	23	0.03	0.08	882	35	-0.11	-0.91	533	22	-0.13	-1.07	99	12
14	191.83	13.83	0.02	0.10	867.17	25	-0.19	-1.99	545.16	15.28	-0.02	-0.18	118.86	15.06
15	164	27	-0.71	-1.92	920	50	0.11	0.69	557	28	0.09	0.64	102	6
16	201	22	0.26	0.85	698	55	-1.13	-6.81	482	35	-0.59	-3.45	167	12
17	102	6	-2.33	-17.80	432	9	-2.60	-37.98	292	7	-2.33	-32.58	-	-
18	190.4	13.2	-0.02	-0.08	862.7	38.1	-0.21	-1.72	536.0	35.1	-0.10	-0.58	79.34	20.9
19	197.2	12.7	0.16	0.83	927.8	39.8	0.15	1.17	557.3	26.9	0.09	0.68	101.6	8.0
20	186	28	-0.13	-0.34	845	175	-0.31	-0.63	531	75	-0.15	-0.42	109	18
21	181	26	-0.26	-0.74	897	100	-0.02	-0.08	539	68	-0.07	-0.23	133	60
22	329.45	76.54	3.62	3.60	61.47	25.30	-4.66	-49.11	26.03	14.64	-4.76	-51.44	123.28	30.84
23	182.0	18.2	-0.24	-0.91	766.0	76.6	-0.75	-3.38	530.0	53	-0.16	-0.62	196.0	19.6
24	73.27	12.86	-3.08	-15.55	545.93	32.91	-1.97	-17.69	498.82	30.17	-0.44	-2.90	123.10	12.7
25	206	25	0.39	1.14	931	101	0.17	0.58	571	53	0.22	0.88	105.9	8.8
26	1454.60	-	33.08	-	12161.5	901.325	62.49	24.98	8641.50	384.18	73.99	42.11	12588.30	1660.6

27	187	24	-0.10	-0.32	880	112	-0.12	-0.37	528	64	-0.17	-0.58	-	-
28	151	18	-1.05	-4.06	748	76	-0.85	-3.85	499	50	-0.44	-1.85	60	10
29	178	14	-0.34	-1.61	797	78	-0.58	-2.56	534	49	-0.12	-0.51	98	11
30	196.6	68.2	0.15	0.16	952.4	85.9	0.29	1.16	591.9	61.9	0.41	1.41	112.3	24.2
31	202	41.1	0.29	0.53	901	182	0.00	0.00	547	110	0.00	0.00	100	21.7
32	170	40	-0.55	-1.03	800	50	-0.56	-3.67	470	30	-0.70	-4.65	74	10
33	184.41	28.53	-0.17	-0.44	826.14	24.27	-0.42	-4.48	580.03	38.23	0.30	1.62	214.83	66.98
34	174.0	9.2	-0.45	-2.79	890	26	-0.06	-0.63	512	16	-0.32	-3.29	121	17
35	190	20	-0.03	-0.09	957	90	0.31	1.21	563	50	0.15	0.62	-	-
36	197	28	0.16	0.41	870	122	-0.17	-0.50	522	73	-0.23	-0.67	104	15
37	545.3	68.1	9.27	10.33	195.7	24.8	-3.91	-41.70	865.6	99.8	2.91	6.32	-	-
38	198.9	43.0	0.21	0.36	895.2	75	-0.03	-0.15	546.5	48.6	0.00	-0.02	102.5	15.4
39	174.56	14.0	-0.43	-2.04	807.80	47.98	-0.52	-3.50	481.73	27.62	-0.60	-4.22	99.44	16.98
40	140.59	5.57	-1.32	-10.34	694.41	8.43	-1.15	-16.87	473.91	7.78	-0.67	-9.13	101.05	16.27
41	204	24	0.34	1.03	813	25	-0.49	-5.18	528	18	-0.17	-1.67	100	17
42	197.70	21.7	0.18	0.58	922.23	135.2	0.12	0.31	550.24	71.1	0.03	0.09	116.71	16.4
43	195	56	0.10	0.14	882	300	-0.11	-0.13	560	140	0.12	0.18	71	53
44	520	151	8.61	4.35	936	44	0.19	1.41	534	28	-0.12	-0.83	-	-
45	228	54	0.97	1.36	1179	101	1.54	5.37	622	81	0.69	1.82	57	8
46	193	56.35	0.05	0.09	812	59.5	-0.49	-3.45	509	25.5	-0.35	-2.24	-	-
47	190	22	-0.03	-0.09	890	80	-0.06	-0.26	540	70	-0.06	-0.20	107	21
48	196	20	0.13	0.46	880	90	-0.12	-0.45	535	55	-0.11	-0.42	94	14
49	1882.57	152.09	44.28	22.21	7779.27	576.84	38.17	23.83	5781.48	158.57	47.85	65.77	5852.79	126.36
50	182	23	-0.23	-0.74	877	102	-0.14	-0.47	526	62	-0.19	-0.65	112	40
51	167	19	-0.63	-2.33	761	76	-0.78	-3.53	533	55	-0.13	-0.49	117	14
52	123	17	-1.78	-7.24	574	46	-1.81	-12.72	385	44	-1.48	-7.02	70.8	13.1
53	202.19	7.71	0.29	2.01	770.28	30.97	-0.73	-6.78	561.66	22.24	0.13	1.12	167.51	41.03
54	213	21	0.58	1.96	835	67	-0.37	-1.86	546	45	-0.01	-0.04	152	82
55	163.0	9	-0.73	-4.65	704.0	35	-1.09	-9.41	482.0	25	-0.59	-4.54	-	-
56	177.3830	32.9894	-0.36	-0.80	732.0204	52.7555	-0.94	-5.87	512.3045	62.5416	-0.32	-1.08	101.1189	12.2762
57	194	17	0.08	0.32	879	44	-0.12	-0.89	541	22	-0.05	-0.46	110	8.0
58	223.7	61.0	0.86	1.06	1012	274	0.62	0.81	620	169	0.67	0.86	118.5	37.1
59	158.61	4.07	-0.85	-7.22	609.84	15.13	-1.62	-21.15	427.96	10.47	-1.09	-13.62	68.43	14.07
60	192	19	0.03	0.10	897	90	-0.02	-0.09	535	54	-0.11	-0.42	95	13

61	219.2	11.70	0.74	3.98	871.7	41.59	-0.16	-1.23	604.1	27.98	0.52	3.65	105.4	13.85
62	215	29	0.63	1.60	897	26	-0.02	-0.23	613	26	0.60	4.47	102	31
63	169	13	-0.58	-2.88	757	48	-0.80	-5.41	474	29	-0.67	-4.53	90	14
64	157	24	-0.89	-2.69	776	89	-0.69	-2.72	445	51	-0.93	-3.86	-	-
65	197	20	0.16	0.56	875	37	-0.14	-1.19	539	58	-0.07	-0.27	97.8	10.2
66	264.88	30.15	1.93	4.74	946.47	56.9	0.25	1.48	558.16	96.92	0.10	0.23	619.72	221.43
67	212.16	11.19	0.55	3.08	916.31	51.22	0.08	0.55	557.75	31.62	0.10	0.62	124.73	19.9
68	183	24	-0.21	-0.63	888	108	-0.07	-0.24	547	64	0.00	0.00	103	26
69	180.3	21.5	-0.28	-0.93	810.3	89.7	-0.50	-1.96	493.3	55.2	-0.49	-1.89	79.0	17.1
70	202.37	19.63	0.30	1.07	929.74	45.50	0.16	1.13	562.3	31.38	0.14	0.89	103.92	21.50
71	677.2	45.54	12.73	21.03	252.3	28.01	-3.60	-35.80	1023	105.2	4.35	8.97	191.6	60.2
72	235	22	1.15	3.76	961	103	0.33	1.14	692	71	1.33	4.01	-	-
73	196	32	0.13	0.30	852	64	-0.27	-1.44	527	58	-0.18	-0.67	120	32
74	166.88	39.4	-0.63	-1.20	760.31	68.34	-0.78	-3.90	518.54	125.28	-0.26	-0.45	91.31	35.32
75	204	22	0.34	1.11	873	69	-0.16	-0.77	536	42	-0.10	-0.50	127	27
76	190.35	6.9	-0.02	-0.12	872.47	31.14	-0.16	-1.47	543.95	26.8	-0.03	-0.20	103.12	17.28
77	244.35	31.22	1.40	3.31	941.09	59.25	0.22	1.26	608.76	37.54	0.56	3.08	185.54	64.94
78	192	22	0.03	0.09	882	95	-0.11	-0.39	534	58	-0.12	-0.44	100	21
79	220.32	19.09	0.77	2.83	854.62	51.44	-0.26	-1.65	585.97	39.32	0.36	1.87	145.06	39.33
80	206	4	0.39	3.35	908	9	0.04	0.57	612	9	0.59	7.81	120	6
81	195.2	15	0.11	0.49	892	24	-0.05	-0.54	550.0	18.4	0.03	0.26	100	24
82	203	7	0.31	2.26	912	14	0.06	0.82	549	11	0.02	0.22	101	27
83	192	14	0.03	0.12	823	54	-0.43	-2.66	554	36	0.06	0.36	140	28
84	183	61	-0.21	-0.26	857	284	-0.24	-0.31	503	72	-0.40	-1.20	97	22
85	163.07	17.2	-0.73	-2.94	787.18	31.51	-0.63	-5.84	510.22	60.15	-0.34	-1.19	97.49	14.53
86	193.167	35.156	0.06	0.12	877.12	119.788	-0.13	-0.39	531.176	71.76	-0.14	-0.43	93.3	39.23
87	199	18	0.21	0.81	899	76	-0.01	-0.05	543	52	-0.04	-0.15	103	17
88	200	30	0.24	0.58	900	110	-0.01	-0.02	530	60	-0.16	-0.55	80	30
89	195	31	0.10	0.25	909	88	0.04	0.18	560	35	0.12	0.69	60	16
90	193	22	0.05	0.17	880	94	-0.12	-0.43	524	62	-0.21	-0.72	92	20
91	188	26	-0.08	-0.22	929	119	0.16	0.46	529	68	-0.16	-0.52	135	31
92	193	12	0.05	0.28	902	54	0.01	0.03	550	34	0.03	0.16	110	12
93	189.16	48.40	-0.05	-0.08	816.69	30.37	-0.47	-4.43	562.23	19.23	0.14	1.28	116.96	21.28
94	186	35	-0.13	-0.28	810	130	-0.50	-1.38	515	61	-0.29	-1.02	91	19

95	167.15	21.84	-0.62	-2.05	716.22	50.85	-1.03	-6.62	478.32	33.75	-0.63	-3.76	102.64	20.65
96	210.9	13.6	0.52	2.52	986.1	38.1	0.47	3.82	602.1	30.1	0.51	3.32	104	25
97	206.76	37.25	0.41	0.83	835.28	23.83	-0.37	-3.99	578.51	33.26	0.29	1.77	112.31	28.65
98	165	33	-0.68	-1.53	781	109	-0.67	-2.15	472	66	-0.68	-2.21	91	13
99	207	9	0.42	2.66	998	34	0.54	4.73	564	13	0.16	1.78	104	19
100	212.08	30.6	0.55	1.33	813.18	37.9	-0.49	-3.96	571.63	33.5	0.23	1.36	139.5	58.9
101	178.10	39.54	-0.34	-0.64	850.09	53.48	-0.28	-1.75	518.56	33	-0.26	-1.59	109.81	23.65
102	190.2	7.8	-0.02	-0.14	838.4	11.6	-0.35	-4.86	518.0	10.2	-0.27	-3.35	99.6	7.8
103	232.8	10.8	1.09	6.22	957.2	6.6	0.31	4.70	653.8	8.2	0.98	13.17	127.8	13.4
104	184	18	-0.18	-0.71	863	32	-0.21	-1.93	550	20	0.03	0.25	99	20
105	200	20	0.24	0.84	884	90	-0.09	-0.37	537	55	-0.09	-0.35	98	15
106	201	16	0.26	1.12	879	38	-0.12	-0.99	576	24	0.27	2.09	117	40
107	188	15	-0.08	-0.35	888	78	-0.07	-0.33	541	42	-0.05	-0.27	104	12
108	194	12	0.08	0.42	888	53	-0.07	-0.45	538	32	-0.08	-0.52	-	-
109	143	14	-1.26	-5.95	611	42	-1.61	-12.11	409	28	-1.26	-8.82	78	16
110	195	30	0.10	0.26	876	120	-0.14	-0.41	524	72	-0.21	-0.63	169	58
111	175.5	30.5	-0.41	-0.98	767.4	90.7	-0.74	-2.86	500.0	62.2	-0.43	-1.47	208.5	47
112	196	24	0.13	0.40	793	50	-0.60	-3.92	552	38	0.05	0.25	144	30
113	210.45	23.65	0.51	1.56	821.78	51.61	-0.44	-2.80	574.97	33.40	0.26	1.54	-	-
114	197	22	0.16	0.51	881	104	-0.11	-0.38	547	59	0.00	0.00	93	16
115	150	15	-1.07	-4.82	940	94	0.22	0.81	550	60	0.03	0.10	102	11
116	210.2	42.9	0.50	0.88	874.6	90.4	-0.15	-0.57	575.4	61.6	0.26	0.90	102.0	34.5
117	197	12.2	0.16	0.82	808	2.46	-0.52	-8.04	570	2.62	0.21	3.23	131	16
118	210	50	0.50	0.75	970	100	0.38	1.34	590	60	0.39	1.40	90	30
119	195	24	0.10	0.32	786	16	-0.64	-8.21	524	11	-0.21	-2.58	114	23
120	163.0	6.4	-0.73	-5.47	715.4	25.8	-1.03	-10.74	431.0	45.4	-1.06	-4.88	111.8	12.6

Annex 10. Results of the homogeneity study

Massic activity of ^{131}I (Bq/kg)			
Bottle number	Replicate 1	Replicate 2	Replicate 3
8	194.4	206.9	201.0
30	199.7	201.6	206.1
41	196.4	203.8	205.0
64	202.8	200.8	202.0
77	200.3	205.0	197.2
90	207.6	207.5	204.2
111	201.7	195.2	206.2
114	204.7	203.2	203.9
141	204.0	206.4	200.5
157	206.3	206.7	203.6
Mean	202.8		
StDev	3.6		
Relative StDev	1.8%		

Source: JRC Geel

One way ANOVA calculation for ^{131}I

Source of Variation	SS	d.f.	MS	StDev	F	F-crit 95%	F-crit 99%
Between Units	106.97	9	11.89	MSB<MSW	0.88	2.39	3.46
Within Units	270.27	20	13.51	3.68			
Total	377.24	29					
Homogeneity Results	Mean	Sbb	Sbb (%)	Swb	Swb (%)	Ubb*	Ubb* (%)
	202.81	MSB<MSW	MSB<MSW	3.68	1.80%	1.193	0.60%

Source: JRC Geel

Differences between units statistically significant? (a=95%): No

Differences between units statistically significant? (a=99%): No

Massic activity of ^{134}Cs (Bq/kg)			
Bottle number	Replicate 1	Replicate 2	Replicate 3
8	933.4	932.4	937.8
30	937.5	945.8	942.8
41	944.7	941.6	925.2
64	934.0	939.7	927.0
77	947.2	937.9	926.2
90	943.3	939.9	950.3
111	930.4	921.8	942.0
114	930.9	931.0	948.0
141	937.4	937.4	938.3
157	944.8	937.9	942.7
Mean	937.6		
StDev	7.2		
Relative StDev	0.8%		

Source: JRC Geel

One way ANOVA calculation for ^{134}Cs

Source of Variation	SS	d.f.	MS	StDev	F	F-crit 95%	F-crit 99%
Between Units	452.265	9	50.252	MSB<MSW	0.953	2.393	3.457
Within Units	1,054.14	20	52.707	7.26			
Total	1,506.40	29					
Homogeneity Results	Weight.Avg.	Sbb	Sbb (%)	Swb	Swb (%)	Ubb*	Ubb* (%)
	937.638	MSB<MSW	MSB<MSW	7.26	0.80%	2.357	0.30%

Source: JRC Geel

Differences between units statistically significant? (a=95%): No

Differences between units statistically significant? (a=99%): No

Massic activity of ^{137}Cs (Bq/kg)			
Bottle number	Replicate 1	Replicate 2	Replicate 3
8	573.4	574.5	570.2
30	574.7	576.2	569.3
41	577.6	573.7	576.5
64	567.4	575.3	567.2
77	577.4	571.6	558.7
90	579.6	571.4	577.4
111	566.6	558.9	580.5
114	578.1	568.1	570.6
141	572.7	574.3	564.2
157	572.7	576.3	573.1
Mean	572.3		
StDev	5.4		
Relative StDev	1.0%		

Source: JRC Geel

One way ANOVA calculation for ^{137}Cs

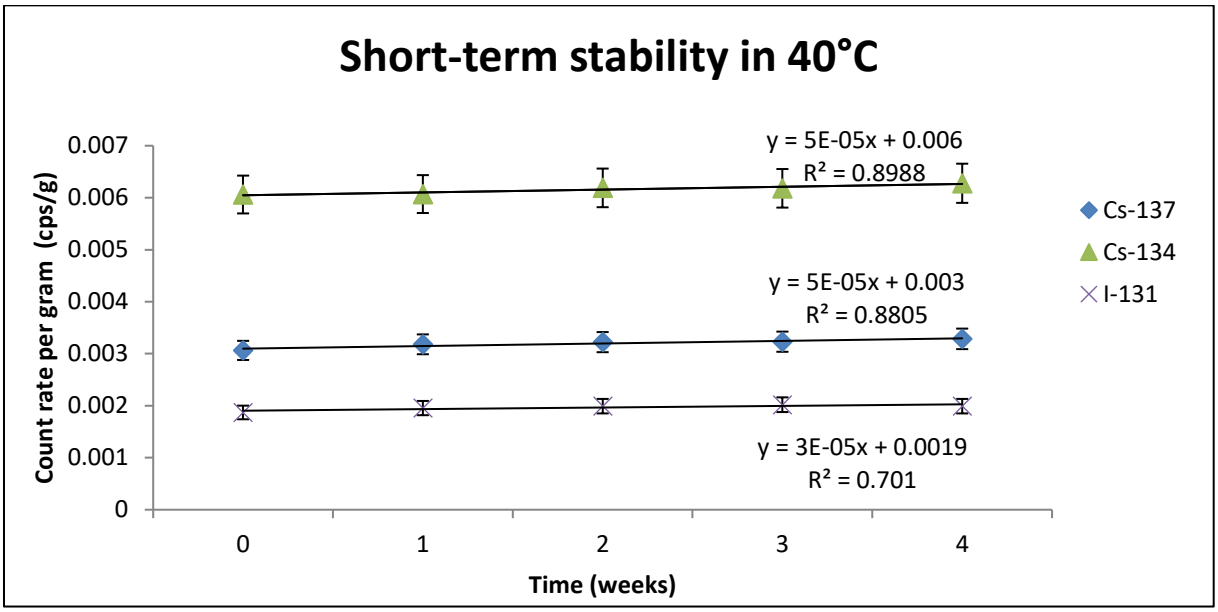
Source of Variation	SS	d.f.	MS	StDev	F	F-crit 95%	F-crit 99%
Between Units	191.516	9	21.28	MSB<MSW	0.638	2.393	3.457
Within Units	667.273	20	33.364	5.776			
Total	858.789	29					
Homogeneity Results	Weight.Avg.	Sbb	Sbb (%)	Swb	Swb (%)	Ubb*	Ubb* (%)
	572.263	MSB<MSW	MSB<MSW	5.776	1.00%	1.875	0.30%

Source: JRC Geel

Differences between units statistically significant? (a=95%): No

Differences between units statistically significant? (a=99%): No

Annex 11. Results of the short-term stability study

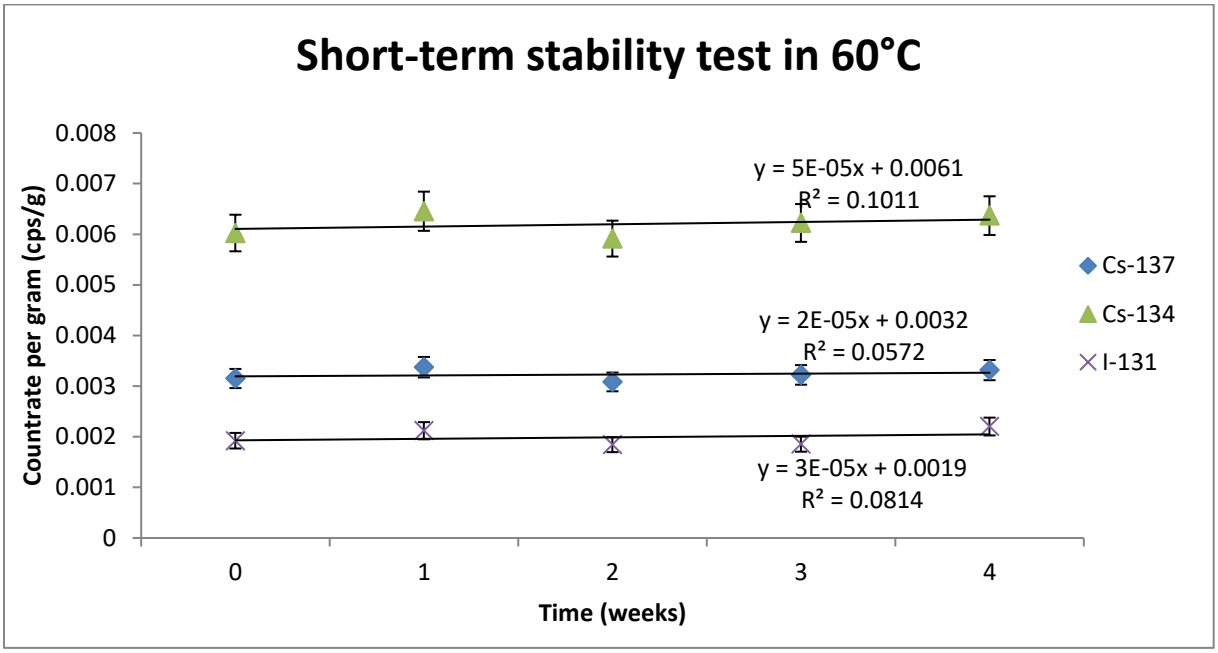


Source: JRC Geel

Table A1. Results of measurements for the short-term stability study in 40°C

	Countrate per mass (cps/g)		
Time (weeks)	Cs-137	Cs-134	I-131
0	0.003150299	0.006023900	0.001922051
1	0.003373622	0.006454323	0.002119166
2	0.003082863	0.005913943	0.001843770
3	0.003221634	0.006223087	0.001853515
4	0.003315840	0.006367836	0.002201835
Mean	0.003228852	0.006196618	0.001988068
Slope	0.000017909	1.905894495	0.577283629
Error on the slope	0.000041974	0.122608076	0.246108236
t	0.426680748	0.58100451	0.515485962
t_{crit}	5.840909310	5.84090931	5.84090931
u_{stab}	4.19735E-05	7.8595E-05	5.70172E-05
relative u_{stab} (%)	0.012999516	0.01268346	0.028679723

Source: JRC Geel



Source: JRC Geel

Table A2. Results of measurements for the short-term stability study in 60°C

	Countrate per mass (cps/g)		
Time (weeks)	Cs-137	Cs-134	I-131
0	0.00315	0.00602	0.00192
1	0.00337	0.00645	0.00211
2	0.00308	0.00591	0.00184
3	0.00322	0.00622	0.00185
4	0.00331	0.00636	0.00220
Mean	0.00322	0.00619	0.00198
Slope	0.00001790	1.90589449	0.57728362
Error on the slope	0.00004197	0.12260807	0.24610823
t	0.426680748	0.58100451	0.515485962
t_{crit}	5.840909310	5.84090931	5.84090931
u_{stab}	4.19735E-05	7.8595E-05	5.70172E-05
relative u_{stab} (%)	0.012999516	0.01268346	0.028679723

Source: JRC Geel

Annex 12. The PomPlot

The PomPlot, a graphical method, is used for producing a summary overview of the participants' results. It displays the relative deviations (*D/MAD*) of the individual results A_i from the reference value A_0 on the horizontal axis and relative uncertainties (*u/MAD*) on the vertical axis (*Fig. A1*). For both axes, the variables are expressed as multiples of *MAD*, which is defined as the median of the absolute deviation from the reference value

$$MAD = Median|D_i|, (i = 1, \dots, n) \quad (1)$$

where

D_i is the difference between the reported and the reference activity concentration:

$$D_i = \frac{A_i}{A_0} - 1 \quad (2)$$

where

A_i activity value reported by laboratory i

A_0 assigned reference value

The median absolute deviation *MAD* is used because of its robustness.

For every data point the uncertainty is calculated as an independent sum of the reported combined uncertainties on A_i and A_0

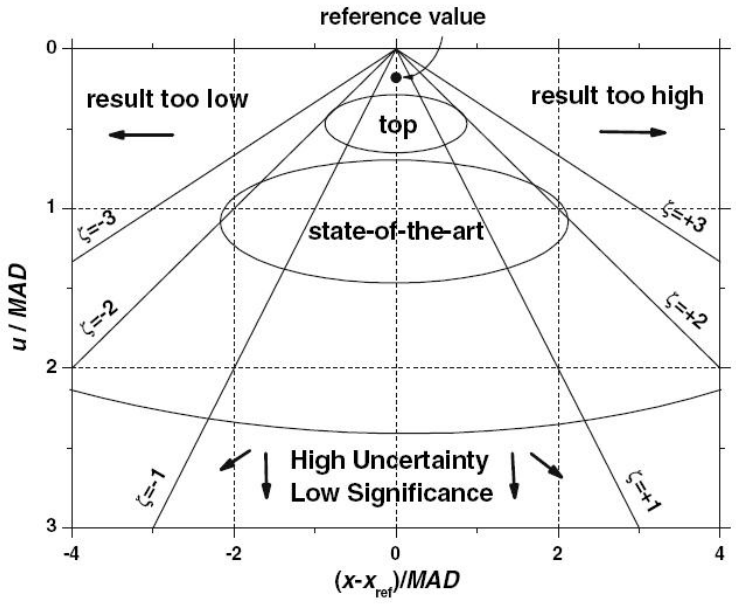
$$u_i^2 = u_c^2(A_i) + u_c^2(A_0) \quad (3)$$

where

$u(A_i)$ standard uncertainty of activity value reported by Laboratory i (k=1)

$u(A_0)$ standard uncertainty of assigned activity reference value for Laboratory i (k=1)

Figure A1. Interpretation of a PomPlot (Spasova et al., 2007)



Source: JRC Geel

The ζ-scores, where $|\zeta| = |D/u|$, with values 1, 2 and 3, are represented by diagonal solid lines, creating the aspect of a pyramidal structure. The ζ-score is a measure of the deviation between laboratory result and reference value relative to the total uncertainty (ISO, 2015). The points on the right-hand side of the graph correspond to results that are higher than the reference value whereas lower values are situated on the left. When the reported uncertainty is small, the corresponding point is situated high in the graph. The most accurate results should be situated close to the top of the pyramid. Points outside of the ζ=±3 lines are probably inconsistent with the reference value.

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Ref. Ares(2018)1156045 - 01/03/2018

Results of EC PT 2017 on I-131, Cs-134 and Cs-137 in maize powder

My lab code: 69

Reference values, all in Bq/kg dry mass

Measurand	Ref Date	Activity X_{ref}	U_{ref} (k=2)	defined σ_{PT} rel.	defined σ_{PT}	u_{ref} (k=1)>0.3*σ_{PT}
Cs-134	01/06/2017	***901***	***23***	20%	180.2	No
Cs-137	01/06/2017	***547***	***14***	20%	109.4	No
I-131	01/06/2017	***191***	***8***	20%	38.2	No
K-40	01/06/2017	***101***	***9***	20%	20.2	No

Global participants data, all in Bq/kg dry mass

Measurand	# reported values	Median activity M̃	MAD	Average activity X	std dev σ
Cs-134	***120***	***873.80***	***54.00***	***980.25***	***1220.07***
Cs-137	***120***	***538.5***	***23***	***648.62***	***883.77***
I-131	***120***	***193.08***	***12.50***	***222.96***	***204.39***
K-40	***105***	***104.00***	***10.90***	***317.34***	***1359.28***

My data in Bq/kg dry mass

Lab code	Measurand	Ref. Date	Value X	Uncertainty U_X	Rel Uncertainty U_X (%)	Coverage_factor
69	Cs-134	01/06/2017	810.3	89.7	11.07	2
69	Cs-137	01/06/2017	493.3	55.2	11.19	2
69	I-131	01/06/2017	180.3	21.5	11.92	2
69	K-40	01/06/2017	79.0	17.1	21.65	2

My results in Bq/kg dry mass

Lab code	Measurand	Deviation X-X_{ref}	% difference	*(zed) z-score*	(zeta) ζ-score	En-score
69	Cs-134	-90.7	-10.07	-0.50	-1.96	-0.98
69	Cs-137	-53.7	-9.82	-0.49	-1.89	-0.94
69	I-131	-10.7	-5.60	-0.28	-0.93	-0.47
69	K-40	-22	-21.78	-1.09	-2.28	**-1.14**

Warning signals: Yellow Action signals: **Red**

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Results of EC PT 2017 on I-131, Cs-134 and Cs-137 in maize powder

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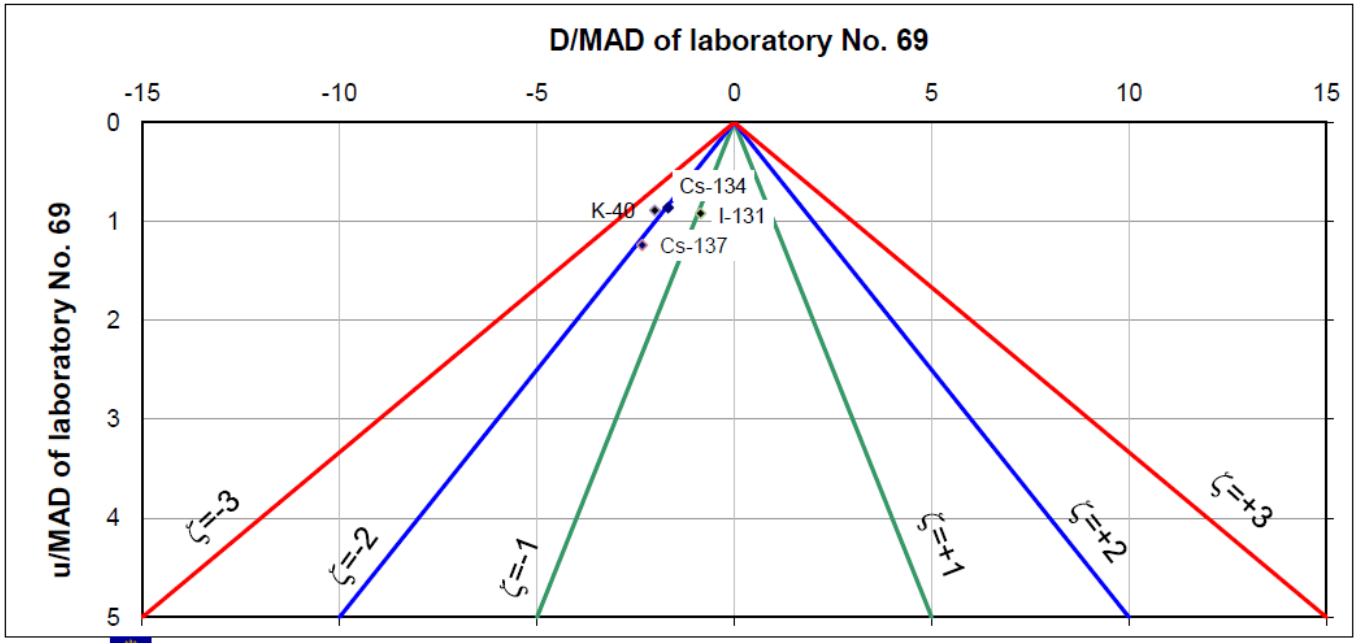
Deviation (D) = My value - Ref. value (X-X_{ref})
u_X and u_{ref} are standard uncertainties (k=1).
U_X and U_{ref} are expanded uncertainties (mostly k=2).
MAD = Median of Absolute Deviations from the reference value

Definitions (ISO 13528:2015)

$$z = \frac{X - X_{ref}}{\sigma_{PT}} \qquad \zeta = \frac{X - X_{ref}}{\sqrt{{u_X}^2 + {u_{ref}}^2}} \qquad En = \frac{X - X_{ref}}{\sqrt{{U_X}^2 + {U_{ref}}^2}}$$

[My place in the famous Pomplot]



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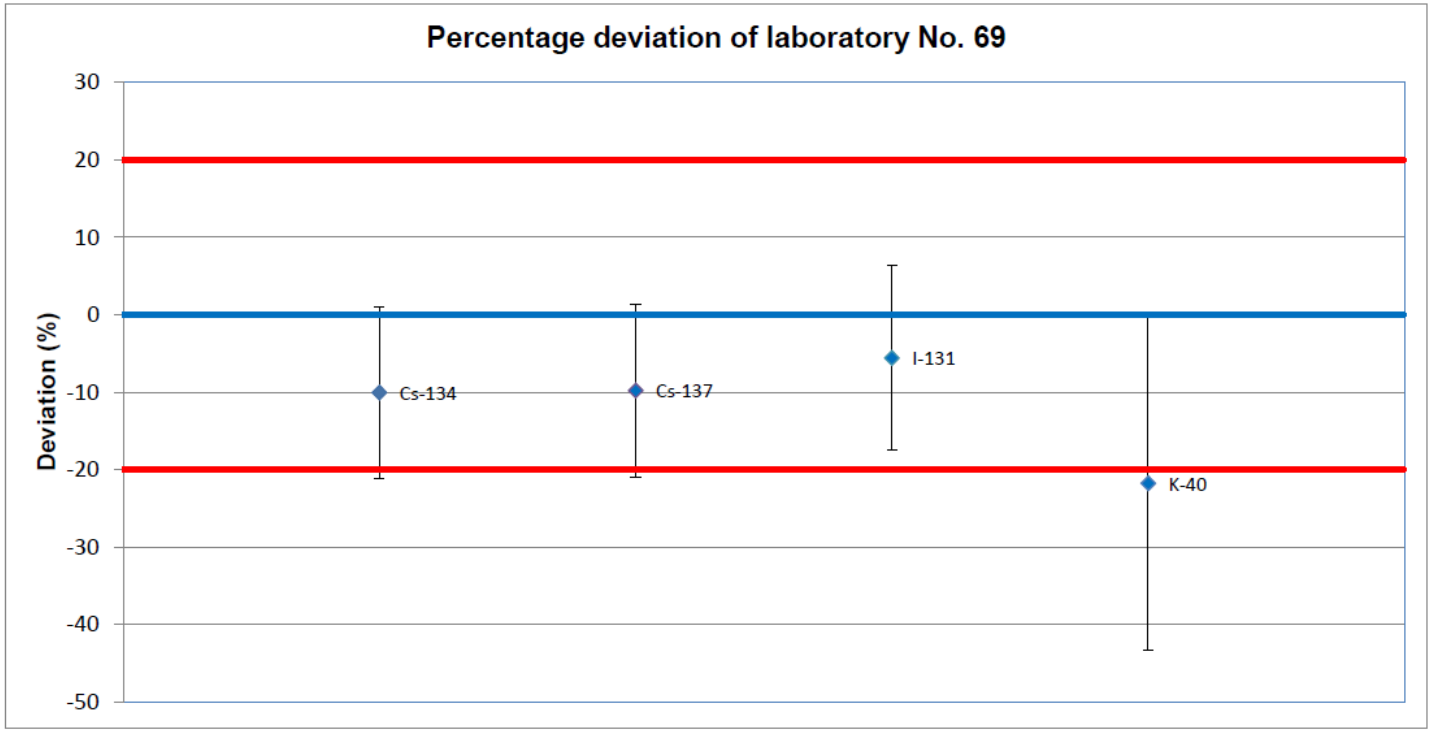
Results of EC PT 2017 on I-131, Cs-134 and Cs-137 in maize powder



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