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Evaluation of EC Comparison on the Determination of ^{40}K, ^{90}Sr and ^{137}Cs in Bilberry Powder

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Summary

An interlaboratory comparison (ILC) within the framework of the Euratom Treaty was conducted by the Institute for Reference Materials and Measurements (IRMM) in 2011. The performance of 88 laboratories in measurement of ^{40}K, ^{90}Sr and ^{137}Cs activity concentrations in dried wild bilberry powder was compared with reference values.

The candidate reference material IRMM-426 Wild Berries was used in this ILC as a testing material. The berries were collected in the region affected by the Chernobyl accident. Due to the natural uptake from elevated levels in the environment, the radionuclides were metabolised by the plants and, hence, the material has elevated concentrations of ^{137}Cs and ^{90}Sr. No spiking was applied. It was reprocessed, homogenized and bottled at IRMM. The reference values traceable to SI units were established in a CCRI(II) supplementary comparison in which nine laboratories from national metrology institutes (NMIs) and international organisations (IAEA and IRMM) participated. Homogeneity as well as short-term stability of the material were demonstrated at IRMM.

The methods of the sample preparation and measurement techniques used by the laboratories participating in the interlaboratory comparison are described. A robust evaluation of the performance of laboratories is performed using three different approaches: relative deviations, E_n numbers and ‘PomPlots’.

The activity concentrations of ^{137}Cs and ^{40}K were determined almost exclusively by gamma-ray spectrometry with 9 % and 17 % of results deviating more than 20 % from the reference values, respectively. These results are worse in comparison to previous ILCs. This may be due to the food matrix and inappropriate use of corrections for differences in density and/or geometry between berry sample and standard sources of activity. In the case of ^{90}Sr, about 77 % of results lie within 20 % from the reference value, much more favourable than observed in previous exercises. Contrary to this, based on the E_n criterion, only 56 % of reported values are satisfactory (compatible with E_n). With one exception, no particular separation or counting method provides results better than the other techniques used. Separation of strontium by extraction chromatography without specified details was performing significantly worse than all other methods used, including extraction chromatography on Sr resin with 100 % success rate.

This comparison demonstrates that several laboratories have difficulties to determine activity concentrations of ^{137}Cs and ^{40}K in food samples. The good laboratory performance in the ^{90}Sr determination (when using the relative deviation as criterion) may be attributed to the easier separation of strontium from the dried fruit matrix compared to milk powder or soil in earlier comparisons. The bad evaluation results with respect to the E_n criterion (44 % incompatible) demonstrate, however, the challenges of realistic uncertainty estimations in particular for ^{90}Sr determination.

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Glossary

AC	accession and pre-accession countries
A_{lab}	laboratory result of activity concentration
A_{ref}	reference value of activity concentration
BIPM	Bureau International des Poids et Mesures
CCRI(II)	Comité Consultatif des Rayonnements Ionisants, Section 2
$D_{\%}$	relative difference between the reported and the reference activity concentration
EC	European Commission
E_n	performance statistic E_n number
Euratom	European Atomic Energy Community
GM	Geiger-Müller counting
GUM	Guide to the Expression of Uncertainty in Measurement
HPGe	high-purity germanium detector
IRMM	Institute for Reference Materials and Measurements
IAEA	International Atomic Energy Agency
ICP-MS	inductively coupled plasma mass spectrometry
ILC	interlaboratory comparison
ISO	International Organization for Standardization
k	coverage factor according to GUM
LOD	limit of detection
LSC	liquid scintillation counter, liquid scintillation counting
MAD	median absolute deviation
MS	member states
NIM	nuclear instrument module
NMI	national metrology institute
SI	Système International d'Unités, International System of Units
SIR	Système International de Référence, International Reference System for radionuclides
u	standard uncertainty according to GUM
u_c	combined standard uncertainty according to GUM
U	expanded uncertainty according to GUM
U_{lab}	expanded uncertainty of average laboratory result
U_{ref}	expanded uncertainty of reference value

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 and to report the results to the European Commission (EC). In order to verify the performance of the monitoring laboratories and to ensure comparability of reported results, regular interlaboratory comparisons (ILCs) were introduced by the EC. Since 2003, the JRC Institute for Reference Materials and Measurements (IRMM) has had the responsibility for their organization.

The metrological approach of IRMM in conducting comparisons relies on its participation in key comparisons among national metrology institutes (Wätjen et al., 2008) as described in Fig. 1. This allows IRMM to work with intercomparison samples for which it determines reference values that are traceable to SI units and the International Reference System (SIR) for gamma-ray emitting radionuclides (Ratel, 2007). In terms of physical properties as well as radioactivity concentration levels, IRMM comparison samples are generally closer to the real samples measured in monitoring laboratories than calibration standards and, therefore, they give a realistic estimate of the performance of these laboratories in their monitoring tasks.

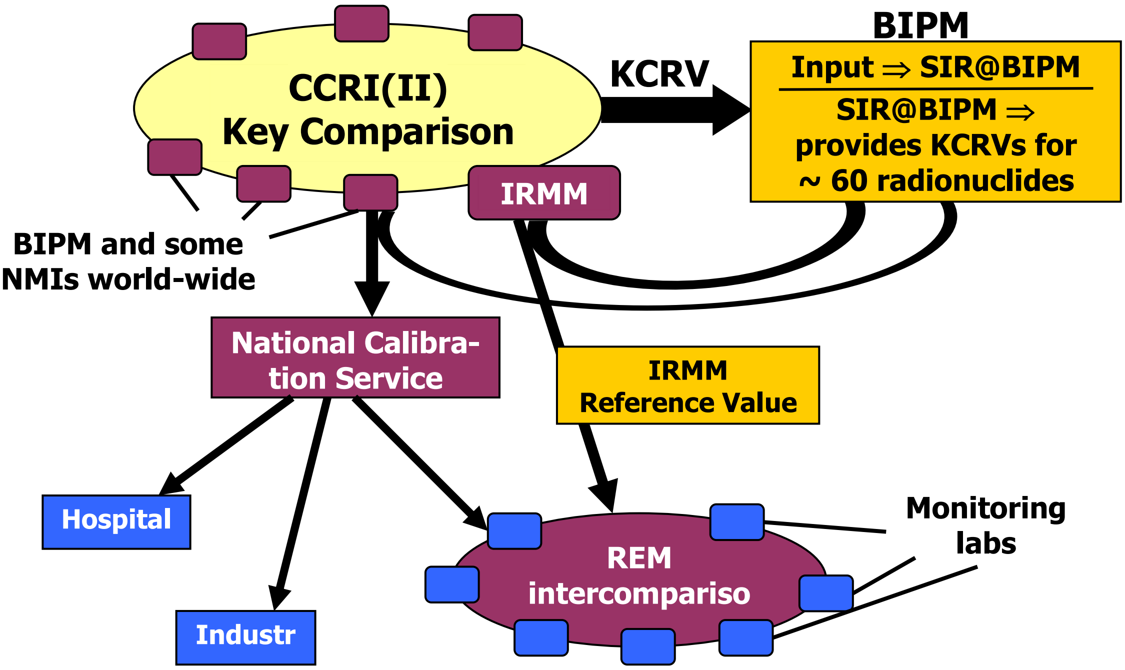

Fig. 1. Key comparisons of CCRI(II) and traceability of the reference values for samples provided by IRMM for the intercomparisons amongst monitoring laboratories (KCRV = key comparison reference value)

The aim of this ILC was to verify the performance of EU MS monitoring laboratories for the determination of ^{90}Sr, ^{137}Cs and ^{40}K in a dried fruit matrix. The candidate reference material IRMM-426 Wild Berries was used as a test item.

This report describes in detail all phases of the ILC organised in 2011, from the description of the analytical methods used at the laboratories, the treatment of the data reported by the participants and, finally, the evaluation and comparison of the participants' results with the reference values. A robust evaluation of the performance of individual laboratories is performed using three different approaches: relative deviations, E_{n} numbers (ISO, 2005a) and ‘PomPlots’ (Spasova et al., 2007).

1.1 Reporting of results

All results of activity concentrations were reported normalised to dry mass (Bq·kg^{-1}) with the associated expanded uncertainty U ($U = k \cdot u_c$, where U is determined from the combined standard uncertainty u_c with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %). The necessary correction to dry mass had to be determined on separate small subsamples. To be representative for their water content, these should be taken from the bottles at about the same time as the samples for radionuclide analysis. The participants were instructed to determine the water content by Karl-Fischer titration or by one of the oven-drying procedures (Appendix 3) which were sent together with the ILC instructions.

The reference date for all results was 1 January 2009. The Monographie BIPM-5 (2004 and 2006) was recommended as source of nuclear decay data. The deadline for results reporting was 15 September 2011.

The reporting of the results was realized via Excel files which served also as a questionnaire. Participants were asked to answer all relevant questions regarding the used measurement procedures. Information given in the questionnaire is essential in order to evaluate the results of the intercomparison. Moreover, it allows us to find out the sources of possible drawbacks and to get an overview of the methods used among the laboratories.

Timetable of ILC:

31 Mar 2011	invitation letter (Appendix 1) sent to the national representatives
15 Apr 2011	the laboratories are nominated by the national representatives
Apr-May 2011	the soil samples are sent to the participants via express mail (DHL) together with the information on ILC (Appendix 2)
Aug-Sep 2011	the laboratories submit their results and questionnaire to IRMM
6 Dec 2011	preliminary results sent to participants (Appendix 9)

1.2 Participating laboratories

The participating laboratories were mainly national research institutes, authorities and monitoring laboratories. From EU member states, 79 laboratories were nominated by the national representatives in the expert group according to the Euratom Treaty Art. 35/36. Traditionally, laboratories from accession and pre-accession countries (AC) and other European countries are also invited by IRMM to participate. This time, 12 laboratories from AC joined in the ILC.

In total 88 laboratories (76 from MS, 8 from AC, 2 from Switzerland and 2 from Norway) reported results. The list of all participating laboratories is shown in Appendix 8. Since the anonymity is a requirement in this programme of ILC, 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 Appendix 8.

2 Reference values

In this ILC, the candidate reference material IRMM-426 Wild Berries was used as the test item. The berries were collected in the region affected by the Chernobyl accident in 2005. The radionuclides were metabolised by the plants due to the natural uptake from higher levels in the environment and, therefore, the material has elevated concentrations of ^{137}Cs and ^{90}Sr. No spiking was applied.

The material was processed by oven-drying at 55 ˚C and cryo-milled to a free-flowing powder with a top grain size of 1.4 mm Ø and a median Ø of 300 µm. It was sieved, homogenized and bottled in units of approximately 100 g into 280 mL amber glass jars. The water content of the material after bottling was 3.6 %. The material was sterilized by gamma-irradiation to enhance its long-term stability and to facilitate its transport across borders.

The property values of the material, traceable to SI units, were established in the CCRI(II)-S8 supplementary comparison "wild berries" (Wätjen et al., 2012 and 2014). Nine NMIs and international organisations participated in this supplementary comparison, in which IRMM was the pilot laboratory. IRMM distributed six bottles of the bilberry material to each of the nine participating laboratories between July and September 2010. After analysis, each NMI was to submit individual results of the activity concentrations normalised to dry mass for the six samples supplied and corresponding mean values. A large variety of efficiency calibration and efficiency transfer methods were used for the determination of the gamma-ray emitting radionuclides (^{40}K and ^{137}Cs), completely independent of each other (Wätjen et al., 2012). Likewise, the determination of ^{90}Sr was achieved with very different radiochemical separation methods and counting techniques (Wätjen et al., 2014). This large variety of procedures makes the property values derived from the CCRI(II)-S8 comparison very robust within their associated uncertainties.

The reference values (Table 1) are composed of the property values A_{ref} and an expanded uncertainty U_{ref} taking into account the standard uncertainty contributions u_{char} from the characterisation study, CCRI(II)-S8, as well as the heterogeneity u_{hom} and instability u_{sts} of the material under transport conditions:

$$U_{ref} = k \cdot u_{ref} = k \cdot \sqrt{u_{char}^2 + u_{hom}^2 + u_{sts}^2} \qquad (1)$$

where k = 2 and, assuming that the results of all n laboratories in CCRI(II)-S8 are independent of each other (Pauwels et al., 1998)

$$u_{char}^2 = \frac{\sum_{i=1}^{n} u_{c,i}^2}{n^2} \qquad (2)$$

As example of the measurement uncertainties $u_{\mathrm{c,i}}$ obtained in the characterisation study, CCRI(II)-S8, Appendix 11 lists the uncertainty budgets, $u_{\mathrm{c,IRMM}}$, for the contributions of IRMM to the reference value characterisation.

In analogy to ISO Guides 34 and 35 (ISO, 2009 and 2006) on reference material production and certification, the heterogeneity and instability (under transport conditions) of the analytes in the comparison material were studied at IRMM. Contrary to requirements for a reference material, however, the long-term instability during storage of the material was not taken into account in the uncertainty of the reference values for the comparison samples, since the reference values had been determined parallel in time by supplementary comparison. For an aliquot mass of 50 g, the homogeneity u_{hom} of ^{137}Cs, ^{40}K and ^{90}Sr in the samples was determined to be 0.9 %, 2.1 % and 2.1 %, respectively (Szántó, 2006). The uncertainty contribution u_{sts} due to instability of the material during transportation at higher temperatures (60°C) was estimated as 0.4 % in the case of ^{137}Cs and ^{40}K. For ^{90}Sr, no instability of the material at higher temperature was observed, hence a higher limit of 0.2 % uncertainty contribution due to dispatch conditions was assumed (Spasova and Vasile, 2011). No special dispatching conditions were required. These contributions to the uncertainties of the reference values are summarised in Table 2, expressed as standard uncertainties u in the unit of activity concentration.

Table 1. Reference values of activity concentrations A_{ref} with expanded uncertainties U_{ref} ($k = 2$) for the comparison samples of IRMM-426 Wild Berries at the reference date 1 January 2009.

Radionuclide	$A_{ref} \pm U_{ref}$ (Bq.kg^{-1})
^{40}K	253 ± 15
^{90}Sr	153 ± 9
^{137}Cs	779 ± 24

Table 2. Uncertainty budgets for the reference values of the comparison samples of IRMM-426 Wild Berries, expressed as standard uncertainties in Bq·kg^{-1} dry mass.

Radionuclide	combined standard uncertainty u_{ref}	u_{char}	u_{hom}	u_{sts}
^{40}K	7.6	5.4	5.3	1.0
^{90}Sr	4.3	2.9	3.2	0.31
^{137}Cs	12	8.8	7.0	3.2

3 Methods used by the participating laboratories

Participants in the ILC were free to use measurement methods of their own choice, preferably the routine procedures used in their laboratories. Depending on their laboratory capabilities, they were asked to determine concentrations of ^{40}K, ^{90}Sr, and ^{137}Cs in the dried bilberry material.

The reporting of the results was realized via Excel files which served also as a questionnaire. The information in this chapter is extracted from these files.

3.1 Water content

All results of activity concentrations were to be reported normalized to dry weight. It was recommended to determine the moisture in separate small samples not undergoing further analysis. The preferable method for the water content determination was Karl-Fischer titration. However, not a single laboratory used this procedure. Ten laboratories out of 88 did not report any information about the method for water content determination.

Since the classical approach – drying until constant weight – could not be used due to the biological nature of the bilberry material, special oven-drying procedures (Appendix 3) were supplied to the participants. Two different temperature-time parameter sets were proposed as alternatives to the Karl-Fischer titration:

a) drying at 90 °C for 40 minutes
b) drying at 60 °C for 12 hours.

Despite the specific oven-drying procedures provided, a few laboratories dried the bilberry material to constant weight. Laboratory no. 77 dried two parallel samples at 100 °C with a result of 10 % of water content, which is a clear overestimation. The temperature used by laboratory no. 87 was 110 °C with a similar result, 10.5 %. Laboratory no. 13 dried the sample until constant mass at a temperature of 105 °C, but, surprisingly, the reported value is 3.3 %.

39 participants used the first alternative (a), with an average result for water content of 3.6 %, varying from 1.9 % to 7 %. Seven laboratories applied the second alternative (b) with an average of 3.3 %, varying from 2.8 % to 4 %. 32 other participants were not specific enough in the information provided on the used method. No significant difference was observed between the two alternative methods. The overall reported values of water content ranged from 1.1 % to 10 %, with an average of 3.7 %.

The mass of bilberry material used for the determination of water content varied from 0.12 g up to 250 g. Some laboratories (2, 8, 22, 28, 31, 44, 45, 49, 86, 87) determined the water content separately for each measurement method, gamma-ray spectrometry and ^{90}Sr determination.

45 participants gave information regarding the traceability of the balance calibration used for the measurements, usually realised via the NMIs. The temperature and humidity is controlled in 42 and 23 laboratories, respectively.

3.2 Gamma-ray spectrometry: ^{137}Cs and ^{40}K

Activity concentrations of ^{137}Cs and ^{40}K were determined by direct gamma-ray spectrometry. Laboratory 17 was an exception; it determined the ^{137}Cs activity via beta emission after radiochemical separation, and the ^{40}K activity using flame spectrophotometry. Most of the participants (72 out of 86) used methods routinely applied in their laboratories.

Out of 85 laboratories applying gamma-ray spectrometry 51 are accredited; 16 laboratories provided no information. In laboratory 17, both methods for determination of ^{137}Cs and ^{40}K are accredited. The management systems ISO/IEC 17025 (ISO, 2005b) or ISO 9001 or both are applied in 57 laboratories. About 30 % of all participants analyse less than 25 foodstuff samples per year, 24 % of the laboratories between 25 and 100 samples of this type, and more than 100 samples are analysed in about 33 % of participating laboratories. Eleven participants (13 %) provided no information. These numbers suggest that this sort of measurement is infrequent in about half of the participating monitoring laboratories, possibly due to their regular task assignments.

Sample preparation

In most of the laboratories, the sample was not specially treated before the gamma-ray spectrometry measurement. Two laboratories (10, 21) tapped the sample in order to compact it and to remove the excess air. The instrument used in laboratory 70 is calibrated to one-litre Marinelli geometry. Therefore, to respect the geometry, they prepared a mixture of equal density with cornmeal. Wet mass, dry mass and density of the bilberry sample and of the cornmeal were determined. Moreover, the activity contribution of cornmeal was determined. Another laboratory (47) homogenised the bilberry powder in agar-agar to obtain 1 L of gel sample. Laboratories 53 and 81 dried/ashed their samples at ~ 400 °C and 450 °C, respectively, prior to the gamma measurement.

As noted before, laboratory 17 applied methods different from gamma-ray spectrometry to determine ^{137}Cs and ^{40}K. Prior to the ^{137}Cs measurement, the sample was ashed for six hours at 700 °C. The sample was mineralized in the presence of strontium, iron and caesium carriers. Afterwards, the sample was completely precipitated as oxalate which was filtered and used for radiochemical separation of ^{90}Sr. Then, the caesium was absorbed from the solution on ammonium phosphomolybdate, whose beta emission was measured. In order to determine the activity concentration of ^{40}K, the sample was ashed at 700 °C. Then it was dissolved in HCl and the potassium concentration was determined using flame spectrophotometry. The activity concentration of ^{40}K was calculated according to its natural content.

The majority of the laboratories prepared just one sample for gamma-ray spectrometry, some participants used, however, up to 5 samples. The sample mass varied from 7.7 g up to 220 g before drying.

The majority of laboratories used cylindrical containers, four laboratories (2, 45, 49, 85), however, used Petri dishes and three (47, 54, 70) Marinelli beakers. The sample containers were mostly made of plastic; laboratory 32 used steel and laboratory 36 aluminium containers. The sample was usually placed directly on the detector end-cap. Only five participants (27, 30, 37, 52, 64) indicated a different source-detector distance; this varied from 1.2 mm to 23.5 mm.

Participants were requested to indicate the density of the bilberry sample they prepared. This varied from 0.35 g·cm^{-3} [a] to 1.18 g·cm^{-3}.

Measurement equipment

The measurements were performed with gamma-ray spectrometry systems consisting of high purity germanium detectors (HPGe). Most of the detectors were coaxial, seven laboratories reported that they used planar detectors. Type p detectors were used in 30 laboratories and type n in 15. Out of 46 laboratories which provided details, 24 used detectors with aluminium detector window, 17 used carbon epoxy, four beryllium and one laboratory used a detector with a magnesium window.

Two participants (20, 31) indicated that NaI-scintillator detectors (with aluminium window) were used.

The acquisition time varied from 4 hours up to 6 days.

Efficiency calibration

Only 41 laboratories provided details on their method of efficiency determination. Most frequently, the efficiency calibration was made with the use of reference material in the measurement geometry identical to the one of the bilberry sample (labs: 2, 15, 16, 18, 19, 24, 32, 33, 40, 45, 54, 58, 60, 71, 76, 85). Laboratories 15, 24 and 71 used in addition the software ISOCS. Whereas using reference material to determine the ^{137}Cs efficiency, laboratories 33 and 58 derived the ^{40}K efficiency from calibration curves.

Five laboratories (1, 10, 57, 75, 79) used standard solutions for calibration of their detectors. In laboratory 12, the calibration source was prepared by addition of reference standard solution into an ash sample similar to the real bilberry sample. Laboratory 8 performed calibration using point sources on a grid of points above the detector cap. Afterwards, the efficiency for a volume source was calculated by integrating the point source response of the detector over the volume of the source taking into account self-absorption.

The software Genie was used for the efficiency calibration in laboratories 43, 48, 73, 74, and 87. In laboratories 41 and 42, GammaVision was applied. Laboratories 27 and 44 used some software without giving further details. Moreover, laboratory 44 stated that the computing process is not transparent for them.

The efficiency for ^{137}Cs varied from 0.34 % to 4.1 %, and for ^{40}K from 0.18 % to 2.3 %.

Corrections

Density corrections were applied in 19 and 15 laboratories for ^{137}Cs and ^{40}K, respectively. The average value of this correction was 1.05 for ^{137}Cs, ranging from 1.02 to 1.23. In the case of ^{40}K, the correction factor varied from 1.01 to 1.12, with an average of 1.03. In case the reverse value of the factor was reported, this was recalculated for the sake of comparison. Few laboratories (36, 43, 48, 73, 74) reported the same value for both of the radionuclides. This is clearly incorrect since the attenuation is different for different gamma-ray energies. Laboratory 45 neglected the effect of self-attenuation due to the relatively thin (16 mm) source measured.

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[a] The reported value is inconsistent with the reported data for the container dimensions and mass.

49 laboratories responded to the question on the source of the nuclear decay data. In the information letter (Appendix 2) the Monographie BIPM-5 (2004, 2006) was recommended. This source or the on-line library of Laboratoire National Henri Becquerel (LNBH): Nucléide-LARA (http://www.nucleide.org/) was used by 29 laboratories. Four participants used Genie or GammaVision libraries. Three laboratories used as source the Lund/LBNL Nuclear Data Search (http://nucleardata.nuclear.lu.se/nucleardata/toi/). Reference data tables of Physikalisch-Technische Bundesanstalt (PTB) were used by four laboratories. The rest of the responding participants used various other resources.

3.3 Determination of ^{90}Sr

In total, 52 laboratories reported results of ^{90}Sr activity concentration. Five different counting methods were applied for the strontium determination. Most of the laboratories (1, 4, 6, 11, 12, 13, 20, 22, 25, 27, 31, 41, 44, 45, 50, 51, 53, 55, 57, 61, 63, 66, 78, 79, 80, 83, 85, 88) used gas flow proportional counting. Ten participants (2, 14, 21, 26, 34, 35, 36, 49, 65, 86) used liquid scintillation counting (LSC) in order to measure ^{90}Sr activity. Cherenkov counting was applied in seven laboratories (23, 28, 30, 38, 62, 67, 87). Plastic scintillation counters were used by four participants (17, 68, 69, 75), and three laboratories (59, 77, 84) used Geiger-Müller (GM) counters. All participants used routine procedures. Only 13 % of all laboratories determine more than 100 foodstuff samples per year. 33 % reported that they analyse between 25 and 100, and another 33 % less than 25 food samples per year. No information was given by 11 laboratories (21 %). Again, these numbers suggest that this sort of measurement is infrequent in about half of the participating monitoring laboratories, possibly due to their regular task assignments.

Sample preparation

A majority (24) of laboratories used one sample, 12 laboratories used two and seven used three parallel samples for the strontium determination. One participant used four parallel samples. The amounts of bilberry powder varied from 0.6 g up to 100 g. Prior to the chemical separation of ^{90}Sr from the bilberry material, the samples were ashed in most of the laboratories with temperatures varying from 400 °C up to 700 °C. Three laboratories (12, 14, 27) used only acid digestion of the sample and 16 laboratories (11, 13, 22, 30, 34, 38, 44, 45, 50, 51, 53, 55, 59, 63, 77, 84) did not provide this particular information. The ashed sample was dissolved by various acid treatments, usually depending on the precipitation and separation procedures applied further on. Four laboratories (26, 41, 61, 66) used pressurised micro-wave digestion, and one laboratory (57) applied aqua regia.

Most often, strontium and yttrium were separated from the sample by several consecutive precipitation (mainly oxalate) and centrifugation steps (in 11 laboratories). Extraction with di-(2-ethylhexyl)phosphoric acid (HDEHP) in toluene was applied in eight laboratories (1, 23, 28, 30, 62, 67, 68, 86). Extraction chromatography on Eichrom or Triskem Sr resin, often in combination with precipitation, was used by seven participants (2, 14, 26, 35, 41, 66, 88). Unspecified extraction chromatography methods for strontium/yttrium separation (incl. those on an unspecified crown ether, or on a home-made resin – laboratory 61) were applied in six laboratories (12, 21, 25, 57, 61, 87). Separation based on isolation of strontium from calcium with fuming nitric acid was applied in two laboratories (45, 83).

The source preparation depended on the measurement method. In the case of proportional, plastic scintillation and GM counting, two precipitation procedures were used: oxalate (labs 1, 4, 6, 25, 31, 44, 45, 57, 61, 63, 66, 68, 69, 75, 77, 79, 88) and carbonate (labs 20, 22, 83, 85). The precipitate was filtered and, in some cases, the filter was then deposited on a metal disk.

For LSC and Cherenkov counting, liquid samples were prepared in vials. Five participants provided details on the LSC source preparation. Laboratory 14 dissolved the precipitate on a filter placed in the counting vial by addition of acid and mixed it with the scintillation cocktail. In laboratories 26, 35, 36 and 86, the solution containing strontium was mixed with the cocktail and measured. Only few laboratories specified the type of scintillation cocktail used, e.g. Ultima Gold AB or LLT cocktails were mentioned.

For the Cherenkov measurement, hydroxide or carbonate precipitates on filter were dissolved in HNO_3 in the vials (laboratories 23 and 87). Laboratories 28, 62, and 67 used HNO_3 in the source preparation process as well. After the separation, laboratory 30 mixed the sample with water. No detailed information was provided by laboratory 38.

Measurement equipment

Most of the laboratories applying the proportional counting method used gas flow proportional counters. About half of these were, according to the information provided, pressurized. Only one laboratory (20) used a permanently sealed proportional detector. Most often, the mixture of argon and methane (90:10), also known as P-10, was used. Laboratories 84 and 20 used an argon/isobutene mixture and argon/CO_2 mixture, respectively. The maximum detection efficiency for ^{90}Sr varied from 3.87 % to 75 %. The acquisition time varied from 200 seconds to 5.3 days.

The laboratories using LSC or the Cherenkov counting method utilized commercial counting instruments: Quantulus (eight laboratories), Tri-Carb (five laboratories), Wallac Guardian (one laboratory) and Beckman (one laboratory). The measurement time ranged from 100 minutes to 13.3 hours. Only nine participants reported the efficiency, five for LSC and four for Cherenkov counting. The efficiency of the LSC determination of ^{90}Sr varied from 60 % to 99 %. In the case of Cherenkov counting, the ^{90}Sr efficiency ranged from 34 % to 73 %.

Four laboratories used a solid scintillation counting method, but only two provided some details in the reporting form. Both indicated that the scintillation material was plastic. The reported counting time was 50 minutes (lab 69) and 17 hours (lab 75) with ^{90}Sr efficiencies of 50 % and 45.2 %, respectively.

Of the three laboratories using a GM detector, only laboratory 77 gave some more information. They used a low level GM beta counter and measured the sample for more than 83 hours. The detection efficiency was 58 %.

Calibration and corrections

The chemical recovery was determined either gravimetrically or by elemental analysis (both methods using stable carriers) or by using radioactive tracers. Carriers of stable strontium (labs 2, 8, 11, 12, 14, 22, 25, 27, 41, 49, 50, 65, 85, 86, 87) or yttrium (labs 1, 4, 17, 23, 28, 30, 31, 61, 62, 67) or both (labs 44, 68, 69, 75) were added to the sample, depending on the radiochemical separation and counting methods applied. Stable yttrium in combination with a ^{85}Sr tracer was used in five laboratories (13, 45, 63, 77, 88). The majority of laboratories determined the chemical yield gravimetrically. However, seven participants (23, 28, 30, 61, 62, 63, 67) used titration with Titriplex III or EDTA. Five laboratories (14, 65, 68, 75, 85) applied atomic absorption spectrometry (AAS) or inductively coupled plasma mass spectrometry (ICP-MS).

Four participants spiked the sample with a reference solution of ^{85}Sr (6, 21, 35, 57) and ^{90}Sr+^{90}Y was added in two laboratories (20, 26). When ^{85}Sr was used as a tracer, the chemical recovery was determined by gamma-ray spectrometry of its peak at 514 keV.

Laboratory 36 determined the chemical recovery on the basis of an unspecified standard solution. Laboratory 78 used Al as tracer for the gravimetrical determination of the chemical recovery of yttrium. Laboratory 66 used the chemical recovery value, that it had established during the validation of the method, namely 99 % ± 6 % for ^{90}Sr in organic material.

The chemical recovery of strontium varied from 20 % to 102 %. These values are very similar to the recovery factors observed in the previous ILC on soil.

4 Reported results

The activity concentrations A_{lab}, as reported by the participants, with corresponding expanded uncertainties U_{lab} (k = 2) are plotted in ascending order in the S-plots (Fig. 2 to Fig. 4). The solid red lines indicate the reference activity concentrations A_{ref} and the dashed red lines their expanded uncertainties U_{ref} (k = 2). Laboratory codes are indicated with the results. The tables with all reported values are presented in Appendix 10.

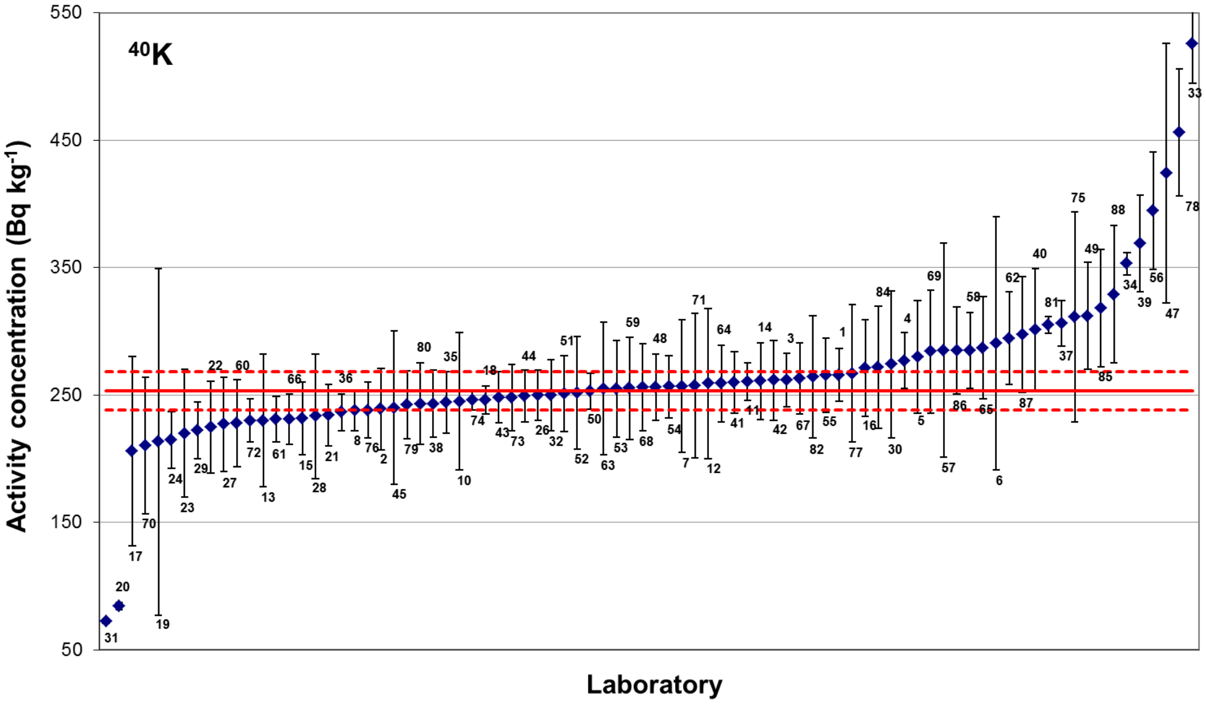

Fig. 2. Activity concentrations of ^{40}K with expanded uncertainties (k = 2) (uncertainty bars) as reported by the participants. Red lines represent the reference value A_{ref} ± U_{ref} (k = 2).

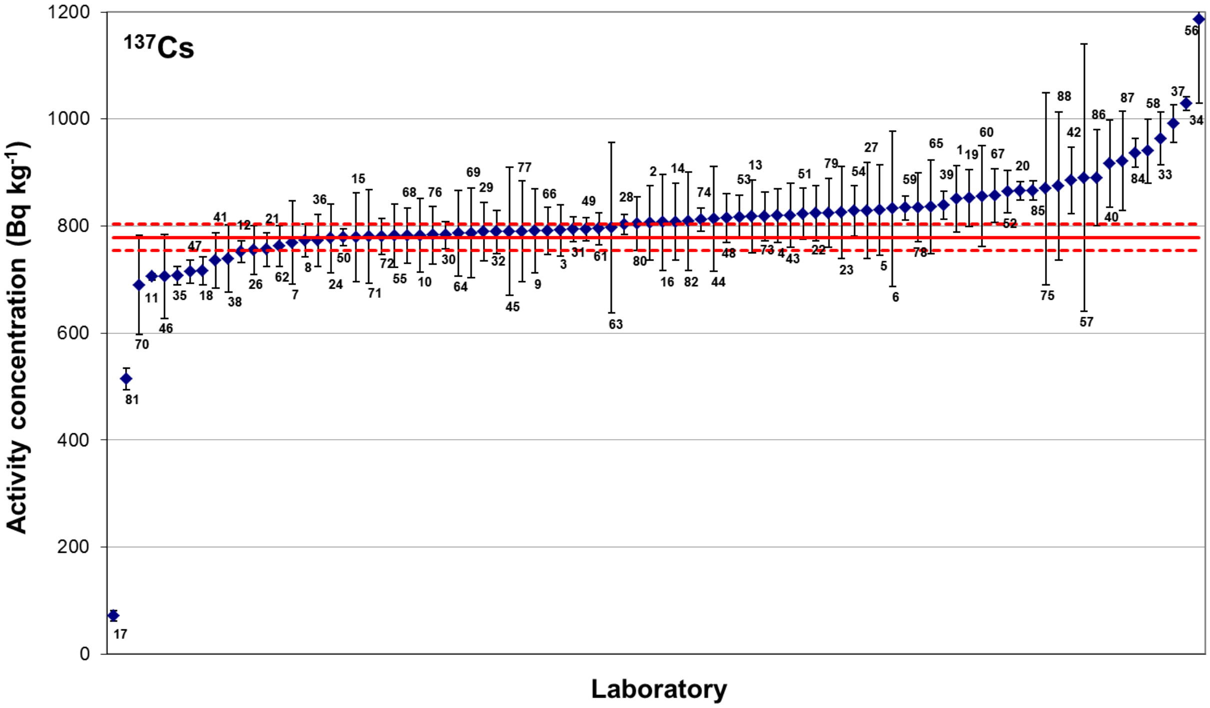

Fig. 3. Activity concentrations of ^{137}Cs with expanded uncertainties (k = 2) (uncertainty bars) as reported by the participants. Red lines represent the reference value $A_{ref} \pm U_{ref}$ (k = 2).

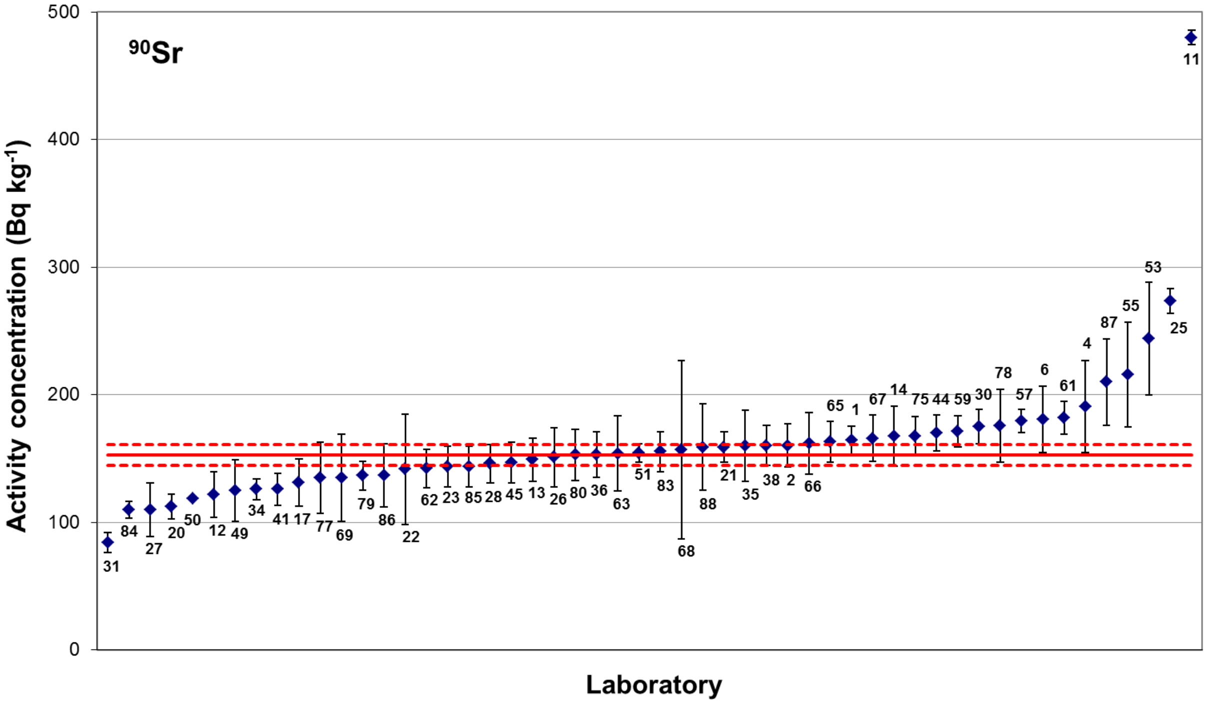

Fig. 4. Activity concentrations of ^{90}Sr with expanded uncertainties (k = 2) (uncertainty bars) as reported by the participants. Red lines represent the reference value $A_{ref} \pm U_{ref}$ (k = 2).

4.1 Uncertainty budgets

The participants were asked to provide the uncertainty budget of their measurement results as part of the reporting sheets for each radionuclide. The submitted uncertainty budgets were analysed. In case a simple recalculation of the relative combined standard uncertainty u_C according to the submitted uncertainty contributions did not comply with the stated value of u_C in the same table, the budget was considered as inconsistent. Sometimes, participants presented an uncertainty budget, but without the associated final value of combined standard uncertainty. Now and then, the combined standard uncertainty was consistent with the one, reported together with the measurement result, but not with the data from the uncertainty budget table. Often, the uncertainty budgets were incomplete and no further conclusions could be drawn. The cases where the effect of rounding could play a role were considered as consistent.

An overview of the submitted uncertainty budgets - consistent or inconsistent - per radionuclide is given in Table 3. The high number of discrepant uncertainty budgets is probably due to the fact that the determination of uncertainties is not well understood in these laboratories or not enough attention is paid to the calculations.

Table 3. Overview of the uncertainty budgets per radionuclide

Radionuclide	Submitted uncertainty budget		Consistent uncertainty budget	
	Yes	No	Yes	No
^{40}K	66 (79 %)	18 (21 %)	36 (55 %)	30 (45 %)
^{137}Cs	69 (80 %)	17 (20 %)	44 (64 %)	25 (36 %)
^{90}Sr	46 (88 %)	6 (12 %)	26 (57 %)	20 (43 %)

To clarify the requested uncertainty reporting, an e-mail was sent to all participants in the course of the ILC (Appendix 5). In particular, standard uncertainty and combined standard uncertainty were to be used following the GUM (2008). Many participants confused combined standard uncertainty and expanded uncertainty. Therefore, after the reporting deadline, another e-mail was sent in order to identify what kind of uncertainty was reported (Appendix 7). Several laboratories responded that they reported expanded uncertainty with a coverage factor $k = 2$, though combined standard uncertainty ($k = 1$) was requested. In these cases, the results were corrected accordingly.

For gamma-spectrometric determinations, the relative uncertainty of weighing, counting statistics, background, decay data, extra-/interpolation of efficiency, calibration factor, half-life, self-absorption and water content were included most often in the calculation of the combined standard uncertainty. An overview of the reported minimum and maximum values is presented in Tables 4 and 5.

The relative uncertainties of the counting statistics varied from 0.04 %[b] and 0.14 %[b] to 27 % and 20 % for ^{137}Cs and ^{40}K, respectively. Since these values depend on the counting time and sensitivity of the used detector, a large variation is reasonable. The same reasoning is valid also for relative uncertainties of background. However, in the case of weighing, the minimum relative values $1.2 \cdot 10^{-6}$ % and $4.2 \cdot 10^{-5}$ %[c] are unrealistically low and are certainly significantly underestimated. Although the participants were requested to report the results normalized to dry mass, only about half of the laboratories accounted for the uncertainty of the water content in the combined uncertainty estimation. However, some laboratories performed the measurements with dried samples, therefore, no additional correction was

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[b] Not feasible based on about 90 000 s counting time; if 4 % were intended to be stated, then the uncertainty budget would contain a mixture of percentage and proportinal values (laboratories 48 and 74).

[c] Even in case a proportion and not a percentage were meant to be stated.

necessary. It is important to note that the lower values of relative uncertainties of calibration and extra-/interpolation of efficiency for ^{137}Cs are unrealistically low and most probably are significantly underestimated.

Table 4. Minimum and maximum relative uncertainties (%) of partial quantities contributing to the combined relative uncertainty as reported by participants for ^{137}Cs. The last column shows the number of laboratories reporting the particular quantity.

^{137}Cs	Minimum	Maximum	Number of labs
Counting statistics	0.04	27	59
Weighing	$1.2 \cdot 10^{-6}$	3.3	58
Background	0.02	19	23
Decay data	0.001	2	28
Extra-/interpolation of efficiency	0.002	5	29
Calibration factor	0.05	7	32
Half life	0.001	2	26
Self-absorption	0.25	10	19
Water content	0.001	3	35

Table 5. Minimum and maximum relative uncertainties (%) of partial quantities contributing to the combined relative uncertainty as reported by participants for ^{40}K. The last column shows the number of laboratories reporting the particular quantity.

^{40}K	Minimum	Maximum	Number of labs
Counting statistics	0.14	20	56
Weighing	$4.2 \cdot 10^{-5}$	3.3	52
Background	0.05	10	32
Decay data	0.01	2	22
Extra-/interpolation of efficiency	0.05	6	30
Calibration factor	0.04	6	31
Half life	0.24	2	20
Self-absorption	0.2	10	18
Water content	0.001	3	30

For the calculation of combined uncertainty of ^{90}Sr activity concentration, the following parameters were included: weighing, counting statistics, background, decay data, amount of tracer, calibration, half-life, water content, sample treatment and chemical recovery. An overview of relative values of partial uncertainties for the determination of ^{90}Sr as reported by the participants is shown in Table 6. Again, some minimum values are clearly underestimated (weighing, background, tracer, calibration, water content).

Table 6. Minimum and maximum relative uncertainties (%) of partial quantities contributing to the combined relative uncertainty as reported by participants for ^{90}Sr. The last column shows the number of laboratories reporting the particular quantity.

^{90}Sr	Minimum	Maximum	Number of labs
Counting statistics	0.06	13	43
Weighing	0.0005	5	38
Background	0.002	26	23
Decay data	0.049	3	10
Tracer	0.04	5	10
Calibration factor	0.03	8.5	30
Half life	0.04	0.26	8
Water content	0.001	5	19
Sample treatment	0.1	12	8
Chemical recovery	0.1	9	38

5 Evaluation of data

Initially, the results were tested for normality and presence of outliers. However, the outlying values were not discarded but were included in the further evaluations. In order to allow a more detailed analysis, several statistical tests – taking the measurement uncertainty and that of the reference values into account – have been applied. Individual laboratory performance is expressed in terms of relative deviations and E_n numbers (ISO, 2005a). The PomPlot graphical method is used for producing a summary overview of the results (Spasova et al., 2007).

5.1 Identification of outliers and normal distribution check

The presence of statistical outliers among the reported results was investigated using the Grubbs' test at a level of significance $\alpha = 1$ %, as suggested in ISO/IEC 5725-2 (ISO, 1994). Moreover, the normal distribution of the data was tested using normal probability plots and frequency histograms.

For ^{40}K, eight results were indicated as outliers in five consecutive runs of the Grubbs' test. In the first run, the values submitted by laboratories 31, 33 and 78 were indicated, in the second, those of participants 20 and 47. In the third, fourth and fifth run, the results of laboratories 56, 39 and 34 were indicated. Out of the eight outlying values, six were too high. The normal probability and frequency histogram plot (Fig. 5) showed that the data of ^{40}K are normally and unimodally distributed.

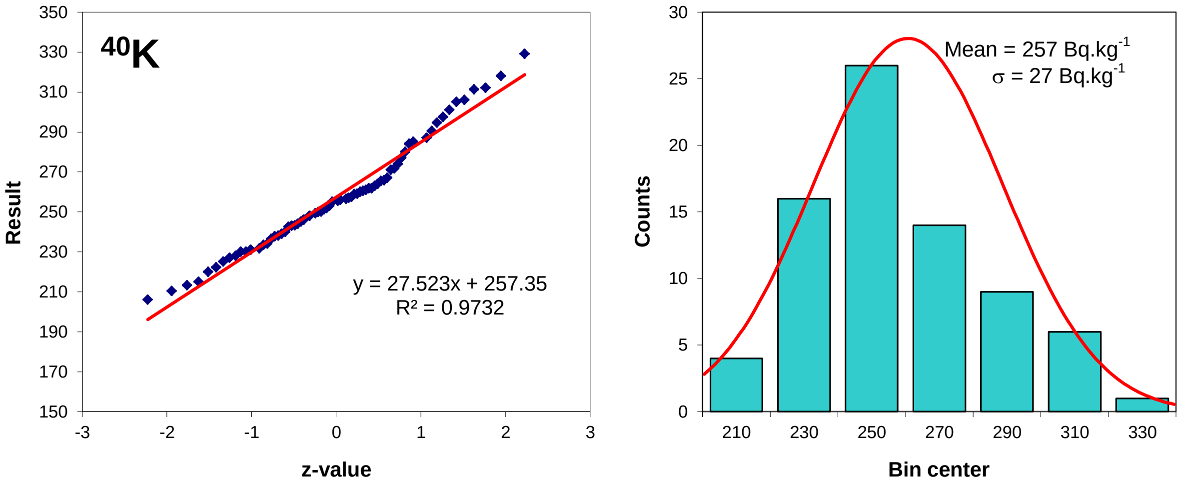

Fig. 5. Normal probability plot and frequency histogram of the ^{40}K data after exclusion of 8 outliers.

In the dataset of ^{90}Sr, in the first run of the Grubbs' test, one outlier was identified (lab 11). In the second and third run, laboratories 25 and 53, respectively, were indicated. All three outlying values were significantly higher than the reference value. As seen in Fig. 6, the data are normally and unimodally distributed. Moreover, the mean of the dataset is very close to the reference value.

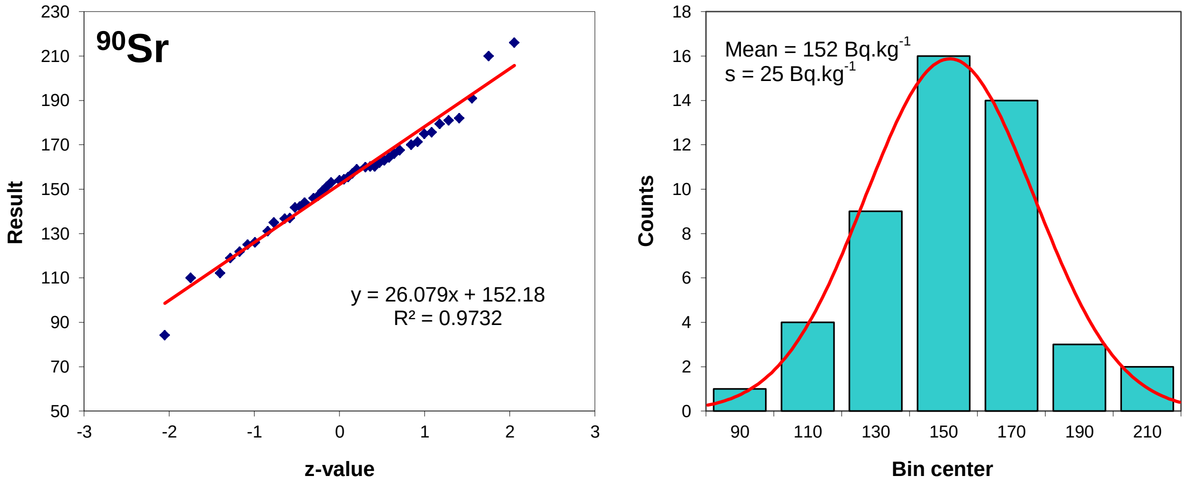

Fig. 6. Normal probability plot and frequency histogram of the ^{90}Sr data after exclusion of 3 outliers.

In the ^{137}Cs data, four outliers were identified in three runs of the Grubbs' test. First, the values of laboratories 17 and 56 were indicated as outliers. In the second and third run, the values submitted by laboratories 81 and 34, respectively, were tagged as outlying values. According to the normal probability plot and frequency histogram (Fig. 7), the ^{137}Cs data are distributed normally and unimodally.

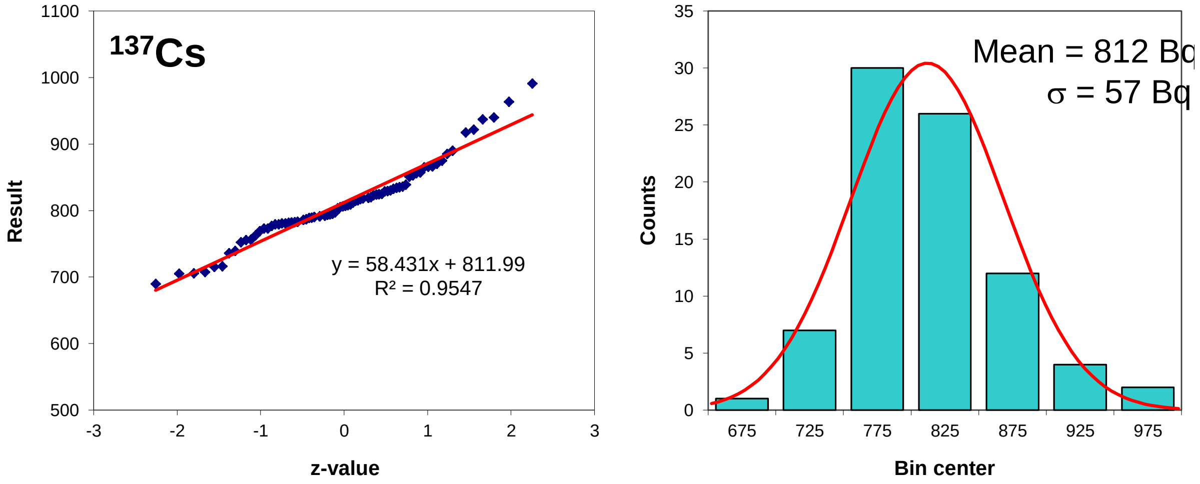

Fig. 7. Normal probability plot and frequency histogram of the ^{137}Cs data after exclusion of four outliers.

5.2 Scores and evaluation criteria

The results of the participating laboratories were evaluated against the reference values using three different approaches: relative deviations, E_n numbers and PomPlots. Details on these methods are described in this chapter.

Relative deviations

Relative deviations (percentage differences in ISO/IEC 13528 (ISO, 2005a)) are calculated as

$$D_{\%} = 100 \frac{A_{lab} - A_{ref}}{A_{ref}} \quad (3)$$

where A_{lab} is the participant’s result;
A_{ref} is the reference value.

These values are plotted in ascending order in deviation charts and the laboratories reporting too low or too high values become more visible. The criterion $|D_{\%}| < 20\ \%$ is used for acceptance. In principle, this is an arbitrarily chosen level, but based on the perception that, at least, routine gamma-spectrometric analysis is achievable within this level of deviation.

E_n numbers

The E_n number takes into account the absolute deviation of the activity concentration value reported by each laboratory (A_{lab}) from the reference value (A_{ref}) together with the combination of expanded uncertainties associated to them (U_{lab} and U_{ref}) (ISO, 1997; ISO, 2005a). Strictly speaking, tests including measurement uncertainty should be used with caution when participants may have poor understanding of uncertainty estimation. We have already observed (chapter 4.1) that this is applicable for about 50 % of the participating laboratories (incl. those not submitting any uncertainty budget at all). Nevertheless, the selected performance test using E_n numbers proves to be robust enough justifying its use in this evaluation. Moreover, incorporating information on uncertainty into the interpretation of results can play a major role in improving the understanding of this difficult subject (ISO, 2005a).

The performance statistic E_n number is calculated as

$$E_n = \frac{A_{lab} - A_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}} \quad (4)$$

where A_{lab} is the participant’s result;
A_{ref} is the reference value;
U_{lab} is the expanded uncertainty of the participant’s result;
U_{ref} is the expanded uncertainty of the reference value.

According to the Guide to the Expression of Uncertainty in Measurement (GUM, 2008), the measurement result should be expressed as $A_{lab} \pm U_{lab}$, where U_{lab} represents the expanded uncertainty with a coverage factor k ($U_{lab} = k \cdot u_c$). For a coverage factor of $k = 2$, corresponding to a level of confidence of 95 %, there is a probability of 95 % for the measurement result A_{lab} with its expanded uncertainty U_{lab} to overlap with the reference value A_{ref} with its expanded uncertainty U_{ref} . Therefore, E_n numbers are interpreted in the following way:

$|E_n| \leq 1$, satisfactory, the laboratory value is compatible with the reference value (green colour in E_n number charts);
$1.5 \geq |E_n| > 1$, unsatisfactory, “warning signal”, the laboratory value differs significantly from the reference value, sources of deviation should be investigated and corrected (orange colour in E_n number charts);
In analogy to the interpretation of zeta-scores, a second level of critical value can be defined:
$|E_n| > 1.5$, “action signal”, there is urgent need to investigate and find the sources of the large deviation (red colour in E_n number charts).

PomPlots

In order to summarize the results, a graphical representation – PomPlot – that underlines the importance of the assigned uncertainties is applied (Spasova et al., 2007). It displays the relative deviations, D/MAD, of the individual results A_{lab} from the reference value A_{ref} on the horizontal axis and relative uncertainties, u/MAD, on the vertical axis (Fig. 8). For both axes, the variables are expressed as multiples of MAD, which is defined as the median of absolute deviation from the reference value

$$MAD = Median\left|D_i\right|, (i = 1, \ldots, n) \tag{5}$$

where D_i is the difference between the reported and the reference value

$$D_i = A_{lab,i} - A_{ref} \tag{6}$$

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

For every data point the uncertainty was calculated as independent sum of the reported combined uncertainties on $A_{lab,i}$ and A_{ref}

$$u_i^2 = u_c^2\left(A_{lab,i}\right) + u_c^2\left(A_{ref}\right) \tag{7}$$

where $u_c\left(A_{lab,i}\right) = U_{lab,i}/k$ and $u_c\left(A_{ref}\right) = U_{ref}/k$ (8)

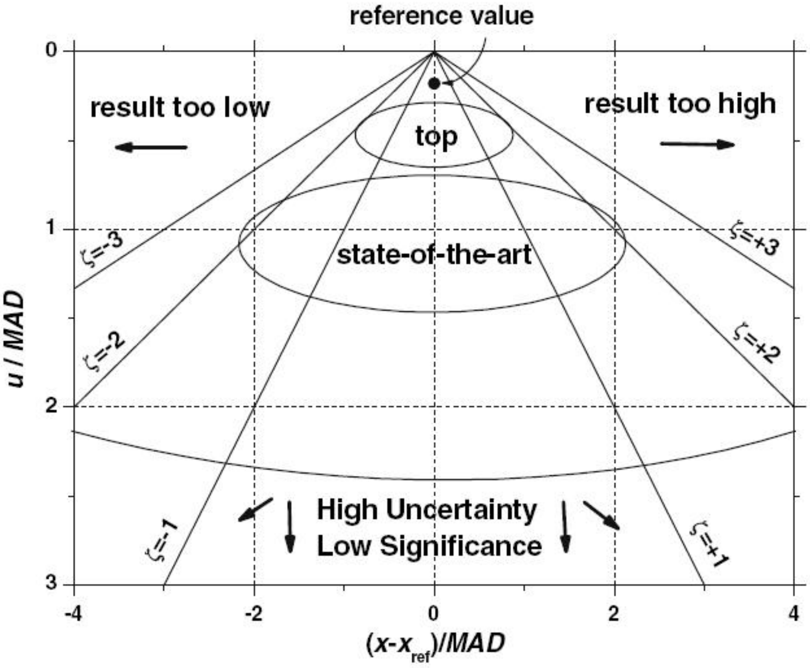

Fig. 8. Interpretation of a PomPlot (Spasova et al., 2007).

The ζ-scores, $\left|\zeta\right| = \left|D/u\right| =$ 1, 2 and 3, are represented by diagonal solid lines, creating the aspect of a pyramidal structure. The ζ-score is a measure for the deviation between laboratory result and reference value relative to total uncertainty (ISO, 2005a). 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 uncertainty is low, 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.

5.3 Evaluation of laboratory performance

Above mentioned statistical tools were used to evaluate the performance of participating laboratories. This section is divided into sub-sections corresponding to the radionuclides.

^{40}K and ^{137}Cs

In general, ^{137}Cs is the most often determined radionuclide in environmental samples. Out of 88 participants, 84 and 86 reported results for ^{40}K and ^{137}Cs, respectively. Both radionuclides are commonly determined via gamma-ray spectrometry. Only one laboratory (no. 17) determined the activities applying different measurement techniques (cf. chapter 3.2).

The vast majority of the laboratories obtained satisfactory results for the gamma-emitting radionuclides, nevertheless 17 % of the ^{40}K results and 9 % of the ^{137}Cs results deviated more than 20 % from the reference values (Table 7). A few laboratories (33, 34, 37 and 56) reported too high values for both radionuclides (Fig. 9 and Fig. 12). Apparently, for these four laboratories, there are sources of error in their general gamma-spectrometric procedures, rendering too high results for both radionuclides. The result of laboratory 33 for ^{40}K deviates by 108 % from the reference value. The methods used in these laboratories should be thoroughly investigated and the source of error identified. Laboratory 17 reported for ^{137}Cs a value lower by 91 % from the reference value, which might suggest that the method is not fit for purpose. In general, the gamma-spectrometric results are comparable with the previous soil ILC (Meresova et al., 2012), but are worse in comparison to the milk ILC (Wätjen et al., 2008), where 93 % and 98 % of laboratories reported results within 20 % for ^{40}K and ^{137}Cs, respectively.

In terms of E_n numbers, 80 % of the ^{40}K results are compatible with the reference value, 8 % of results trigger the warning signal and 12 % the action signal. For ^{137}Cs, the numbers are worse: 72 %, 6 % and 22 %, respectively. This discrepancy in performance of about 20 % of the laboratories between E_n criterion and deviation from the reference value can only be explained by an underestimation of uncertainties. 16 % of all laboratories claim combined standard uncertainties for the determination of ^{137}Cs of less than 1.5 %, most of which (11 laboratories or 13 %) declare an uncertainty contribution from the efficiency calibration either as dominant (at such too low values) or non-existant compared to counting uncertainty.

Table 7. Overview of the laboratory performances regarding the relative deviation and E_n numbers for ^{40}K, ^{137}Cs and ^{90}Sr. The number in parentheses indicates number of laboratories.

	^{40}K (84)	^{137}Cs (86)	^{90}Sr (52)
Within ± 20 %	83 % (70)	91 % (78)	77 % (40)
Outside ± 20 %	17 % (14)	9 % (8)	23 % (12)
Compatible $\vert E_n \vert < 1$	80 % (67)	71 % (61)	56 % (29)
Warning sig. $\vert E_n \vert > 1$	8 % (7)	7 % (6)	15 % (8)
Action sig. $\vert E_n \vert > 1.5$	12 % (10)	22 % (19)	29 % (15)

The relatively unsatisfactory results can probably be attributed to difficulties dealing with the matrix material, in particular the reproducible preparation and mechanical characterisation of volume sources for gamma-ray spectrometry, and the efficiency calibration for such volume sources together with the necessary corrections for differences in density and geometry between standard sources and the bilberry powder samples. Similar difficulties were observed also among laboratories participating in the supplementary comparison CCRI(II)-S8 for the characterisation of the bilberry material (Wätjen et al., 2012). Moreover, the high number of ^{40}K results deviating more than 20 % from the reference value suggests difficulties with the evaluation of the background.

PomPlots are presented in Fig. 11 and Fig. 14. The results for ^{40}K and ^{137}Cs show many points outside $|\zeta| = 1$ and 2, and few outside $|\zeta| = 3$, which are mainly shifted to the right side, thus representing too high results.

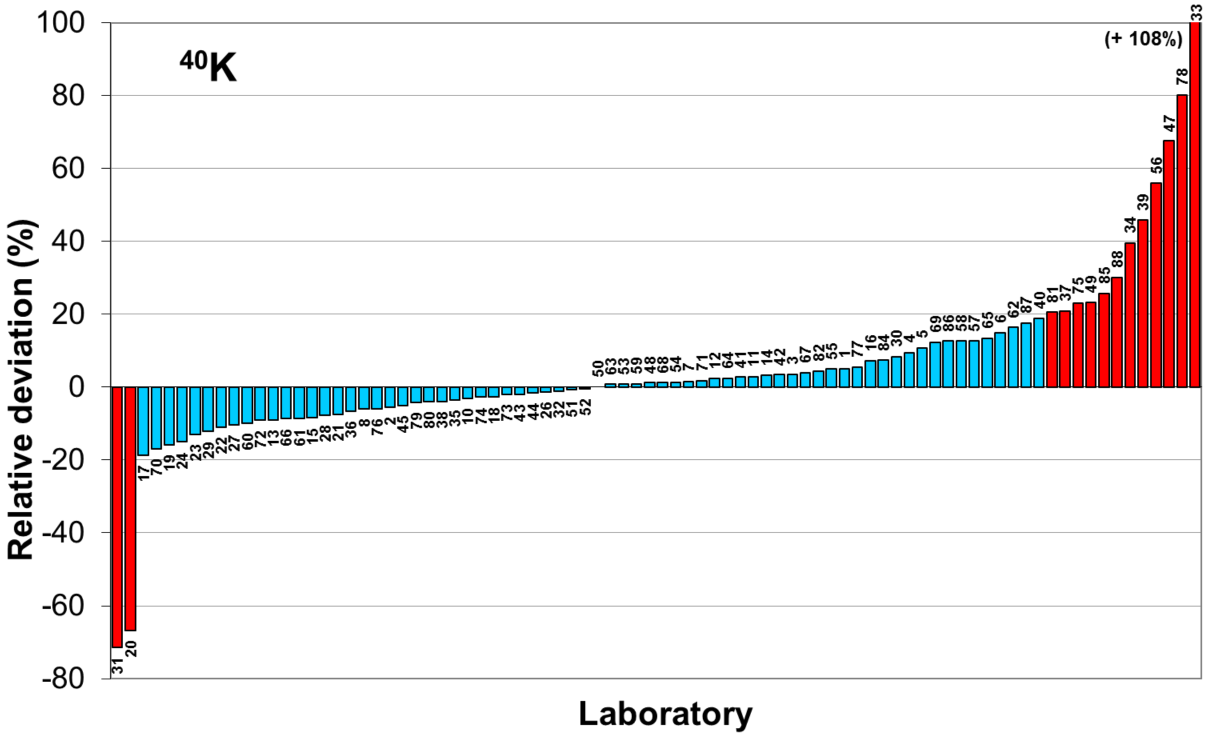

Fig. 9. Deviation chart of the participants' results for ^{40}K plotted in ascending order. Blue colour indicates results within the range ± 20 % from the reference value and red indicates unsatisfactory results outside this range. Numbers show the laboratory code.

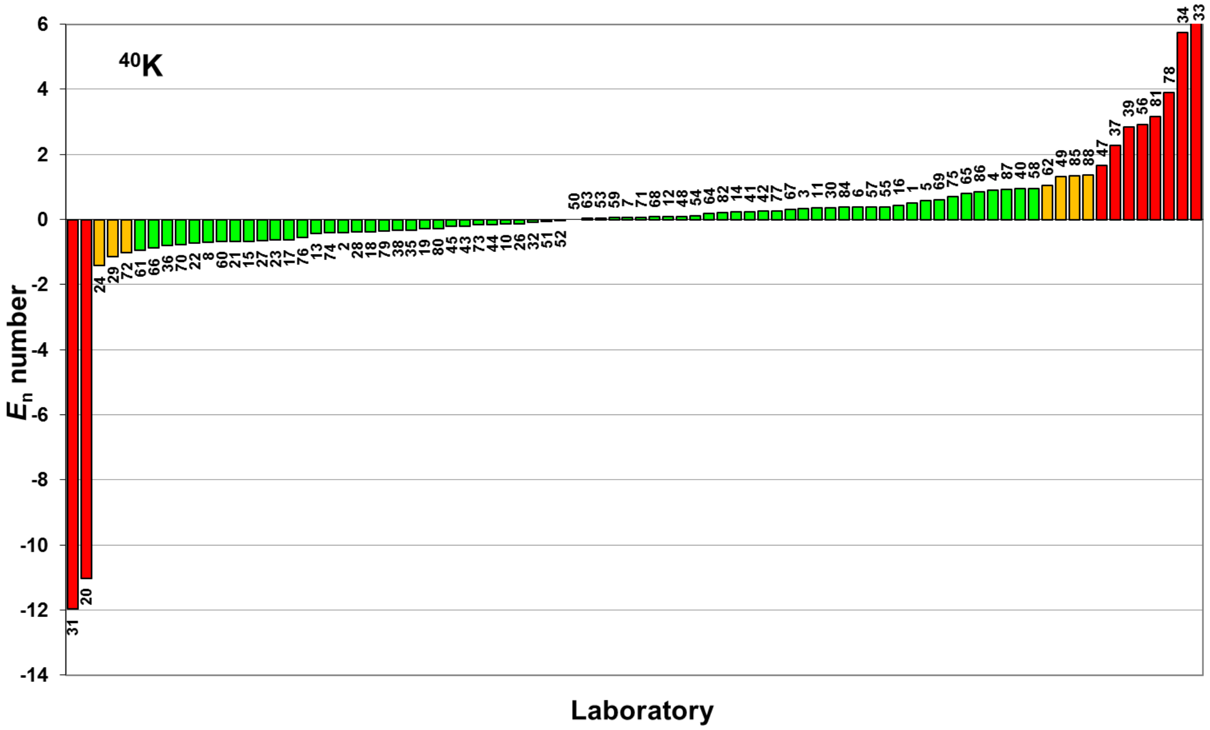

Fig. 10. E_n number chart for ^{40}K activity concentration plotted in ascending order. Green colour indicates compatible results, orange indicates incompatible results with warning signal and red indicates action signal. Numbers show the laboratory code.

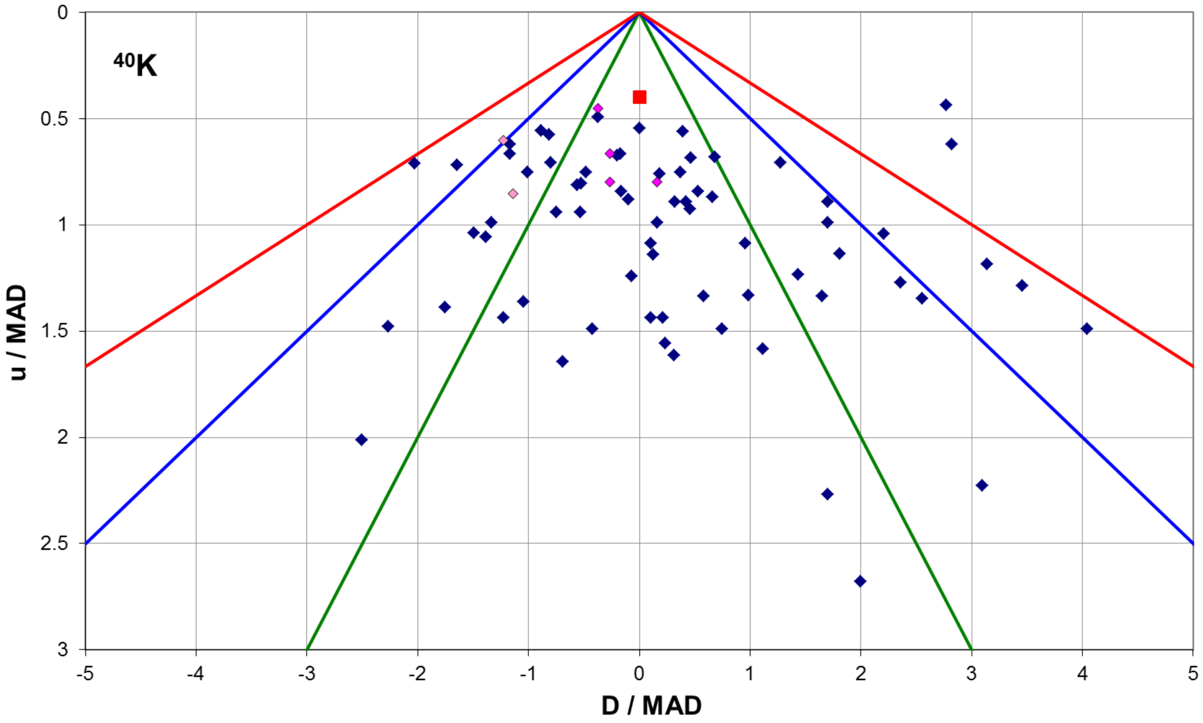

Fig. 11. PomPlot of the ^{40}K data. The red square indicates the reference value. Green, blue and red solid lines indicate ζ-scores = 1, 2 and 3, respectively.

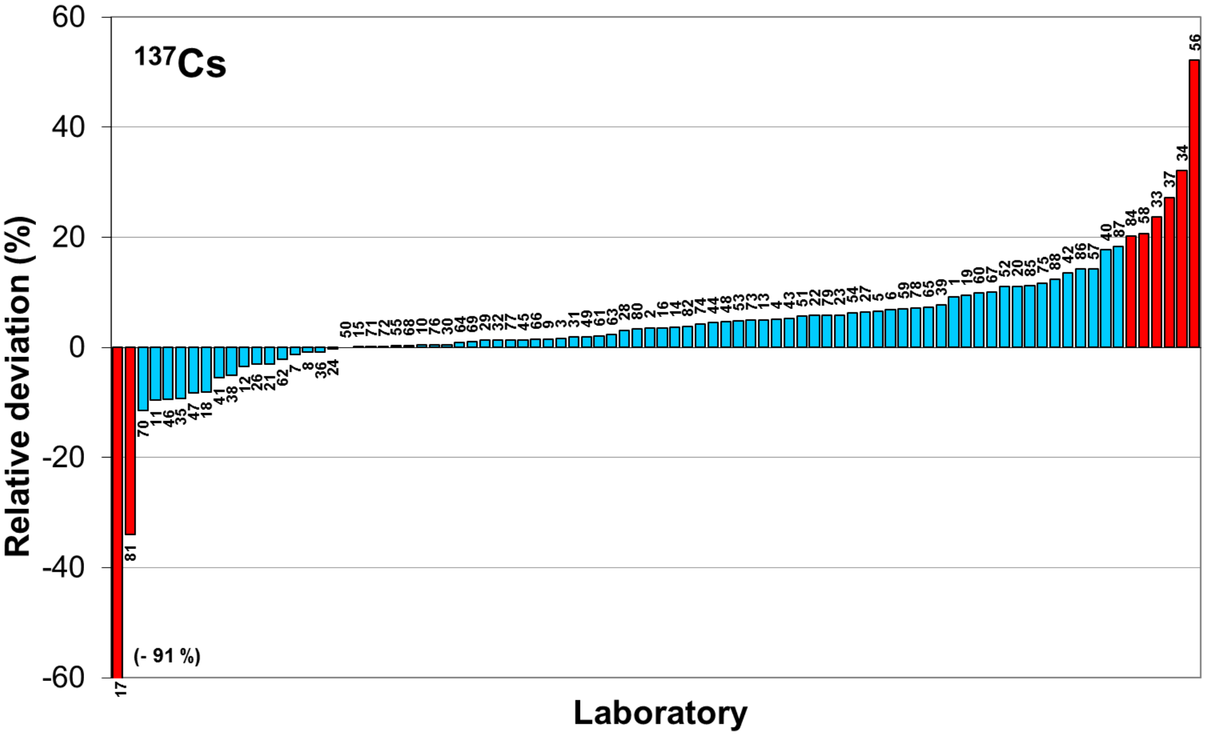

Fig. 12. Deviation chart of the participants' results for ^{137}Cs plotted in ascending order. Blue colour indicates results within the range ± 20 % from the reference value and red indicates unsatisfactory results outside this range. Numbers show the laboratory code.

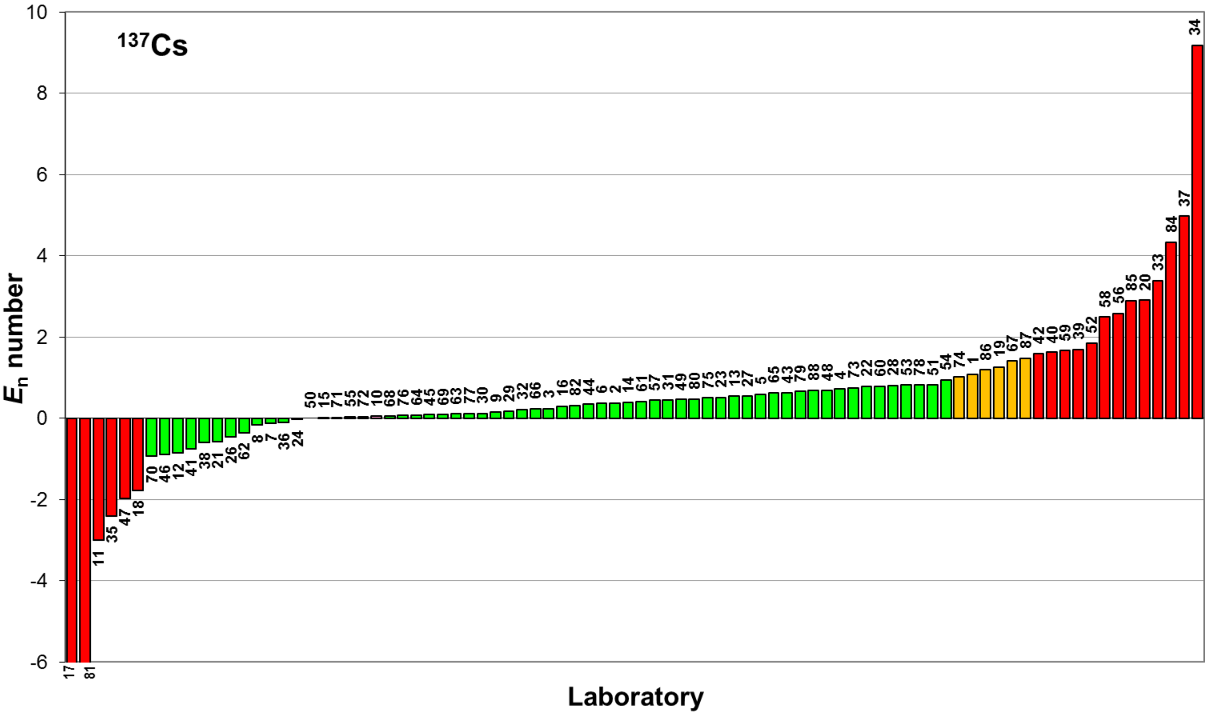

Fig. 13. E_n number chart for ^{137}Cs activity concentration plotted in ascending order. Green colour indicates compatible results, orange indicates incompatible results with warning signal and red indicates action signal. Numbers show the laboratory code.

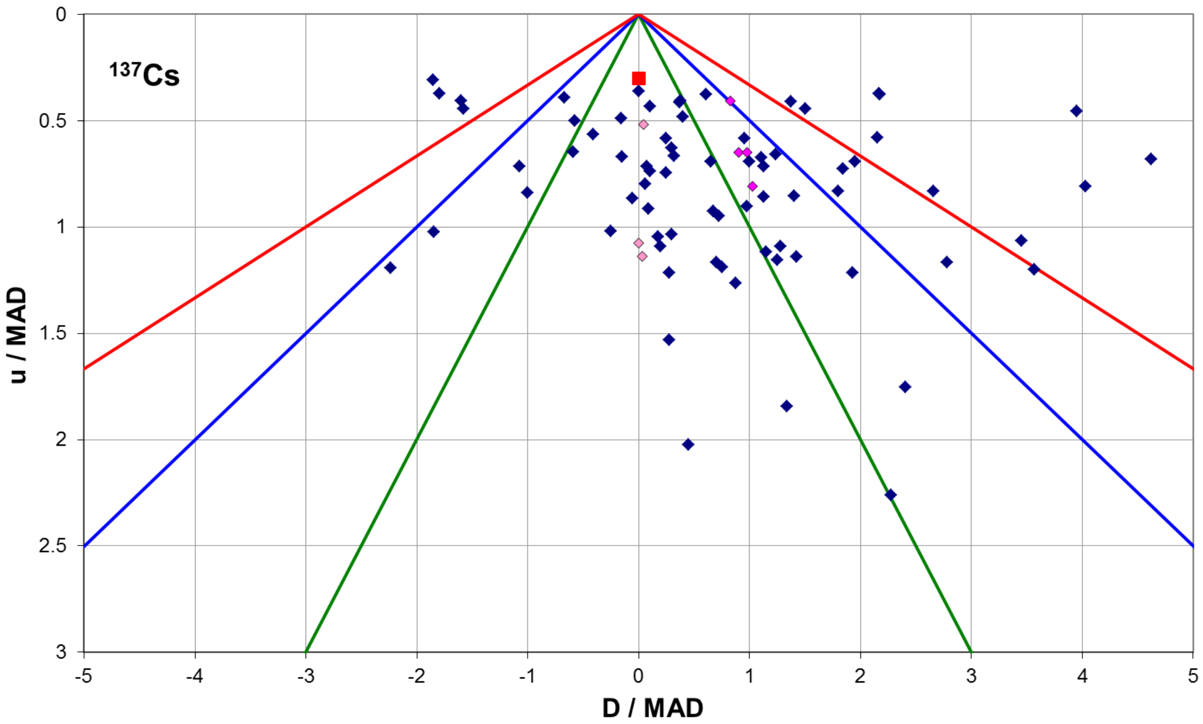

Fig. 14. PomPlot of the ^{137}Cs data. The red square indicates the reference value. Green, blue and red solid lines indicate ζ-scores = 1, 2 and 3, respectively.

^{90}Sr

About 23 % of the ^{90}Sr results deviate more than 20 % from the reference value as can be seen in Table 7 and Fig. 15, and are thus not compliant. If one did take into account the high complexity of the analysis procedures, and used a less strict criterion (30 %), 88 % of the participants' results would fall within 30 % from the reference value. This performance (regardless of the criterion applied) is much better than what was observed in the previous ILCs, where about 23 % and 35 % of laboratories deviated more than 30 % from the reference value for the milk ILC (Spasova et al., 2008) and the soil ILC (Meresova et al., 2012), respectively. Nevertheless, laboratory 11 reported an extreme result 214 % higher than the reference value. Otherwise, the unsatisfactory laboratory results were evenly distributed on both sides – too low and too high (Fig. 15).

Based on the E_n criterion (Table 7, Fig. 16), 56 % of the reported values are compliant. Incompatibility with warning and action signals are triggered by 15 % and 29 % of the results, respectively. These results are worse than those of the milk ILC (68 % satisfactory) and better in comparison to the soil ILC (35 % satisfactory).

The fair outcome for ^{90}Sr determination in this ILC, at least in terms of relative deviations from the reference value, is remarkable in itself and also compared to previous ILCs. Contrary to this, the poor performance based on the E_n criterion (only 56 % of results compatible) indicates – by comparison to the favourable results based on relative deviations – that, in ^{90}Sr determination, difficulties with a correct estimation of measurement uncertainty are even larger than in gamma-ray spectrometry.

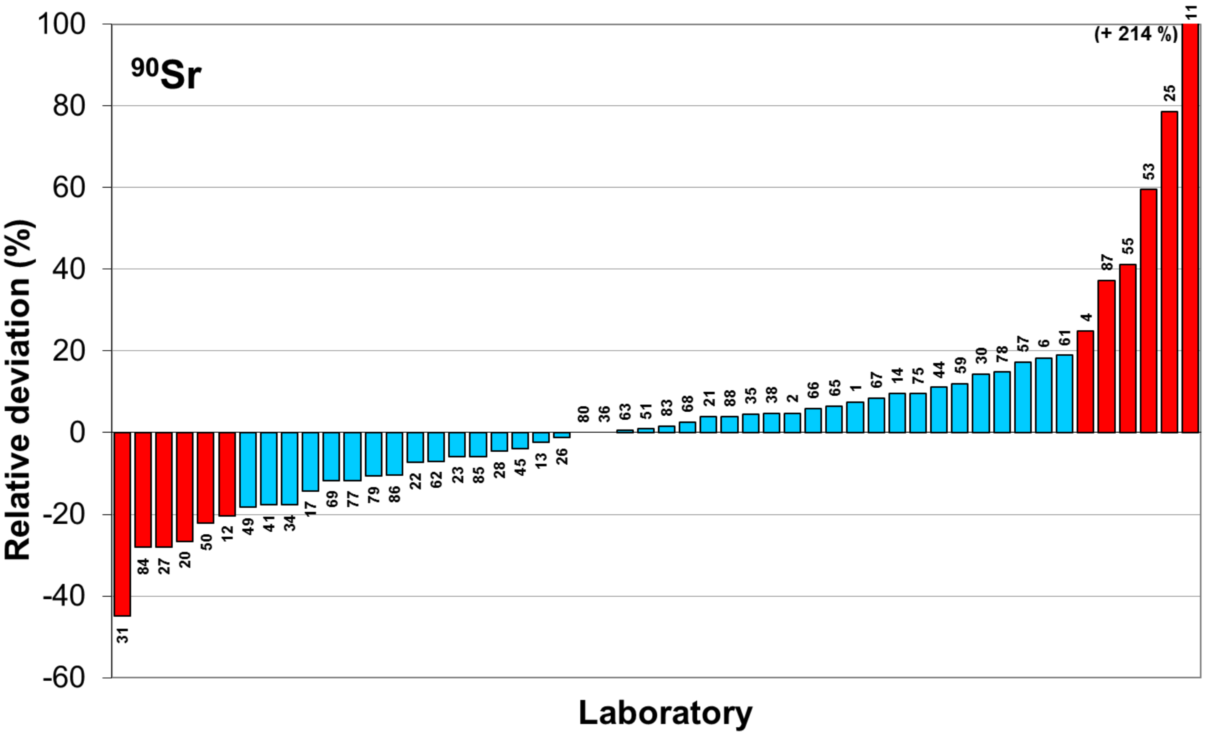

Fig. 15. Deviation chart of the participants' results for ^{90}Sr plotted in ascending order. Blue colour indicates results within the range ± 20 % from the reference value and red indicates unsatisfactory results outside this range. Numbers show the laboratory code.

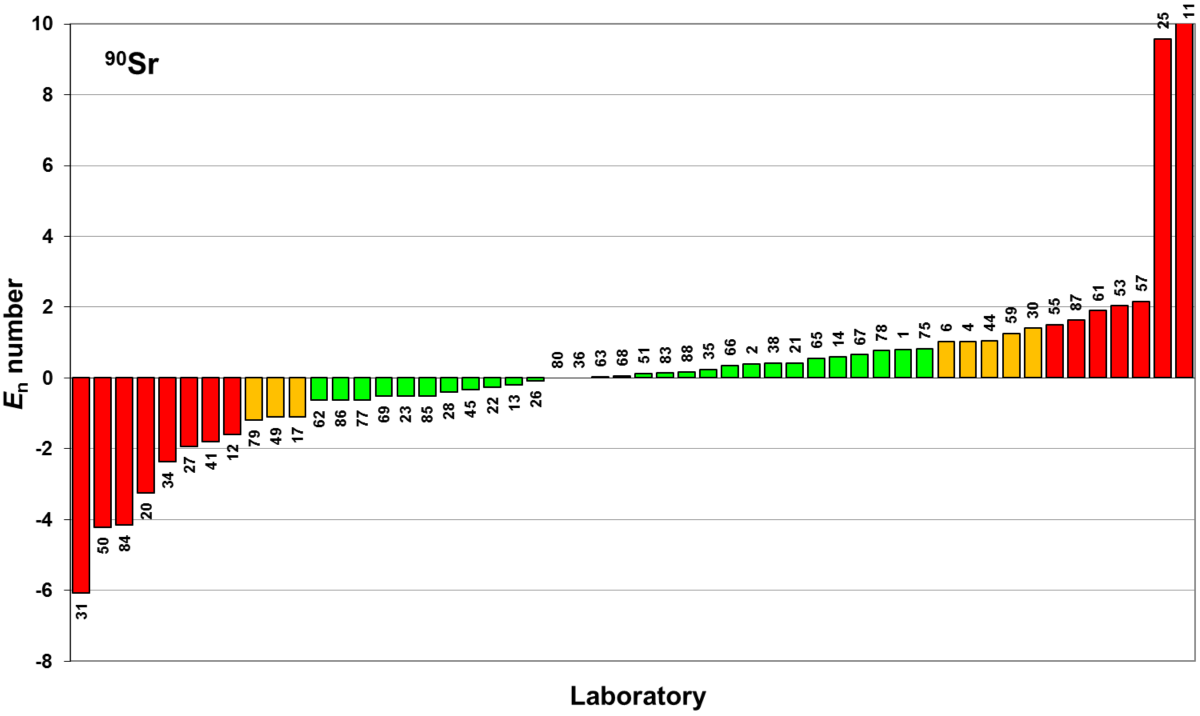

Fig. 16. E_n number chart for ^{90}Sr activity concentration plotted in ascending order. Green colour indicates compatible results, orange indicates incompatible results with warning signal and red indicates action signal. Numbers show the laboratory code.

As expected, ^{90}Sr was determined by a high number of different analysis procedures. Although difficult due to the variety and complex nature of the used methods, an attempt was made to group the results by separation methods (Fig. 17, Fig. 18 and Table 8). The best results - accurate and E_n compatible - are obtained with the HDEHP method, the use of extraction chromatography on Eichrom or Triskem Sr resin, and the fuming nitric acid method. Also precipitation techniques rendered, at least in terms of relative deviation, rather fair results. Extraction chromatography without any specified details provided by far the most discrepant results with 50 % of them deviating more than 20 % and 33 % more than 30 % from the reference value, while 5 of the 6 results (83 %) are triggering the action signal based on E_n incompatibility. The result of laboratory 61, which used a home made resin (in Table 8, included in group "unspecified"), triggered an action signal and it was 19 % higher than the reference value. Unfortunately, 18 laboratories did not provide relevant information on the used separation methods and, therefore, were not included in this evaluation.

Table 8. Overview of the laboratories' performances regarding the relative deviation and E_n numbers sorted according to separation methods of ^{90}Sr. The number in parentheses indicates number of laboratories. Eighteen laboratories did not provide relevant information.

	Precipitation (11)	HDEHP (8)	Extraction chromatography		Fuming nitric acid (2)
			unspecified (6)	Sr resin (7)	
Within ± 20 %	82 % (9)	100 % (8)	50 % (3)	100 % (7)	100 % (2)
<20 %, 30 %>	9 % (1)	0 % (0)	17 % (1)	0 % (0)	0 % (0)
Outside ± 30 %	9 % (1)	0 % (0)	33 % (2)	0 % (0)	0 % (0)
Compatible $\lvert E_n \rvert < 1$	55 % (6)	87.5 % (7)	17 % (1)	86 % (6)	100 % (2)
Warning sig. $\lvert E_n \rvert > 1$	27 % (3)	12.5 % (1)	0 % (0)	0 % (0)	0 % (0)
Action sig. $\lvert E_n \rvert > 1.5$	18 % (2)	0 % (0)	83 % (5)	14 % (1)	0 % (0)

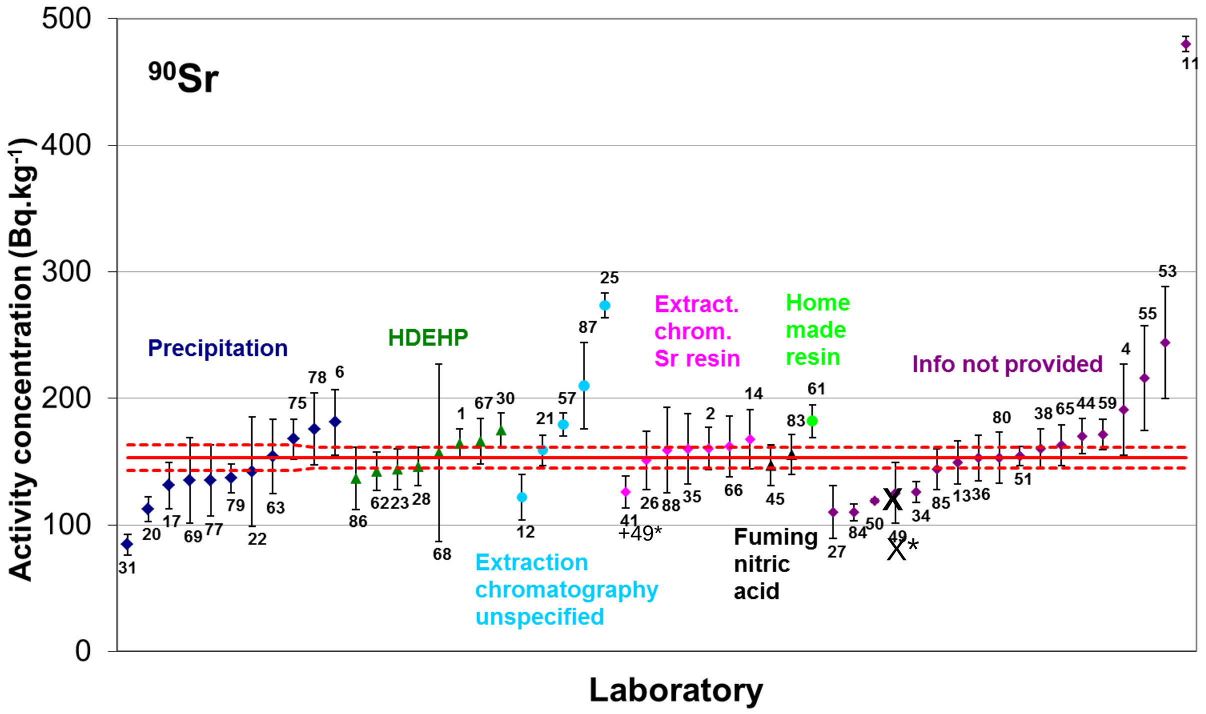

Fig. 17. Laboratory results of ^{90}Sr activity concentration with expanded uncertainties (k = 2) (uncertainty bars) sorted according to separation methods. Red lines represent the reference value $A_{ref} \pm U_{ref}$ (k = 2). Numbers indicate the laboratory code. (*corrected in proof)

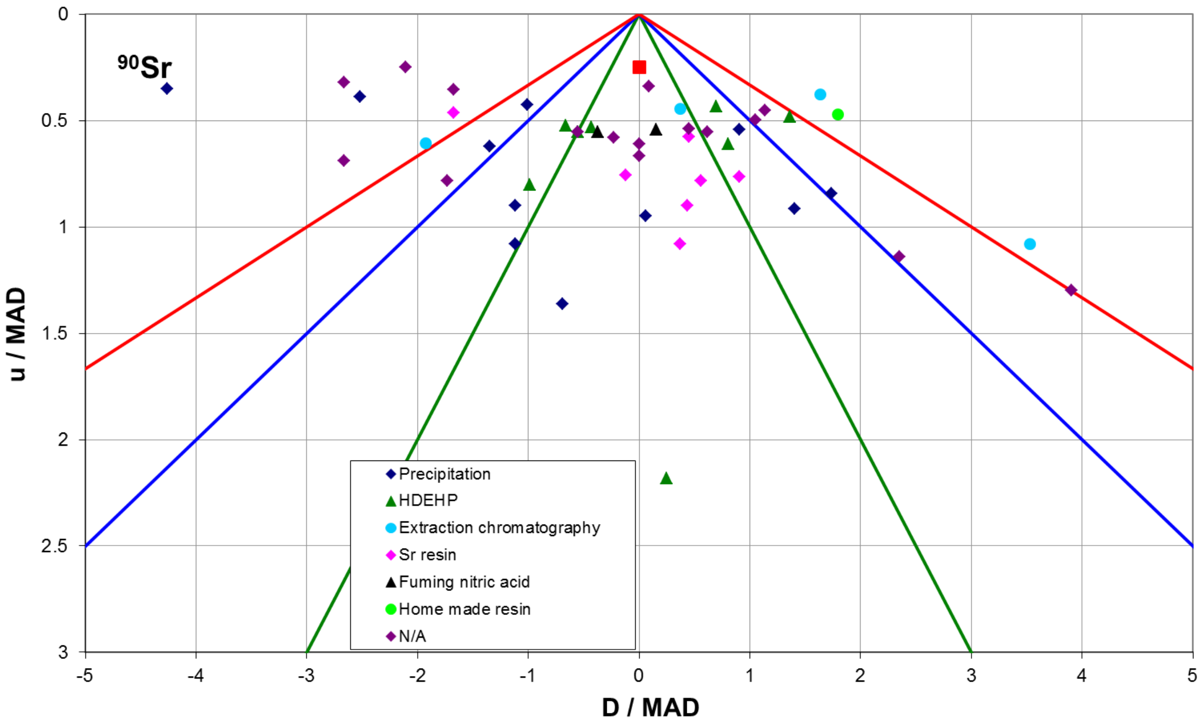

Fig. 18. PomPlot of the ^{90}Sr data sorted according to separation methods. The red square indicates the reference value. Green, blue and red solid lines indicate ζ-scores = 1, 2 and 3, respectively.

A similar comparison was done for the counting methods. The most frequently used counting method was gas proportional counting; 28 participants applied this technique. However, with 36 % of results outside 20 % from the reference value and only 43 % E_n compatible results (Table 9), this method performed worst. Also Geiger Müller counting gives evidence (only 3 laboratories involved do not allow a more definite conclusion) of poor performance: one of the three results more than 20 % relative deviation, only one result E_n compatible. LSC and plastic scintillation counting rendered the most consistent results: all submitted results deviated less than 20 % from the reference value, and 80 % or 75 % were E_n compatible. Also Cherenkov counting proved to be satisfactory with 71 % E_n compatible results and 86 % within 20 % from the reference value. Fig. 19 and the PomPlot (Fig. 20) corroborate the finding that gas flow proportional and Geiger Müller counting performed worse than liquid or plastic scintillation counting or the Cherenkov counting method.

Table 9. Overview of the laboratories' performances regarding the relative deviation and E_n numbers sorted according to counting methods of ^{90}Sr. The number in parenthesis indicates the number of laboratories.

	Gas flow proportional counter (28)	LSC (10)	Cherenkov counting (7)	Plastic scintillation counting (4)	Geiger Müller counting (3)
Within ± 20 %	64 % (18)	100 % (10)	86 % (6)	100 % (4)	67 % (2)
<20 %, 30 %>	18 % (5)	0 % (0)	0 % (0)	0 % (0)	33 % (1)
Outside ± 30 %	18 % (5)	0 % (0)	14 % (1)	0 % (0)	0 % (0)
Compatible $\lvert E_n \rvert < 1$	43 % (12)	80 % (8)	71 % (5)	75 % (3)	33 % (1)
Warning sig. $\lvert E_n \rvert > 1$	14 % (4)	10 % (1)	14 % (1)	25 % (1)	33 % (1)
Action sig. $\lvert E_n \rvert > 1.5$	43 % (12)	10 % (1)	14 % (1)	0 % (0)	33 % (1)

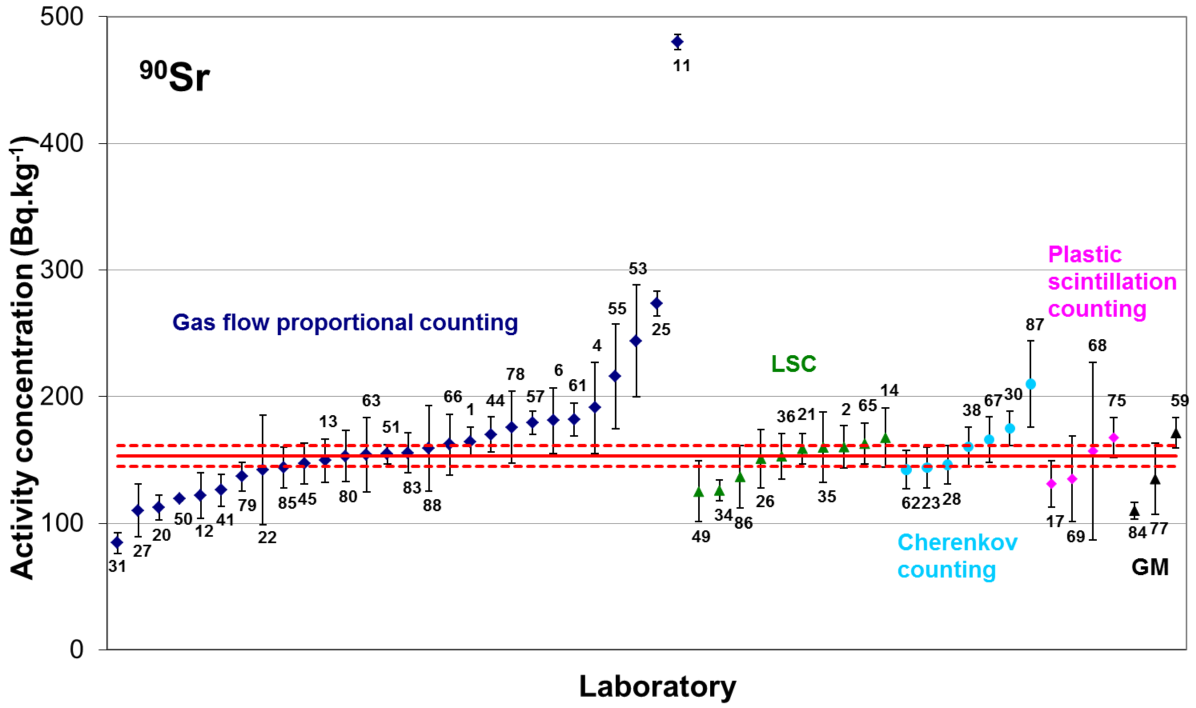

Fig. 19. Laboratory results of ^{90}Sr activity concentration with expanded uncertainties (k = 2) (uncertainty bars) sorted according to counting methods. Red lines represent the reference value $A_{ref} \pm U_{ref}$ (k = 2). Numbers indicate the laboratory code.

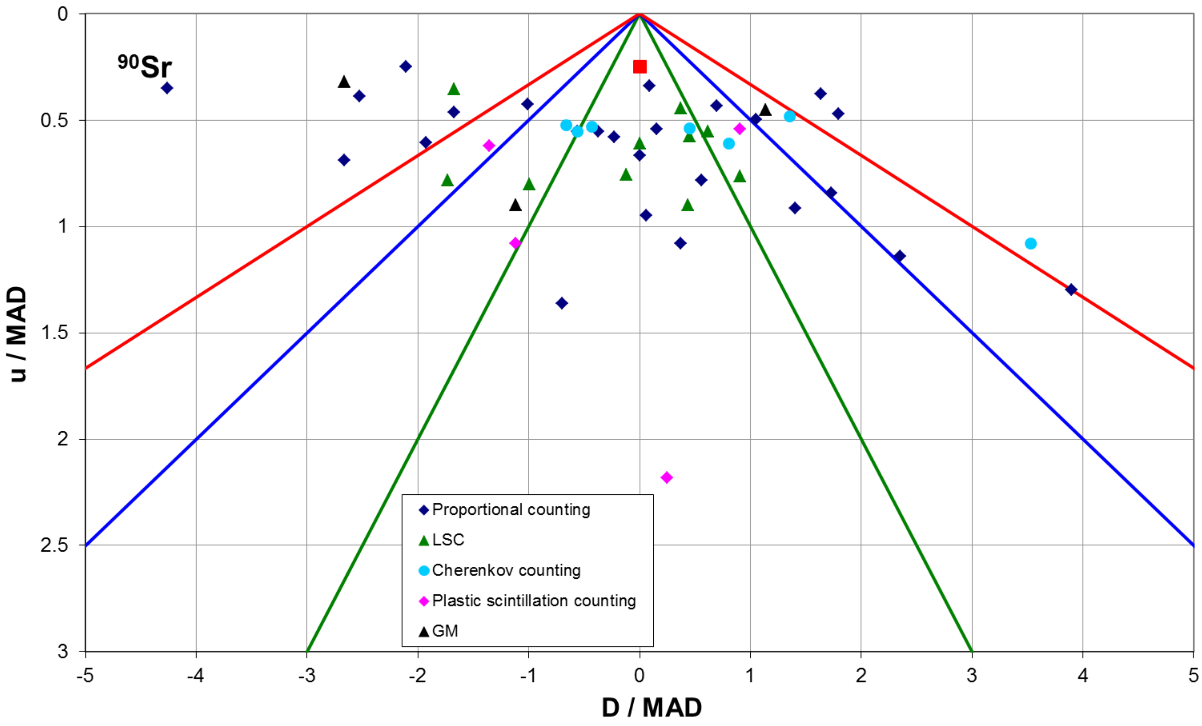

Fig. 20. PomPlot of the ^{90}Sr data sorted according to counting methods. The red square indicates the reference value. Green, blue and red solid lines indicate ζ-scores = 1, 2 and 3, respectively.

6 Conclusions

In 2011, IRMM conducted an interlaboratory comparison (ILC) within the framework of the Euratom Treaty, Articles 35-36. The certified reference material IRMM-426 Wild Berries was used as a testing material. The berries were collected from the region affected by the Chernobyl accident. Due to the natural uptake from elevated levels in the environment, the radionuclides ^{137}Cs and ^{90}Sr were metabolised by the plants; thus, the comparison samples represent a natural matrix. The material was processed at IRMM and the reference values traceable to SI units and the SIR were established in a CCRI(II) supplementary comparison in which nine national metrology institutes and international organisations participated.

In total, 88 laboratories participated in this ILC. They were free to use methods of their own choice, preferably the routine procedures used in their laboratories. A robust evaluation of the performance of individual laboratories was performed using relative deviations and E_n numbers.

The activity concentrations of ^{137}Cs and ^{40}K were determined almost exclusively by gamma-ray spectrometry with 9 % and 17 % of the results deviating more than 20 % from the reference values, respectively. These results are worse in comparison to the previous ILCs. This performance may be due to difficulties in dealing with the fruit matrix and inappropriate use of corrections necessary for gamma-ray spectrometry of volume sources. One laboratory using beta counting for ^{137}Cs determination after radiochemical separation reported a result which is a factor of 10 lower than the reference value, which suggests that the method is not fit for purpose. Such extreme underevaluation of radioactivity in a food sample is not acceptable.

On the other hand, the performance in the determination of ^{90}Sr, seen the complexity of radiochemical procedures, is remarkably good with only 12 % of results lying outside ± 30 % from the reference value, better than what was observed in previous exercises. Based on the E_n criterion, however, 44 % of the ^{90}Sr results are not compatible, demonstrating the great difficulties of many laboratories with realistic uncertainty estimations. Among the counting methods used for ^{90}Sr determination, there is evidence (albeit with small numbers) that gas flow proportional and Geiger Müller counting perform worse than the other detection methods, namely liquid and plastic scintillation counting and Cherenkov counting.

This comparison demonstrates that several laboratories have difficulties to determine activity concentrations of ^{137}Cs, ^{40}K and ^{90}Sr in berry samples. The use of two fundamentally different performance criteria, leading to discrepant evaluation results depending on the criterion, revealed the enormous difficulties that many laboratories have with correct uncertainty estimations, in particular, in this ILC, for ^{137}Cs and ^{90}Sr. All laboratories with unsatisfactory results with respect to relative deviation or to the E_n criterion, whether in gamma-ray spectrometry or in the determination of ^{90}Sr, are urged to investigate and improve their analysis procedures and/or their estimation of uncertainties.

Acknowledgements

This work was possible only with the active participation of 88 laboratories from 35 countries (listed in Appendix 8). The authors would like to thank Z. Szanto for the selection of the material among other candidate materials and the realization of the homogeneity study, as well as Y. Spasova and M. Vasile for the assessment of the short-term stability under transport conditions. Moreover, we are grateful to the participants of the CCRI(II)-S8 supplementary comparison for their contributions to the determination of SI-traceable reference values. We extend great thanks to our colleagues from the Reference Materials Unit of IRMM for processing of the wild bilberries and for determining the water content of the packaged samples.

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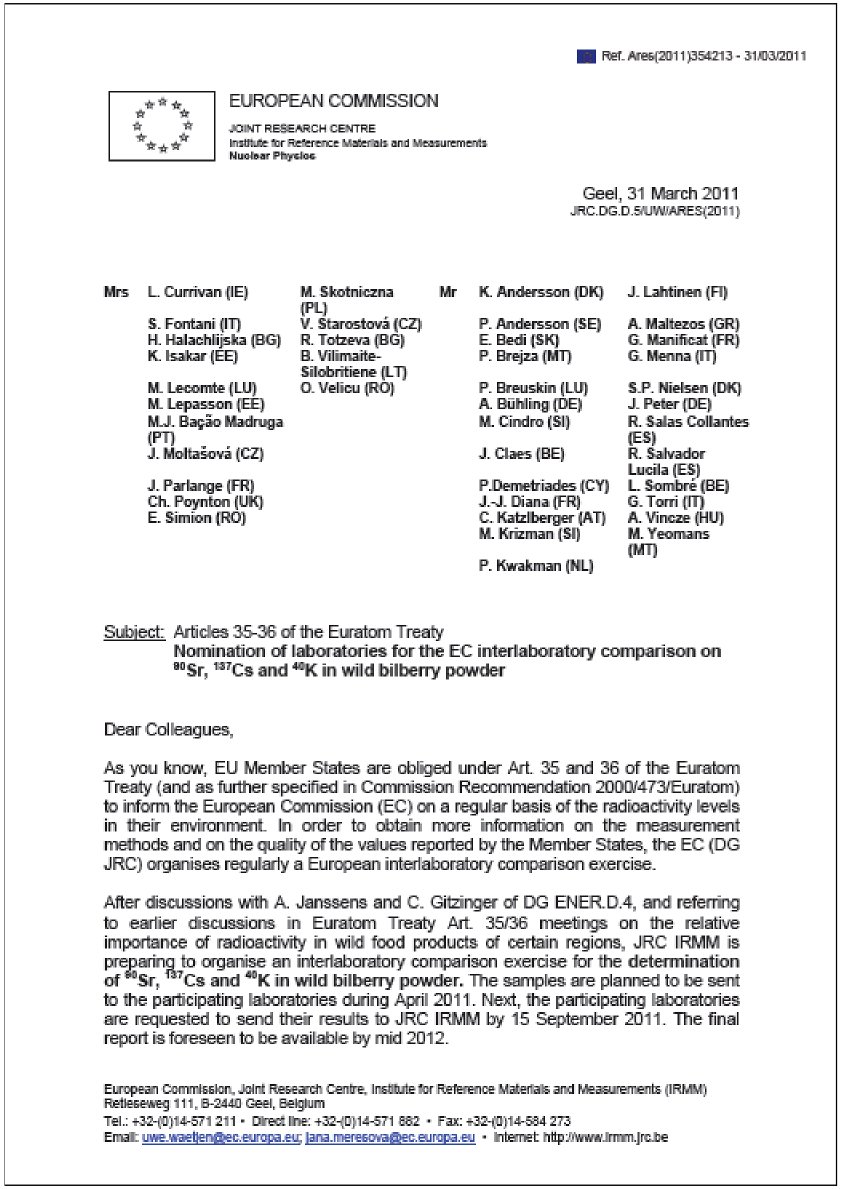
Appendix 1: Invitation letter



Ref. Ares(2011)354213 - 31/03/2011

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements
Nuclear Physics

Geel, 31 March 2011
JRC.DG.D.5/UW/ARES(2011)

Mrs
L. Currivan (IE)

S. Fontani (IT)
H. Halachlijska (BG)
K. Isakar (EE)

M. Lecomte (LU)
M. Lepasson (EE)
M.J. Bação Madruga (PT)
J. Moltašová (CZ)

J. Parlange (FR)
Ch. Poynton (UK)
E. Simion (RO)

M. Skotniczna (PL)
V. Starostová (CZ)
R. Totzeva (BG)
B. Vilimaite-Silobritiene (LT)
O. Velicu (RO)

Mr
K. Andersson (DK)

P. Andersson (SE)
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J. Claes (BE)

P.Demetriades (CY)
J.-J. Diana (FR)
C. Katzlberger (AT)
M. Krizman (SI)

P. Kwakman (NL)

J. Lahtinen (FI)

A. Maltezos (GR)
G. Manificat (FR)
G. Menna (IT)

S.P. Nielsen (DK)
J. Peter (DE)
R. Salas Collantes (ES)
R. Salvador Lucila (ES)
L. Sombré (BE)
G. Torri (IT)
A. Vincze (HU)
M. Yeomans (MT)

Subject: Articles 35-36 of the Euratom Treaty
Nomination of laboratories for the EC interlaboratory comparison on ^{90}Sr, ^{137}Cs and ^{40}K in wild bilberry powder

Dear Colleagues,

As you know, 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 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 (DG JRC) organises regularly a European interlaboratory comparison exercise.

After discussions with A. Janssens and C. Gitzinger of DG ENER.D.4, and referring to earlier discussions in Euratom Treaty Art. 35/36 meetings on the relative importance of radioactivity in wild food products of certain regions, JRC IRMM is preparing to organise an interlaboratory comparison exercise for the **determination of ^{90}Sr, ^{137}Cs and ^{40}K in wild bilberry powder.** The samples are planned to be sent to the participating laboratories during April 2011. Next, the participating laboratories are requested to send their results to JRC IRMM by 15 September 2011. The final report is foreseen to be available by mid 2012.

European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM)
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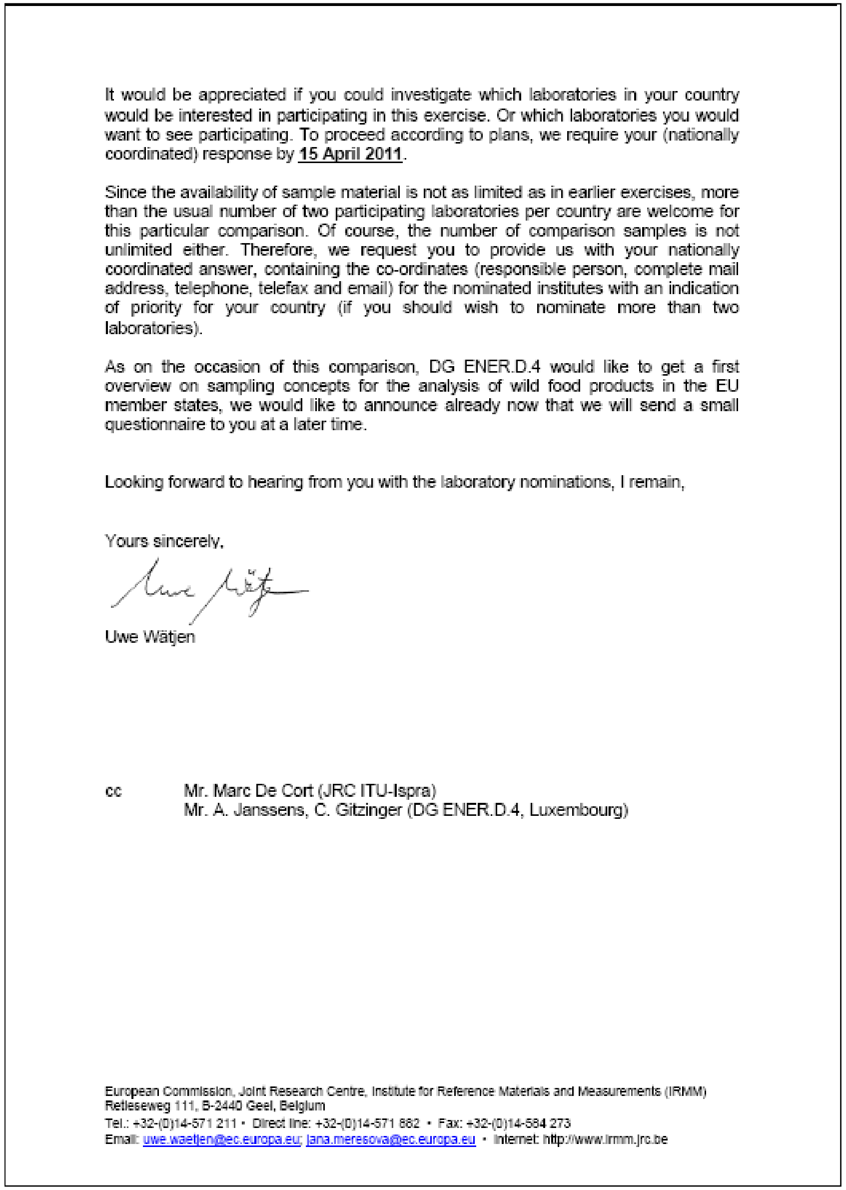
Email: uwe.waetjen@ec.europa.eu; jana.meresova@ec.europa.eu • Internet: http://www.irmm.jrc.be



It would be appreciated if you could investigate which laboratories in your country would be interested in participating in this exercise. Or which laboratories you would want to see participating. To proceed according to plans, we require your (nationally coordinated) response by **15 April 2011**.

Since the availability of sample material is not as limited as in earlier exercises, more than the usual number of two participating laboratories per country are welcome for this particular comparison. Of course, the number of comparison samples is not unlimited either. Therefore, we request you to provide us with your nationally coordinated answer, containing the co-ordinates (responsible person, complete mail address, telephone, telefax and email) for the nominated institutes with an indication of priority for your country (if you should wish to nominate more than two laboratories).

As on the occasion of this comparison, DG ENER.D.4 would like to get a first overview on sampling concepts for the analysis of wild food products in the EU member states, we would like to announce already now that we will send a small questionnaire to you at a later time.

Looking forward to hearing from you with the laboratory nominations, I remain,

Yours sincerely,

Uwe Wätjen

cc Mr. Marc De Cort (JRC ITU-Ispra)
Mr. A. Janssens, C. Gitzinger (DG ENER.D.4, Luxembourg)

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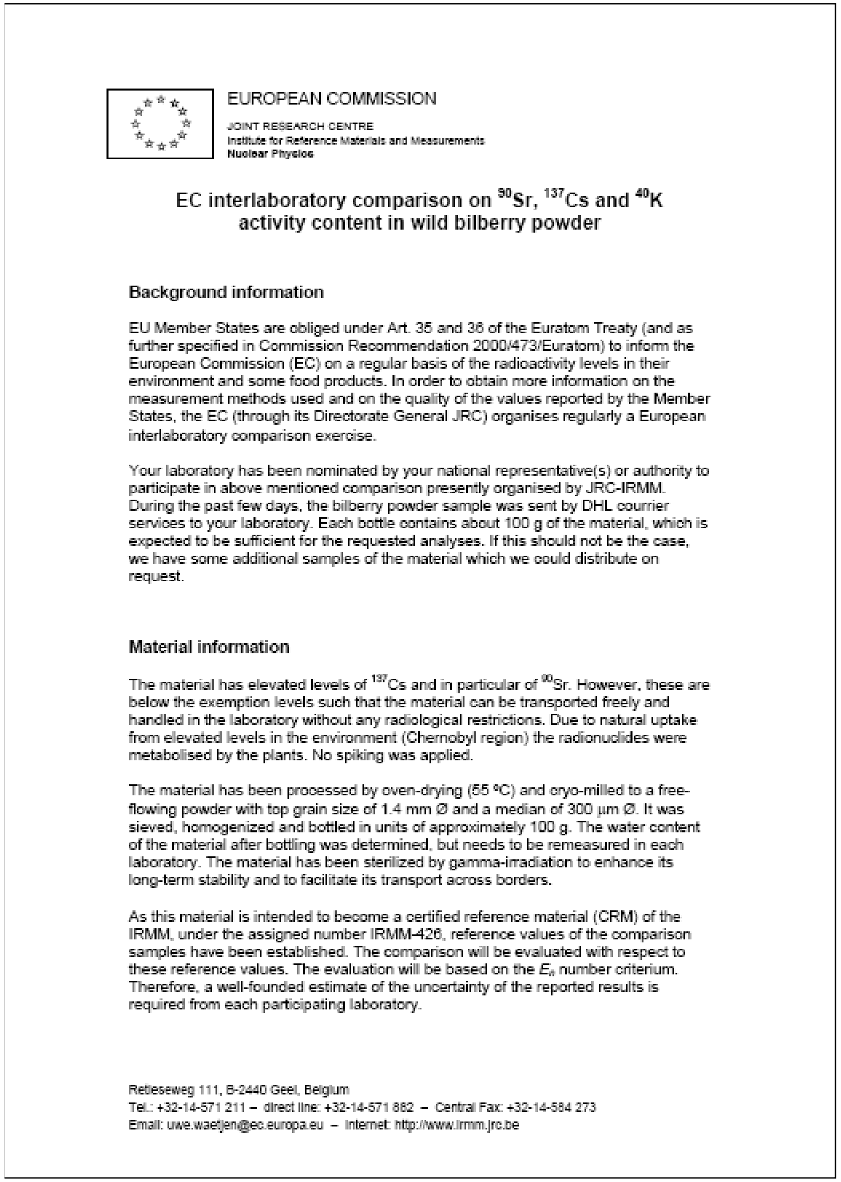
Appendix 2: Information letter



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Nuclear Physics

EC interlaboratory comparison on ^{90}Sr, ^{137}Cs and ^{40}K activity content in wild bilberry powder

Background information

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 of the radioactivity levels in their environment and some food products. In order to obtain more information on the measurement methods used and on the quality of the values reported by the Member States, the EC (through its Directorate General JRC) organises regularly a European interlaboratory comparison exercise.

Your laboratory has been nominated by your national representative(s) or authority to participate in above mentioned comparison presently organised by JRC-IRMM. During the past few days, the bilberry powder sample was sent by DHL courrier services to your laboratory. Each bottle contains about 100 g of the material, which is expected to be sufficient for the requested analyses. If this should not be the case, we have some additional samples of the material which we could distribute on request.

Material information

The material has elevated levels of ^{137}Cs and in particular of ^{90}Sr. However, these are below the exemption levels such that the material can be transported freely and handled in the laboratory without any radiological restrictions. Due to natural uptake from elevated levels in the environment (Chernobyl region) the radionuclides were metabolised by the plants. No spiking was applied.

The material has been processed by oven-drying (55 ºC) and cryo-milled to a free-flowing powder with top grain size of 1.4 mm Ø and a median of 300 µm Ø. It was sieved, homogenized and bottled in units of approximately 100 g. The water content of the material after bottling was determined, but needs to be remeasured in each laboratory. The material has been sterilized by gamma-irradiation to enhance its long-term stability and to facilitate its transport across borders.

As this material is intended to become a certified reference material (CRM) of the IRMM, under the assigned number IRMM-426, reference values of the comparison samples have been established. The comparison will be evaluated with respect to these reference values. The evaluation will be based on the E_n number criterium. Therefore, a well-founded estimate of the uncertainty of the reported results is required from each participating laboratory.

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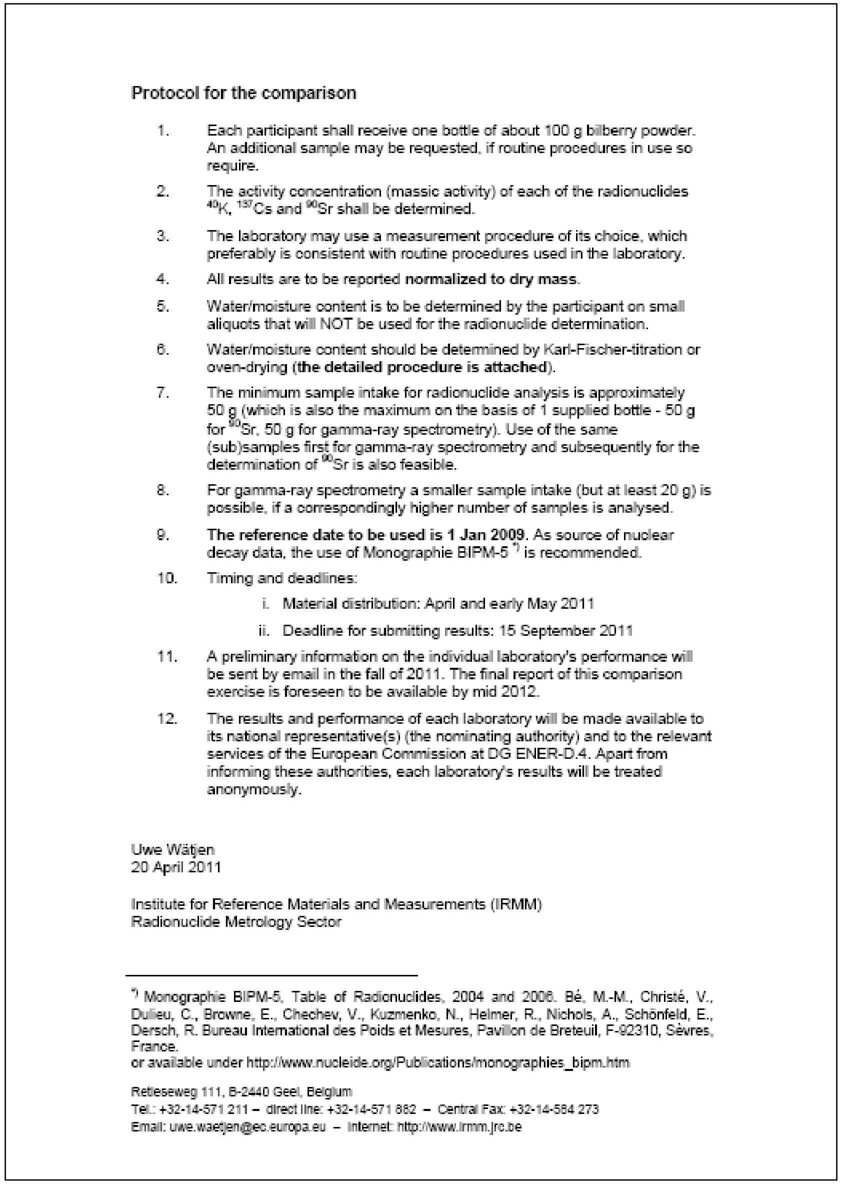

Protocol for the comparison

1. Each participant shall receive one bottle of about 100 g bilberry powder. An additional sample may be requested, if routine procedures in use so require.
2. The activity concentration (massic activity) of each of the radionuclides ^{40}K, ^{137}Cs and ^{90}Sr shall be determined.
3. The laboratory may use a measurement procedure of its choice, which preferably is consistent with routine procedures used in the laboratory.
4. All results are to be reported **normalized to dry mass**.
5. Water/moisture content is to be determined by the participant on small aliquots that will NOT be used for the radionuclide determination.
6. Water/moisture content should be determined by Karl-Fischer-titration or oven-drying (**the detailed procedure is attached**).
7. The minimum sample intake for radionuclide analysis is approximately 50 g (which is also the maximum on the basis of 1 supplied bottle - 50 g for ^{90}Sr, 50 g for gamma-ray spectrometry). Use of the same (sub)samples first for gamma-ray spectrometry and subsequently for the determination of ^{90}Sr is also feasible.
8. For gamma-ray spectrometry a smaller sample intake (but at least 20 g) is possible, if a correspondingly higher number of samples is analysed.
9. **The reference date to be used is 1 Jan 2009.** As source of nuclear decay data, the use of Monographie BIPM-5 [*)] is recommended.
10. Timing and deadlines:
    - i. Material distribution: April and early May 2011
    - ii. Deadline for submitting results: 15 September 2011
11. A preliminary information on the individual laboratory's performance will be sent by email in the fall of 2011. The final report of this comparison exercise is foreseen to be available by mid 2012.
12. The results and performance 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 DG ENER-D.4. Apart from informing these authorities, each laboratory's results will be treated anonymously.

Uwe Wätjen
20 April 2011

Institute for Reference Materials and Measurements (IRMM)
Radionuclide Metrology Sector

[*)] Monographie BIPM-5, Table of Radionuclides, 2004 and 2006. 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.
or available under http://www.nucleide.org/Publications/monographies_bipm.htm

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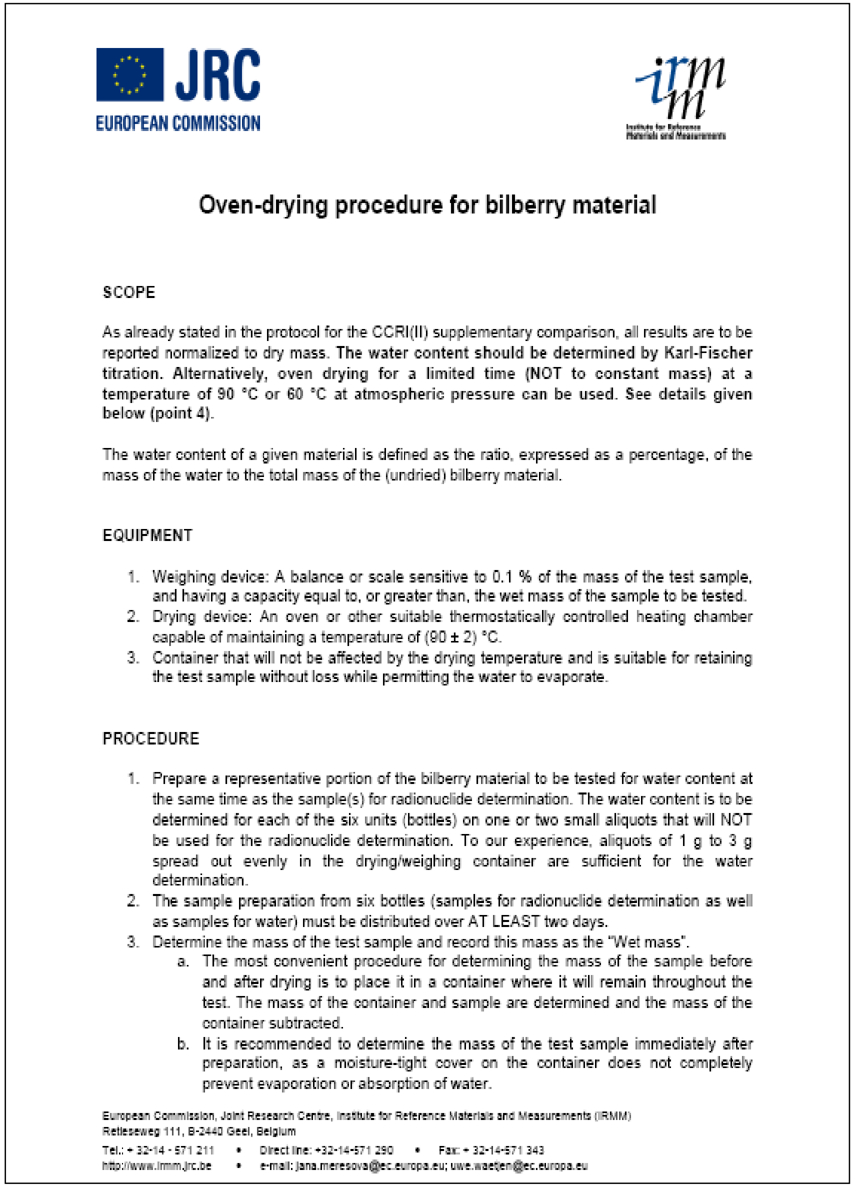
Appendix 3: Drying procedures



Oven-drying procedure for bilberry material

SCOPE

As already stated in the protocol for the CCRI(II) supplementary comparison, all results are to be reported normalized to dry mass. **The water content should be determined by Karl-Fischer titration. Alternatively, oven drying for a limited time (NOT to constant mass) at a temperature of 90 °C or 60 °C at atmospheric pressure can be used. See details given below (point 4).**

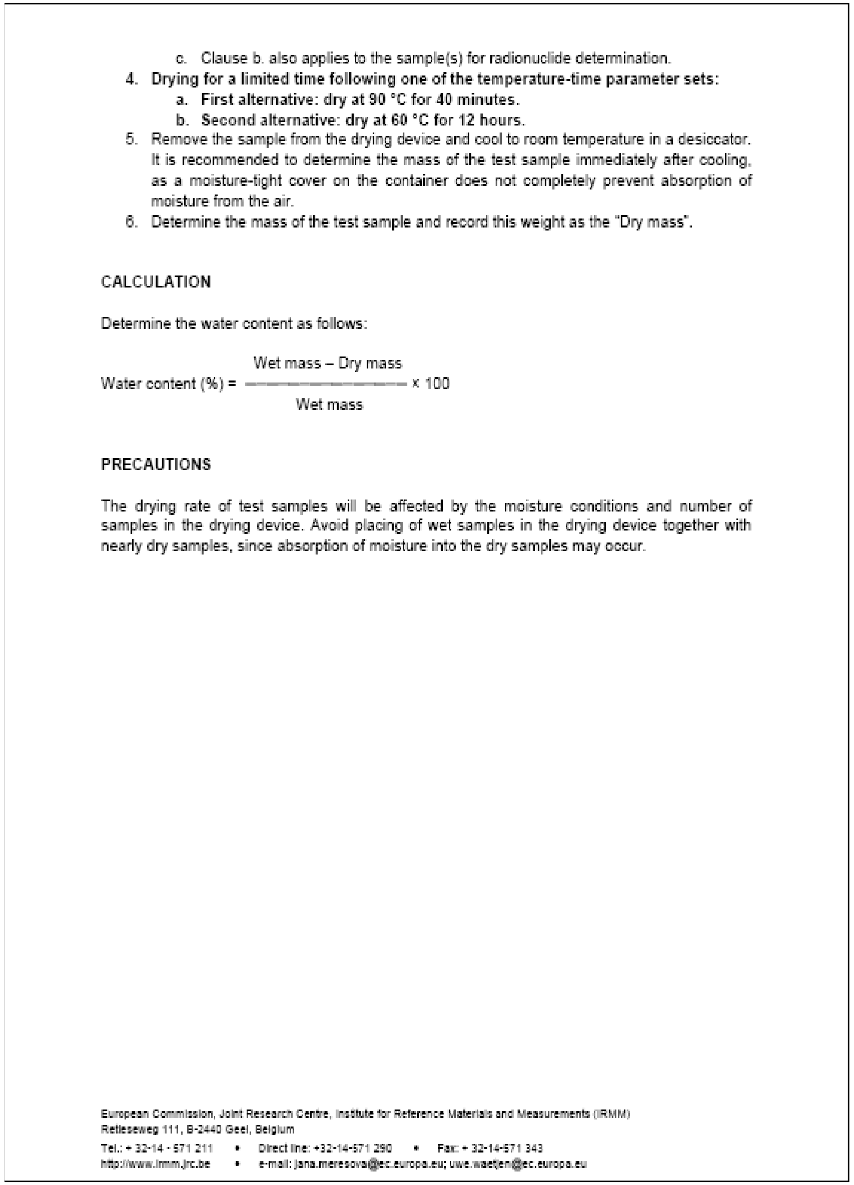
The water content of a given material is defined as the ratio, expressed as a percentage, of the mass of the water to the total mass of the (undried) bilberry material.

EQUIPMENT

1. Weighing device: A balance or scale sensitive to 0.1 % of the mass of the test sample, and having a capacity equal to, or greater than, the wet mass of the sample to be tested.
2. Drying device: An oven or other suitable thermostatically controlled heating chamber capable of maintaining a temperature of (90 ± 2) °C.
3. Container that will not be affected by the drying temperature and is suitable for retaining the test sample without loss while permitting the water to evaporate.

PROCEDURE

1. Prepare a representative portion of the bilberry material to be tested for water content at the same time as the sample(s) for radionuclide determination. The water content is to be determined for each of the six units (bottles) on one or two small aliquots that will NOT be used for the radionuclide determination. To our experience, aliquots of 1 g to 3 g spread out evenly in the drying/weighing container are sufficient for the water determination.
2. The sample preparation from six bottles (samples for radionuclide determination as well as samples for water) must be distributed over AT LEAST two days.
3. Determine the mass of the test sample and record this mass as the "Wet mass".
    a. The most convenient procedure for determining the mass of the sample before and after drying is to place it in a container where it will remain throughout the test. The mass of the container and sample are determined and the mass of the container subtracted.
    b. It is recommended to determine the mass of the test sample immediately after preparation, as a moisture-tight cover on the container does not completely prevent evaporation or absorption of water.



    c. Clause b. also applies to the sample(s) for radionuclide determination.
4. **Drying for a limited time following one of the temperature-time parameter sets:**
    a. **First alternative: dry at 90 °C for 40 minutes.**
    b. **Second alternative: dry at 60 °C for 12 hours.**
5. Remove the sample from the drying device and cool to room temperature in a desiccator. It is recommended to determine the mass of the test sample immediately after cooling, as a moisture-tight cover on the container does not completely prevent absorption of moisture from the air.
6. Determine the mass of the test sample and record this weight as the "Dry mass".

CALCULATION

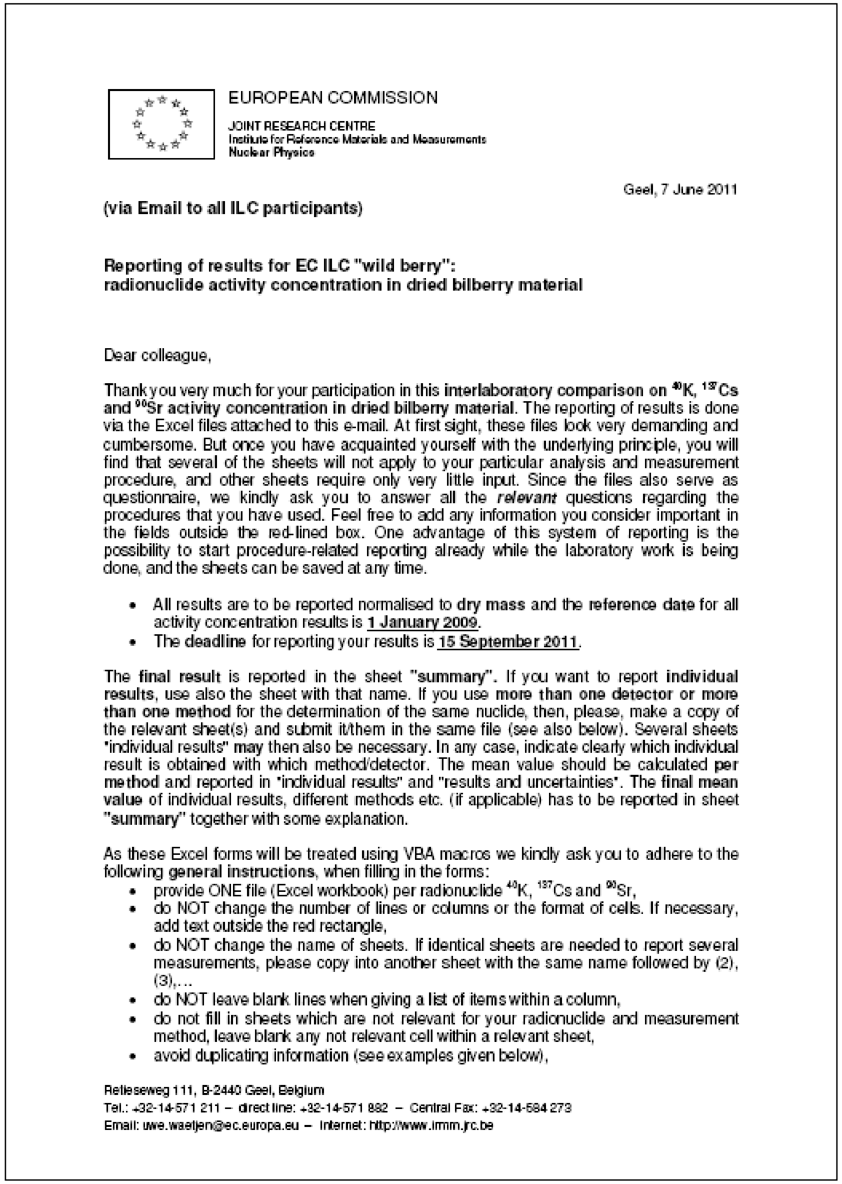
Determine the water content as follows:

$$\text{Water content (\%)} = \frac{\text{Wet mass} - \text{Dry mass}}{\text{Wet mass}} \times 100$$

PRECAUTIONS

The drying rate of test samples will be affected by the moisture conditions and number of samples in the drying device. Avoid placing of wet samples in the drying device together with nearly dry samples, since absorption of moisture into the dry samples may occur.

Appendix 4: Reporting of results



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements
Nuclear Physics

Geel, 7 June 2011

(via Email to all ILC participants)

Reporting of results for EC ILC "wild berry":
radionuclide activity concentration in dried bilberry material

Dear colleague,

Thank you very much for your participation in this **interlaboratory comparison on ^{40}K, ^{137}Cs and ^{90}Sr activity concentration in dried bilberry material**. The reporting of results is done via the Excel files attached to this e-mail. At first sight, these files look very demanding and cumbersome. But once you have acquainted yourself with the underlying principle, you will find that several of the sheets will not apply to your particular analysis and measurement procedure, and other sheets require only very little input. Since the files also serve as questionnaire, we kindly ask you to answer all the ***relevant*** questions regarding the procedures that you have used. Feel free to add any information you consider important in the fields outside the red-lined box. One advantage of this system of reporting is the possibility to start procedure-related reporting already while the laboratory work is being done, and the sheets can be saved at any time.

- All results are to be reported normalised to **dry mass** and the **reference date** for all activity concentration results is **1 January 2009**.
- The **deadline** for reporting your results is **15 September 2011**.

The **final result** is reported in the sheet **"summary"**. If you want to report **individual results**, use also the sheet with that name. If you use **more than one detector or more than one method** for the determination of the same nuclide, then, please, make a copy of the relevant sheet(s) and submit it/them in the same file (see also below). Several sheets "individual results" **may** then also be necessary. In any case, indicate clearly which individual result is obtained with which method/detector. The mean value should be calculated **per method** and reported in "individual results" and "results and uncertainties". The **final mean value** of individual results, different methods etc. (if applicable) has to be reported in sheet **"summary"** together with some explanation.

As these Excel forms will be treated using VBA macros we kindly ask you to adhere to the following **general instructions**, when filling in the forms:

- provide ONE file (Excel workbook) per radionuclide ^{40}K, ^{137}Cs and ^{90}Sr,
- do NOT change the number of lines or columns or the format of cells. If necessary, add text outside the red rectangle,
- do NOT change the name of sheets. If identical sheets are needed to report several measurements, please copy into another sheet with the same name followed by (2), (3),…
- do NOT leave blank lines when giving a **list** of items within a column,
- do not fill in sheets which are not relevant for your radionuclide and measurement method, leave blank any not relevant cell within a relevant sheet,
- avoid duplicating information (see examples given below),

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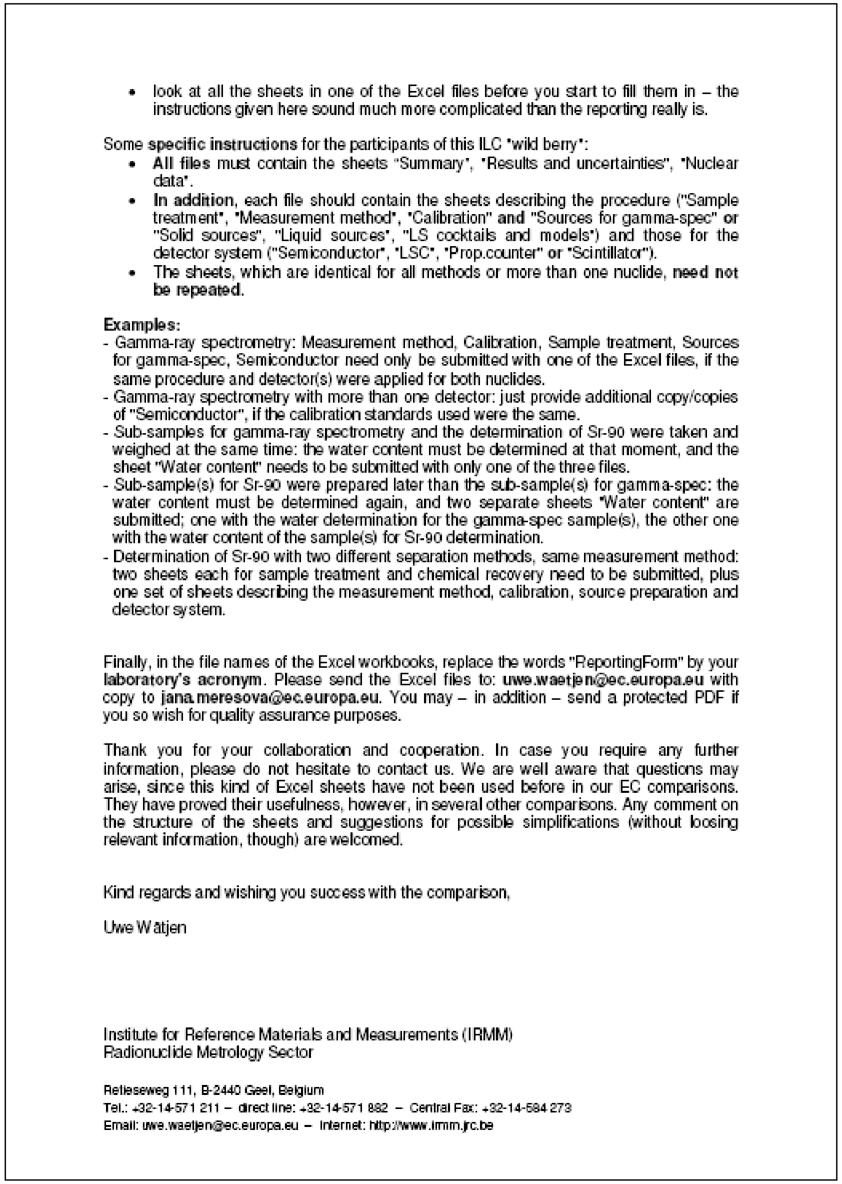

- look at all the sheets in one of the Excel files before you start to fill them in – the instructions given here sound much more complicated than the reporting really is.

Some **specific instructions** for the participants of this ILC "wild berry":

- **All files** must contain the sheets "Summary", "Results and uncertainties", "Nuclear data".
- **In addition**, each file should contain the sheets describing the procedure ("Sample treatment", "Measurement method", "Calibration" **and** "Sources for gamma-spec" **or** "Solid sources", "Liquid sources", "LS cocktails and models") and those for the detector system ("Semiconductor", "LSC", "Prop.counter" **or** "Scintillator").
- The sheets, which are identical for all methods or more than one nuclide, **need not be repeated.**

Examples:

- Gamma-ray spectrometry: Measurement method, Calibration, Sample treatment, Sources for gamma-spec, Semiconductor need only be submitted with one of the Excel files, if the same procedure and detector(s) were applied for both nuclides.
- Gamma-ray spectrometry with more than one detector: just provide additional copy/copies of "Semiconductor", if the calibration standards used were the same.
- Sub-samples for gamma-ray spectrometry and the determination of Sr-90 were taken and weighed at the same time: the water content must be determined at that moment, and the sheet "Water content" needs to be submitted with only one of the three files.
- Sub-sample(s) for Sr-90 were prepared later than the sub-sample(s) for gamma-spec: the water content must be determined again, and two separate sheets "Water content" are submitted; one with the water determination for the gamma-spec sample(s), the other one with the water content of the sample(s) for Sr-90 determination.
- Determination of Sr-90 with two different separation methods, same measurement method: two sheets each for sample treatment and chemical recovery need to be submitted, plus one set of sheets describing the measurement method, calibration, source preparation and detector system.

Finally, in the file names of the Excel workbooks, replace the words "ReportingForm" by your **laboratory's acronym**. Please send the Excel files to: **uwe.waetjen@ec.europa.eu** with copy to **jana.meresova@ec.europa.eu**. You may – in addition – send a protected PDF if you so wish for quality assurance purposes.

Thank you for your collaboration and cooperation. In case you require any further information, please do not hesitate to contact us. We are well aware that questions may arise, since this kind of Excel sheets have not been used before in our EC comparisons. They have proved their usefulness, however, in several other comparisons. Any comment on the structure of the sheets and suggestions for possible simplifications (without loosing relevant information, though) are welcomed.

Kind regards and wishing you success with the comparison,

Uwe Wätjen

Institute for Reference Materials and Measurements (IRMM)
Radionuclide Metrology Sector

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Tel.: +32-14-571 211 – direct line: +32-14-571 882 – Central Fax: +32-14-584 273
Email: uwe.waetjen@ec.europa.eu – Internet: http://www.irmm.jrc.be

Appendix 5: Clarification on uncertainty reporting (e-mail)

Subject: Clarification on uncertainty reporting - EC "wild berry" comparison
Sent: 9 June 2011

Dear colleague,

I have been asked by one participant, whether uncertainties should be reported at the one or two sigma level. I would like to forward my answer to all participants:

According to the GUM, the terms

standard uncertainty, combined *standard* uncertainty or relative *standard* uncertainty and combined relative *standard* uncertainty (and only these terms are used in the reporting forms)

all refer to the **"one sigma" or s** level.

GUM 2.3.1 standard uncertainty

uncertainty of the result of a measurement ***expressed as a standard deviation***

GUM 2.3.4 combined standard uncertainty

standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the ***positive square root of a sum of terms, the terms being the variances or covariances*** *of these other quantities weighted according to how the measurement result varies with changes in these quantities*

Another clarification, but probably self-evident:

Whenever results are to be reported (in the sheets Summary, Individual results, top of Results and uncertainties), the corresponding uncertainty is **combined** (relative) standard uncertainty.

Kind regards,

Uwe Wätjen

Appendix 6: Clarification on question in reporting forms (e-mail)

Subject: EC "wild berry" comparison - clarification on question in reporting forms
Sent: 11 August 2011

Dear participants,

One of you pointed out the mistake we have made in the reporting forms (Excel files). In the sheet “measurement method” is question: “How many measurements of this type your laboratory perform per year in soil samples?” Of course, this question is related to the foodstuff samples (not to soil samples). We are interested in the number of foodstuff samples that your laboratory performs per year?

We are sorry for this inconvenience and looking forward to receiving your results.

Best regards,

Uwe Wätjen and Jana Meresova

Appendix 7: Clarification about uncertainties (e-mail)

Subject:
Sent: 28 September 2011

Dear participants,

In the reporting sheets for the present interlaboratory comparison on 90Sr, 137Cs and 40K in wild bilberry powder you have been asked to report **standard uncertainty** and **relative standard uncertainty** (all refer to the **"one sigma" or 1s** level).

In June, another e-mail was also sent to you in order to clarify this topic. However, since the uncertainty is playing a crucial role in the evaluation of your results, we want to make sure what kind of uncertainty you submitted.

In case you have reported **the expanded uncertainty**, please let us know together with the value of **the coverage factor k** you have used.

If you have reported the standard uncertainty (not expanded), please disregard this e-mail.

Thank you for your participation in the intercomparison.

Kind regards,
Jana Meresova

Some definitions from **Evaluation of measurement data — Guide to the expression of uncertainty in measurement (GUM)**

GUM 2.3.1 **standard uncertainty**
- uncertainty of the result of a measurement expressed as a standard deviation

GUM 2.3.4 **combined standard uncertainty**
- standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

GUM 2.3.5 **expanded uncertainty**
- quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand.

GUM 2.3.6 **coverage factor**
- numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty
NOTE A coverage factor, k, is typically in the range 2 to 3.

Appendix 8: List of laboratories, nominated to participate

ALBANIA

Dr Elida Bylyku
Centre of Applied Nuclear Physics
Qesarake street, 1000 Tirana

AUSTRIA

Mag Claudia Landstetter
Austrian Agency for Health and Food Safety
CC Radiation Protection and Radiochemistry
Spargelfeldstrasse 191, 1220 Vienna

Arno Achatz
Austrian Agency for Health and Food Safety
CC Radioecology and Radon
Wieningerstrasse 8, 4020 Linz

Bernd Obenaus
Austrian Agency for Health and Food Safety
Institute for Food Control
Beethovenstrasse 8, 8010 Graz

Alan Tessadri — Did not participate – no response given
Austrian Agency for Health and Food Safety
Institute for Food Control, Innsbruck
Technikerstrasse 70, 6020 Innsbruck

BELGIUM

Peter Vermaercke
SCK•CEN
Low-Level Radioactivity Measurements
Boeretang 200, 2400 Mol

Benoit Deconninck
IRE ELIT
Avenue de l'espérance, 1, 6220 Fleurus

BOSNIA - HERZEGOVINA

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

BULGARIA

Rositza Totzeva
National Center of Radiobiology and Radiation Protection
Radiation Monitoring Department
Public Exposure Monitoring Laboratory
3 "Georgi Sofiiski" Blvd, 1606 Sofia

Mihail Shishenkov
Executive Environment Agency
Radioactivity Measurements Laboratory
136 Tzar Boris III Blvd., 1618 Sofia

Rumyana Mitkova
Executive Environment Agency
Vraca Regional Laboratory Department
81 “Ekzarh Josif" Str., 3000 Vratza

Any Krasteva
Executive Environment Agency
Montana Regional Laboratory Department
4 “Julius Irasec” Str., 3400 Montana

Marieta Vasileva
Executive Environment Agency
Pleven Regional Laboratory Department
Storgoziya District, “Measures and Measuring Devices”, RO Building, 5800 Pleven

CROATIA

Dr Zeljko Grahek
Rudjer Boskovic Institute
Laboratory for radioecology
Bijenicka 54, 10002 Zagreb

Tomislav Bituh
Inst. for Medical Research and Occupational Health
Radiation Protection Unit
Ksaverska cesta 2, 10001 Zagreb

CYPRUS

Anastasia Caballero — Did not participate – problems with detector
State General Laboratory of Cyprus
Radioactivity Laboratory
Kimonos 44, 1451 Nicosia

CZECH REPUBLIC

Dr Petr Rulík
National Radiation Protection Institute
Bartoskova 28, 140 00 Prague

Jan Rosmus
State Veterinary Institute Prague
Sidlistni 24, 165 03 Prague

DENMARK

Henrik Roed
National Institute of Radiation Protection
Knapholm 7, 2730 Herlev

Dr Sven Nielsen
Riso National Laboratory for Sustainable Energy
Technical University of Denmark
Radiation Research,
Frederiksborgvej 399, 4000 Roskilde

ESTONIA

Kadri Isakar
University of Tartu, Institute of Physics
Laboratory of Environmental Physics
Riia 142, 51014 Tartu

Eia Jakobson
Environmental Board
Radiation Safety Department
Kopli 76, 10416 Tallinn

FINLAND

Pia Vesterbacka
STUK
Laboratory of Radionuclide Analytics
Laippatie 4, 00881 Helsinki

FRANCE

Dr Cédric Aubert, Roselyne Ameon
IRSN/DEI/STEME
Laboratoire des Etalons et Intercomparaisons
31 rue de l'ecluse, 78116 Le Vesinet

GERMANY

Dr David Tait
Max Rubner-Institut
Bundesforschungsinstitut für Ernährung und Lebensmittel
Institut für Sicherheit und Qualität bei Milch und Fisch
Leitstelle für die Überwachung der Radioaktivität in der Umwelt
Hermann-Weigmann-Str. 1, 24103 Kiel

GREECE

Dr Panagiotis Kritidis
National Centre for Scientific Research "DEMOKRITOS"
Environmental Radioactivity Laboratory
Patriarchou Grigoriou & Neapoleos, 15310 Aghia Paraskevi

Dr Constantinos Potiriadis
Greek Atomic Energy Commission
Environmental Radioactivity Monitoring Department
Patriarchou Grigoriou & Neapoleos, 15310 Aghia Paraskevi

HUNGARY

Dr Sándor Tarján
Central Agricultural Office
Food and Feed Safety Directorate
Radioanalytical Reference Laboratory
Fogoly str 13-15, 1182 Budapest

IRELAND

Jennie Wong
Radiological Protection Institute of Ireland
Radiation Monitoring
3 Clonskeagh Square, Dublin 14

ITALY

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Dr Leandro Magro
Istituto Superiore per la Protezione e la Ricerca Ambientale (ISPRA)
Servizio Misure Radiometriche
Settore Sorgenti Naturali
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Dr Rosella Rusconi
ARPA Lombardia
U.O. Agenti Fisici
Via Juvara, 22, 20129 Milano

Dr Roberto Sogni
ARPA Emilia Romagna
Sezione Provinciale di Piacenza
Via XXI Aprile, 82, 29121 Piacenza

Dr Luigi Vitucci
ARPA Puglia - DAP Bari
U.O.S. Polo di Specializzazione Radiazioni Ionizzanti
Via Piccinni, 164, 70024 Bari

Massimo Cappai
ARPA Sardegna
Direzione Tecnico Scientifica
Servizio progettazione ricerca e sviluppo
Via Palabanda, 9, 09123 Cagliari

Dr Corrado Pantalone
ARPA delle Marche
Servizio Radiazioni/Rumore
Dipartimento Prov.le di Ancona
via Colombo, 106, 60127 Ancona

Dr.ssa Carmela P. Fortunato
ARPA Basilicata
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c/o Dipartimento Provinciale
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Silvia Bucci
ARPA Toscana
Dipartimento Provinciale di Firenze
via Ponte alle Mosse, 211, 50144 Firenze

Raffaella Trozzo
ARPA Calabria
Dipartimento Provinciale di Cosenza
Servizio Laboratorio Fisico
Via Trento, 21, 87100 Cosenza

Paola Sabatini
ARPA Umbria
U.O Laboratorio Perugia
Via Pievaiola, 207 B-3, 06132 San Sisto (Perugia)

Laura Porzio
ARPA Piemonte
Dipartimento Tematico Radiazioni
Struttura Semplice Siti Nucleari
Via Trino, 89, 13100 Vercelli

Luca Amendola
ARPA Lazio
Unità Alimenti
Via Saredo, 52, 00173 Roma

LATVIA

Konstantins Bavrins
State Ltd "Latvian Environment
Geology and Meteorology Centre"
Environmental Laboratory
Maskavas street 165, 1019 Riga

Jelizaveta Cernihovica
Institute of Food Safety
Animal Health and Environment "BIOR"
Laboratory of Food and Environmental Investigations
Lejupas street 3, 1076 Riga

LITHUANIA

Dr Vladimir Vlaskin
Ignalina Nuclear Power Plant
Radiation Safety Service
Laboratory of Environment Monitoring
Druksiniu kaimas, 31500 Visaginas

Rima Ladygiene
Radiation Protection Centre
Department of Expertise and Exposure Monitoring
Division of Public Exposure Monitoring
Kalvarijų 153, 08221 Vilnius

Pranas Drulia
National Food and Veterinary Risk Assessment Institute
Laboratory Department
Radiology unit
J. Kairiukscio st. 10, 08409 Vilniuis

LUXEMBOURG

Dr Marielle Lecomte
Ministère de la Santé
Division de la Radioprotection
Villa Louvigny, Allée Marconi, 2120 Luxembourg

FYR of MACEDONIA

Zdenka Stojanovska, Jovan Janusevski
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Evans Building, Lower Merchants Street, VLT1179 Valletta

MONTENEGRO

Tomislav Andjelic
Center for Eco-Toxicological Research of Montenegro
Radiation Protection and Monitoring Department
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THE NETHERLANDS

Dr Gerard Krijger, Mark van Bourgondien
RIKILT - Instituut voor Voedselveiligheid
Akkermaalsbos 2, 6708WB Wageningen

Dr Pieter Kwakman Did not participate – not fit for food samples
RIVM
Laboratorium voor Stralingsonderzoek
Anthonie van Leeuwenhoeklaan 9, 3720MA Bilthoven

NORWAY

Mr Bredo Møller
Norwegian Radiation Protection Authority (NRPA)
Svanhovd Emergency Preparedness Unit
9925 Svanvik

Anna Nalbandyan
Norwegian Radiation Protection Authority
The Fram Centre
Hjalmar Johansensgate 14, 9296 Tromsø

POLAND

Dr Zbigniew Haratym
National Centre for Nuclear Research
former Institute of Atomic Energy POLATOM
Radiation Protection Measurement Laboratory
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Dr hab Jerzy Wojciech Mietelski
The Henryk Niewodniczanski Institute of Nuclear Physics,
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Dr Tamara Zalewska
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ROMANIA

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Centrul Regional de Sanatate Publica Cluj-Napoca
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Camino de Vera s/n, 46071 Valencia

Catalina Gascó Leonarte
Unidad de Radiactividad Ambiental y Vigilancia Radiológica
Departamento de Medio Ambiente, CIEMAT
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Departamento de Ingeniería Civil II: Hidráulica y Energética
Universidad Politécnica de Madrid
ETS de Ingenieros de Caminos, Ca-nales y Puertos, 28040 Madrid

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Carmen Pérez Marín
Cátedra de Física Atómica, Molecular y Nuclear
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Bundesamt für Gesundheit
Abteilung Verbraucherschutz
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Dr Pascal Froidevaux
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TURKEY

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Turkish Atomic Energy Authority
Cekmece Nuclear Research and Training Center
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Paul Smedley
CEFAS
Radiological and Chemical Risk
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Tony Dell
Veterinary Laboratories Agency
Radiochemistry Unit (LS6)
Woodham Lane, New Haw, Addlestone, Surrey, KT15 3NB

Appendix 9: Information letter on preliminary results

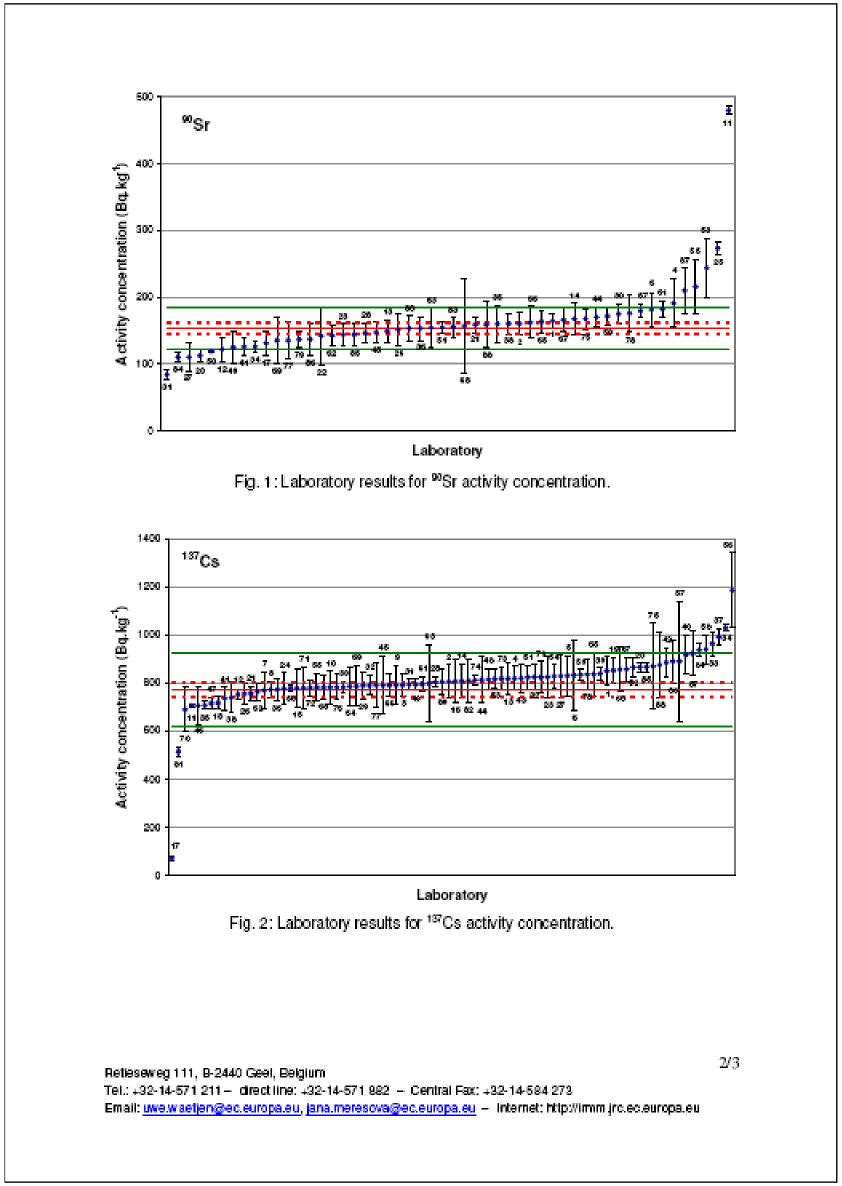

Fig. 1: Laboratory results for ^{90}Sr activity concentration.

Fig. 2: Laboratory results for ^{137}Cs activity concentration.

2/3

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Tel.: +32-14-571 211 – direct line: +32-14-571 882 – Central Fax: +32-14-584 273
Email: uwe.waetjen@ec.europa.eu, jana.meresova@ec.europa.eu – Internet: http://irmm.jrc.ec.europa.eu

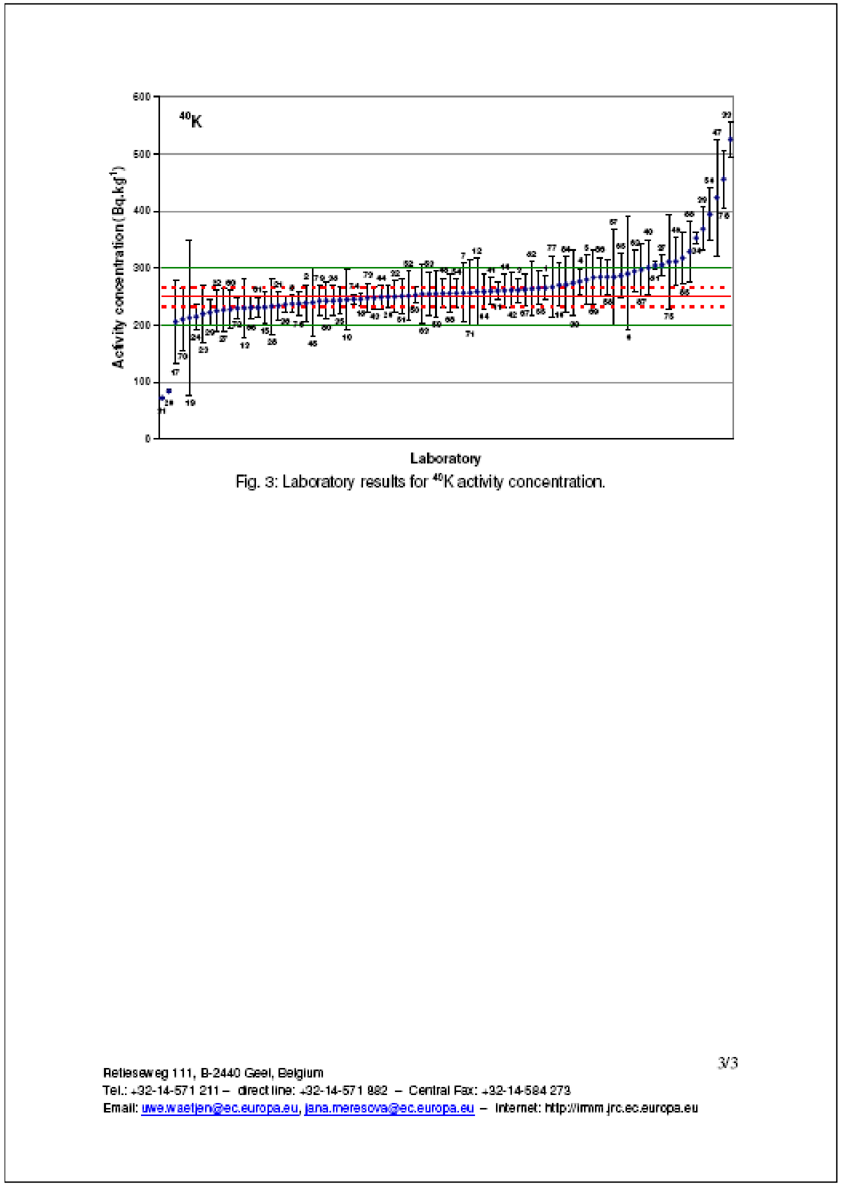
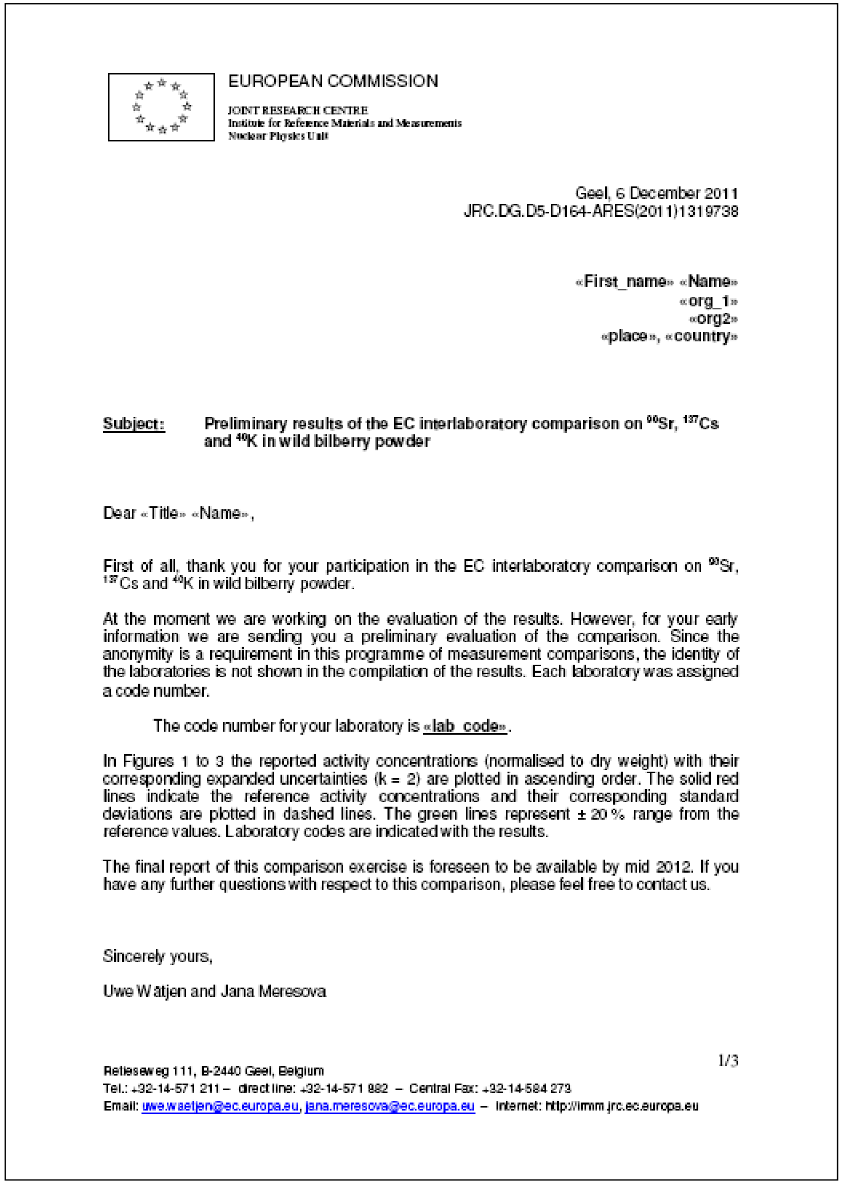


Fig. 3: Laboratory results for ^{40}K activity concentration.



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements
Nuclear Physics Unit

Geel, 6 December 2011
JRC.DG.D5-D164-ARES(2011)1319738

«First_name» «Name»
«org_1»
«org2»
«place», «country»

Subject: **Preliminary results of the EC interlaboratory comparison on ^{90}Sr, ^{137}Cs and ^{40}K in wild bilberry powder**

Dear «Title» «Name»,

First of all, thank you for your participation in the EC interlaboratory comparison on ^{90}Sr, ^{137}Cs and ^{40}K in wild bilberry powder.

At the moment we are working on the evaluation of the results. However, for your early information we are sending you a preliminary evaluation of the comparison. Since the anonymity is a requirement in this programme of measurement comparisons, the identity of the laboratories is not shown in the compilation of the results. Each laboratory was assigned a code number.

The code number for your laboratory is «lab_code».

In Figures 1 to 3 the reported activity concentrations (normalised to dry weight) with their corresponding expanded uncertainties (k = 2) are plotted in ascending order. The solid red lines indicate the reference activity concentrations and their corresponding standard deviations are plotted in dashed lines. The green lines represent ± 20 % range from the reference values. Laboratory codes are indicated with the results.

The final report of this comparison exercise is foreseen to be available by mid 2012. If you have any further questions with respect to this comparison, please feel free to contact us.

Sincerely yours,

Uwe Wätjen and Jana Meresova

Appendix 10: Results, methods and scores of laboratories

Tables 1 to 3 present the laboratory results A_{lab} with their expanded uncertainties U_{lab} (k = 2). The values are listed as reported by the participants, but standard uncertainties were recalculated to expanded uncertainties. Information on the method used is included as well as information on whether the result was indicated by the Grubbs' test (α = 1 %) as an outlier. The performance evaluation by relative deviation $D_{\%}$ and E_n criterion are given in the last two columns. Red colour indicates results deviating more than 20 % from the reference value or E_n incompatible results (with "action signal"). E_n numbers in yellow colour indicate incompatible results at the "warning level".

Table 1. Activity concentrations of ^{40}K and their expanded uncertainties (k = 2) together with the laboratory scores $D_{\%}$ and E_n.

Lab. code	Laboratory's result		Used method	Outlier	$D_{\%}$ (%)	E_n
	A_{lab} ± U_{lab} (Bq kg^{-1})	$U_{\%}$ (%)				
1	265.78 ± 20.74	7.8	Direct gamma-spec.	-	**5**	**0.5**
2	239 ± 32	13.4	Direct gamma-spec.	-	**-6**	**-0.4**
3	261.71 ± 20.94	8.0	Direct gamma-spec.	-	**3**	**0.3**
4	277 ± 22	7.9	Direct gamma-spec.	-	**9**	**0.9**
5	280 ± 44	15.7	Direct gamma-spec.	-	**11**	**0.6**
6	290.5 ± 99.54	34.3	Direct gamma-spec.	-	**15**	**0.4**
7	257 ± 52	20.2	Direct gamma-spec.	-	**2**	**0.1**
8	237.7 ± 15.6	6.6	Direct gamma-spec.	-	**-6**	**-0.7**
10	245 ± 54	22.0	Direct gamma-spec.	-	**-3**	**-0.1**
11	260.41 ± 14.92	5.7	Direct gamma-spec.	-	**3**	**0.4**
12	258.92 ± 58.84	22.7	Direct gamma-spec.	-	**2**	**0.1**
13	230 ± 52	22.6	Direct gamma-spec.	-	**-9**	**-0.4**
14	261 ± 30	11.5	Direct gamma-spec.	-	**3**	**0.2**
15	231.56 ± 28.36	12.2	Direct gamma-spec.	-	**-8**	**-0.7**
16	271 ± 38	14.0	Direct gamma-spec.	-	**7**	**0.4**
17	205.93 ± 74.14	36.0	Flame spectrophotometry	-	**-19**	**-0.6**
18	246 ± 11	4.5	Direct gamma-spec.	-	**-3**	**-0.4**
19	213.2 ± 136	63.8	Direct gamma-spec.	-	**-16**	**-0.3**
20	84.25 ± 3	3.6	Direct gamma-spec.	yes	**-67**	**-11.0**
21	234 ± 24	10.3	Direct gamma-spec.	-	**-8**	**-0.7**
22	225 ± 36	16.0	Direct gamma-spec.	-	**-11**	**-0.7**
23	220 ± 50	22.7	Direct gamma-spec.	-	**-13**	**-0.6**
24	214.89 ± 22.2	10.3	Direct gamma-spec.	-	**-15**	**-1.4**
26	249.82 ± 20.0	8.0	Direct gamma-spec.	-	**-1**	**-0.1**
27	227 ± 36.8	16.2	Direct gamma-spec.	-	**-10**	**-0.7**
28	233.4 ± 49	21.0	Direct gamma-spec.	-	**-8**	**-0.4**
29	222.13 ± 22.43	10.1	Direct gamma-spec.	-	**-12**	**-1.1**
30	274 ± 57.6	21.0	Direct gamma-spec.	-	**8**	**0.4**
31	72.42 ± 1.65	2.3	Direct gamma-spec.	yes	**-71**	**-12.0**
32	250 ± 28	11.2	Direct gamma-spec.	-	**-1**	**-0.1**
33	525.7 ± 30.91	5.9	Direct gamma-spec.	yes	**108**	**7.9**
34	353 ± 8.9	2.5	Direct gamma-spec.	yes	**40**	**5.7**
35	244 ± 24	9.8	Direct gamma-spec.	-	**-4**	**-0.3**
36	236.4 ± 14.6	6.2	Direct gamma-spec.	-	**-7**	**-0.8**
37	306 ± 17.9	5.8	Direct gamma-spec.	-	**21**	**2.3**
38	243.1 ± 26.4	10.9	Direct gamma-spec.	-	**-4**	**-0.3**
39	369 ± 38	10.3	Direct gamma-spec.	yes	**46**	**2.8**

40	301 ± 48.4	16.1	Direct gamma-spec.	-	**19**	**0.9**
41	260 ± 24	9.2	Direct gamma-spec.	-	**3**	**0.2**
42	261.67 ± 31.4	12.0	Direct gamma-spec.	-	**3**	**0.2**
43	248 ± 20	8.1	Direct gamma-spec.	-	**-2**	**-0.2**
44	249.2 ± 20.4	8.2	Direct gamma-spec.	-	**-2**	**-0.2**
45	240 ± 60	25.0	Direct gamma-spec.	-	**-5**	**-0.2**
47	424 ± 102	24.1	Direct gamma-spec.	yes	**68**	**1.7**
48	256 ± 26	10.2	Direct gamma-spec.	-	**1**	**0.1**
49	312 ± 42	13.5	Direct gamma-spec.	-	**23**	**1.3**
50	253 ± 14	5.5	Direct gamma-spec.	-	**0**	**0.0**
51	251.10 ± 29.61	11.8	Direct gamma-spec.	-	**-1**	**-0.1**
52	251.74 ± 44.17	17.5	Direct gamma-spec.	-	**0**	**0.0**
53	255 ± 38	14.9	Direct gamma-spec.	-	**1**	**0.0**
54	256.5 ± 24.34	9.5	Direct gamma-spec.	-	**1**	**0.1**
55	265.49 ± 29	10.9	Direct gamma-spec.	-	**5**	**0.4**
56	394.5 ± 46	11.7	Direct gamma-spec.	yes	**56**	**2.9**
57	285 ± 84	29.5	Direct gamma-spec.	-	**13**	**0.4**
58	285 ± 30	10.5	Direct gamma-spec.	-	**13**	**1.0**
59	255.43 ± 40.14	15.7	Direct gamma-spec.	-	**1**	**0.1**
60	228 ± 34	14.9	Direct gamma-spec.	-	**-10**	**-0.7**
61	231 ± 18	7.8	Direct gamma-spec.	-	**-9**	**-0.9**
62	294.53 ± 36.25	12.3	Direct gamma-spec.	-	**16**	**1.1**
63	255 ± 52	20.4	Direct gamma-spec.	-	**1**	**0.0**
64	259 ± 30	11.6	Direct gamma-spec.	-	**2**	**0.2**
65	287 ± 40	13.9	Direct gamma-spec.	-	**13**	**0.8**
66	231 ± 20	8.7	Direct gamma-spec.	-	**-9**	**-0.9**
67	263 ± 28	10.6	Direct gamma-spec.	-	**4**	**0.3**
68	256 ± 34	13.3	Direct gamma-spec.	-	**1**	**0.1**
69	284 ± 48	16.9	Direct gamma-spec.	-	**12**	**0.6**
70	210.36 ± 53.58	25.5	Direct gamma-spec.	-	**-17**	**-0.8**
71	257.4 ± 56.6	22.0	Direct gamma-spec.	-	**2**	**0.1**
72	229.937 ± 16.97	7.4	Direct gamma-spec.	-	**-9**	**-1.0**
73	248 ± 26	10.5	Direct gamma-spec.	-	**-2**	**-0.2**
74	246 ± 8	3.3	Direct gamma-spec.	-	**-3**	**-0.4**
75	311.2 ± 82.4	26.5	Direct gamma-spec.	-	**23**	**0.7**
76	238 ± 22	9.2	Direct gamma-spec.	-	**-6**	**-0.6**
77	267 ± 54	20.2	Direct gamma-spec.	-	**6**	**0.2**
78	456 ± 50	11.0	Direct gamma-spec.	yes	**80**	**3.9**
79	242.5 ± 26.6	11.0	Direct gamma-spec.	-	**-4**	**-0.3**
80	243 ± 32	13.2	Direct gamma-spec.	-	**-4**	**-0.3**
81	305.03 ± 6.61	2.2	Direct gamma-spec.	-	**21**	**3.2**
82	264 ± 48	18.2	Direct gamma-spec.	-	**4**	**0.2**
84	271.6 ± 47.8	17.6	Direct gamma-spec.	-	**7**	**0.4**
85	318 ± 46	14.5	Direct gamma-spec.	-	**26**	**1.3**
86	285 ± 34	11.9	Direct gamma-spec.	-	**13**	**0.9**
87	297.4 ± 45.4	15.3	Direct gamma-spec.	-	**18**	**0.9**
88	329 ± 54	16.4	Direct gamma-spec.	-	**30**	**1.4**

Table 2. Activity concentrations of ^{90}Sr and their expanded uncertainties (k = 2) together with the laboratory scores $D_{\%}$ and E_n.

Lab. code	Laboratory's result		Used method	Outlier	$D_{\%}$ (%)	E_n
	$A_{lab} \pm U_{lab}$ (Bq kg^{-1})	$U_{\%}$ (%)				
1	164.27 ± 11.42	7.0	Proportional counting	-	7	0.8
2	160.3 ± 16.8	10.5	LSC	-	5	0.4
4	191 ± 36	18.8	Proportional counting	-	25	1.0
6	181 ± 26	14.4	LSC	-	18	1.0
11	480.02 ± 5.92	1.2	LSC	yes	214	32.9
12	121.89 ± 17.92	14.7	Proportional counting	-	-20	-1.6
13	149.3 ± 17.0	11.4	Proportional counting	-	-2	-0.2
14	167.6 ± 23.4	14.0	LSC	-	10	0.6
17	131.17 ± 18.36	14.0	Solid scintillation detector	-	-14	-1.1
20	112.267 ± 9.72	8.7	Proportional counting	-	-27	-3.2
21	159 ± 12	7.5	LSC	-	4	0.4
22	141.79 ± 43.32	30.6	Proportional counting	-	-7	-0.3
23	144 ± 16	11.1	Cherenkov counting	-	-6	-0.5
25	273.308 ± 9.68	3.5	Proportional counting	yes	79	9.6
26	151.1 ± 23.07	15.3	LSC	-	-1	-0.1
27	110 ± 20.8	18.9	Proportional counting	-	-28	-1.9
28	146 ± 15.2	10.4	LSC	-	-5	-0.4
30	174.93 ± 13.4	7.7	LSC	-	14	1.4
31	84.213 ± 8.05	9.6	Proportional counting	-	-45	-6.1
34	126 ± 8.2	6.5	LSC	-	-18	-2.4
35	160 ± 28	17.5	LSC	-	5	0.2
36	153 ± 18	11.8	LSC	-	0	0.0
38	160.24 ± 15.5	9.7	Cherenkov counting	-	5	0.4
41	126 ± 12.6	10.0	Proportional counting	-	-18	-1.8
44	170 ± 14	8.2	Proportional counting	-	11	1.1
45	147 ± 16	10.9	Proportional counting	-	-4	-0.3
49	125 ± 24	19.2	LSC	-	-18	-1.1
50	119 ± 1.02	0.9	Proportional counting	-	-22	-4.2
51	154.4 ± 7.433	4.8	Proportional counting	-	1	0.1
53	244 ± 44	18.0	Proportional counting	yes	59	2.0
55	216 ± 41.2	19.1	Proportional counting	-	41	1.5
57	179.4 ± 9.2	5.1	Proportional counting	-	17	2.2
59	171.36 ± 12.26	7.2	GM	-	12	1.3
61	182 ± 13	7.1	Proportional counting	-	19	1.9
62	142.27 ± 14.96	10.5	Cherenkov counting	-	-7	-0.6
63	154 ± 29.6	19.2	Proportional counting	-	1	0.0
65	163 ± 16	9.8	LSC	-	7	0.6
66	162 ± 24	14.8	Proportional counting	-	6	0.4
67	166 ± 18	10.8	Cherenkov counting	-	8	0.7
68	157 ± 70	44.6	Solid scintillation detector	-	3	0.1
69	135 ± 34	25.2	Solid scintillation detector	-	-12	-0.5
75	167.6 ± 15.6	9.3	Solid scintillation detector	-	10	0.8
77	135 ± 28	20.7	GM	-	-12	-0.6
78	175.7 ± 28.4	16.2	Proportional counting	-	15	0.8
79	136.7 ± 11.2	8.2	Proportional counting	-	-11	-1.2
80	153 ± 20	13.1	Proportional counting	-	0	0.0
83	155.5 ± 15.6	10.0	Proportional counting	-	2	0.1
84	110 ± 6.6	6.0	GM	-	-28	-4.1
85	144 ± 16	11.1	Proportional counting	-	-6	-0.5
86	137 ± 24.6	18.0	LSC	-	-10	-0.6
87	210 ± 34	16.2	Proportional counting	-	37	1.6
88	159 ± 34	21.4	Proportional counting	-	4	0.2

Table 3. Activity concentrations of ^{137}Cs and their expanded uncertainties (k = 2) together with the laboratory scores $D_{\%}$ and E_n.

Lab. code	**Laboratory's result**		**Used method**	**Outlier**	**$D_{\%}$ (%)**	**E_n**
	$A_{lab} \pm U_{lab}$ (Bq kg^{-1})	**$U_{\%}$ (%)**				
1	850.77 ± 62.08	7.3	Direct gamma-spec.	-	**9**	**1.1**
2	806 ± 70	8.68	Direct gamma-spec.	-	**3**	**0.4**
3	791.89 ± 47.52	6	Direct gamma-spec.	-	**2**	**0.2**
4	819 ± 50	6.11	Direct gamma-spec.	-	**5**	**0.7**
5	830 ± 84	10.1	Direct gamma-spec.	-	**7**	**0.6**
6	832.4 ± 145.4	17.5	Direct gamma-spec.	-	**7**	**0.4**
7	769 ± 78	10.1	Direct gamma-spec.	-	**-1**	**-0.1**
8	772.7 ± 31	4.01	Direct gamma-spec.	-	**-1**	**-0.2**
9	791 ± 79	9.99	Direct gamma-spec.	-	**2**	**0.1**
10	782.6 ± 69.2	8.84	Direct gamma-spec.	-	**0**	**0.0**
11	705.1 ± 5.72	0.81	Direct gamma-spec.	-	**-9**	**-3.0**
12	752.18 ± 20.08	2.67	Direct gamma-spec.	-	**-3**	**-0.9**
13	818 ± 68	8.31	Direct gamma-spec.	-	**5**	**0.5**
14	808 ± 72	8.91	Direct gamma-spec.	-	**4**	**0.4**
15	779.019 ± 82.53	10.6	Direct gamma-spec.	-	**0**	**0.0**
16	807 ± 90	11.2	Direct gamma-spec.	-	**4**	**0.3**
17	71.653 ± 10.142	14.2	Beta counting after radiochemical separation	yes	**-91**	**-27.1**
18	716 ± 26	3.63	Direct gamma-spec.	-	**-8**	**-1.8**
19	852.5 ± 52.8	6.19	Direct gamma-spec.	-	**9**	**1.3**
20	865.61 ± 17.68	2.04	Direct gamma-spec.	-	**11**	**2.9**
21	756 ± 32	4.23	Direct gamma-spec.	-	**-3**	**-0.6**
22	824 ± 52	6.31	Direct gamma-spec.	-	**6**	**0.8**
23	825 ± 86	10.4	Direct gamma-spec.	-	**6**	**0.5**
24	776.83 ± 64.86	8.35	Direct gamma-spec.	-	**0**	**0.0**
26	755.38 ± 45.95	6.08	Direct gamma-spec.	-	**-3**	**-0.5**
27	829 ± 89.2	10.8	Direct gamma-spec.	-	**6**	**0.5**
28	803.3 ± 18.4	2.29	Direct gamma-spec.	-	**3**	**0.8**
29	789 ± 54.50	6.91	Direct gamma-spec.	-	**1**	**0.2**
30	783 ± 25	3.19	Direct gamma-spec.	-	**1**	**0.1**
31	793.77 ± 22.7	2.86	Direct gamma-spec.	-	**2**	**0.4**
32	789 ± 40	5.07	Direct gamma-spec.	-	**1**	**0.2**
33	963.55 ± 48.96	5.08	Direct gamma-spec.	-	**24**	**3.4**
34	1029 ± 12.8	1.24	Direct gamma-spec.	yes	**32**	**9.2**
35	707.2 ± 17.8	2.52	Direct gamma-spec.	-	**-9**	**-2.4**
36	773 ± 48	6.21	Direct gamma-spec.	-	**-1**	**-0.1**
37	991 ± 35.2	3.55	Direct gamma-spec.	-	**27**	**5.0**
38	739.1 ± 62.8	8.5	Direct gamma-spec.	-	**-5**	**-0.6**
39	839 ± 26	3.1	Direct gamma-spec.	-	**8**	**1.7**
40	917 ± 81.6	8.9	Direct gamma-spec.	-	**18**	**1.6**
41	736 ± 52	7.07	Direct gamma-spec.	-	**-6**	**-0.8**
42	885.06 ± 61.98	7	Direct gamma-spec.	-	**14**	**1.6**
43	820 ± 60	7.32	Direct gamma-spec.	-	**5**	**0.6**
44	814 ± 98	12	Direct gamma-spec.	-	**4**	**0.3**
45	790 ± 120	15.2	Direct gamma-spec.	-	**1**	**0.1**
46	705.36 ± 78.3	11.1	Direct gamma-spec.	-	**-9**	**-0.9**
47	715 ± 22	3.08	Direct gamma-spec.	-	**-8**	**-2.0**
48	815 ± 46	5.64	Direct gamma-spec.	-	**5**	**0.7**
49	794 ± 22	2.77	Direct gamma-spec.	-	**2**	**0.5**
50	779 ± 16	2.05	Direct gamma-spec.	-	**0**	**0.0**
51	823 ± 48.17	5.85	Direct gamma-spec.	-	**6**	**0.8**
52	864.88 ± 39.79	4.6	Direct gamma-spec.	-	**11**	**1.8**
53	817 ± 40	4.9	Direct gamma-spec.	-	**5**	**0.8**

54	828.5 ± 46.74	5.64	Direct gamma-spec.	-	**6**	**0.9**
55	781.42 ± 59.06	7.56	Direct gamma-spec.	-	**0**	**0.0**
56	1185.3 ± 155.2	13.1	Direct gamma-spec.	yes	**52**	**2.6**
57	890 ± 250	28.1	Direct gamma-spec.	-	**14**	**0.4**
58	940 ± 60	6.38	Direct gamma-spec.	-	**21**	**2.5**
59	833.72 ± 22.38	2.68	Direct gamma-spec.	-	**7**	**1.7**
60	856 ± 94	11	Direct gamma-spec.	-	**10**	**0.8**
61	795 ± 30	3.77	Direct gamma-spec.	-	**2**	**0.4**
62	762.78 ± 38.21	5.01	Direct gamma-spec.	-	**-2**	**-0.4**
63	797 ± 160	20.1	Direct gamma-spec.	-	**2**	**0.1**
64	786 ± 80	10.2	Direct gamma-spec.	-	**1**	**0.1**
65	836 ± 88	10.5	Direct gamma-spec.	-	**7**	**0.6**
66	791 ± 44	5.56	Direct gamma-spec.	-	**2**	**0.2**
67	857 ± 50	5.83	Direct gamma-spec.	-	**10**	**1.4**
68	782 ± 52	6.65	Direct gamma-spec.	-	**0**	**0.1**
69	787 ± 84	10.7	Direct gamma-spec.	-	**1**	**0.1**
70	689.76 ± 92.3	13.4	Direct gamma-spec.	-	**-11**	**-0.9**
71	780.3 ± 87.7	11.2	Direct gamma-spec.	-	**0**	**0.0**
72	780.72 ± 33.73	4.32	Direct gamma-spec.	-	**0**	**0.0**
73	818 ± 46	5.62	Direct gamma-spec.	-	**5**	**0.8**
74	812 ± 22	2.71	Direct gamma-spec.	-	**4**	**1.0**
75	870 ± 179.2	20.6	Direct gamma-spec.	-	**12**	**0.5**
76	783 ± 54	6.9	Direct gamma-spec.	-	**1**	**0.1**
77	790 ± 94	11.9	Direct gamma-spec.	-	**1**	**0.1**
78	835 ± 64	7.66	Direct gamma-spec.	-	**7**	**0.8**
79	824.2 ± 64.2	7.79	Direct gamma-spec.	-	**6**	**0.7**
80	805 ± 50	6.21	Direct gamma-spec.	-	**3**	**0.5**
81	514.56 ± 20.02	3.89	Direct gamma-spec.	yes	**-34**	**-8.5**
82	809 ± 92	11.4	Direct gamma-spec.	-	**4**	**0.3**
84	936.8 ± 27.4	2.92	Direct gamma-spec.	-	**20**	**4.3**
85	866 ± 18	2.08	Direct gamma-spec.	-	**11**	**2.9**
86	890 ± 90	10.1	Direct gamma-spec.	-	**14**	**1.2**
87	921.5 ± 93	10.1	Direct gamma-spec.	-	**18**	**1.5**
88	875 ± 138	15.8	Direct gamma-spec.	-	**12**	**0.7**

Appendix 11: Measurement uncertainties of IRMM in the characterisation study

As example of measurement uncertainty budgets, this appendix lists the measurement uncertainties obtained by IRMM for its contributions to the characterisation study, CCRI(II)-S8, for the reference values (cf. section 2).

Table 1. Uncertainty budget of IRMM measurements for determination of ^{40}K in CCRI(II)-S8.

QUANTITY *Q*	Relative uncertainty of *Q* / %	Relative uncertainty in activity concentration / %	Type (A/B)	Comment
counting statistics	3	3	A	including background
weighing	0.2	0.2	B	includes buoyancy
background	-	-		included above
dead/live time	0.005	0.005	B	
decay data	1.1	1.1	B	
extra-/inter-polation of efficiency curve	2.5	2.5	B	experimental calibration and transfer with MC codes GEOLEP and GEANT
half-life	0.24	< 0.0001	B	
water content	0.3	0.3	B	
sample treatment	-	-		see next 3 lines
filling height	??	1.2	B	included in efficiency curve
density	??	0.2	B	included in efficiency curve
positioning (geometry)	0.6	0.6	A	
combined standard uncertainty u_c (single measurement)		4.2		

Table 2. Uncertainty budget of IRMM measurements for determination of ^{90}Sr in CCRI(II)-S8.

QUANTITY *Q*	Relative uncertainty of *Q* / %	Relative uncertainty in activity concentration / %	Type (A/B)	Comment
counting statistics	0.28	0.3	A	including background
weighing	0.2	0.2	B	includes buoyancy
background	1.8	-	A	included in counting stats
quenching	0.9	0.9	B	
tracer	0.1	-	B	due to buoyancy (incl in chem recov)
extra-/inter-polation of efficiency curve	-	-		included in quenching
calibration factor	-	-		included in quenching
water content	0.3	0.3	B	
sample treatment	-	-		see details next 5 lines
chemical recovery	2.1	2.1	B	B type dominates
weighing of carrier	0.4	-		incl buoyancy (incl in chemical recovery)
weighing of oxalate	0.3	-		incl buoyancy (incl in chemical recovery)
mass loss/gain oxalate	2	2	B	included in chemical recovery
ash transfer loss	1.3	1.3	A	
natural Sr in sample	9	0.4	B	included in chemical recovery
combined standard uncertainty u_c (single measurement)		2.7		

Table 3. Uncertainty budget of IRMM measurements for determination of ^{137}Cs in CCRI(II)-S8.

QUANTITY *Q*	Relative uncertainty of *Q* / %	Relative uncertainty in activity concentration / %	Type (A/B)	Comment
counting statistics	0.35	0.35	A	including background
weighing	0.2	0.2	B	includes buoyancy
background	-	-		included above
dead/live time	0.005	0.005	B	
decay data	0.24	0.24	B	
extra-/inter-polation of efficiency curve	2.5	2.5	B	experimental calibration and transfer with MC codes GEOLEP and GEANT
half-life	0.1	0.001	B	
water content	0.3	0.3	B	
sample treatment	-	-		see next 3 lines
filling height	??	1.2	B	included in efficiency curve
density	??	0.2	B	included in efficiency curve
positioning (geometry)	0.6	0.6	A	
combined standard uncertainty u_c (single measurement)		2.7		

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Abstract

The evaluation is presented of a laboratory comparison (LC) on ^{90}Sr, ^{40}K and ^{137}Cs in dried bilberries organised in 2011 by the IRMM. The activity concentrations reported by 88 participant laboratories are compared to the reference values of the new reference material IRMM-426 Wild Berries. 9 % and 17 % of activity concentration results for ^{137}Cs and ^{40}K, respectively, deviate more than 20 % from the reference values, a result worse than that obtained in previous LCs. For ^{90}Sr, about 88 % of results lie within 30 % of the reference value, better than observed in previous LCs. But only 56 % of ^{90}Sr results are satisfactory in terms of the E_n criterion, indicating difficulties with a complete uncertainty estimation.

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