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Spiked air filters for the 2014 EC measurement comparison on simulated airborne particulates

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Abstract

In 2014, the European Commission Directorate-General Joint Research Centre, Institute for Reference Materials and Measurements (JRC-IRMM) organized on request of the Directorate-General Energy an interlaboratory comparison (EC ILC) exercise on ^{137}Cs measurement in air filters; a similar exercise was organized in 2003.

This report describes the production and distribution of the reference material to the 76 European laboratories monitoring radioactivity in the environment, which participated at the ILC. The JRC-IRMM provided the comparison samples which were prepared individually for each laboratory using a gravimetrically diluted ^{137}Cs solution standardised by JRC-IRMM. The samples were made by gravimetrically dispensing the necessary activity amounts close to those the laboratories routinely measure.

Introduction

In 2014, the European Commission Directorate-General Joint Research Centre, Institute for Reference Materials and Measurements (JRC-IRMM) organized on request of the Directorate-General Energy an interlaboratory comparison (EC ILC) exercise on ^{137}Cs measurement in air filters; a similar exercise was organized in 2003.

This report describes the production and distribution of the reference material to the 76 European laboratories monitoring radioactivity in the environment, which participated at the ILC. The JRC-IRMM provided the comparison samples which were prepared individually for each laboratory using a gravimetrically diluted ^{137}Cs solution standardised by JRC-IRMM. The samples were made by gravimetrically dispensing the necessary activity amounts close to those the laboratories routinely measure.

Standardisation of the spike solution

For this ILC exercise ^{137}Cs has been chosen as it is easy to measure and requires no specific corrections. The standardisation of the ^{137}Cs solution used for the spiking of the air filters was performed by liquid scintillation counting (LSC). Gamma-ray spectrometry was used as a secondary method and as an additional link to the standard solution used for the ILC of 2003.

For the LSC, the efficiency tracing method developed by CIEMAT/NIST (Grau Malonda and Garcia-Toraño, 1982) was used. All sources were prepared gravimetrically using a Mettler AX26 (Mettler-Toledo, Greifensee, Switzerland) mass comparator, calibrated using traceable weights. All samples were measured using a Packard 3100 TR/AB (PerkinElmer, Boston, MA, USA) liquid scintillation counter and a Wallac Quantulus 1220 (PerkinElmer, Boston, MA, USA) LSC.

For the CIEMAT/NIST efficiency tracer method, the computer code CN2005 (Günther, 2002) was employed to calculate the radionuclide beta-particle counting efficiencies. As tracer, the IRMM tritiated water standard was used (Spernot and Denecke, 1964; Makepeace et al, 1998) and the maximum efficiency for ^{137}Cs was 1.15 for both LSC instruments used. The overall uncertainty of the method was determined to be 0.85% ($k=1$). In the calculations nuclear data as proposed by the Decay Data Evaluation Program (DDEP, 2015) were used, like the ^{137}Cs half-life of 30.05 (8) a or 10975 (29) d.

The activity concentration of the mother solution was found to be 3.60 (3) MBq g⁻¹ on the reference date 1 January 2015 0:00 UTC. As usually, the numbers in parentheses are the numerical values of the combined standard uncertainties u_c expressed in the unit of the quoted result.

Traceability was guaranteed by using the exact same method as for participation at the ongoing comparison BIPM.RI(II)-K1.Cs-137 (Ratel et al., 2005), following the standardisation of ^{137}Cs for the EC ILC organised in 2003; the standardised solution of ^{137}Cs of the 2003 campaign was submitted together with the results of the described standardisation at JRC-IRMM as entry into the International Reference System (SIR) of the Bureau International des Poids et Mesures (BIPM) in order to establish traceability of the activity values of the spiking solutions and subsequently of the reference values of spiked activities on filters.

In addition, a gamma-ray spectrometric study was performed with the sources prepared gravimetrically using a Mettler AX26 mass comparator, calibrated using traceable weights. Two high-purity germanium (HPGe) detector systems were used for the measurements, one with a 36% relative efficiency co-axial detector (Detector A) and the other with a 92% low-background co-axial detector (Detector B) (Canberra Industries, Inc., Meriden, CT, USA). The spectra analysis was performed using the GammaVision-32 software program (ORTEC, Oak Ridge, TN, USA) and the data analysis was done with

custom made spreadsheets. The sources were measured at two different distances from the detectors, a close one and a further one, against similar sources prepared during the 2003 EC ILC campaign and the discrepancy was determined to be less than 0.5%.

Dilutions

In order to approximate the activity level measured at each participating laboratory under routine conditions with the appropriate amount of spiked ^{137}Cs on the filters, four different diluted solutions (D1, D2, D3, D4) were prepared from the standardised mother solution (A1) via two intermediate dilutions (B1 and C1). The diluent was a solution of $50 \mu\text{g mL}^{-1} \text{Cs}^+$ (as CsCl) in 0.1M HCl. In the final dilutions methylene blue, used to visualise the spikes on the filters, was gravimetrically added.

It should be noted that all dilutions were prepared gravimetrically for utmost traceability and small uncertainty on the dilution factors. In addition, quantitative sources were prepared from all dilutions and measured for quality control by both liquid scintillation counting and gamma-ray spectrometry.

In *Figure 1* the dilution scheme is shown schematically together with the approximate dilution factors and the activity concentration of each of the solutions prepared.

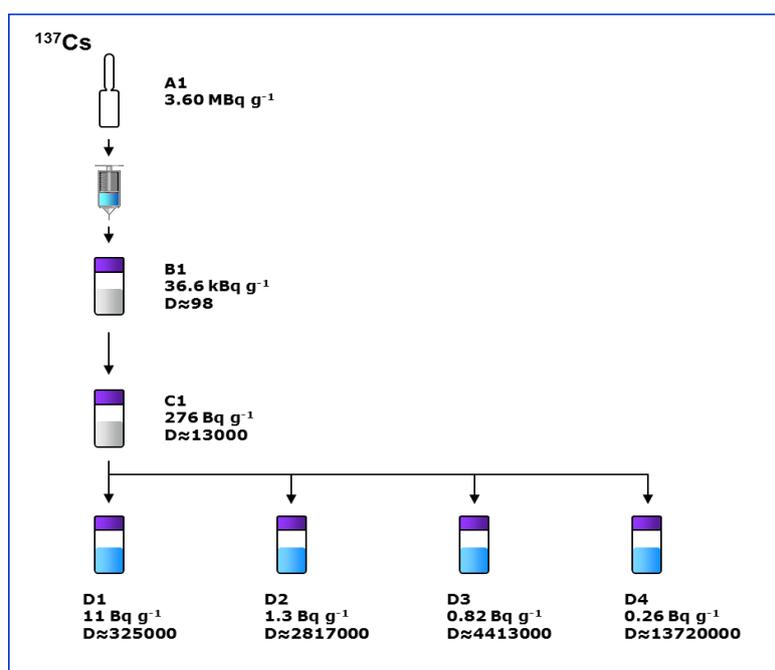


Fig. 1. Preparation scheme of the ^{137}Cs dilutions.

Spiking of the air filters

On the basis of the information provided by the participating laboratories in the relevant questionnaire, the ^{137}Cs activity per filter and the spiking pattern was determined for each filter individually. The amount of ^{137}Cs spiked onto each filter was chosen to resemble the activity routinely measured by the corresponding laboratory in a whole filter (or set of filters if it measures several at the same time).

The JRC-IRMM Radionuclide Metrology Sector prepared 76 air filters by depositing on each of them gravimetrically an amount of ^{137}Cs from one of the standard solutions D1, D2, D3 and D4. Each participating laboratory in this ILC exercise had sent two blank

filters of the type it is routinely using and after spiking one of them at IRMM with the ^{137}Cs solution, the filters were returned to the participants in order to be measured according to their routine procedure. The second blank filter was kept in reserve.

Since a uniform distribution of spikes was not feasible, depending on the filter size about 10 to 100 droplets were dispensed gravimetrically using a pycnometer, in a more or less symmetric pattern on the air filter. In any case, the spiked spots could easily be distinguished due to their methylene blue color, which allowed accounting for their discrete distribution when preparing the filters for measurement or when calculating counting efficiency corrections.

Where the filter was large enough, the filter was folded up in a way that the active part came into the centre of the pack such that any substance falling off the filter during transport by regular mail would still be caught in the surrounding filter and thus would not be lost. In the case of small filters this solution was not possible, therefore, the laboratories were asked to also measure the empty plastic bag in which the samples were shipped (or to measure the filter inside the bag) in order to verify that no losses from the filter had occurred.

Various spiked filters are shown in *Figure 2*. In many of the filters (e.g. those made of polypropylene or glass fibres) spiking became difficult due to the hydrophobic nature of the filters. The drops of radioactive solution were not absorbed into the filter material, therefore, the spiking had to be followed by several hours of drying at room temperature.

The spiked air filters, after drying, were sealed in plastic bags and dispatched to the participating laboratories. The relevant correspondence to the laboratories is given at the end of this report.

Reference values

The reference activity values, i.e. the spiked activities on the filters were calculated using the activity concentration of the mother solution determined by primary standardisation and the gravimetrically determined dilution factors of the spiking standard solutions D1, D2, D3 and D3. The mass and activity of the dispensed solution on each filter was determined once per filter by weighing the pycnometer before and after depositing the total number of drops on the corresponding filter. The lowest ^{137}Cs activity spiked on an air filter was 0.069 (1) Bq and the highest value was 2.31 (2) Bq. The reference date for reporting the activity (and likewise of the reference values) is 1 January 2015 0:00 UTC.

Conclusion

Air filters were spiked with the appropriate amount of ^{137}Cs , individually for each laboratory and dispatched to the 76 participating laboratories for analysis. The standardisation of the mother solution of ^{137}Cs , the dilutions prepared and the spiking of the air filters are presented in this report. A complete report on the 2014 EC measurement comparison on simulated airborne particulates is available.

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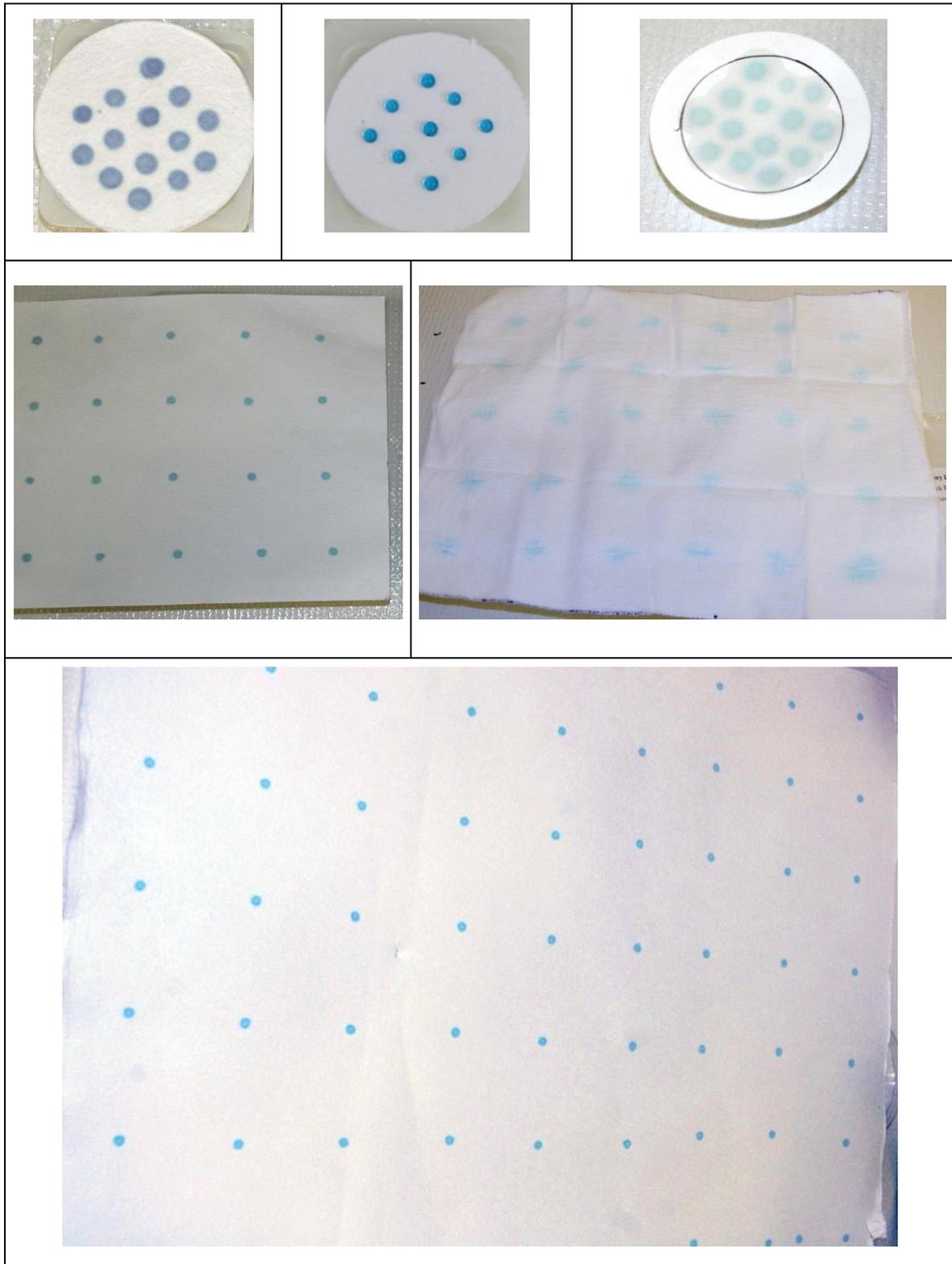


Fig. 2. Examples of spiked air filters.

Spiked air filter dispatch (email) and accompanying letter

Subject: EC interlaboratory comparison on Cs-137 measurement in air filters – spiked air filter dispatch

Dear colleague,

The parcel containing the spiked air filter was dispatched to your laboratory by DHL courier from our site (IRMM).

Please confirm the receipt of the sample by e-mail to JRC-IRMM-REM-COMPARISONS@ec.europa.eu. Please check the spiked air filter and in case of any damage report to the above e-mail address.

If you have already confirmed the receipt of the sample you don't have to confirm it again.

The parcel contains:

- a) Test item: spiked air filter
- b) Accompanying letter

The reporting of the results is done via the login page using the following URL: <https://web.jrc.ec.europa.eu/ilcReportingWeb>

To report your results you need a password key which is unique to this interlaboratory comparison and your laboratory. You will find your password key in the accompanying letter. Keep the accompanying letter for further reference!

The deadline for reporting results and completing the questionnaire is Friday, 27 February 2015.

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

We wish you success with your measurements.

Kind regards,

Timotheos ALTZITZOGLU
ICS-REM Project Leader

Borbala MATE
ICS-REM Project Coordinator



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Geel, 20 January 2015
IRC.D.4TA/our/ARES(2015)

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

Participation in the EC interlaboratory comparison on ^{137}Cs measurement in air filters

Dear «Title» «Surname»,

Thank you for participating in the EC interlaboratory comparison on ^{137}Cs measurement in air filters.

Enclosed, you will find one of your blank filters spiked with ^{137}Cs activity either at a level you normally measure or higher than the threshold you indicated in the questionnaire.

Please confirm the receipt of the sample by e-mail to IRC-IRMM-REM-COMPARISONS@ec.europa.eu. Please check the spiked air filter and in case of any damage report to the above e-mail address.

This parcel contains:

- a) Test item: spiked air filter
- b) This accompanying letter.

Please keep this letter, you will need it to report your results.

On the filter you can easily distinguish the active spots (light blue colour). This should make further preparation of the filter for measuring easier. Large filters had to be folded; the activity is on the inner side.

For the measurement and analysis, you should follow the procedure you routinely use. In case you do not measure the filter in the inner plastic bag, it is advisable to measure also the bag to make sure that no activity from the filter is left in the plastic bag.

Reporting of the results:

The reporting of the results is done via the login page using the following URL:
<https://web.jrc.ec.europa.eu/jrcReportingWeb>

Réserveweg, B-2440 Geel - Belgium, Telephone: (32-14)571-211.
Office: 010 000055, Telephone: direct line (32-14)571-4251.
E-mail: IRC-IRMM-REM-COMPARISONS@ec.europa.eu

To report your results you need a password key which is unique to this interlaboratory comparison and your laboratory.

Your password key is: «Part_key»

Please note that only submitted results will be taken into account, therefore, do not only save your results but also click on the *Submit* button. Once you have submitted your results and questionnaire, please remember to send us a signed copy by e-mail (IRC-IRMM-REM-COMPARISONS@ec.europa.eu) or to fax us (Fax no. +32 14 584 273).

As you have been informed, the description of your analytical and measurement procedures will be collected via questionnaire using the same URL link as for reporting the results. We kindly ask you to answer all relevant questions regarding the procedures you employed for the measurement of the filter sample. Disregard questions which are not related to the methods used in your laboratory. The uncertainty of the result must be reported in the same units as the activity concentration (Bq).

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

The deadline for reporting results and completing the questionnaire is **Friday, 27 February 2015**.

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

We wish you success with your measurements.

Kind regards,

Timotheos ALITZOGLOU
ICS-REM Project Leader

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