COOMET Project № 386/RU/07

"Key comparison of Cs-137 radioactivity measurements"

Identifier in the Key and Supplementary Comparisons section of the Key Comparisons Data Base (KCDB):

COOMET.RI(II)-K2.Cs-137

Technical Protocol

(Version 2007-08-30)

Pilot laboratory:

D.I. Mendeleyev Institute for Metrology (VNIIM)

Responsible person:

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19 Moskovsky pr., 190005

St.Petersburg, Russia

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Contents

1. Description of the Project

COOMET Project N 386/RU/07 makes provision for realizing the regional key comparisons of national measurement standards of the unit of radionuclide activity, COOMET.RI(II)-K2. Cs-137.

The objective of this project is to make a COOMET key comparison of national measurement standards of the activity concentration of a standardized solution of radionuclide Cs-137 to enable the traceability of the measurement results to the reference value of key comparison BIPM.RI(II)-K1.Cs-137 and to demonstrate their traceability to the SI. Two of the comparison participants are those national measurement institutes (NMIs) of the COOMET that did not participate in the CIPM comparisons. The pilot laboratory and linking institute for the comparison COOMET.RI(II)-K2.Cs-137 to the BIPM.RI(II)-K1.Cs-137 is the D.I. Mendeleyev Institute for Metrology (VNIIM).

2. Participants

Participant	NMI	Country	Responsible person	E-mail
1	VNIIM	Russia	I.A.Kharitonov	khia@vniim.ru
2	CENTIS	Cuba	Pilar Oropesa Verdecia	poropesa@centis.edu.cu
3	SMU	Slovakia	Anton Svec	svec@smu.gov.sk
4	BelGIM	Belarus	V.Milevsky	milevsky@belgim.by

Note: Full addresses are given in Appendix A. The VNIIM will be the pilot and linking laboratory.

3. Procedure of the comparisons

3.1. Object of the comparisons

Measurement of activity concentration of a radionuclide Cs-137 in the same solution with available measurement methods.

3.2. Travelling measurement standard

As a travelling measurement standard for the comparison, the solution of Cs-137 in 2 moles of nitric acid, placed in a NIST glass ampoule will be used. The value of radionuclide Cs-137 activity concentration in the solution is about 800 kBq/g. The mass of the solution in an ampoule is indicated in the passport (certificate) that will be directed to the participants simultaneously with the solution. The solution is provided for the comparison by the VNIIM.

3.3. Procedure of measuring and reporting measurement results

The participants of comparisons determine the activity concentration of Cs-137 in the solution, using available measurement methods, fill in a form for reporting their comparison results (Appendix 2 to the present Technical Protocol) and submit it to the VNIIM.

This form of the Report is the same as that dispatched by the BIPM to the participants of the SIR comparisons. It includes a description of the applied measurement method, total budget of measurement uncertainty according to [1] and final results of measurements with a combined uncertainty value for the coverage factor $k = 2$.

If a participant of the COOMET.RI(II)-K2.Cs-137 comparisons uses a number of measurement methods rather that one method , then in accordance with CCRI(II)/05-01[2], this participant has to make a choice and indicate in the report only one value as the final result. This should be the value obtained with one of the methods applied or the mean (the weighted average value) of the results obtained for each applied method with indication of a corresponding uncertainty.

3.4. Assumed time schedule

The comparison is planned to be performed within the period from May until the end of November, 2007. The solution will be dispatched by the VNIIM to the participants, as soon as the documents authorizing transportation of the above radioactive solution from the VNIIM to a particular participant become executed, but not later than on October 20, 2007. The participants of the comparison should send the report to the VNIIM not later than in a month after receiving the solution.

3.5. Link to the SIR KCRV

The pilot laboratory will perform the activity concentration measurements before dispatching the solution to the comparison participants. The report about the comparison together with a solution aliquot of 3.6 g in mass in a standard ampoule will be sent by the VNIIM to the BIPM to provide the traceability of the results obtained by the comparison participants to the KCRV in the SIR system. When the VNIIM receives from the BIPM the results obtained in SIR, the VNIIM report and BIPM message will be sent confidentially to the Chairman of the TC-IR COOMET.

3.6. Evalution of measurement results

The pilot laboratory will evaluate the comparison results on the basis of the reports (see item 3.3) submitted by the participants. A detailed description of the evaluation procedure will be given in Draft A of the report on the results of comparisons, which the pilot laboratory will prepare after completion of the program by all the participants.

Draft A of the report will be dispatched to the participants confidentially to obtain their remarks and discussions, and after its agreement, it will be sent to the BIPM/SIR and the CCRI. After editing by the BIPM, the report will be sent as a report B to the CCRI(II) KCWG and then, after approval, it will be directed for publication in the *Technical Supplement of Metrologia*.

In accordance with the CCRI(II)/05-01, the degrees of equivalence of the NMIs national measurement standards participating in the comparisons, will be established with respect to the KCRV of the BIPM.RI(II)-K2.Cs-137 key comparisons and published as a separate BIPM/SIR report in the *Technical Supplement of Metrologia*. A link between the KCRV and participant results will be established on the basis of the measurement result obtained by the VNIIM in the SIR comparison made within the frame of the given KOOMET N 386/RU/07 Project.

3.7. Safety precautions in transporting the solution and handling it in the process of measurements

The solution is transported in a glass ampoule packaged in a leaded container, placed in a box of corrugated board with dimensions of side 20 cm. The total weight is 0,5 kg.

After removing a cover of the container, it is necessary to examine the ampoule in order to be convinced that there is no damage due to its transportation, and to check with the smear method whether the ampoule has not become depressurized.

3.8. Financial aspects and insurance documents

The participants of comparisons themselves pay for all the expenses bound with the comparison, within their own countries. The VNIIM grants the Cs-137 solution for the comparison free of charge.

3.9. Schedule of carrying out the comparisons

The assumed schedule dates of carrying out the Project are May – November, 2007.

REFERENCES:

1. ISO International Organisation for Standardisation (1993). “Guide to the Expression of Uncertainty in Measurement. Geneva, ISBN 92-67-10188-9.
2. CCRI(II), guidelines for CCRI(II) key comparisons,CCRI(II)/05-01

Appendix A: Full addresses of the Participants

1. D.I.Mendeleyev Institute for Metrology (VNIIM)
   19 Moskovsky pr.,
   St.Petersburg, 190005, Russia
   Responsible person:
   I.A.Kharitonov
   E-mail: khia@vniim.ru

2. Centro de Isotópos (CENTIS)
   P.O. Box 3415. San José de las Lajas,
   Habana, Cuba
   Responsible person:
   Pilar Oropesa Verdecia
   E-mail: poropesa@centis.edu.cu

3. Belarussian State Institute of Metrology (BelGIM)
   Division of ionizing radiation measurements
   93 Starovilenski tract,
   Minsk, 220053, Republic of Belarus
   Responsible person:
   Valeri Milevski
   E-mail: milevsky@belgim.by

4. Slovak Institute of Metrology (SMU),
   Centre for Ionizing Radiations
   63, Karloveska str., 84255 Bratislava,
   Slovak Republic
   Responsible person: Dr. Anton Svec
   E-mail: svec@smu.gov.sk

COOMET Project № 386/RU/07

"Key comparison of Cs-137 radioactivity measurements"

Identifier in Appendix B of the Key Comparisons Data Base (KCDB):

COOMET.RI(II)-K2.Cs-137

Participating laboratory :

$T_{1/2}$ = (30.03 years; u = 0.01 years)*
1 year= 365.25636 days

Ampoule number _ _ _ _ _ _

Mass of solution, according to distributing laboratory _ _ _ _ _ g

Name(s) of the person(s) who carried out the measurements:

Date:

Please send the filled-in form and any additional information to the VNIIM
not later than ***November 1st, 2007.***

* NUBASE Evaluation of Nuclear and Decay Properties , G.Audi, O.Bersillon, J.Blachot,and A.H.Wapstra, Nuclear Physics, A 279,129,(2003)

A. Preliminary measurements

A.1. Method used for preliminary measurements

- calibrated ionization chamber YES □ NO □
- well crystal YES □ NO □
- other method YES □ NO □

A.2. Results obtained

Radioactivity concentration, in kBq g^{-1} (2007-06-01, 0 h UTC)

- before opening the original ampoule _ _ _ _ _
  date of this measurement _ _ _ _ _

- after transfer to another ampoule _ _ _ _ _
  date of this measurement _ _ _ _ _

Total mass of solution found in the ampoule_ _ _ _ _ g

A.3. Adsorption tests

Please take into account the adsorption tests in the evaluation of the final results.

A.3.1. *Adsorption tests carried out with liquid-scintillation counting*

Please keep in mind the following:

- No rinsings are necessary,
- Use a water-immiscible cocktail to measure the residual activity.

Activity remaining in the "empty" original ampoule _ _ _ _ Bq.

Date of this test _ _ _ _ _ _ _ _ _ _ _.

Please explain the measuring procedure used:

A.3.2. *Adsorption tests carried out with proportional counting*

Please rinse the ampoule with an aggressive solution to remove most of the activity and prepare solid source(s) to measure this residual activity.

Activity remaining in the "empty" original _ _ _ _ _ Bq.
Date of this test _ _ _ _ _.

A.4. Impurity checks* :

Method of measurement _,
Nuclide _ _ _ _ _ _ _ ,
Impurity to ^{137}Cs ratio _ _ _ _ _ _ _ _ _ _ and its uncertainty _ _ _ _ _ _ _ _ _ _
at reference date (2007-06-01, 0 h UTC).

* Please give this information for each impurity found.

B. Source preparation

B.1. Methods used for source preparation:

Possible remarks about drying, precipitation, foils used (gold-coated or not, number, etc.), type of balance used…

B.2. Solutions, sources

B.2.1. For *photon counting and beta counting (if relevant)*

Diluent:

	dilution number		
	1	2	3 ...
- dilution factor	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _
possible remarks	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _
- number of sources prepared	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _
- disposed mass of solution (approx.)	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _

B.2.2. *For liquid-scintillation counting*

Diluent	_ _ _ _ _	
Dilution factor	_ _ _ _ _	
Scintillator used to prepare the sources	_ _ _ _ _	
Volume of scintillator used	_ _ _ _ _	cm^3
Chemicals used to stabilize the solution	_ _ _ _ _	
Substances used as quenching agent	_ _ _ _ _	
Type of vials used	_ _ _ _ _	

C. Procedures used for the activity measurements

C.1. Method of measurement used and code as explained in Appendix

(e.g. 2π(PC)α-γ counting, 4π(PC)α-γ counting, α absolute counting with defined solid angle, α-x or α-γ coincidence with defined solid angle, liquid-scintillation counting or other)

C.2. ^{137}Cs nuclear data

Please list the values for all the decay-scheme parameters (transition energies, transition probabilities, etc.) relevant to your measurements.

In case you used more than one method, please assemble the relevant information on separate sheets.

D. Detectors, counting equipment

D.1. Beta counting (channel 1)

D.1.1. Semiconductor *detector*

Nature	_ _ _ _ _ _ _ _	Solid angle	_ _ _ _ _ sr
Number of detectors	_ _ _ _ _ _ _ _		
Type	_ _ _ _ _ _ _ _	Coaxial □	Planar □
Diameter	_ _ _ _ _ mm	Volume	_ _ _ _ _ cm^3
Window material	_ _ _ _ _	Window thickness	_ _ _ _ _ mm
Distance between photon counter and source			_ _ _ _ _ mm
Resolution at	_ _ _ _ _ keV,	FWHM*_ _ _ _ _ %,	_ _ _ _ _ keV

Please add a typical pulse-height spectrum and an efficiency curve.

D.1.2. *Proportional counter* _ _ _ _ _

Solid angle _ _ _ _ _ sr

Wall material _ _ _ _ _ Height of each half _ _ _ _ _ mm

Anode

Nature _ _ _ _ _

Wire diameter _ _ _ _ _ mm Wire length _ _ _ _ _ mm

Distance from source _ _ _ _ _ mm

Voltage applied _ _ _ _ _ kV

Gas

Nature _ _ _ _ _

Pressure

(above atmospheric pressure) _ _ _ _ _ MPa

Discrimination range _ _ _ _ _ keV

Remarks

D.1.3. *Liquid-scintillation method (LSC)*

D.1.3.1.*Characterization of the liquid-scintillation equipment*

Type of the counter _ _ _ _ _

Age _ _ _ _ _

Quench parameter _ _ _ _ _

Nuclide used as external standard _ _ _ _ _

Efficiency obtained with an unquenched standard of ^{3}H _ _ _ _ _

Background (unquenched standard in toluene scintillator, 0 to 2000 or more keV) _ _ _ _ _

Options used (e.g. low-level counting) _ _ _ _ _

Type of phototubes _ _ _ _ _

Operating temperature _ _ _ _ _

Coincidence resolving time _ _ _ _ _

Type of dead time extending □ non-extending □

Minimum dead-time length _ _ _ _ _ μs

Efficiency variation method:

- defocusing _ _ _ _ _
- grey filters _ _ _ _ _
- chemical quenching _ _ _ _ _
- other ones (please describe) _ _ _ _ _

External standard (^{3}H or other) used for the determination of the figure of merit _ _ _ _ _

D.1.3.2. *Characterization of the tracer* (e. g. ^{3}H)

Standard used and its origin _ _ _ _ _

Uncertainty on the standard _ _ _ _ _

Date of preparation of the tracer samples _ _ _ _ _

Chemical composition of the tracer samples _ _ _ _ _

D.1.4. *Other method(s)*

D.2. Photon counting (channel 2)

D.2.1. *Scintillator detector*

Crystal material	_ _ _ _ _ _ _ _	Solid angle	_ _ _ _ _ sr
Number of crystals	_ _ _ _ _ _ _ _	Well type YES □	NO □
Crystal diameter	_ _ _ _ _ mm	Crystal height	_ _ _ _ _ mm
Well diameter	_ _ _ _ _ mm	Well depth	_ _ _ _ _ mm
Window material	_ _ _ _ _ _	Thickness	_ _ _ _ _ mm
Distance between photon counter and source			_ _ _ _ _ mm
Resolution at	_ _ _ _ _ keV,	FWHM* _ _ _ _ _ %,	_ _ _ _ _ keV

Please add a typical pulse-height spectrum.

∗ full width at half maximum

D.2.2. *Semiconductor detector*

Nature	_ _ _ _ _ _ _ _	Solid angle	_ _ _ _ _ sr
Number of detectors	_ _ _ _ _ _ _ _	Well type YES □ NO □	
Type	_ _ _ _ _ _ _ _	Coaxial □ Planar □	
Diameter	_ _ _ _ _ mm	Volume	_ _ _ _ _ cm^3
Window material	_ _ _ _ _	Window thickness	_ _ _ _ _ mm
Distance between photon counter and source			_ _ _ _ _ mm
Resolution at	_ _ _ _ _ keV,	FWHM*_ _ _ _ _ %,	_ _ _ _ _ keV

Please add a typical pulse-height spectrum and an efficiency curve.

Radionuclides used for an efficiency determination (if relevant)

	$P\gamma$ (%)
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

D.2.3. *Other detectors used*

D.3. Parameters of counting equipment

(Give a brief description and/or a block diagram of the experimental arrangement.)

D.3.1. Channel *1(betas)*

a) Discrimination level _ _ _ _ _ keV
(or window)

b) Dead times and their uncertainties (standard deviation)

Dead time τ_1 = _ _ _ _ _ _ μs; u = _ _ _ _ _ _ μs

Type of dead time extending □ non-extending □

Method used for measurement _

Live time clock Yes □ No □

Pulser technique Yes □ No □

Loss free counting Yes □ No □

c) Pile-up rejector Yes □ No □

D.3.2. Channel *2 (photons)*

a) Discrimination level _ _ _ _ _ keV
(or window)

b) Dead times and their uncertainties (standard deviation)

Dead time τ_2 = _ _ _ _ _ _ μs; u = _ _ _ _ _ _ μs

Type of dead time extending □ non-extending □

Method used for measurement _

D.3.3. *Coincidence unit (if relevant)*

Coincidence resolving time τ_R = _____ μs; u = _____ μs

Method used for measurement _____

_____

_____

_____

D.3.4. *Other modules used*

(for LSC see section D.1.3.)

E. Relevant data, corrections and uncertainties

E.1. Date of measurement of 137Cs

(Mean date on which your measurements were carried out)

E.2. Measuring data

E.2.1. *Channel 1 (betas)*

Dead time	_ _ _ _ _ μs	Number of sources measured	_ _ _ _ _
Background rate	_ _ _ _ _ s^{-1}	Typical count rate	_ _ _ _ _ s^{-1}
Typical time for one measurement		_ _ _ _ _ s	
Discrimination threshold or window		_ _ _ _ _ keV	

E.2.2. *Channel 2 (photons)*

Dead time	_ _ _ _ _ μs	Number of sources measured	_ _ _ _ _
Background rate	_ _ _ _ _ s^{-1}	Typical count rate	_ _ _ _ _ s^{-1}
Typical time for one measurement		_ _ _ _ _ s	
Discrimination threshold or window		_ _ _ _ _ keV	

E.2.3. *Extrapolation of efficiency function*

Maximum achieved efficiency _ _ _ _ _ %

Method used for varying the efficiency

Number of degrees of freedom _ _ _ _ _

Please add a figure, if possible.

E.2.4. *Calculated data for the liquid-scintillation method*
Total efficiency ^{137}Cs

– – – – –

E.2.5. *Corrections applied*

E.2.6. *Uncertainty components**, in % of the activity concentration, due to

		Type A or B method	Remarks
counting statistics	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
weighing	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
dead time	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
background	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
pile-up	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
resolving time	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
Gandy effect	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
counting time	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
adsorption	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
impurities	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
tracer	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
input parameters and statistical model	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
quenching	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
interpolation from calibration curve	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
decay-scheme parameters	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
half life ($T_{1/2}$ = 30,03 years; u = 0,01 years)	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _	
self absorption	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
extrapolation of efficiency curve	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
other effects (if relevant) (explain)	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
combined uncertainty (as quadratic sum of all uncertainty components)	_ _ _ _ _	_ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _

* The uncertainty components are to be considered as approximations of the corresponding standard deviations (see also *Metrologia*, 1981, **17**, 73 and *Guide to expression of uncertainty in measurement,* ISO, corrected and reprinted 1995).

F. Combination of individual results

(obtained from the individual dilutions, source preparation, etc.)

How have the individual results been used for arriving at the final result (statistical weights) ?

G. Final result

The radioactivity concentration of the ^{137}Cs solution on the reference date*

(2007-06-01, 0 h UTC) is

_ _ _ _ _ _ _ _ kBq g^{-1},

and the combined uncertainty is

_ _ _ _ _ _ _ _ kBq g^{-1}, _ _ _ _ %

Remarks

* For adjusting your result to the reference date, please use the half-life value given on page 1.

Appendix - Acronyms used to identify different measurement methods

Geometry	acronym	Detector	acronym
4π	4P	proportional counter	PC
defined solid angle	SA	pressurized proportional counter	PP
2π	2P	liquid scintillation counting	LS
undefined solid angle	UA	NaI(Tl)	NA
		Ge(HP)	GH
		Ge-Li	GL
		Si-Li	SL
		CsI	CS
		ionisation chamber	IC
		bolometer	BO
		calorimeter	CA
		PIPS detector	PS
		Grid ionisation chamber	GC

Radiation	acronym	Mode	acronym
positron	PO	efficiency tracing	ET
beta particle	BP	internal gas counting	IG
Auger electron	AE	CIEMAT/NIST	CN
conversion electron	CE	sum counting	SC
bremsstrahlung	BS	coincidence	CO
gamma ray	GR	anti-coincidence	AC
x - rays	XR	coincidence counting with efficiency tracing	CT
alpha - particle	AP	anti-coincidence counting with efficiency tracing	AT
mixture of various radiation e.g. x and gamma	MX	triple-to-double coincidence ratio counting	TD
		selective sampling	SS
		high efficiency	HE

Examples	
method	**acronym**
4π(PC)β–γ-coincidence counting	4P-PC-BP-NA-GR-CO
4π(PPC)β–γ-coincidence counting efficiency. tracing	4P-PP-MX-NA-GR-CT
defined solid angle α-particle counting with a PIPS detector	SA-PS-AP-00-00-00
4π(PPC)AX-γ(GeHP)-anticoincidence counting	4P-PP-MX-GH-GR-AC
4π CsI-β,AX,γ counting	4P-CS-MX-00-00-00
calibrated IC	4P-IC-GR-00-00-00
internal gas counting	4P-PC-BP-00-00-IG