Validity of Ionizing Radiation Comparisons under the CIPM MRA

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Executive Summary

The CCRI has followed the spirit of the CIPM MRA recommendations with regard to a 10-year period for which metrology comparisons can be considered as valid support for the measurement capabilities of a laboratory, with some dispensation accorded by the CIPM. This presumes that there have been no significant changes in staff, equipment or measurement methods, each of which would need some form of validation even if the previous comparison falls within the period of validity. It should be noted that the date of the comparison is the date the measurements have been completed by the participating laboratory.

Each NMI (or designated institute) may have one result in each comparison, the most recent replacing any earlier participation. Results that are older than 10 years (presently 15 years for radionuclides) are normally coloured black in the KCDB and no result is removed from the KCDB without prior notice to the laboratory.

In the area of ionizing radiation dosimetry, comparison results are valid for 10 years, being replaced as new comparisons are held, and results are not removed from the KCDB for up to 15 years as long as the comparison is rescheduled within this period. The BIPM programme contains eight ongoing comparisons to support the Member States in this work. The CCRI and the RMOs hold linked comparisons and also run supplementary comparisons to support their CMCs for other ranges and quantities, and to include NMIs that do not hold primary standards in the particular area.

In the area of radionuclide metrology, comparison results are valid for 10 years, will be coloured black after 15 years but will not be removed from the KCDB for up to 20 years. This is to enable the Measurement Methods Matrix (MMM) to be developed to support a rolling 10-year programme of valid comparisons and for the International reference system for gamma emitters (SIR) to be fully extended to include pure beta emitters and pure alpha emitters. The MMM can already be used to support CMCs that are traceable to other primary standardizations through their measurement method. The BIPM programme contains the SIR and its extension through the SIRTI for short-lived radionuclides that are measured at the NMI using the BIPM transfer instrument (SIRTI). The BIPM programme covers 69 radionuclides at present. The CCRI and the RMOs run comparisons that link into the SIR and they also run other key or supplementary comparisons, particularly for radionuclides or matrices that cannot be measured in the SIR at present.

In the area of neutron metrology, comparison results are valid for 10 years but will remain in the KCDB for up to 15 years to enable these complex comparisons to be organized and completed within a practicable time-frame. There are only 5 NMIs world-wide that can host comparisons that need neutron beams, and circulating neutron sources for emission rate comparisons is increasingly difficult, requiring much advanced planning.

The history of how these decisions have been reached is presented in this paper.

1. INTRODUCTION

International comparisons in the field of ionizing radiation have been conducted by the BIPM through the CCRI since 1967. Consequently, when the CIPM MRA [1] was signed in 1999, many comparisons were already in existence. At that time the CCRI decided that as so many comparisons had been established before the CIPM MRA these should not be discarded but could be used for provisional equivalence until such time as a planned replacement programme was fully operational.

Some of the requirements of the CIPM MRA, such as fully detailed uncertainty budgets, had not always been fulfilled at the time of the earlier comparisons. This resulted in decisions of the CCRI, such as the arithmetic mean would be used for the KCRV in preference to the weighted mean specifically for radionuclide activity comparisons. Indeed, each area of ionizing radiation metrology developed slightly different criteria for determination of the KCRV and for the approval and validity of the comparisons.

As a result of these differences, it has not always been clear for reviewers of the calibration and measurement capabilities (CMCs) to identify which comparisons are still valid in the traceability chain of measurements. The CCRI has decided to adopt this paper that brings together the decisions on the validity of ionizing radiation comparisons under the auspices of the CIPM MRA.

2. NORMAL PERIOD OF VALIDITY OF COMPARISONS

When the CIPM MRA was established, a Technical Supplement [2] was also written that provided more detail for the comparison procedure. For example paragraph T.8 b) states:

Technical Supplement to the CIPM MRA

T.8 CCs have the responsibility :
b) to initiate and organize, with the collaboration of the BIPM, the execution of key comparisons at intervals to be decided individually for each comparison.

Consequently, the CCRI decided that for ionizing radiation comparisons, the normal default period of validity should be 10 years, unless the NMI staffing and/or facilities change within such a period, in which case a new comparison is recommended. However, as there were many comparisons that dated prior to the CIPM MRA in 1999, a grace period was accepted that differs according to the field of the comparison. The following sections describe the recommendations of each CCRI Section followed by a summary of the present situation and the way in which the comparisons and other information can be used to support the CMCs.

It should be noted that the CIPM has approved a new document that provides more detail on the operation and validity of comparisons in general under the CIPM MRA [3].

3. CCRI(I) COMPARISONS FOR X- AND GAMMA-RAY DOSIMETRY

The CCRI(I) has made a series of recommendations over the years concerning dosimetry comparisons and these are reproduced here for convenience. These individual series of recommendations are followed by a summary of the situation as at the present time.

3.1. CCRI(I) Recommendations 1999 [4]

3.1.1 NMIs will be required to repeat key comparisons at least once every 10 years.

3.1.2 Comparisons over 10 years old will normally be removed from the database, unless their retention is agreed by the CCRI(I).

3.1.3 The published equivalence database will show only current results. Old values to be available "on request".

3.2. CCRI(I) Recommendations 2001 [5]

3.2.1 After ten years – each NMI will be consulted on the validity of its result.

3.2.2 If declared valid, the result is included in the KCDB with the proviso that a new comparison be made within the next two years.

3.2.3 If a new comparison is planned, the entry for that NMI will be marked with an asterisk (*).

3.2.4 If a new comparison is not conducted during that time, the results of the old comparison will expire at the next CCRI(I) meeting - the last period in which such older results will be included.

3.2.5 When a NMI changes its standard, either a document that can be referenced is prepared, or a new comparison with the BIPM is conducted.

3.2.6 The results are submitted for approval by the CCRI(I), and – if approved – are then used to update the data in the KCDB.

3.3. CCRI(I) Recommendations 2003 [6]

3.3.1 To use Monte Carlo calculations for wall correction factors.

3.3.2 To update and publish BIPM.RI(I)-K4 results.

3.4. CCRI(I) Recommendation 2005 [9]

The CCRI(I), considering that there is

- likely to be a considerable work load of key comparisons that will fall on the BIPM in the years from 2005 to 2010;
- a current requirement to repeat a key comparison at least every ten years;
- a need for transparency in the KCDB;

recommends that an extension of five years is allowed in which to repeat a comparison on condition that

- the next comparison is clearly scheduled to occur within the extension period;
- the results that are more than ten years old are identified as such in the KCDB.

3.5. Dosimetry comparisons in summary

Dosimetry comparisons are normally valid for 10 years, assuming no critical changes in personnel, in the standards themselves or in measurement methods or equipment.

The date of the comparison is defined as the date of the measurements. The *Draft A* Report is normally produced within a couple of months following the comparison after assurance that the transfer chambers have maintained their integrity. Once approved by the participants the *Draft A* becomes the basis of the *Draft B* report, at which stage the NMI may use the report to support their CMCs until the final report is approved by the CCRI(I) and published.

Only published results for NMIs and designated institutes of the CIPM MRA will be included in the key comparison database (KCDB) of equivalences. Equivalence will normally be based on the most recent comparison, unless exceptional circumstances justify a previous value and this is approved by the CCRI(I).

The NMIs will normally be required to repeat key comparisons at least once every 10 years to maintain a result in the KCDB. Comparisons over 10 years old will be indicated in a different colour, normally black, and removed from the database 15 years after the comparison, unless their retention is agreed by the CCRI(I). A comparison result over 10 years remains valid up to 15 years only if a new comparison has been scheduled within this time frame. This is demonstrated by the future comparison date being recorded in the KCDB against the participant NMI.

For low and medium energy x-ray key comparisons, all the CCRI qualities should be used if possible, but this is not mandatory. Indeed there are a large number of x-ray beam qualities in use for different purposes and holding comparisons for all of them would not be practical. The KCWG(I) recommended in 2011, and the CCRI delegates accepted, that there is no requirement to define new beam qualities for key comparisons to support CMCs [8]. The RMOs are free to choose those qualities that best support their CMCs. Equivalence will be determined for individual qualities; there is no averaging of results over several qualities.

New results (plus full uncertainty budgets) for inclusion in the equivalence data base will be circulated to all CCRI(I) members in *Draft B* reports for approval. Publication and inclusion in the KCDB will be automatic if no objections are received, normally within one month. Minor points of detail will be dealt with by the BIPM but significant objections or amendments will be referred to the KCWG(I) and if necessary to the next CCRI(I) meeting.

In accordance with the CCRI(I) recommendation R(I)-2 (2005) [9], if an NMI wishes to change its standard for some reason, this will normally require a repeat comparison unless the change relates to calculated correction factors only, or an agreed change to some physical constant value. In the latter cases, advance notification and a publication of the change in the open literature will be followed by an update in the KCDB of the previous comparison result for both its value and uncertainty.

3.6 Using dosimetry comparisons to support CMCs

Published comparisons of air kerma and absorbed dose will normally support the CMCs at the relevant photon quality. The CCRI(I) decided in 2011 [8] that the range of agreed CCRI x-ray qualities provide adequate coverage for all diagnostic x-ray qualities. At the same time, the CCRI(I) decided that that there was no justification for holding a key comparison for ambient dose equivalent, $H^*(10)$, because the standard of air kerma at the relevant energy together with published conversion coefficients would be sufficient to support this radiation protection level quantity.

Any CMCs related to other quantities will normally need to be supported by regional supplementary comparisons, published bilateral comparisons or other supporting publications.

4. CCRI(II) COMPARISONS FOR ACTIVITY MEASUREMENTS

International comparisons of activity measurements have been held since the late 1960s and the SIR was launched in 1976 to enable NMIs to make comparisons at their convenience for the gamma-emitting radionuclides of their choice [10]. The KCRVs defined by the measurements in the SIR that are designated as BIPM.RI(II)-K1 comparisons are used to link associated CCRI(II)-K2 and RMO.RI(II)-K2 comparisons for the same radionuclide. When there is no SIR comparison, the CCRI(II)-K2 comparisons provide the KCRV and the appropriate links for any associated RMO.RI(II)-K2 comparisons. When a comparison has been run using a transfer well-type ionization chamber it has been designated as a CCRI(II)-K3 comparison. Comparisons using the BIPM SIR transfer instrument (a NaI(Tl) well detector) directly linked to the SIR are part of the BIPM.RI(II)-K4 ongoing comparisons. To support CMCs of activity measurements in complex matrices, international supplementary comparisons may also be organized.

4.1 Validity of SIR results

At the end of 2011, there were 710 independent results of activity measurements in the SIR for 66 different radionuclides displayed in the KCDB, with some still dating back to pre-1980. Although the original CIPM MRA proposal was to have results no older than 10 years in the KCDB, the long-term stability of radionuclide activity measurements has been well demonstrated in the NMIs and at the BIPM. Consequently, at the CCRI(II) in 2003, it was agreed to extend the time-frame for displaying results in the KCDB from 10 years to 20 years, at least until 2020.

Proposals for a gradual phasing to achieve the 20 year target by 2011 were agreed by the CCRI(II) in 2003 [11] as follows:

- *at the end of 2003, when the transitional period of the CIPM MRA ends, the notation "approved for provisional equivalence" no longer applies. All the SIR results in the KCDB that are more than 20 years old (pre-1983) and currently indicated in red, will be re-coloured black but will remain in the KCDB. Similarly for CCRI(II) and RMO comparisons that are currently coloured in blue and green;*
- *at the end of 2005, pre-1985 results will be re-coloured in black;*
- *at the end of 2007, pre-1987 results will be re-coloured in black and pre-1977*

results (i.e. more than 30 years old) will no longer be visible in the degrees of equivalence;

- *at the end of 2009, pre-1989 results will be re-coloured in black and pre-1984 results (i.e. more than 25 years old) will no longer be visible in the degrees of equivalence;*
- *at the end of 2011, pre-1991 results will no longer be visible in the degrees of equivalence;*
- *all the earlier results will remain in the original published reports and may still be used in the KCRV if relevant.*

4.2 Present situation

There are currently about 160 activity measurement results in the KCDB that are over 20 years old and have been coloured black, pending their removal. Results over 20 years old are now being removed from the KCDB at the same time as comparisons are being updated with new results. As results are removed, each affected NMI is informed of the fact and given the opportunity to check whether their related CMCs are covered by another comparison result, and, if necessary, they then send a new SIR ampoule to maintain a result in the KCDB. This new ampoule does not need to be of a primary standardized solution to update their KCDB value. However, only primary standardized solutions can be considered for inclusion in an update of the KCRV.

In addition to almost eighty SIR results that are due for removal, there are four international comparisons that are now over 25 years old, for activity measurements of Ba-133, Cs-134, Cd-109 and I-125 CCRI(II)-K2 comparisons. However, the possibility to submit SIR ampoules and the planned programme of CCRI(II) and BIPM key comparisons present opportunities for the NMIs to participate and make up for these losses.

As part of this planned programme, it is anticipated that by 2020 the BIPM and CCRI(II) will have held appropriate comparisons to cover every CMC currently listed for activity measurements by the judicious use of the *Measurement methods grouping criteria for radionuclides, to support CMCs* [12] commonly known as the *Measurement Methods Matrix* (MMM). Thus in 2020, consideration may be given by the CCRI(II) to reducing the validity period for activity comparisons, perhaps again by phasing, to the more usual 10 year target as for other CIPM key comparisons. In the meantime, comparison results that are over 15 years old are now being coloured black in the KCDB to signal their advancing age.

4.3 Activity comparisons in summary

Activity comparison results in the KCDB are coloured black when they are more than 15 years old unless they are updated with new comparison results. Results more than 20 years old are now being removed when each comparison is updated with ~~other~~ new results. The NMIs concerned are informed in advance of such removals.

If an SIR submission has been made and the SIR result is complete with the NMI activity value and uncertainty budget, the KCDB is updated with the year of the submission against the participating NMI and the *Draft A* report (SIR Master-file table) is issued. Once approved by the participants the results are included in the *Draft B* report and the NMI may then use their result to support their CMCs.

As soon as the *Draft B* report is approved by the CCRI(II), the Final report is published and the new result is entered into the KCDB as the new BIPM.RI(II)-K1 value, replacing any earlier result in the graph for the NMI whether this was in a BIPM comparison or a linked CCRI or RMO comparison. Results in tables are updated systematically only for the BIPM comparisons.

4.4 Using activity comparisons to support CMCs

Valid activity comparison results, published in the KCDB may be used to support CMCs for the same radionuclide, or a corresponding radionuclide as indicated in the *Measurement methods matrix* (MMM) [12] that is available through the BIPM website.

If a KCDB result is coloured black and there is a more recent date for the NMI participation listed in the KCDB, the published KCDB result remains valid although the NMI may prefer to use the SIR Master-file result to support their CMCs while awaiting publication of the results in the KCDB.

5. CCRI(III) COMPARISONS FOR NEUTRON MEASUREMENTS

5.1 Summary of CCRI(III) recommendations

The CCRI(III) has adopted the concept of 10 years as being the valid period for a neutron measurement comparison [13].

However, in this field, many of the comparisons were started well in advance of the CIPM MRA and are consequently now more than 10 years old. All the earlier data were "approved for provisional equivalence" by the CCRI(III) [14] although the data themselves do not appear in the KCDB in terms of degrees of equivalence. Consequently, the CCRI(III) formulated a special resolution to the CIPM [15] that was accepted, to maintain the validity of comparisons until they can be repeated in a planned programme for neutron measurements.

At the CCRI(III) meeting in April 2009, the committee decided that comparisons used to support CMCs should be the most recent and not the earlier “approved for provisional equivalence” comparisons [16]. Indeed, the recently published CCRI(III)-K10 neutron fluence comparison held in 2001 now supersedes many of the earlier results. The CIPM accepted the recommendation of the CCRI in 2007 [17] that the K10 neutron dosimetry comparison would remain valid for 15 years, which would give time for the K11 comparison to be completed and published.

It is presumed that interpolation between energies in comparisons is valid when the same measurement procedures are used for both these effective energies.

5.2 Neutron comparisons in summary

The validity of CCRI(III) comparisons, endorsed by the CCRI(III) 2011, is as follows:

Comparison	Topic	Energy	Year	Status
CCRI(III)-K1	Neutron fluence	24.5 keV	1993 to 1996	Valid until K11 is published
CCRI(III)-K2.series	Neutron fluence	0.144 MeV	1983 to1986	Superseded by K10
CCRI(III)-K3.series	Neutron fluence	0.250 MeV	1973 to 1978	Superseded by K10
CCRI (III) K4.series	Neutron fluence	0.565 MeV	1973 to 1986	Valid until 2013
CCRI(III)-K5 series	Neutron fluence	2.5 MeV	1973 to 1988	Valid until K11 is published
CCRI(III)-K6 series	Neutron fluence	5.0 MeV	1981 to 1988	Superseded by K10
CCRI(III)-K7 series	Neutron fluence	14.8 MeV	1973 to 1988	Superseded by K10
CCRI(III)-K8	Neutron fluence	Thermal neutrons	2005 to 2008	Valid (replaces K8.Au)
CCRI(III)-K9.AmBe	Emission rate	AmBe source	1999 to 2005	Valid (replaces K9.Cf-252)
CCRI(III)-K10	Neutron fluence	0.144 MeV, 1.2 MeV, 5.0 MeV, 14.8 MeV	2001	Valid until K11 is published
CCRI(III)-K11	Neutron fluence	27.4 keV 565 keV 2.5 MeV 17 MeV	2012	Valid once published

6 GENERAL POLICY ON THE USE OF COMPARISONS TO SUPPORT CMCs

The CCRI first produced a set of *International rules for completing CMC tables for ionizing radiation* in 2000 and this is reviewed and updated regularly [18]. The rules concerning the use of comparisons to support CMCs are presented here in full (shown in *italic* style) together with some additional guidelines.

Comparisons used for such support should be completed and at least the *Draft A* approved by the participants e.g. when the *Draft B – in progress* is indicated in the KCDB.

To facilitate the task of the reviewers, the CCRI has decided that, particularly for supplementary comparisons, there should be a clear statement in the protocol, repeated in the introduction of the comparison report, as to what type of CMCs the comparison can support. This is particularly relevant for reference materials.

Column P of the CMC Excel files is the column in which comparisons to support the given CMC line may be listed. This information is to help the reviewer of the CMC to verify the level of uncertainty claimed. It is no longer acceptable for this column to be left blank after 2010. One of the easiest ways to fill Column P is to cite a supporting comparison. However, although comparisons are the simplest way to support CMCs, other information may also be used in Column P, such as referenced publications [18].

Comparisons not held under the CIPM MRA, such as the EURADOS comparisons can be used to support CMCs as long as the comparison report is an open publication. However, it is recommended that all comparisons that are planned to be used as support for CMCs, be registered in the KCDB. This is a way to ensure the equivalence of the comparisons and to avoid some problems that have occurred in the past. It also ensures that the technical protocol has been appropriately reviewed.

Supporting comparisons can be BIPM, CCRI or RMO comparisons. Bi-lateral comparisons may be used if no other comparison exists for the NMI, as long as the partner NMI or the IAEA as bilateral partner has taken part in a BIPM, CCRI or RMO comparison.

If no directly related comparison exists, a similar type of comparison can be used to support the CMC but the reference should be preceded by the words "similar to" or some other indication as to the indirect nature of the comparison.

- *If an NMI holds primary standards, supporting comparisons will include key comparisons. There should be a supporting comparison for the CMC unless the type of calibration is unique to that NMI.*
- *If an NMI receives its traceability from another laboratory, supporting comparisons referring to this other laboratory should not be listed here. The regional comparison supporting the NMI's own calibration capability should be cited.*
- *Any comparison supporting the measurement capability of the NMI with secondary standards should be given in this column*
- *If an NMI has more than one comparison supporting its capability, only one of these should be quoted and the expanded uncertainty of the calibration (column I) should reflect that used in the comparison chosen.*

For BIPM, CCRI or RMO key and supplementary comparisons, only the reference is required. For other comparisons, the reference of the publication (or internal report) and the year of the comparison should be indicated.

- If the type of calibration is unique to that NMI, a supporting scientific publication should be cited.
- Activity CMCs may be supported by a comparison directly for the radionuclide in question or indirectly by reference to the appropriate radionuclide comparison given in the *Measurement Methods Matrix* [12].

On the expiry of a period established for the validity of the comparison results, these comparison results can continue to be used, if necessary to cover the claimed CMC line, until the comparison result is removed from the KCDB in accordance with the CCRI rules on validity and phasing as stated in this paper.

REFERENCES

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[5] *Comité Consultatif des Rayonnements Ionisants*, 17th meeting (2001), 2001, 15th meeting of the CCRI(I), 167-169 (in English).

[6] *Comité Consultatif des Rayonnements Ionisants*, 18th meeting (2003), 2004, 16th meeting of the CCRI(I), 22-25.

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[8] *Comité Consultatif des Rayonnements Ionisants*, 22nd meeting (2011), 2012, 20th meeting of the CCRI(I), 28.

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[15] *Comité Consultatif des Rayonnements Ionisants*, 20th meeting (2007), 2009, Recommendation R 2 2007, 18 (in English).

[16] *Comité Consultatif des Rayonnements Ionisants*, 21st meeting (2009), 2009, 18th meeting of the CCRI(III).

[17] *Procès-Verbaux des Séances du Comité International des Poids et Mesures*, 96th meeting (2007), 2008, 206 pp

[18] *International rules for completing CMC tables for ionizing radiation*, 2010, 12 pp.