Guidelines for CCRI(II) key comparisons

25 May 2005

1. Introduction

The procedures used by the CCRI(II) for selecting, conducting and evaluating key comparisons, including the detailed technical protocols and periodicity of the comparisons, are designed to ensure that:

- the comparisons test all the principal measurement methods in the field of activity measurements;
- the results are clear and unequivocal;
- the results are robust;
- the results are easy to compare with those of corresponding comparisons carried out by regional metrology organizations;
- overall, the comparisons are sufficient in range and frequency to demonstrate and maintain equivalence between the participating laboratories.

This note lays out the broad guidelines to be followed in conducting key comparisons. It is supplemented by the detailed technical protocols written for the individual key comparisons, and, if necessary, by additional guidelines prepared by the CCRI(II) that apply to particular areas of work.

During key comparisons, it is important that up-to-date information on the progress of the comparison be readily available. This implies that the participants, the chairman and executive secretary of the CCRI(II) be regularly informed by the pilot institute as to the status of each comparison. It is the task of the executive secretaries at the BIPM to maintain, for consultation, a central record of the status of key comparisons. The status is transmitted to the KCDB manager who updates the relevant web page on the BIPM site.

Note that formally, most members of the CCRI(II) are national metrology institutes (NMIs) but at meetings they are represented by designated delegates. Between meetings of the Consultative Committee it is understood that the member institutes continue to be represented by the delegate at the last meeting. In the event that this delegate is no longer available it is the responsibility of the member institute to inform the executive secretary of the Consultative Committee of the name of his or her replacement. This does not apply to individual members of CCRI(II), which are appointed by the CIPM for their expertise and are not representing a NMI.

2. Types of key comparison

There are two broad types of key comparison: in the first are those comparisons for which a transfer instrument having long-term stability is used, as for the SIR. SIR comparisons are normally carried out unilaterally and on a continuing basis at the convenience of the participating institute. The procedures used at the BIPM in carrying out this type of ongoing comparison are described in a separate note entitled *Procedures used in ongoing key comparisons carried out by the BIPM* and in the *BIPM monograph on the SIR*.

Other comparisons are carried out under a strict time schedule. This requires for all the participating institutes to make their measurements within a fixed period of time. These comparisons could require travelling standards having good short-term stability and stability during transport. Much of the detail in what follows applies mainly to this type of comparison.

3. Responsibilities for choosing key comparisons

The CCRI(II) is responsible for choosing the key comparisons. A set of key comparisons is identified which covers a range of standardization methods so as to test the principal techniques in the field. In deciding on the list of key comparisons, the Consultative Committee takes into account views expressed by the Regional Metrology Organizations (RMOs). These views can be expressed by the RMO to the Director of the BIPM or through RMO members who are members of the Consultative Committee.

On the basis of the results of the key comparisons, statements of equivalence can be made covering a wide range of measurements using these techniques, not just the measurements directly tested by a key comparison. The periodicity of the comparisons is set to ensure continuity of the equivalence without overloading the participating laboratories.

The procedure for choosing and updating the list of key comparisons is the following:

- The Consultative Committee appoints a small working group, that may be one of its permanent Working Groups, to draw up a list of proposed key comparisons and their periodicity, or to propose modifications to an existing list.
- At a meeting of the Consultative Committee the working group proposal is discussed and a list adopted. This list which is published in the report of the meeting appears in Appendix D of the MRA, in the key comparison data base of the MRA and elsewhere as required.

4. Initiating a key comparison

Key comparisons are initiated by a decision of the Consultative Committee.

- The Consultative Committee examines the needs for comparisons and decides which ones from the list of key comparisons should be initiated. In deciding this, the committee takes into account, among other things, the views of regional metrology organizations. For each comparison, a pilot institute is identified to take the main responsibility for running the CCRI(II) key comparison.
- In drawing up the provisional list of participants and an approximate timetable, the Consultative Committee ensures, as far as possible, that an adequate number of participants from each of the main RMOs take part so that subsequent corresponding regional comparisons can be linked properly to the CIPM comparison.
- In some key comparisons the number of participants may be limited for technical reasons (short half life).
- Two or three institutes from the provisional list are nominated by the Consultative Committee or the key comparison working group (KCWG) to help the pilot institute in drawing up the technical protocol and timetable for the comparison.
- The timetable of this and any other comparisons decided by the Consultative Committee is discussed to ensure that the work load of the whole set is not too great for the participating and pilot institutes, and that the results will be available for the next meeting, normally in two years time.

5. Organization of a key comparison

The organization of a CCRI(II) key comparison is the responsibility of the pilot institute helped by the two or three nominated participants. The first task of this small group is to draw up the detailed technical protocol for the comparison (see paragraph 6 below) and its dispatch, inviting participation as defined by the Consultative Committee. The invitation to participate is normally sent directly to those NMIs with primary standardization techniques available in their radioactivity laboratories. For rules on eligibility for participation in Consultative Committee key comparisons see the Note in Appendix 1 (paragraph 6 of the MRA). The protocol agreed by the participants is sent to the BIPM executive secretary of the Consultative Committee to circulate through the CCRI(II) members for approval.

The main points decided by the small group headed by the pilot institute are the following:

- the list of participants with full details of mailing and electronic addresses;
- the solution to be used in the comparison;
- whether or not a trial comparison or any other preliminary work needs to be carried out among a restricted number of participants to verify the homogeneity and measurability of the solution;
- the pattern of the full scale comparison: this ranges from the sending of individual samples of the solution directly to each participant by the pilot institute to the simple circulation of a single travelling standard or transfer instrument around all the participants, or some combination of these. The need to send some or all of the samples to the BIPM to link the CCRI comparison to the SIR should be considered, taking into account the limitations of the SIR (gamma emitters of sufficient energy and with a minimal level of activity concentration);
- the starting date, detailed timetable and deadlines, means of transport and, if relevant, the itinerary to be followed by each travelling standard;
- the procedure in the case of failure of a travelling standard. The detection of unexpected impurities in the solution by some participants is not considered as a failure. Indeed the measurement of impurities is part of the comparison. Consequently, this information must not be revealed to the other participants until after the dead-line for reporting results has passed;
- the procedure in the case of an unexpected delay at a participant institute;
- the customs documents to accompany the travelling standards, either ATA Carnet or some other for those participants not qualifying for the ATA scheme, and documents related to the transportation of radioactive material (Pro forma invoice, dangerous material certificate, authorisation of importing radioactive material,…)

The executive secretary of the CCRI(II) must be informed at each stage of the key comparison, preferably by filling in the form shown in Appendix 2 to these guidelines.

6. The technical protocol for a key comparison

The pilot institute together with two or three nominated participants draws up the detailed technical protocol. The technical protocol is an important part of the comparison and specifies in detail the procedure to be followed for the comparison. It is important to remember, however, that the purpose of a key comparison is to compare the standardizations made in the participating institutes, not to require each participant to adopt precisely the same standardization method or entire set of parameters. The protocol should, therefore, specify the procedures necessary for the comparison, but not detailed procedures for the standardization methods to be used. However the protocol should include a reporting form where the detail of the measurement method and experimental conditions, or a reference to these, are given by the participants.

Among the points treated in the protocol are the following:
- Detailed description of the samples: radionuclide, recommended half-life value, reference date, chemical composition, source production mode, sample identifier, solution mass, packaging etc…
- The radioactive impurities in the solution identified by the pilot laboratory are listed. It is part of the comparison to determine and report their activity. It should be noted that the absence of impurity data in the protocol is not a guarantee that none is present.
- Actions to be taken on receipt of the samples in a participating institute.
- Any tests to be carried out before measurement.
- Advice on measuring the residual activity in the ampoule.
- The conditions of use of the travelling transfer instrument during measurement and technical data needed for its operation, if relevant.
- Instructions for reporting the results.
- A list of the principal components of the uncertainty budget to be evaluated by each participant, and any necessary advice on how uncertainties are estimated (this is based on the principles laid out in the Guide to the Expression of Uncertainty in Measurement, published by ISO). In addition to the principal components of the uncertainty, common to

all of the participants, individual institutes must add any other components they consider appropriate. Uncertainties are evaluated at a level of one standard uncertainty and information must be given on the number of effective degrees of freedom, required for a proper estimate of the level of confidence, where this is appropriate.
- The traceability to the SI of a standard or standardization method used in the comparison.
- A timetable for the communication of the results to the pilot institute. Early communication helps to reveal problems during the comparison.
- Financial aspects of the comparison, noting that in general each participating institute is responsible for its own costs for the measurements, transportation and any customs charges as well as any damage that may occur within its country. Overall costs of the organization of the comparison including the supply of the radioactive solution are normally born by the pilot institute.
- Insurance of transfer devices is decided by agreement among the participants taking account of the responsibility of each participant for any damage within its country.

The protocol is sent to the CCRI(II) members for approval before the start of the comparison (see paragraph 5).

7. Circulation of the samples and customs formalities

The pilot institute is responsible for organizing the transport of the samples in appropriate packages (e.g. type A package for radioactive samples) and ensuring that the participants make proper arrangements for local customs formalities. Sample details such as sample identifier, mass of solution, chemistry and impurities should accompany the sample inside the package.
The equipment must be handled with care, i.e., only by qualified metrology personnel. As goods are usually delivered to a shipping department in an institute, a warning note should be attached to the package indicating that the package should be opened only by laboratory personnel.
After arrival of the package, the participating institute shall inform the pilot institute of this. Immediately after receipt, the participating institute shall check for any damage of the samples, and report this to the pilot institute.
If a delay occurs the pilot institute shall inform the participants, the KCWG and the executive secretary and revise, if necessary, the time schedule.

8. Reporting the results of a comparison

The participating institutes must report the results of a comparison to the pilot institute as soon as possible and in any case before the deadline. Participants who have reported their result are entitled to revise their value up to the deadline. If for some technical reason (customs delay, unexpected impurity in the solution, ...), the deadline needs to be postponed, all the participants must be informed immediately.

The measurement results together with the uncertainties and any additional information required should be reported in the format given in the instructions as part of the protocol. This is usually achieved by completing the standard forms annexed to the instructions. In particular, the participants should describe the mathematical model used in their measurement analysis.

9. Preparation of the report on a key comparison

The pilot institute is responsible for the preparation of a report on the comparison. The report passes through a number of stages before publication, and these are referred to here as drafts A and B.

The following schedule and actions/consultations are required:

- During the comparison, as the results are received by the pilot institute, they are kept confidential by the pilot institute until all the participants have completed their measurements and all the results have been received, or until the deadline for receipt of results.
- Two weeks before the end date of the comparison, the participants are reminded of the approaching deadline for the results, to include the activity concentration value and the associated uncertainty budget.
- A result from a participant is not considered complete without an associated uncertainty, and is not included in the draft report unless it is accompanied by an uncertainty supported by a complete uncertainty budget. Uncertainties are drawn up following the guidance given in the technical protocol.
- If a participant submits several results corresponding to different standardization methods, the participant is asked to also give the single value and uncertainty (e.g. one of the results or a weighted mean of some or all results) that represents its national reference, and which will be used to calculate the degrees of equivalence for the KCDB.
- Participants that do not provide their results and uncertainty budgets by the deadline are assumed to have withdrawn from the comparison.
- On examination of the complete set of results, if the pilot institute finds results that appear to be anomalous (i.e. one that could be identified as an "outlier" as defined by the KCWG), the corresponding institute(s) is (are) contacted but without being informed as to the magnitude or sign of the apparent anomaly. A one-month deadline is given to check their results for numerical errors. If no numerical error is found the result stands and the complete set of results is sent to all participants.
- The first draft, draft A, is prepared as soon as all the results have been received and anomalous results have been checked by the participants. It includes a brief description of the comparison, the results with the uncertainty budget, the standardization methods (and the codes as defined by the KCWG) and perhaps a graph of the results identified by participant. The draft A could be more complete if there is time to do this.
- Draft A of the report is sent not later than 5 weeks after the deadline to all the participants for comment, with a one-month deadline for replies. The date at which this draft is sent to the participants is taken to be the end date for the comparison and is subsequently referred to as such.
- Draft A is considered as confidential to the participants. Copies are not given to non-participants, and graphs or other parts of the draft are not used in oral presentations at an external Conference without the specific agreement of all the participants.
- Note that once all participants have been informed of the results, individual values and uncertainties may be changed or removed, or the complete comparison abandoned, only with the agreement of all participants and on the basis of a clear failure of a travelling standard or some other phenomenon that renders the comparison or part of it invalid. The participants may ask for additional information if this is needed to determine whether a comparison should be abandoned or not.
- On receipt of final comments from participants on the draft A, the first version of draft B is distributed to the participants and the KCWG and is no longer confidential. Subsequently, the status of the comparison is indicated as “Draft B report in progress” in the relevant KCDB web page on the BIPM site. At this stage, a participant may publish experimental techniques of special interest or new developments of a measurement method made in the frame of the comparison, as long as no interpretation is made of the comparison results.

In the case when the comparison is linked to the SIR, all the experimental details are incorporated by the pilot laboratory in a full report that, once agreed by the participants and the CCRI(II), is published often as a *Rapport BIPM*. At the same time, the BIPM produces a draft B wherein the link between the CCRI comparison and the SIR is presented for the KCWG. The key comparison reference value (KCRV) is defined in the frame of the SIR and the degrees of equivalence are evaluated for the participants of the CCRI comparison through the link, and added to the matrix of any pre-existing SIR results of non-participants of the CCRI comparison.

In the case when the comparison is not linked to the SIR, the draft B, is prepared within two months by the pilot laboratory incorporating all the experimental details. The pilot institute

produces an Appendix to draft B containing proposals for a reference value (KCRV) and degrees of equivalence in accordance with the recommendations of the CCRI(II).

In both cases the following instructions apply:

- The draft B including the Appendix is circulated through the participants and the KCWG normally not later than 3 months after the end date of the comparison with a one-month deadline for comments. If any controversial or contradictory comments are received by the pilot institute, they are circulated to all participants and discussion continues until a consensus is reached. Once agreed, the content of draft B may be the subject of publication, with the exception of the Appendix containing proposals for the reference value and degrees of equivalence. (see paragraph 11 below on publication)
- The draft B including the Appendix is sent to the CCRI(II) for review and approval. The final approval by the Consultative Committee may be given by electronic correspondence on the recommendation of the working group on key comparisons. The CCRI(II) needs to set its own procedures for approving the results of key comparisons in the most efficient and timely way possible.
- The entry of the results, including the degrees of equivalence, into Appendix B of the MRA (the key comparison data base) must wait until draft B has been formally reviewed and approved by the Consultative Committee when it becomes the Final Report.
- An institute that considers its result unrepresentative of its standards may request a subsequent separate bilateral comparison with the pilot institute or another of the participants. This should take place as soon as possible after the completion of the comparison in progress. The subsequent bilateral comparison is considered as a new and distinct comparison (see paragraph 10).
- In the event that there is disagreement concerning the results or the interpretation of the results of a key comparison, and the disagreement cannot be resolved by the participants, by the KCWG or by the Consultative Committee, the matter is referred to the CIPM for decision.

10. Bilateral key comparisons

A bilateral key comparison, referred to in paragraphs T.8 and T.9 of the MRA and in paragraph 9 above, may be carried out by two institutes meeting the following conditions:

(a) one of them must have already participated in the relevant CIPM or RMO key comparison; this institute acts as pilot for the bilateral comparison which must use the same or a similar protocol as for the key comparison;

(b) the other must be an NMI that meets the requirements for participation in a key comparison given in paragraph 6 of the MRA.

A bilateral comparison must be carried out following the parts of these Guidelines that are appropriate for bilateral key comparisons.

The executive secretary of the CCRI(II) and the chairman of the KCWG must be informed of a bilateral key comparison before it takes place.

The BIPM ongoing key comparisons, the SIR and its extension to beta-emitters, being a continuing series of bilateral comparisons, is treated separately, see paragraph 2 above.

11. Publication of the results of a key comparison and entry into Appendix B of the MRA and the key comparison data base (KCDB).

For all key comparisons, the Final Report approved by the Consultative Committee forms the basis for the entry of results into Appendix B of the MRA (the key comparison data base). Publication of the results in draft B, with the exception of the Appendix containing the proposed reference value and degrees of equivalence, may take place, if so desired, as soon as draft B is agreed by the participants.

There are different forms in which the results of a key comparison may be published, depending on the wider significance of the information. The main publication channels are the following:

- publication of the Final Report *in extenso* as a BIPM Report, or in electronic form in the *Metrologia Technical Supplement* on the web.
- publication of an extended paper in *Metrologia* or some other journal;
- publication in a shortened form in *Metrologia* or some other journal;
- publication in a Conference Proceedings following presentation at a Conference;

A combination of more than one of these channels is possible.

The reference to the Final Report or another extended publication in which details of the comparison can be found is given in the KCDB.

12. Supplementary comparisons

Supplementary comparisons whose results are intended to be included in Appendix B.3 of the MRA should be carried out following these guidelines. For more information about Supplementary comparisons, see document CCRI(II)/03-05.

APPENDIX 1

Note on eligibility for participation in a CIPM or RMO key comparison

The following is an extract from the agreement on mutual recognition of national measurement standards and calibration certificates issued by national metrology institutes:

6. Participation in key and supplementary comparisons

6.1 Participation in a CIPM key comparison is open to laboratories having the highest technical competence and experience, normally the member laboratories of the appropriate Consultative Committee. Those laboratories that are not members of a Consultative Committee and not NMIs must be nominated by the designated national metrology institute referred to in paragraph 1.4. as being responsible for the relevant national measurement standards. In choosing participants, the Consultative Committees must take proper account of regional representation. The number of laboratories participating in CIPM key comparisons may be restricted for technical reasons.

6.2 Participation in key comparisons organized by an RMO is open to all RMO members and to other institutes that meet the rules of the regional organization (including institutes invited from outside the region) and that have technical competence appropriate to the particular comparison.

6.3 Participation in RMO supplementary comparisons is open to those institutes meeting the requirements specified in paragraph 6.2.

APPENDIX 2

STATUS REPORT ON CCRI OR RMO COMPARISON

1. CCRI Section:	2. CCRI Ref No (to be completed by the BIPM):

3. Type of comparison: CCRI ☐ RMO ☐
Key ☐ Supplementary ☐ Pilot study ☐

4. Subject area:

5. Participating institutes (*and countries*):

Bilateral ☐

6. Pilot laboratory:

7. Measurand / unit (nominal value(s)):

8. Description:

9. Progress: *(Please note date and tick appropriate box to indicate current status)*

Date	Status	Pilot	Supplementary	Key
	Proposed to CCRI	☐	☐	☐
	Accepted and registered	☐	☐	☐
	Protocol submitted to CCRI	☐	☐	☐
	Protocol agreed	☐	☐	☐
	Measurements in progress	☐	☐	☐
	Measurements completed	☐	☐	☐
	Report agreed	☐	Draft A ☐	Draft A ☐
			Draft B ☐	Draft B ☐
	Report submitted to CCRI	☐	☐	☐
	Results approved	☐	☐	☐
	Approved for Equivalence			☐
	Progression to Key Comparison	☐		
	Abandoned	☐	☐	☐

Comments:

Publication reference:

10. Measurement start date:	11. Expected measurement completion date:

12. Contact person's name:
Address:

Telephone: Fax:
e-mail : Web address:

13. Contact Person's signature:	14. Date:

Completed copy to be forwarded to a) CCRI Executive Secretary, b) Regional coordinator and c) relevant Key Comparison WG Chairman