The BIPM key comparison database (KCDB): linkage of key comparison results

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Abstract

The Appendix B of the BIPM key comparison database (KCDB) maintains records of some 600 key and supplementary comparisons conducted by the BIPM, Consultative Committees (CCs) of the CIPM, and Regional Metrology Organizations (RMOs). Results of key comparisons are interpreted in terms of equivalence and published via the KCDB after formal approval by the appropriate CC. It now displays the results of 170 comparisons. In April 2005, the results of 24 RMO key comparisons were linked to those of the corresponding CC key comparisons, and the full sets of degrees of equivalence were published via the KCDB. The same type of linkage is also carried out for nine CCRI key comparisons of radionuclide activity, linked to the corresponding ongoing BIPM SIR key comparisons, and for another four CC bilateral comparisons subsequent to full-scale CC key comparisons. This paper summarizes the different methods that were used to establish the linkages currently published via the KCDB, and shows some typical examples.

Introduction

In 1999, the Comité International des Poids et Mesures (CIPM) drew up an arrangement for the "*mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes*": the <u>CIPM MRA</u> (Mutual Recognition Arrangement) [1]. Its objectives are to provide international recognition of the realization of national standards, and to provide confidence in the measurement capabilities of participating laboratories for all users, including the regulatory and accreditation communities, thus reducing technical barriers to trade arising from lack of traceability and equivalence.

The principal output of the CIPM MRA is the BIPM key comparison database (KCDB), maintained at the Bureau International des Poids et Mesures (BIPM). The KCDB Website is publicly available at <u>http://kcdb.bipm.org/</u>. All the information it contains is internationally recognized through the procedures described in the text of the CIPM MRA, and always kept up to date.

The information in the KCDB is divided into four parts corresponding to the appendices of the text of the CIPM MRA (*Figure 1*): 1) the Appendix A is the list of the

participating institutes, 2) the Appendix B contains information on key and supplementary comparisons, 3) the Appendix C contains Calibration and Measurement Capabilities (CMCs) declared by participants, and 4) the Appendix D lists the key comparisons.



Figure 1. KCDB Website Home Page: access to the four appendices.

This paper is focused on the content of the KCDB Appendix B, more specifically on the different types of comparisons that are recorded, and how their results are interpreted in terms of equivalence.

<u>1. International comparisons</u>

Appendix B keeps records of international (key and supplementary) comparisons selected and managed by the Consultative Committees (CCs) of the CIPM and by the Regional Metrology Organizations (RMOs).

<u>1.1. Types of comparisons</u>

1.1.1. Key comparisons

A "*key comparison*" is one of the set of comparisons selected by a Consultative Committee (CC) to test the principal techniques and methods in the field. Its protocol is agreed by the CC, and includes information such as the nominal values of the measurand and of the influence parameters, the dates of measurement, the methods, the transfer standards, the pilot laboratory, etc.

Key comparisons are denoted "*CIPM key comparisons*" if carried out by one of the CCs or by the BIPM, and "*RMO key comparisons*" if carried out by one of the RMOs within its region. An RMO key comparison can be initiated only if a CIPM (CC or BIPM) key comparison with the same protocol has already been decided.

1.1.2. Supplementary comparisons

A "supplementary comparison" is one of the set of comparisons conducted by the RMOs to meet specific needs not covered by key comparisons, including comparisons to support confidence in calibration and measurement certificates. Laboratories from countries outside the region may be invited to participate.

1.1.3. Bilateral comparisons

An issue often raised in CC meetings relates to the case of bilateral comparisons.

- The CIPM MRA has foreseen the case of "subsequent bilateral comparisons to key comparisons" for those participants, which need to redo the measurements. It may happen that such subsequent key comparisons have more than two participants. These are recorded as CC or RMO key comparisons, and a comment is added stating which key comparison they follow up.
- Some RMO or CC key comparisons are bilateral ones because there are only two participants. This often happens after the full-scale comparison is completed simply because the new comer was not ready in time.
- The "*BIPM key comparisons*" are series in time of bilateral comparisons between a National Metrology Institute (NMI) and the BIPM. These are special cases of CIPM key comparisons.

1.2. Registration of comparisons

1.2.1. Identifier and information

A key or supplementary comparison is registered into Appendix B only when the BIPM KCDB office receives from the appropriate body (a CC or an RMO) a formal authorization to do so.

On 13 April 2005, 489 key comparisons and 117 supplementary comparisons were effectively registered.

Note: All numbers given in the following reflect the content of Appendix B as on 13 April 2005.

The Appendix B has a search engine for the user at http://kcdb.bipm.org/AppendixB/KCDB ApB search.asp

allowing the access to the list of comparisons corresponding to a selection of search criteria. A unique identifier is attributed to each of them according to an agreed <u>nomenclature</u> [2]. Clicking on the identifier returns the page of information on the comparison, together with the list of participants, the pilot laboratory and the contact person's name and address details. A link is provided to its Final Report and to its results (for key comparisons only) when they become available.

1.2.2. Status

The status of a comparison is an important feature given in the information page of all comparisons. It indicates the progress of the work from the beginning, status "Planned", to the end of the exercise, status Approved for equivalence, Results available" for key comparisons or "Approved" for supplementary comparisons. The case of BIPM key comparisons is different since they last as long as the need exists. They are thus attributed the status "Ongoing" or "Ongoing, Results available".

At the beginning of the implementation of the CIPM MRA, CCs were requested to choose a number of comparisons already completed in order to set up a basis for the first sets of CMCs [3]. The corresponding key and supplementary comparisons received the status

"Approved for provisional equivalence" and "Published", respectively. An open-literature reference is inserted in the information page, but no values or graphs are displayed.

This status is attributed to 88 key comparisons (over a total of 489). Provision is made in the KCDB to turn these key comparisons to archives (returned by the Web on request only), as soon as new results become available. It follows that 401 key comparisons are conducted according to the <u>CIPM MRA guidelines</u> [4].

1.2.3. Appendix B content

The Appendix B content is illustrated on Figure 2.

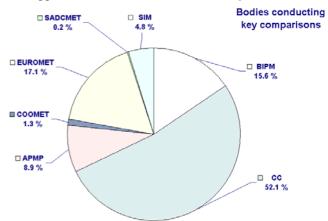


Figure 2. Appendix B: repartition of the key comparisons recorded in the Appendix B, according to the body under the auspices of which they are conducted [5].

1.3. Families of key comparisons

Key comparisons are organized to form families of comparisons, as shown in *Figure 3*. Each family is centred on a CIPM key comparison, which we will designate as "*master CIPM key comparison*" in the following, to which RMO key comparisons and bilateral comparisons are linked.

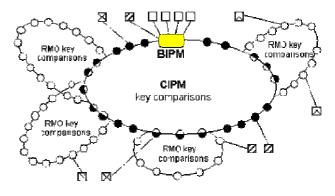


Figure 3. Appendix B: overall organization of CIPM and RMO key comparisons.

- Full circles: participants in CC key comparisons,
- Half full circles: participants in CIPM and RMO key comparisons,
- Empty circles: participants in RMO key comparisons only,
- Empty squares: participants in BIPM key comparisons,
- Crossed and hachured squares: participants in bilateral key comparisons,

This grouping is generally made apparent in the KCDB Appendix B by the use of a common part in the identifiers of all comparisons belonging to the family (*Table 1*).

Identifier	Organizing body	Status
CCAUV.A-K1	CCAUV full-scale CIPM key comparison	Approved for equivalence, Results available
APMP.AUV.A-K1	APMP	In progress
COOMET.AUV.A-K1	COOMET	Report in progress, Draft A
COOMET.AUV.A-K1.1	COOMET Subsequent bilateral to the previous one	Measurements completed
EUROMET.AUV.A-K1	EUROMET	Approved for equivalence
SIM.AUV.A-K1	SIM	Report in progress, Draft B
SIM. <mark>AUV.A-K1</mark> .PREV	SIM	Approved for provisional equivalence

Table 1. List of key comparisons forming a family, example of "AUV.A-K1", together with their status. The family is composed of seven key comparisons in the field of acoustics on calibration of laboratory microphones type LS1P. (See [5] for information on the RMO full names.)

Common participation between the master CIPM key comparison and any of the other key comparisons of the family is required for interpreting the results in terms of global equivalence (see section 4).

2. Results of CIPM key comparisons

Measurements obtained by laboratories participating in a master CIPM key comparison are interpreted in terms of equivalence as foreseen in the text of the <u>Technical</u> <u>Supplement to the CIPM MRA</u> [6], which introduces the notion of "degrees of equivalence" among the measurement standards of laboratories participating in the key comparison. The resulting numbers and graphs (often designated as "*results*") are inserted in the Final Report, and also displayed in Appendix B.

2.1. Paragraphs T.1 and T.2 of the Technical Supplement to the CIPM MRA

2.1.1. Paragraph T.1

It states that "CIPM key comparisons lead to reference values, known as key comparison reference values".

Key comparison reference values are thus deduced exclusively from measurements obtained in CC and BIPM key comparisons.

2.1.2. Paragraph T.2

It states that "The degree of equivalence of each national measurement standard is expressed quantitatively by two terms: its deviation from the key comparison reference value and the uncertainty of this deviation (at a 95 % level of confidence). The degree of equivalence between pairs of national measurement standards is expressed by the difference of their deviations from the reference value and the uncertainty of this difference (at a 95 % level of confidence)".

Results are thus given as degrees of equivalence, but the performance of one participant relative to the others is not judged on a scale from "worst to best".

2.2. Practical realization

In practice, the results of a master CIPM key comparison, obtained for one value of the measurand or one value of an influence parameter, generally take the form of a four-sheet folder, including: 1) the laboratory individual measurements, 2) the equivalence statements, 3) the matrix of equivalence, and 4) the graph of equivalence.

2.2.1. Laboratory individual measurements

The measurements and combined standard uncertainties obtained by the participants are presented in a table, together with the date of measurement, and any other information of interest (method, transfer instrument serial number, etc. (see an example in *Figure 4*).

X	relative difference between the result of measurement
	of laboratory <i>i</i> and that of the BIPM
<i>u</i> ::	combined standard uncertainty of x:

	complice standard uncertainty of x7					
Lab i	x _i	u _i	Date			
	/ 10 ⁻⁸	/ 10 ⁻⁸	of measurement			
CEM	-8.0	9.0	96-06			
JV	2.7	6.2	97-06			
NML(IE)	3.0	20.0	98-04			
CMI	11.0	10.0	98-06			
INPL	18.0	15.0	98-12			
NML(IE)	-3.0	21.0	00-04			
NML(IE)	-7.0	23.0	02-03			
KRISS	3.1	4.2	03-06			
NML(IE)	-7.1	23.0	04-04			

Laboratory using a quantum Hall resistance standard

Figure 4. Example of the laboratory individual measurements obtained in the ongoing BIPM key comparison <u>BIPM.EM-K13.a</u> of 1 ohm resistance standards. Full names of the participants are available from <u>Appendix A</u>.

2.2.2. Equivalence statements

The process used for computing the key comparison reference value, it combined standard uncertainty and the degrees of equivalence is explained in a few sentences (see *Figure 5*).

```
Key comparison reference value: the BIPM value.

Its standard uncertainty is evaluated to be 1.5 \times 10^{-8}

and is included in the u_i's values.

The degree of equivalence of each laboratory with respect

to the reference value is given by a pair of terms:

D_i = x_i and its expanded uncertainty (k = 2), U_i,

both expressed in 10^{-8}.

The degree of equivalence between two laboratories i and

j is given by two terms:

D_{ij} = D_i \cdot D_j and its expanded uncertainty (k = 2), U_{ij},
```

Figure 5. Example of the equivalence statements (truncated on the bottom) stated for BIPM.EM-K13.a.

2.2.3. Matrix of equivalence

It is a table composed of two parts:

- the "blue part" including the degrees of equivalence relative to the key comparison reference value, and
- the "yellow part" including the pair-wise degrees of equivalence.

An example is shown on Figure 6.

Lab j ===>										
Lab <i>i</i> []				CEM		JV		CMI		
4	D_i	Ui		Dij	U_{ij}	Dij	Uij	Dij	Uij	E
	/ 10 ⁻⁸	/ 10 ⁻⁸		/ 10 ⁻⁸	/ 1					
CEM	-8.0	18.0				-10.7	21.8	-19.0	26.9	-2
JV	2.7	12.4		10.7	21.8			-8.3	23.5	-1
CMI	11.0	20.0		19.0	26.9	8.3	23.5			-7
INPL	18.0	30.0		26.0	35.0	15.3	32.4	7.0	36.0	
KRISS	3.1	8.4		11.1	19.8	0.4	14.9	-7.9	21.6	-1
NML(IE)*	-7.1	46.0		0.9	49.4	-9.8	47.6	-18.1	50.1	-2
* For this I	* For this laboratory, only the most recent comparison is retained									

* For this laboratory, only the most recent comparison is retained.

Figure 6. Example of the matrix of equivalence (truncated on the right side) obtained in BIPM.EM-K13.a.

2.2.4. Graph of equivalence

A graphical representation of the blue part of the matrix of equivalence is drawn up: the zero horizontal axes corresponds to the key comparison reference value and each point represents an individual degree of equivalence relative to the reference value (see *Figure 7*).

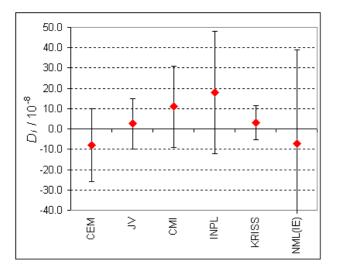


Figure 7. Example of the graph of equivalence obtained for BIPM.EM-K13.a.

2.3. Results already published for master CIPM key comparisons

Among the 401 CIPM and RMO key comparisons registered in Appendix B and conducted according to the CIPM MRA guidelines, 171 have their Final Reports approved by the appropriate CC and posted in the database.

Among these, 134 are master CIPM key comparisons, 50 % of which are BIPM on-going key comparisons. Their measurements are generally obtained for several nominal values of the measurand or of an influence parameter, leading to a total of about 350 sets of results interpreted in terms of equivalence as explained in section 2.2. They are all entered in Appendix B, and thus made publicly available via internet under the form of html folders (*Figure 8*) and .pdf files.

The laboratory individual measurements are not explicitly displayed when they are too complex. In such a

case, a summary is available together with a link to the corresponding tables and pages of the Final Report.

APPENDIX B	APPENDIX C	A	PPENDIX D	
Search form > Results of	the search > BIPM.	EM-K13.a results		
			KCDI	
BIPM.EM-K13.a				
۲ Results				
Laboratory individual measurements	Equivalence statements	Degrees of equivalence	Graph(s) of equivalence	
MEASURAND : Resista NOMINAL VALUE : 1 0				

Figure 8. Access to the folder of a set of results as presented in Appendix B (example of BIPM.EM-K13.a, measurand value: 1 ohm). It is composed of four html pages containing the information organized as explained in section 2.2.

The equivalence statements constitute the core information, and are always available from the second page of the folder. The colour code (red characters, light blue and yellow-beige) shown in *Figure 5* is respected.

In some exceptional cases (paragraph T.3 of the <u>Technical Supplement to the CIPM MRA</u> [6]), a CC may conclude that for technical reasons a reference value for a particular set of results is not appropriate. In those cases the key comparison has no reference value. An explanatory sentence should be inserted in the equivalence statements page. This applies at present for only 7 sets of results (see paragraph 3.1.8).

The matrix of equivalence is available from the html page "Degrees of equivalence".

The degrees of equivalence relative to the key comparison reference value are always computed (except in the very few cases where there are no key comparison reference values) and presented under the form of a "blue table" (*Figure 9*).

The pair-wise degrees of equivalence (yellow-beige part of the matrix) are not always given. If not, the equations are inserted is the page "Equivalence statements". Among the 350 sets of results published, 45 do not display pair-wise degrees of equivalence.

The pair-wise degrees of equivalence are given as "Lab *i* - Lab *j*" Select Lab *j* CMI

Lab i	D _i	U_j	D _{ij}	U _{ij}
	/ 10 ⁻⁸	/ 10 ⁻⁸	/ 10 ⁻⁸	/ 10 ⁻⁸
CEM	-8.0	18.0	-19.0	26.9
JV	2.7	12.4	-8.3	23.5
CMI	11.0	20.0		
INPL	18.0	30.0	7.0	36.0
KRISS	3.1	8.4	-7.9	21.6
NML(IE)*	-7.1	46.0	-18.1	50.1

Figure 9. Presentation of the matrix of equivalence in Appendix B (degrees of equivalence of each participant relative to the key comparison reference value and pair-wise degrees of equivalence relative to the Czech institute of metrology CMI for key comparison BIPM.EM-K13.a; same numbers as in Figure 6).

The "yellow table" is generally too large to be easily readable. It is thus split into partial tables, each of which displays the degrees of equivalence of laboratory i relative to a selected laboratory j (*Figure 9*).

Clicking on "Graph(s) of equivalence" in a results folder gives access to the graph of equivalence. About 350 graphs of equivalence are currently published.

If the graph contains too much information, it is presented under the form of a stamp on which the user can click to access a full-screen image (*Figure 10*).

Click on the stamp to get the corresponding graph of equivalence when available



Figure 10. Presentation of the graphs of equivalence as stamps (example of key comparison CCTF-K2001.UTC on the computation of the reference time scale Coordinated Universal Time, UTC, the graph for March 2005 is not yet made available).

3. Published reference values and degrees of equivalence

The aim of this section is to give an overview of the methods used for computing the key comparison reference values and degrees of equivalence that are currently published. It is by no means a comprehensive catalogue of the recipes that could be found in the open literature.

3.1. Principal methods

An examination of the equivalence statements stated for the 134 master key comparisons published in Appendix B shows that the key comparisons reference values and degrees of equivalence have been so far computed as follows.

<u>Note</u>: Only typical well-known formulas are evoked here. Detailed equations can be found through the links to the KCDB Appendix B given from the specific examples mentioned below.

3.1.1. Arithmetic mean

The key comparison reference value is computed as the arithmetic mean of the laboratory individual measurements, outliers excluded (if identified).

Its combined standard uncertainty is generally the standard deviation of the mean, and thus does not depend on the laboratory individual uncertainties (among others, this is the case of the on-going BIPM key comparisons of activity of radionuclide carried out in the framework of the International system of Reference, SIR; for example BIPM.RI(II)-K1.Co-57, SIR equivalent activity of ⁵⁷Co). In some cases its standard uncertainty is, however, computed according to the external consistency concept, and does take into account the individual uncertainties (example of key comparison <u>CCQM-K9</u> on pH measurements for 17 pH nominal values).

The degrees of equivalence are computed as the offsets of each laboratory individual measurement to the reference value, and as the expanded uncertainty of this offset. The correlation between the uncertainty of the mean and the individual uncertainty of the laboratory is generally taken into account. The pair-wise degrees of equivalence are computed as the offset between the two laboratory individual measurements, and as its expanded uncertainty, often approximated by twice the root mean square of the individual uncertainties.

Note that the pair-wise degrees of equivalence do not depend upon the value and the uncertainty of the reference value. In addition, the expanded uncertainties at a 95 % level of confidence are generally estimated from a coverage factor equal to 2.

3.1.2. Weighted mean

A mean is computed using weights proportional to the inverse square of the individual uncertainties. The combined standard uncertainty of the reference value is usually the standard deviation of the weighted mean, and has a value smaller than any of the individual uncertainties (example of <u>CCEM-K6.c</u> on AC/DC voltage transfer difference for five frequency values). In a few cases, the uncertainty of the reference value has been set at the value zero by definition (example of <u>CCT-K2</u>, key comparison of CSPRT, for seven nominal values of the temperature).

The degrees of equivalence are generally computed as for the arithmetic mean, the expanded uncertainty of those relative to the reference value being equal to twice the difference of the squared uncertainties of the laboratory and of the reference value. Note that this sign "minus" is sometimes replaced by a sign "plus", which gives a conservative estimate.

3.1.3. Weighted mean with a maximum weight

This method is used when the CC recognizes that some individual uncertainties are smaller than the state-of-theart (example of the key comparison <u>CCPR-K3.b</u> on luminous responsivity of photometers). These are artificially turned to the minimal uncertainty value normally achievable, which gives to the corresponding measurements a maximum weight. A dedicated formula is used for computing the combined standard uncertainty of the reference value and the expanded uncertainties included in the degrees of equivalence.

3.1.4. Median

The median is generally used when a robust statistic is needed. The combined standard uncertainty is the standard deviation of the median (example of <u>CCM.M-K3</u> on mass standards of nominal value 50 kg).

3.1.5. Value of an international standard

The reference values of the BIPM key comparisons in electricity and length are set to be equal to the value delivered by the standard maintained at the BIPM. Since the individual measurements are made relative to the BIPM value, the uncertainty of the reference value is included in the uncertainties of individual measurements. These thus represent the degrees of equivalence relative to the reference value. The pair-wise degrees of equivalence are obtained directly from the degrees of equivalence relative to the reference value to the BIPM standard is taken into account (example of <u>BIPM.EM-K13.a</u>).

3.1.6. Global value known a priori

The measurand is a ratio that should be equal to 1 with an uncertainty equal to zero (example of $\underline{\text{BIPM.RI(I)-K2}}$ on air-kerma for five values of the radiation quality). The

uncertainties included in the degrees of equivalence involve the individual uncertainties and their correlations.

3.1.7. Individual values known *a priori*

The key comparison is organized as a distribution of samples that are measured by the pilot laboratory using a high-order technique (case of most of the chemical measurements obtained by gravimetry, specifically when gas cylinders are used, example of <u>CCQM-K3</u> on automotive emission gases). It follows that each participant is attributed a different reference value. The pair-wise degrees of equivalence depend upon the individual gravimetric values and uncertainties.

3.1.8. No reference value

The comparison <u>CCT-K3</u> (realization of the ITS-90, seven temperature fixed points) is the unique case where it was decided that no key comparison reference value could safely be estimated. Only pair-wise degrees of equivalence can thus be calculated. The graphs are drawn up by reference to each of the participants. There thus does not exist one unique graph of equivalence for one temperature value, but as many graphical representations as there are participants in the comparison.

3.2. Usage of the principal methods

Figure 11 give the repartition of the methods used for the computation of the key comparison reference values published in Appendix B (the case of no reference value is excluded from this study).

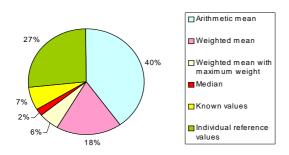


Figure 11. Methods used to compute the key comparison reference values published in Appendix B. The different items given in the caption are explained above; "Known values" correspond to values of international standards maintained by the BIPM and to global values known a priori; "Individual reference values" correspond to individual values known a priori.

The method based on the arithmetic mean of the individual measurements dominates. This is a consequence of the publication of 56 results from the 59 BIPM radioactivity key comparisons.

Gravimetric measurement are carried out for many different gases and analytes contained in samples used for key comparisons in Chemistry. This explains the large occurrence of individual reference values in Appendix B.

4. Published linkages

In this section we give an overview on the linking procedures found in Appendix B. Again, no attempt is

made to present a comprehensive catalogue of all possible techniques.

4.1. Situation

As already mentioned previously, 134 master CIPM key comparisons have their results published in Appendix B, and another 37 RMO and CC key comparisons have received the status "*Approved for equivalence, results available*". This set is composed of:

- 15 EUROMET key comparisons,
- 9 APMP key comparisons, and

• 13 CC key comparisons, among which 4 are bilateral. Their results expand those of the master CIPM key comparisons that constitute the centres of 29 families:

- 23 duos (one linkage to the master, *Table 2*),
- 3 trios (two linkages to the same master, Table 3), and

• 3 quartets (three linkages to the same mater, *Table 4*), leading to 47 expanded graphs of equivalence.

Master CIPM key	Linked	Ν
comparison	key comparisons	
CCAUV.V-K1	APMP.AUV.V-K1	1
CCEM-K4	EUROMET.EM-K4	1
BIPM.EM-K11.b	EUROMET.EM.BIPM-K11	1
CCM.M-K1	APMP.M.M-K1	1
CCM.P-K4	EUROMET.M.P-K1.a	6
CCQM-K1.c	EUROMET.QM-K1.c	2
CCQM-K17	EUROMET.QM-K17	3
BIPM.RI(II)-K1.Ba-133	CCRI(II)-K2.Ba-133	1
BIPM.RI(II)-K1.Ce-139	CCRI(II)-K2.Ce-139	1
BIPM.RI(II)-K1.Cd-109	CCRI(II)-K2.Cd-109	1
BIPM.RI(II)-K1.Co-58	APMP.RI(II)-K2.Co-58	1
BIPM.RI(II)-K1.Cs-143	CCRI(II)-K2.Cs-143	1
BIPM.RI(II)-K1.Cs-137	CCRI(II)-K2.Cs-137	1
BIPM.RI(II)-K1.Eu-152	CCRI(II)-K2.Eu-152	1
BIPM.RI(II)-K1.Ho-166m	APMP.RI(II)-K2.Ho-166m	1
BIPM.RI(II)-K1.I-123	EUROMET.RI(II)-K2.I-123	1
BIPM.RI(II)-K1.Ir-192	CCRI(II)-K2.Ir-192	1
BIPM.RI(II)-K1.Sc-47	EUROMET.RI(II)-K2.Sc-47	1
BIPM.RI(II)-K1.Sc-75	CCRI(II)-K2.Sc-75	1
BIPM.RI(II)-K1.Y-88	APMP.RI(II)-K2.Y-88	1
BIPM.RI(II)-K1.Y-90	CCRI(II)-K2.Y-90	1
BIPM.RI(II)-K1.Yb-169	EUROMET.RI(II)-K2.Yb-169	1
CCT-K2	CCT-K2 .1	7

Table 2. List of the 23 duos of linked key comparisons published in Appendix B on 13 April 2005. N is the number of expanded sets of results obtained from linkage. In blue: acronym of the body conducting the key comparison. In red: the common part of the identifier, featuring the family to which the duo belongs.

Master CIPM key	Linked	N
comparison	key comparisons	
CCM.P-K1.c	APMP.M.P-K1.c	2
	EUROMET.M.P-K2	
CCQM-K3	APMP.QM-K3	3
	EUROMET.QM-K3	
CCQM-K4	APMP.QM-K4	1
	EUROMET.EM-K4	

Table 3. List of the three trios of linked key comparisons published in Appendix B on 13 April 2005 (same colour code than in Table 2).

Master CIPM key	Linked	Ν
comparison	key comparisons	
CCEM-K8	CC.EM-K8.1	2
	EUROMET.EM-K8	
	EUROMET.EM-K8.1	
CCM.P-K4	EUROMET.M.P-K1.a	1
	EUROMET.M.P-K1.b	
	SIM.EUROMET.M.P-BK3	
CCPR-K3.b	CCPR-K3.b.1	1
	CCPR-K3.b.2	
	APMP.PR-K3.b	

Table 4. List of the 3 quartets of linked key comparisons published in Appendix B on 13 April 2005 (same colour code than in Table 2).

Tables 2, 3, and 4 call for two remarks: 1) the identifiers including the characters ".1" and ".2" are subsequent bilateral comparisons; and 2) the key comparison CCM.P-K4 is the centre of 1 duo for 6 nominal values of the pressure ranging from 3 Pa to 1000 Pa, and the centre of 1 quartet for the nominal pressure of 1 Pa (key comparison EUROMET.M.P-K1.a, carried out in the range 1 Pa to 1000 Pa appears thus both in *Table 2* and in *Table 4*).

4.2. Linkage methods used in Appendix B

The linkage between two key comparisons of the same family, and thus the expansion of the results of its master CIPM key comparison, can be established only if there exists a common participation. The linkage is stronger if the common participants show similar performance in both exercises.

The linkage does not modify the value and the uncertainty of the master CIPM key comparison reference value, which remains unique and non-altered for the whole family. It simply extends the matrix of equivalence and the graph of equivalence in order to give evidence on the comparability between institutes that have participated in one of the exercises only. The expanded uncertainty included in their degrees of equivalence is however generally higher than if they had been compared directly.

The linkages reported in Appendix B are all deduced from a case-by-case examination of the situation. The different methods used are thoroughly related to the techniques put in place to conduct the comparisons and to the way the key comparison reference values are computed. In the following, the most typical cases are presented and illustrated with examples.

4.2.1. Individual reference values known a priori

As mentioned above, the gravimetric values attributed to the samples distributed in chemical key comparisons constitute individual reference values for each participant. This applies to the linked key comparison as well as to the master CIPM key comparison. Degrees of equivalence relative to the reference value are thus computed in the same way for all participants.

The linkage could, however, be safely established only if the nominal value of the measurand is the same in both comparisons. It is thus necessary to check that all gravimetric values are very close to each other. Note that this condition is normally fulfilled when the pilot laboratory is the same for both comparisons because all samples are usually made at the same time and in the same environment: splitting the exercise in a CC and an RMO key comparison is here simply artificial. In addition, the common participants (there can be only one: the common pilot laboratory that has prepared the samples) should have obtained similar degrees of equivalence relative to the gravimetric values in both exercises.

The next step is to place all the individual degrees of equivalence in the same table and on the same graph of equivalence with no corrections brought to the offsets and uncertainties.

The family "QM-K4" (amount content of ethanol in air, nominal value of 120 μ mol/mol) is a good example of this process (trio included in *Table 3*). NPL (United Kingdom) is the common pilot laboratory to <u>CCQM-K4</u> and <u>EUROMET.QM-K4</u>, and the linkage is further supported by the performance of LNE (France). NMIJ (Japan) is the pilot laboratory of <u>APMP.QM-K4</u> and also participated in CCQM-K4. The three sets of degrees of equivalence are collected on the same graph of equivalence shown in *Figure 12*.

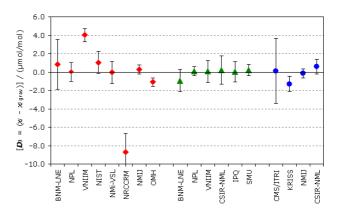


Figure 12. Graph of equivalence of the "QM-K4" trio as published in Appendix B.

Red diamonds: participants in CCQM-K4. Green triangles: participants in EUROMET.QM-K4. Blue circles: participants in APMP.QM-K4.

Note that the common participants have two degrees of equivalence whereas one could consider that only those obtained in CCQM-K4 should be retained. This practice may have, however, an advantage. VNIIM (Russia) participated in CCQM-K4 and EUROMET.QM-K4 with quite different results. The more recent measurement, obtained in EUROMET.QM-K4, may be regarded as the result of a subsequent bilateral key comparison to CCQM-K4, and thus be retained for any further use.

4.2.2. Direct transfer

The family "<u>EM-K8</u>" (DC voltage ratio, nominal values 1000 V / 10 V and 100 V / 10 V) provides another example of direct transfer though the key comparison reference value is not known *a priori*.

The data obtained in both key comparisons CCEM-K8 and EUROMET.EM-K8 are treated separately: two arithmetic means and two sets of degrees of equivalence relative to these means are computed.

The two means are considered as equivalent because it is unlikely that they are affected by systematic errors. This statement is supported by the argument that a significant number of participants (respectively 15 and 19 participants in the CC and EUROMET comparisons), measured the same travelling standard using different methods.

It follows that the degrees of equivalence obtained in the EUROMET exercise are assumed to be the same as if the participants had directly taken part in the CC key comparison.

CCEM-K8.1 and EUROMET.EM-K8.1 are further linked via the common pilot laboratory of all four comparisons, IEN (Italy).

Note that it was judged that pair-wise degrees of equivalence have no meaning (and thus are not computed) for voltage ratio comparisons, which involve a transfer device and no national standards.

4.2.3. Organization of key comparisons in radioactivity

In the field of radioactivity, the linkage of CC and RMO key comparisons to the corresponding BIPM key comparison, carried out in the framework of the <u>SIR</u>, is intrinsic to the organization of the exercises.

Take the example of radionuclide ⁸⁸Y.

Since 1976, 14 laboratories have submitted 35 samples of known activity of ⁸⁸Y to the BIPM for measurements in the SIR. The activities measured in individual laboratories range from about 400 kBq to 29 MBq. At the BIPM, these activity values are transformed into comparable equivalent activities using the unique SIR facilities (ionization chambers that are well-characterized and very stable in the long-term, and the BIPM radium-226 sources). The equivalent activities and associated uncertainties accumulated over years constitute the input data to key comparison <u>BIPM.RI(II)-K1.Y-88</u>. The key comparison reference value, obtained as the arithmetic mean, is equal to 6893 kBq with an associated combined standard uncertainty of 5 kBq. Degrees of equivalence are also computed.

The linked comparison <u>APMP.RI(II)-K2.Y-88</u> is organized in such a way that the pilot laboratory, NMIJ (Japan) produced a number of ampoules containing the same solution of radionuclide ⁸⁸Y, and distributed them among the participants. All measurements were carried out over a few months. The NMIJ sent also one sample, after having measured it, to the BIPM for estimation of its equivalent activity in the SIR. NMIJ thus acted as the intermediary which made it possible to convert all APMP measurements to SIR equivalent activities (*Figure 13*).

The equivalent activities and associated uncertainties obtained in the APMP comparison are directly compared to the BIPM key comparison reference value, which extends the matrix of equivalence to all APMP participants.

It may happen that several, or even all, ampoules distributed for the linked comparison are measured in the SIR. The conversion to equivalent activities then involves a mean of these measurements [7].

MEASURAND : Equivalent activity of ⁸⁸Y

• Key comparison BIPM.RI(II)-K1.Y-88

 x_i : result of measurement carried out in the SIR for the sample submitted by laboratory i
 u_i : combined standard uncertainty of x_i

Lab i	x _i / kBq	u _j / kBq	Year of measurement
NPL	6906	24	1977-05-17
VNIIM	6912	23	1993-01-05
OMH	6865	18	1993-04-09
BNM-LNHB	6882	22	1994-04-18
CMI-IIR	6865	26	1999-11-17
РТВ	6877	8	1999-12-01
NMIJ	6903	21	2000-03-15
IRA	6893	16	2001-06-13
BEV	6895	27	2001-09-26
NIST	6913	15	2002-06-19

• Key comparison APMP.RI(II)-K2.Y-88

 \pmb{x}_i : result of measurement carried out at laboratory i converted to the equivalent activity through the NMID (see <u>Final Report</u>) \pmb{u}_i : combined standard uncertainty of \pmb{x}_i

Lab i	- x _i / kBq	u _i / kBq	Date of measurement
ANSTO	6885	31	2000
BARC	6924	106	2000
CNEA	6894	86	2000
INER	6936	25	2000
KRISS	6912	20	2000
LNMRI	6918	33	2000
NIM	6903	44	2000
OAP	6888	103	2000

Figure 13. Laboratory individual measurements for the BIPM and APMP linked key comparisons of activity for the radionuclide ⁸⁸Y. The NMIJ (Japan) ensures the conversion of APMP measurements to equivalent SIR activity. It follows that the NMIJ measurement is inserted in the table of the BIPM.RI(II)-K1.Y-88 key comparison and not in the APMP table. Only the most recent measurement is retained for each laboratory participating in the BIPM key comparison.

4.2.4. Value of an international standard

The reference value of the BIPM key comparison <u>BIPM.EM-K10.a</u> (DC voltage, nominal value: 1.018 V) is set to be equal to the value delivered by the Josephson standard maintained at the BIPM with a combined standard uncertainty of 0.1 nV. The BIPM participated in the corresponding EUROMET key comparison, <u>EUROMET.EM.BIPM-K10.a</u>, and provides the linkage between both. It follows that the graph of equivalence is extended to another 13 EUROMET laboratories.

Note: At the time this paper was written, the results of the EUROMET "EM-K10.a" were not yet published via Appendix B, but its Final report had been approved at the CCEM meeting held on 17 and 18 March 2005.

4.2.5. Statistical treatment of common participation

The basic principle is to examine the reproducibility of the performance of the linking laboratories in both exercises, in order to estimate a correction (offset and uncertainty) to apply to the measurements obtained in the comparison to be linked. The corrected results then represent the best estimates of what would have been the results of the linked laboratories, had they actually participated in the master key comparison.

Take the example of a EUROMET key comparison to be linked to a CC key comparison. The process used is shown on *Figure 14*, and the notations are as follows:

- $x_{\rm R}$ and $u_{\rm R}$ are the value and the combined standard uncertainty of the key comparison reference value obtained from the CC key comparison, and nonaltered by the linkage; $x_{\rm R}$ is represented as an horizontal red line in *Figure 14*.
- $x_{\text{R-EUR}}$ and $u_{\text{R-EUR}}$ are the value and the combined standard uncertainty of a provisional reference value obtained from the data taken in the EUROMET comparison only; $x_{\text{R-EUR}}$ is represented as an horizontal black line in *Figure 14*.

Note that at this stage we don't know the relative position of $x_{\rm R}$ and $x_{\rm R-EUR}$.

• $D_{i:\text{EUR}}$ and $U_{i:\text{EUR}}$ are the two terms (offset and expanded uncertainty) of the provisional degree of equivalence of one laboratory *i* having participated in the EUROMET comparison. $D_{i:\text{EUR}}$ (black arrow on *Figure 14*) and $U_{i:\text{EUR}}$ (black uncertainty bar) are computed from EUROMET data only (laboratory *i* measurement, $x_{i:\text{EUR}}$, laboratory *i* uncertainty, $u_{i:\text{EUR}}$, $x_{\text{R}-\text{EUR}}$, and $u_{\text{R}-\text{EUR}}$, in particular

$$D_{i-\text{EUR}} = x_{i-\text{EUR}} - x_{\text{R-EUR}}.$$

The degree of equivalence of one laboratory *i* that has participated in the EUROMET key comparison *only* relative to the CC key comparison reference value is composed of two terms: the offset D_i and the expanded uncertainty U_i , written as:

$$D_i = x_i - x_R = D_{i-EUR} + (x_{R-EUR} - x_R)$$
, and
 $U_i = (U_{i-EUR}^2 + U_{LINK}^2)^{1/2}$.

where x_i is the virtual measurement of laboratory *i*, had it participated in the CC key comparison, and taken equal to $x_{i-\text{EUR}}$ (the single measurement available for this laboratory), and U_{LINK} is the expanded uncertainty added by the linkage; D_i and U_i are represented in blue on *Figure 14*.

Note that at this stage we don't know the value of U_{LINK} .

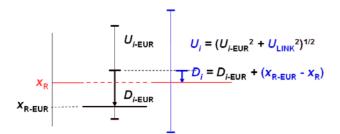


Figure 14. Principle of the linkage based on the examination of common participation in a master CIPM key comparison and the corresponding EUROMET key comparison. The notations are explained in the text. Laboratory i has participated in the EUROMET exercise only.

Each common participant "cp" has two degrees of equivalence: one (D_{cp} and U_{cp}) obtained in the CC key comparison, and one (D_{cp-EUR} and U_{cp-EUR}) obtained in the EUROMET key comparison. It thus provides an estimate of ($x_{R-EUR} - x_R$) equal to ($D_{cp} - D_{cp-EUR}$) with associated uncertainty ($U_{cp}^2 + U_{cp-EUR}^2$)^{1/2}.

The average of these estimates, weighted by their inverse square uncertainties, provides the best value achievable for $(x_{\text{R-EUR}} - x_{\text{R}})$ and associated uncertainty U_{LINK} .

Note that this method can use only those common participants that obtained close values, x_i and x_{i-EUR} , of their measurements in both exercises, and that the uncertainty of the degree of equivalence of a participant in the linked comparison is always increased by the transfer.

This method is applied to the linkage of <u>EUROMET.EM.BIPM-K11</u> to <u>BIPM.EM-K11.b</u> (DC voltage of Zener diode at 10 V), using the results of the common participants that also have an independent realization of the volt by means of a Josephson array voltage standard. The expanded uncertainty U_{LINK} is equal to 0.283 µV, about four times the magnitude of the offset ($x_{R-EUR} - x_R$), and is not negligible compared to the other uncertainties involved ($2u_R = 0.2 \mu V$).

In the case of the link of <u>EUROMET.EM-K4</u> to <u>CCEM-K4</u> (capacitance standards, nominal value: 10 pF), each of the seven common participants was asked if its results should be used for the link and, if so, to provide a one-sigma estimate of the uncertainty corresponding to the imperfect reproducibility of its measurements during the time elapsed between its measurements in the two comparisons. One of the common participants wished that its results not be used for the link, and the uncertainty related to the reproducibility of the six others was used for the estimation of $U_{\rm LINK}$. The corresponding graph of equivalence is shown in *Figure 15*.

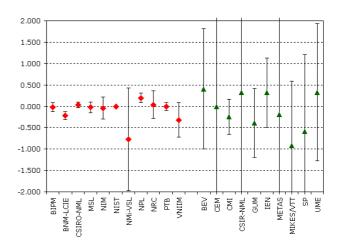


Figure 15. Graph of equivalence of the "EM-K4" duo as published in Appendix B. The values given on the graph are relative and expressed as parts in 10^6 . The expanded uncertainty U_{LINK} brought by the linkage is small (0.02 x 10^6) compared to the uncertainties obtained in the EUROMET comparison.

Red diamonds: participants in CCEM-K4. Green triangles: participants in EUROMET.EM-K4. More generally, the global combination of the data obtained in the linked key comparison with those of the common participants can be carried out using the generalised least-squares (sometimes known as the Gauss-Markov) method. This was successfully applied to extend the matrix and graph of equivalence of key comparison <u>CCM.M-K1</u> to the participants in <u>APMP.M.M-K1</u> (comparison of stainless steel standards, nominal value: 1 kg) [8].

Conclusions

In April 2005, after five and a half years of implementation of the CIPM MRA, more than 600 international comparisons, all tackling the highest level of accuracy achievable, are conducted under its auspices, and, as such, are recorded in the Appendix B of the BIPM key comparison database (KCDB).

The added value of the process is to interpret the results of key comparisons in terms of equivalence. The key comparisons that obey the same technical protocol are arranged as families, centred on master key comparisons able to produce reference values. Common participation among the members of the family makes it possible to extend its use to adjacent exercises, in order to obtain a global overview of the comparability of national standards all around the world.

With about 350 graphical representations of equivalence published in its Website, among which nearly 50 are composed of results of several key comparisons linked among them, the KDCB Appendix B appear as a unique tool, which serves as the ultimate reference for all actors in metrology and as an internationally agreed support for a wide range of calibration and measurement services provided every day by national laboratories.

References

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[2] Nomenclature of key and supplementary comparisons, available at: http://www.bipm.org/utils/en/pdf/nomenclature.pdf

[3] CMCs: Calibration and Measurement Capabilities, information available at: http://kcdb.bipm.org/appendixC/default.asp

[4] Guidelines for key comparisons, information available at: http://www.bipm.org/utils/en/pdf/guidelines.pdf

[5] Regional Metrology Organizations, information available at:

http://www.bipm.org/en/practical info/useful links/rmo. html [6] Technical supplement to the CIPM MRA, text available at: http://www.bipm.org/utils/en/pdf/mra_techsuppl2003.pdf

[7] G. Ratel, *Metrologia*, 2005, **42**, 140-144

[8] V. Tulasombut and C. Sutton, <u>*Metrologia*</u>, 2004, **41**, <u>*Tech. Suppl.*</u>, 07003</u>

The main BIPM Website and the KCDB Website are available at:

<u>http://www.bipm.org</u> and <u>http://www.bipm.org/kcdb</u> (or <u>http://kcdb.bipm.org</u>) respectively.

The full names of the laboratories cited by their acronyms can be found in the KCDB Appendix A at: <u>http://www.bipm.org/utils/en/pdf/signatories.pdf</u>

List of acronyms for fields of metrology: AUV: Acoustics, Ultrasound, and Vibration EM: Electricity and Magnetism L: Length M: Mass and Related Quantities PR: Photometry and Radiometry QM: Amount of Substance (Chemistry) **RI:** Ionizing Radiation T: Thermometry TF: Time and Frequency AUV.V: Acoustics, Ultrasound, and Vibration . Vibration M.M: Mass and Related Quantities . Mass Standards M.P: Mass and Related Quantities . Pressure RI(I): Ionizing Radiation (Dosimetry) RI(II): Ionizing Radiation (Radioactivity) Xy-abc: Radionuclide ^{abc}Xy