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# Application of the CIPM MRA to radionuclide metrology

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### Abstract

The adoption of the mutual recognition arrangement by the CIPM in 1999 had an almost immediate affect on the approach national measurement laboratories took in the measurement of radioactivity and how their abilities in radionuclide metrology were relayed to customers and stakeholders. In order to meet the need presented by the CIPM MRA for validated claims to calibration and measurement capabilities, radionuclide measurement laboratories have taken an accelerated track to international comparisons and expanding quality systems, leading to a more rigorous approach to radionuclide metrology.

### 1. Introduction

In 1910, Marie Curie prepared the first international radium standard, initially maintained by the Bureau of Weights and Measures (BIPM), then the Curie laboratory. However, it was not until the Soviet delegation, at the 9th General Conference on Weights and Measures (CGPM) in 1948, proposed to organize 'comparisons of national standards of radium with international standards of radium, and to maintain these at the Bureau International (BIPM)' that the first international comparison of the activity of a radionuclide was considered [1]. While the resulting 'comparable' measurements would be a step forward in metrology, and as the beginning of an expanding focus on international cooperation in the sciences, there was no mechanism other than scientific satisfaction and peer-reviewed publication to consider the result beyond one of good measurement. Although in and of itself, 'good' measurements are a crucial component to a national measurement institution's (NMI's) raison d'être, governments and regulatory organizations around the world needed a more concrete, and archival, means by which measurement standards for laboratories outside the home economy could be considered in the areas of import/export, manufacturing and international trade and commerce. Therefore, a more systematic and better-defined approach to determine the comparability among NMIs was needed.

Acknowledging the growing need for mutual recognition of measurements and increased cooperation among economies and regions in a shrinking world, the CGPM, in 1995, with

input and support of the International Laboratory Accreditation Cooperation (ILAC), initiated an effort to facilitate traceability of measurement standards around the world, and to formalize the recognition of those standards among the various NMIs. Following discussions among participants from the regional metrology organizations (RMOs) and the BIPM, the BIPM drafted an arrangement reflecting the initiative to support world-wide measurement recognition and traceability. After several iterations, the final agreed-upon version of the arrangement was signed by the directors of NMIs from 38 member states of the Metre Convention (along with representatives of two international organizations, the International Atomic Energy Agency (IAEA) and the Institute for Reference Materials and Measurements (IRMM)) in October 1999 [2]. This International Committee for Weights and Measures Mutual Recognition Arrangement (CIPM MRA) for national measurement standards and for measurement and calibration certificates issued by NMIs is a means to meet the increasing need for an open approach to provide reliable and quantitative information on the comparability of national measurement services to a variety of users (governments, academic institutions, manufacturers, industry, etc). The sixty seven institutions (45 Member States, 20 Associates to the CGPM, and two international organizations), which are current signatories to the most recent version of the CIPM MRA (revised in 2003), and the 117 additional institutions designated by them, can be found on the BIPM web site [3].

The CIPM MRA is an arrangement among NMIs to allow for the mutual recognition of national measurement

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standards, and the validity of NMI issued calibration and measurement certificates [4]. In general, to be a signatory to the arrangement, and to participate directly in the activities of the MRA, an institution must be the nationalgovernment designated NMI (where there are more than one such institutions, the CIPM MRA is signed by a single, representative NMI with the names of the others also attached). International/intergovernmental organizations, designated by the CIPM, may also participate, and NMIs of associates to the CGPM may participate through their RMO or upon specific invitation [5]. In the field of radionuclide metrology, participating NMIs recognize the validity of each others measurements and standards, and the validity of calibrations, for a variety of measurable quantities (including activity, activity per unit mass, emission rate, etc) for an array of radionuclides.

## 2. Operational aspects

The particular nature of radioactive materials (both from a regulatory perspective and from a handling perspective) limits the number of NMIs that perform radionuclide measurements. Signatories to the CIPM MRA with radionuclide metrology capability at the current time are listed in table 1 (several other NMIs and designated laboratories also have radionuclide metrology capabilities, but are not signatories to the CIPM MRA at the time of this writing).

A major consequence of the arrangement has been the increased accessibility to radioactivity measurements for economies whose NMI does not have such a capability. However, there is the potential for the elimination of radionuclide metrology facilities in favour of using more centralized laboratories, leaving fewer potential participants for comparisons and other interactions in the field and potentially reducing the robustness of metrology in this area. In fact, comparisons, while not the sole method by which a radionuclide metrology laboratory can assess its own performance, are considered one of the more optimal approaches in the evaluation of an NMI's capabilities [6] as well as to the improvement of measurement results in the metrology community as a whole.

Not unexpectedly, an increasing interaction among radionuclide metrology laboratories has brought to light the need for a more rigorous approach to the documentation of technical procedures and other aspects of a quality system (QS). The CIPM MRA requires that all CMCs in appendix C be supported by an implemented QS, and that the NMI be declared by either an outside body or itself ('self declared') to be in compliance with this requirement in order for its capability claims for radionuclide measurement services and production of radioactive reference materials to remain in appendix C The complexity of implementing a QS is extensive and a great deal of time (for NIST's QS, about eighteen months; http://physics.nist.gov/Divisions/Div846/QualMan/ index.html) is often devoted to its establishment. radionuclide measurements often require a level of specific technical expertise, and most NMIs have relatively few individuals working in this area, the resources diverted to the preparation of QSs has somewhat limited the flexibility of many NMIs to participate fully in additional measurements

during the time that the QS is being developed. Nevertheless, the effort toward the establishment of a QS enables radionuclide laboratories to be aware not only of one another's bases of measurements, but also of their own systems and how they may possibly be improved.

Finally, the implementation of the CIPM MRA has been particularly timely. Until the widespread accessibility to information and near-instantaneous communication possibilities that the internet affords, the goals of the CIPM MRA would have been exceptionally difficult to achieve in a timely fashion. For radionuclide metrology in particular, a rapid distribution of measurement results can be crucial. A rapid 'feedback' allows an NMI to repeat measurements for short-lived impurities, improving the subsequent activity measurement. Before the capability existed for rapid information exchange, it may have taken days or even weeks (or non-archival telephone communications) before additional measurements would be pursued. Electronic mail and data posting on the world-wide web now allow nearly 'real-time' communications, facilitating immediate (within hours) re-measurements as necessary. In addition, the rapid posting of reports encourages more active participation by all laboratories to meet government requests and regulations, and to justify budgets.

# 3. Impact on radionuclide metrology

Several key aspects of radionuclide metrology have been impacted by the implementation of the CIPM MRA. Previous to the arrangement's implementation, were able to disseminate and inform other organizations of their measurement capabilities through the typical approaches of scientific meetings and publications, and through interactions in organizations as the International Committee on Radionuclide Metrology (ICRM) and the Consultative Committee on Ionizing Radiation, Measurement of Radionuclides Section (CCRI section II). Although these approaches were widely-accepted, they were generally inaccessible to the wider communities of users (in fields such as nuclear medicine, environmental clean-up, geological and industrial applications, and academia) and government (e.g. regulators and policy setting bodies) entities which depended on these same measurement capabilities to support With the establishment of the CIPM their activities. MRA, these capabilities, the measurable quantities, and ranges (including such adjunct information as uncertainty intervals) for radionuclide metrology are indicated in appendix C and are accessible to any user with free and open access to the BIPM key comparison database (KCDB, http://kcdb.bipm.org/default.asp) on the internet. These capabilities, or CMCs, describe the radionuclide measurement capabilities of the NMIs of 21 economies (including intergovernmental bodies), indicated in bold in table 1. The veracity of the claims of capabilities is critically evaluated by radionuclide metrologists at NMIs throughout the RMOs, who depend heavily on the results of comparisons in these types of measurements (listed in appendices B and D of the KCDB) as well as other information (including publications from the NMI claiming the CMC).

Although the CIPM MRA is not prescriptive on the mechanisms by which measurement claims may be justified

**Table 1.** Signatory institutions with radionuclide metrology capability. Those with currently published calibration and measurement capabilities (CMCs) are indicated in bold.

RMO	Country/economy	NMI						
APMP	Australia China Chinese Taipei India Indonesia Japan Republic of Korea Thailand	<ul> <li>a ANSTO—Australian Nuclear Science and Technology Organisation</li> <li>NIM—National Institute of Metrology</li> <li>a INER—Institute for Nuclear Energy Research</li> <li>a BARC—Bhabha Atomic Research Centre</li> <li>PTKMR—Center for Technology of Radiation Safety and Metrology</li> <li>NMIJ/AIST—National Metrology Institute of Japan</li> <li>KRISS—Korean Research Institute for Standards and Science</li> <li>a OAP—Office of Atoms for Peace</li> </ul>						
COOMET	Belarus Cuba Israel <sup>b</sup> Ukraine Russian Federation	BelGIM—Belarussian State Institute for Metrology  a CENTIS-DMR—Centro de Isótopo y Radionúclidos  a CPHR—Centro de Protección e Higiene de las Radiaciones  a MoE-ISR—Israel Ministry of Environment  a NSC IM—National Scientific Centre 'Institute of Metrology'  a VNIIFTRI - All-Russian Research Institute for Physical, Technical and Radiophysical Measurements  a VNIIM - D.I. Mendeleyev Institute for Metrology						
SADCMET	Austria Bulgaria Croatia Czech Republic EU Denmark Finland France Germany Greece Hungary Iceland Ireland Italy Latvia Lithuania Norway Romania Portugal Poland Slovakia Slovenia Spain Sweden Switzerland The Netherlands United Kingdom South Africa	BEV—Bundesamt für Eich- und Vermessungswesen NCM—National Centre of Metrology IRB—Ruder BOSKOVIC Institute CMI—Czech Metrology Institute EC-JRC-IRMM—Institute for Reference Materials and Measurements SIS—Statens Institut for Strålingshygiejne  a STUK—Radiation and Nuclear Safety Authority LNE-LNHB—Laboratoire National d'Essais/Laboratoire National Henri Becquerel, Sacla PTB—Physikalisch -Technische Bundesanstalt (also in COOMET)  a HIRCL/HAEC—Hellenic Ionizing Radiation Calibration Laboratory of the Hellenic Atomic Energy Commission MKEH—Magyar Kereskedelmi és Engedélyezési Hivatal (previously OMH - National Office of Measures) GR—Geislavarnir rikisins – Icelandic Radiation Protection Institute RPII—Radiological Protection Institute of Ireland  a ENEA-INMRI—Ente per le Nuove Tecnologie, l'Energia e l'Ambiente / Instituto Nazionale di Metrologi adelle Radiazioni Ionizzanti RMTC—Radiation Metrology and Testing Centre of the Latvian National Metrology Centre (also participates in COOMET projects) VMT/VMC—Vilniaus Metrologijos Centras NRPA—Norwegian Radiation Protection Authority IFIN-HH—'Horia Hulubei' National Institute of Physics and Nuclear Engineering ITN-LMRIR—Instituto Tecnológico e Nuclear – Laboratório de Metrologia das Radiaçoes Ionizantes e Radioactividade  POLATOM - Radioisotope Centre SMU—Slovak Institute of Metrology CMCs on activity in preparation IIS—J. Stefan Institute—Dosimetry Standards Laboratory  CEIR MAT—Centro de Investigaciones Energéticas Medioambientales y Tecnológicas SSI—Swedish Radiation Protection Institute  NBL—National Physical Laboratory  CSIR NML—National Metrology Laboratory						
SIM	Argentina Brazil Mexico	<ul> <li><sup>a</sup> CNEA—National Commission of Atomic Energy</li> <li><sup>a</sup> LNMRI/IRD—National Laboratory for Metrology of Ionising Radiation/Institute of Radiation Protection and Dosimetry</li> <li><sup>a</sup> ININ—Instituto Nacional de Investigaciones Nucleares</li> </ul>						
International/ intergovernmental	United States European Union International	NIST—National Institute of Standards and Technology IRMM—Institute for Reference Materials and Measurements IAEA—International Atomic Energy Agency						

<sup>&</sup>lt;sup>a</sup> Designated institution.

('supported') by the claimant (in fact, there are several approaches including published results and the recognized technical experience of the NMI) [6], perhaps the most direct and objective is the successful participation in comparisons.

Radionuclide measurement comparisons, whether within an RMO or on a more expansive scale, are an approach by which national metrology institutions are able to compare not only their measurement results with one another, but also

<sup>&</sup>lt;sup>b</sup> Not a member of any RMO, but participates in COOMET projects.

Table 2. Section of generic groupings table for radionuclide metrology. For complete table, including acronym definitions, refer to [8].

Nr	Nuclide	4P-BP/AP-PC/PP/LS-GR-NA/GH-CO/AC	4P-XR/AE-PC/PP/LS-GR-NA/GH-CO/AC	4P-BP/AP-LS-PH-00-CN/TD	4P-XR/AE-LS-PH-00-CN/TD	4P-BP/AP/XR/AE-NA/CS-00-00-HE	4P-PH-NA-00-00-HE	4P-BP/AP-PP-00-00-HE	4P-XR/AE-PP-00-00-HE	SA-AP-PS-00-00-00	4P-BP-PC-00-00-IG	UA-BP-PC/PP/LS-GR-NA-CT/AT	4P-BP-LS-00-00-CN/TD	4P-BP-PP-00-00-HE	4P-BP-PC-00-00-IG	??-XR/AE/PO-??-00-00-??
1	H-3												1		1	
2	Be-7		2													
3	C-11															3
4																
	C-14											3	2		1	
5	C-14 F-18	1		3				2				3	2		1	
		1	0.5	3	2		1	2				3	2		1	
5	F-18	0.5	0.5	3	2		1 0.6	2				3	2		1	
5	F-18 Na-22		0.5		2								2		1	
5 6 7	F-18 Na-22 Na-24	0.5	0.5	2	2							1.5	1.5	1.5	1	
5 6 7 8	F-18 Na-22 Na-24 Al-26 P-32 P-33	0.5	0.5	2	2							1.5 1.5		1.5	1	
5 6 7 8 9	F-18 Na-22 Na-24 Al-26 P-32	0.5	0.5	2	2							1.5	1.5		1	

the methods by which those results are obtained. For the measurement of radioactive materials, this is a key aspect as the physics involved in the measurement method, and the physicochemical properties of the radionuclide itself, often impact the confidence in the result and even its actual value. For this reason, a determination of the uncertainty in a comparison is critical and without it and its inclusion in the final comparison result, the utility of the exercise is partly lost. The need for comparisons has led to increased interactions among NMIs with each other (including with the international bodies IAEA and IRMM) and with the BIPM.

With the establishment of the CIPM MRA, for the first time since the serendipitous discovery of radioactivity by Antoine-Henri Becquerel in 1896, a mechanism is in place by which the results from one radionuclide metrology laboratory can be considered in the same context as a measurement of the same quantity at another NMI. This is not to say that the measurement results of two different laboratories are equal, rather their results are comparable. That is, 'the degree of equivalence of measurement standards is taken to mean the degree to which these standards are consistent with reference values determined from the key comparisons and hence are consistent with one another.' This approach of determining 'degrees of equivalence,' a quantitative measure of comparability, enables customers to evaluate potential providers of measurement services among NMIs, ascertain the validity of measurements from an NMI in meeting regulatory requirements, and assures NMIs of their own capabilities in the face of changing technologies.

Successful participation in comparisons is a crucial component in determining degrees of equivalence, and

wording in the CIPM MRA suggests the necessity for comparisons to support a wide range of measurements and standards, including those in radionuclide metrology. However, with CMCs covering more than 150 individual radionuclides (and the potential for more in the future) already accessible to laboratories and stakeholders, it was apparent early in the implementation of the CIPM MRA that the possibility of carrying out comparisons (key or otherwise) for each radionuclide by all appropriate methods would not be possible. The key comparison working group (KCWG) of the CCRI(II), working closely with the uncertainties working group and experts in radionuclide metrology, generated a 'generic grouping' of all the radionuclides currently in appendix C, based on method of measurement and ease of measurement, for a more efficient approach towards addressing needed comparisons (for a complete table, refer to the CCRI document, 'Grouping Criteria for Radionuclides for Supporting CMCs') [8]. An extract of the structure of these groupings is given in table 2.

Radionuclides are categorized by radiation-type and primary measurement method (along with the minimum expected uncertainty, at k=2, for the measurement) appropriate to the specific nuclide in an approach intended to optimize the number of comparisons that needed to be run over time. The relative difficulty of measuring a specific nuclide by a specific method has been denoted by a colour-coded system: 'red' for the most difficult, 'yellow' for the moderately difficult, and 'green' for the least difficult. Occasionally, a radionuclide indicated as 'red' when measured by one method may be indicated as 'green' by another. In this way, results from a comparison of a single radionuclide by a given primary

**Table 3.** CCRI (II) comparisons proposed by the KCWG to be run through 2010 to support CMCs for radionuclide metrology CMCs (from the generic groupings table for radionuclide metrology).

Nr	Nuclide	4P-BP/AP/XR/AE-NA/CS-00-00-HE	4P-XR/AE-PP-00-00-HE	4P-BP-PC-00-00-IG	UA-BP-PC/PP/LS-GR-NA-CT/AT	4P-BP-LS-00-00-CN/TD	4P-BP-PP-00-00-HE	4P-BP-PC-00-00-IG	??-XR/AE/PO-??-00-00-??	??-GR-??-00-00-??	4P-??-PC/PP/LS-??-NA/GH- CO/AC/CT/AT (DS)*	4P-??-LS-00-00-CN/TD (DS)*	4P-??-PP-00-00-HE (DS)*	SA-??-PS-00-00-00 (DS)*	4P-??-PC-00-00-IG (DS)*	Year of previous comparison / proposal
1	H-3					1		1								1991 EUROMET + CCRI
11	S-35				3	2	3									CCRI
25	Fe-55								4							2006 CCRI
56	Tc-99m									3						SIR + CCRI
61	Cd-109		2								3	1.5				1986 + SIR + <b>CCRI</b>
67	Te-123m									3						CCRI
74	Cs-131								3							CCRI
79	Xe-133			3												SIR + CCRI
81	Cs-137	1.5									1.5	1.2	1.5			1982 + SIR + 2005 COOMET + <b>CCRI</b>
124	Rn-222											2		3	3	SIR + 2004 EUROMET + 2005 COOMET + CCRI
130	Th-228+d										4	1.5	3	3		SIR + CCRI

method may be used to support measurement capability claims for others in the same category. In general, radionuclides indicated as red for a given primary measurement method may be used to support the CMCs for all other nuclides of the same energy type by that measurement method (i.e. all other red, yellow and green-coded nuclides for that method). Similarly, results from a comparison of a nuclide indicated as yellow will support claims for the yellow and green-coded nuclides, and that of a green-coded nuclide will support CMCs of only green-coded nuclides by the same method. A comparison result from a radionuclide measured by a specific primary method generally can not be used to support claims for that radionuclide measured by other primary methods. However, in the case where a nuclide is measured, in the context of a comparison, by a method under which it is indicated as red, a judgment may be made that the result for that comparison may be extended to other methods under which the radionuclide is indicated at a lower level of difficulty after considering the laboratory's established capabilities and other supporting factors. Laboratories are encouraged to use any and various methods appropriate to measuring the radionuclide while participating in a comparison. Secondary methods of measurement, and the expected associated uncertainties for radionuclides measured by them, are not listed in the table. When such a method is used in the context of a comparison, the results can support the CMCs of only that nuclide as measured by that traceable method, so no grouping of nuclides is feasible.

With this grouping of radionuclides, comparisons of only a few (generally, red) can support the CMCs of many. The KCWG of CCRI(II), based on the history of comparisons in radionuclide metrology, proposed to the Section a series of comparisons to be run through 2010 which, when considered with other key and supplementary comparisons from the previous few years, would support the majority of CMCs currently published in appendix C. Aware that some NMIs may not need to support all radionuclides by all methods, the KCWG also proposed comparisons for 'yellow' nuclides which would support a more limited number of CMCs. The proposed comparisons are indicated in table 3.

In addition, CMCs for the measurement of radionuclide activity in reference materials (soils, organic matrices, natural waters, etc) have also been published. In these cases, comparisons relating results back to key comparison reference values, the goal for the CCRI(II) comparisons, is impractical or impossible. While the measurement of their contributing radionuclides (such as <sup>137</sup>Cs in soil) have been compared, and such comparisons are used to support the CMC of the same nuclide even in a reference material, the comparison of the reference materials themselves offers very specific and often recalcitrant difficulties. In addition to the preponderance of a vast variety of reference materials, many of which are considered by only one laboratory, how such material is to be handled (sampling) and prepared for analysis (i.e. procedures used to extract the nuclides of interest from the matrix quantitatively) present potential problems for any kind of comparison. The KCWG, recognizing the advantage of key and supplementary comparisons to provide a consistent foundation for justifying and evaluating all CMCs, has worked

to develop a representative approach, much like the generic groupings, to the comparison of reference materials based on chemical composition and physical characteristics of the matrix material as well as the distribution of radionuclide species within that matrix [9].

# 4. The future CIPM MRA and radionuclide metrology

Despite the current successes of the CIPM MRA, and its efficient implementation for radionuclide metrology, several difficulties still exist. Timeliness for comparisons, to be undertaken in a short enough time period to assure validity for the published CMCs, will be an on-going issue that will require resources (time, personnel, and funding) that may not be available in the long-term for NMIs. Timing is particularly important in radionuclide metrology when comparisons of short-lived radionuclides (such as the planned <sup>99</sup>Tc<sup>m</sup> comparison) are considered. A current solution to this particular issue, a  $4\pi$  NaI(Tl) transfer instrument to extend the international system of reference (SIR) for these types of radionuclides, is under development, and may be extended to other radionuclides [10]. Acceptability of equivalences among economies, including acceptance by regulatory bodies and acceptability of certificates stating measurement or calibration results, could continue to be an issue when there are marketing pressures and changes in political structures. However, the CIPM MRA has led to a more rigorous approach to radionuclide metrology, allowing NMIs to provide more reliably validated measurement results to their stakeholders, governments, and to the metrology community at-large. The increased interactions among NMIs because of the need for comparisons, contrary to a more insular and restricted approach when comparisons are peripheral to an NMI's activities as was the case when that first radionuclide comparison was proposed in 1948, is facilitating not only improved support to users of these measurements, but also to those performing the metrology, allowing a wider base for information exchange and problem solving.

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