RADIOACTIVITY LABORATORY OF LNMRI IN THE FRAMEWORK OF MRA

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Abstract: This work presents the experience of the National Laboratory for Ionizing Radiation Metrology (LNMRI) in the implementation of the requirements of the Mutual Recognition Arrangement (MRA) signed by 38 National Institutes of Metrology (NMI) in 1999. The degree of equivalence of activity reported by Bureau International des Poids et Mesures (BIPM) in the Key Comparison Database (KCDB), the main data of a specific radionuclide listed in the Calibration and Measurement Capabilities (CMC) and the implementation of a quality system are informed.

Keywords: radioactivity, quality system, key comparison.

1. INTRODUCTION

The Institute of Radiation Protection and Dosimetry (IRD), where LNMRI is settled, has been formally designed, in 1989, by Brazilian National Institute of Metrology, Standardization and Industrial Quality (INMETRO) to fulfill, at national level, the mission of the maintenance and dissemination of standards related to the main quantities in the field of ionizing radiation. The word, which describes this field of activity, is “Traceability”, a vertical and hierarchical concept which links the national measurement systems adopted by individual countries. It is defined as: “The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national, through an unbroken chain of comparisons” [1].

In October 1999, the directors of the National Metrology Institutes of 38 Member States of the Metre Convention signed a Mutual Recognition Arrangement [2] for national measurement standards and for calibration and measurement certificates issued by NMs. The MRA aims to establish the metrological degree of equivalence of the standards maintained by the NMs in order to provide a reliable technical base for wide scientific and technological agreements as well as treaties related to international trade. Once INMETRO is signatory of MRA, the LNMRI, as designated laboratory must comply the requirements of MRA.

The database which gives solidity to the metrological degree of equivalence of NMs is kept by Bureau International des Poids et Mesures (BIPM) in which the results of the key comparisons are used as reference.

In this work will be presented the degree of equivalence of the activity (SI unity becquerel, symbol Bq) reported by BIPM in the Appendix B of KCDB for those key and supplementary comparisons that LNMRI has participated since 1987. Besides, the Calibration and Measurement Capabilities for ⁶⁰Co, reported by LNMRI and listed in the Appendix C of KCDB as well as the implementation of a quality system based on the requirements of ISO/IEC 17025 [3] are described.

2. METHODS

The harmonization of the measurements leads to the formulation of the principle of Equivalence [BIPM, 1999], defined as: The condition of being equivalent, i.e. equal for practical purposes, in significance or worth. The Equivalence should quantitatively be demonstrated via key comparison that may be defined as “one of the set of comparisons selected by a Consultative Committee of BIPM to test the principal techniques and methods in the field” [4].

A more specific term is “Degree of Equivalence of a measurement standard”. The degree of equivalence of a given measurement standard is the degree to which this standard is consistent with the key comparison reference value (KCRV). The degree of equivalence of a particular NIM, i, with the KCRV is expressed as the difference between the results

\[ D_i = A_{ei} - \text{KCRV} \] (1)

and the expanded uncertainty (k=2) of this difference, \( U_i \), known as the equivalence uncertainty, hence

\[ U_i = 2u_{D_i} \] (2)

taking correlation into account as appropriate. \( A_{ei} \) means the equivalent activity of a specific radionuclide of i NMI. The KCRV is derived from the unweighted mean of all the results submitted to the International Reference System (SIR) of BIPM with some restrictive provisions [4]. The results of the international comparisons organized by section
II of the Consultative Committee for Ionising Radiation (CCRI(II)) linked to SIR can be used for the KCRV with the additional restriction that the participant must have measured impurities of the radioactive solution. In this work the degree of equivalence means the comparison of a given NMI with the KCRV of activity measurement.

A large number of radionuclides are used in different applications in the field of fundamental research, industry, teaching and medicine. At this moment, the small number of key comparisons organized by BIPM doesn’t allow the validation of the measurement standards of the individual NMIs. Considerable cost in terms of equipment and human resources is involved to support the primary standardization systems necessary in the participation of a key comparison. Hence, most of the NMIs have dedicated to develop and implement restricted group of primary standardization techniques. In the LNMRI the coincidence counting and liquid scintillation counting systems have been implemented for absolute activity measurements of beta-gamma, alpha-gamma, electron capture and pure beta decay radionuclides. By the other hand, the radionuclides may be arranged according to the degree of difficult for the method used in its standardization. For instance, the standardisation of $^{60}$Co by coincidence method is classified as green, while $^{54}$Mn is yellow and $^{152}$Eu is red, representing, respectively, the simple, intermediate and difficult levels of standardization. In this way, the participation in a key comparison of a radionuclide classified as difficult could validate those radionuclides classified as yellow and green in which the same standardisation method has been used.

CMCs of NMIs are part of the BIPM KCDB concerning Appendix C of the MRA. The CMCs are declared by NMIs and refers to the characteristics of the calibration and measurement capabilities for the NMI concerned. The data from NMIs are issued in the form of an EXCEL worksheet where there are at least 21 columns into which data are inserted for each standard or service. In the field of radioactivity the main characteristics are: activity ($Bq$), activity per unit mass ($Bq \ g^{-1}$); emission rate and surface emission rate ($s^{-1}$), measurement system, specification (solid, liquid, gas, container, mass or volume), uncertainty with $k=2$, identifier and source traceability.

The quality system has been implanted based on the requirements of ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories. After the submission to an internal audit to get an overview of the system the radioactivity laboratory has been assessed in a technical Peer Review by two experts from Europe in 2004.

3. RESULTS

At present time, twenty-one radionuclides, in name of LNMRI, are listed in the Appendix B of the KCDB of BIPM. Six more recent comparisons have no results so far and the reports are in progress. Figure 1 depicts the degrees of equivalence of these radionuclides as may be obtained from the Appendix B of KCDB. Almost all these radionuclides present deviation from the KCRV lower than $\pm 1.0\%$, with the exception of $^{152}$Eu (-1.24$\%$), $^{58}$Co (3.08$\%$) and $^{166}$Ho (1.79$\%$) These radionuclides are classified as red and should be measured again and submitted to SIR to improve their accuracies. The results show the reliability of the radioactivity standards and measurement systems implanted at LNMRI.

![Fig. 1. Degrees of equivalence for activity quantity.](image)

The characteristics of the twenty-one radionuclides of Figure 1 plus twelve (alpha, beta and gamma emitters), are presented, in details, in the Appendix C of the KCDB and may be accessed by users. The data show the diversity of radioactive standards available at LNMRI and are continuously updating with the entry of new radionuclides or in reducing some specific stated uncertainty. The summarized data for $^{60}$Co as reported in Appendix C is presented in Table 1 as an example.

<table>
<thead>
<tr>
<th>Activity per unit mass (Bq g$^{-1}$)</th>
<th>Relative uncertainty, in % (k=2)</th>
<th>Method of measurement</th>
<th>Container, volume (mL)</th>
<th>Internal identifier</th>
</tr>
</thead>
<tbody>
<tr>
<td>$5.0E+02$ to $1.0E+05$</td>
<td>5</td>
<td>Germanium spectrometer*</td>
<td>Glass ampoule, 3</td>
<td>SIM-RAD-LNMRI-2048</td>
</tr>
<tr>
<td>$3.7E+04$ to $1.0E+09$</td>
<td>0.4</td>
<td>Pressurized ionisation chamber</td>
<td>Glass ampoule, 3</td>
<td>SIM-RAD-LNMRI-2005</td>
</tr>
<tr>
<td>$5.0E+01$ to $1.0E+06$</td>
<td>3</td>
<td>Germanium spectrometer</td>
<td>Glass ampoule, 3</td>
<td>SIM-RAD-LNMRI-2033</td>
</tr>
<tr>
<td>$7.8E+02$ to $7.4E+05$</td>
<td>2.0</td>
<td>Planar NaI(Tl) crystal</td>
<td>Glass ampoule, 3</td>
<td>SIM-RAD-LNMRI-2062</td>
</tr>
<tr>
<td>$5.0E+03$ to $3.0E+05$</td>
<td>1.0</td>
<td>Liquid scintillation</td>
<td>Glass ampoule, 5</td>
<td>SIM-RAD-LNMRI-2068</td>
</tr>
</tbody>
</table>

*Multinuclide solution

The CMC worksheet should contain the information about the traceability of the measurement value relating to the standard or service of individual NMIs [5]. Table 2 shows that information for some radionuclides declared by LNMRI.
Table 2. Absolute standardization methods and supporting comparisons.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Standard</th>
<th>Source of traceability</th>
<th>Comparison supporting this measurement/calibration service</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba-133</td>
<td>4π(πC)γ-coincidence counting</td>
<td>LNMRI</td>
<td>BIPM.RI(II)-K1.Ba-133</td>
</tr>
<tr>
<td>Ce-139</td>
<td>4π(πC)γ-coincidence counting</td>
<td>LNMRI</td>
<td>BIPM.RI(II)-K1.Ce-139</td>
</tr>
<tr>
<td>Co-60</td>
<td>4π(πC)γ-coincidence counting</td>
<td>LNMRI</td>
<td>BIPM.RI(II)-K1.Co-60</td>
</tr>
<tr>
<td>Am-241</td>
<td>4π(πC)γ-coincidence counting</td>
<td>LNMRI</td>
<td>CCR(II)-K2.Am-241</td>
</tr>
<tr>
<td>Ir-192</td>
<td>4π(πC)γ-coincidence counting</td>
<td>LNMRI</td>
<td>CCR(II)-K2.Ir-192</td>
</tr>
<tr>
<td>Co-58</td>
<td>4π(πC)γ-coincidence counting</td>
<td>LNMRI</td>
<td>APMP.RI(II)-K2.Co-58</td>
</tr>
</tbody>
</table>

Procedures for source preparation and standardization methods implanted as well as corporate procedures in the scope of the quality system were internally audited. Non-conformities were pointed out and corrected later. The system was then submitted to an international Peer Review and approved in the Inter-American Metrology System (SIM) meeting held at Venezuela in September of 2004. This process is dynamic and annual internal audit has been done to improve it.

4. CONCLUSIONS

The radioactivity laboratory of LNMRI has been developed effective methods for radionuclide measurements, which can be demonstrated through the degrees of equivalence in activity listed in Figure 1. With the accomplishment of the MRA, the certificates of calibration issued by LNMRI should be accepted by others NMIs, and will establish a reliable base to overcome technical barriers and the effective recognition of the measurements quality realized at LNMRI. The requirements of the ISO17025 were Peer Reviewed and approved allowing LNMRI to publish its CMC in the Appendix C of KCDB/BIPM.

REFERENCES


