

# SPECIFIC ANALYTICAL CHEMISTRY MEASUREMENTS

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**Abstract** – This paper describes the work currently undertaken in the framework of the Brazilian Network for Chemical Measurements (RBMQ – Rede Brasileira de Medições em Química), in relation to the sub-network of laboratories called LACM. This sub-network's objective is to identify and support laboratories that are capable of conducting specific analytical chemistry techniques applied to different kinds of samples and analytes at higher metrological level, with the intention of providing the Brazilian community with laboratory references prepared for performing chemical analyses for different purposes, thus delivering reliable results that are comparable at national and international levels. At the completion of the first phase, which was an initial nationwide survey, a group of twenty-one laboratories became part of the network, covering a broad range of matrixes, analytes and analytical techniques. The paper describes the situation of the laboratories, with regard to quality control management, submission to internal audits and accreditation. A recent survey applied to the laboratories also revealed their most relevant metrological demands, which is a very important information for the RBMQ and Brazilian financial institutions for research support, providing them with data for better decisions in establishing their priorities.

**Key-words:** RBMQ, LACM, metrological demands, analytical chemistry measurement.

## Introduction

In the framework of the Brazilian Network for Chemical Measurements (RBMQ), some actions were taken in order to identify laboratories, which are capable of conducting specific analytical techniques applied to different kinds of samples and analytes at higher metrological level, with the intention of providing the Brazilian community with laboratory references prepared for performing chemical analyses for different purposes, thus delivering reliable results, which are comparable at national and international levels, with declared uncertainties and traceability.

The RBMQ's Coordination started an initial survey by sending questionnaires to potential participants among Brazilian laboratories, which were answered by about one hundred of them. A number of these laboratories that expressed interest in participating in the network were visited so that more accurate information could be obtained.

After this first phase, the process resulted in the consolidation of twenty-one participating laboratories, located in different regions of the country, comprising the States of São Paulo, Rio de Janeiro, Minas Gerais, Bahia, Rio Grande do Norte, Rio Grande do Sul, Pernambuco and Paraná, thus forming the sub-network called LACM of the RBMQ.

This sub-network of laboratories covers a broad range of activities, since they analyze very different kinds of matrixes, such as: foodstuffs, gases, water, rocks, ores, soils, metals, plastics, fuels, environmental samples, radioactive materials, among others.

These laboratories also employ a diversity of analytical techniques, such as: atomic absorption spectrometry, gas chromatography, ICP OES, ICP MS, neutron activation analysis, infrared spectrophotometry, X-Ray fluorescence, ion chromatography, alpha, beta and gamma-ray spectrometry.

The purposes of the RBMQ in relation with the participation of these laboratories and that were presented to them since the beginning of the formation of the LACM group were:

- to provide the Brazilian chemical community with reliable references for specific analytical techniques and matrixes to be analyzed;
- to assure that the measurement processes are under control, with declared uncertainties and defined traceability;
- to contribute towards the improvement of the laboratories that already present a certain technical and organizational level;
- to contribute towards the validation process of analytical techniques; and
- to stimulate laboratories with high metrological level to apply for accreditation.

## **Interactions and contributions of the RBMQ to the laboratories**

Initially, this group's activities comprised the exchange of knowledge through the use of questionnaires designed by the RBMQ's coordinator and the LACM sub-network, based on meetings and technical visits to the laboratories.

The RBMQ provided the participating laboratories with resources and administrative support for the purchase of several kinds of reference materials and standard solutions and also offered them the opportunity to attend relevant courses related with metrology in chemistry and quality management to be applied in their laboratories, such as: validation of analytical methods, statistics applied to laboratories, inter-laboratory programs.

Another important aspect of the RBMQ's contribution to the laboratories was the opportunity to participate in inter-laboratory comparison studies and proficiency testing, organized by several institutions, such as Senai-Cetind, IRD, IPT, Cientec from Brazil, CCQM, SIM, among others. It was evident that these participations resulted in significant improvement of important aspects, such as the eva-

luation of all uncertainties involved in analytical techniques employed by laboratories and a better understanding of each phase of the measurement process.

A recent questionnaire applied to the laboratories also revealed the most important metrological demands according to them, which is important information for the RBMQ and for Brazilian financial institutions for research support, providing them with data for better decisions in establishing their priorities.

Other questionnaires provided the coordinators with very important information about the situation of the laboratories regarding their quality systems, their internal and external audits and their accreditation or recognition by different Brazilian organizations.

Several papers have been published by the participating laboratories that contain descriptions of uncertainty assessments and other aspects of their quality systems [1-6].

It became clear to the coordinators that many Brazilian laboratories can provide important contributions on demand to authorities concerning the presentation of analytical results of high metrological levels using different kinds of samples, analytes and analytical techniques.

The last survey conducted by LACM/RBMQ aiming at evaluating the current situation of the laboratories has shown that most of them has implemented quality systems and that they are submitted to internal audits. Many of these laboratories are accredited for several assays – following the RBMQ's guidelines and principles.

## Data of the Laboratories

Table 1 presents a summary of the data of the laboratories pertaining to this sub-network of the RBMQ.

**Table 1** – Summary of the data of the laboratories taking part in the LACM sub-network of RBMQ.

| Institution     | Laboratory                                    | Matrix/Analytes  | Analytical techniques                        |
|-----------------|---|--|--|
| 1. CETEC        | Trace Metals Laboratory                       | Trace metals in water and biological fluids                                  | Atomic Absorption<br>ICP OES                 |
| 2. CTGAS        | Chemical Characterization Laboratory          | Mercury in natural gas   | Gas Chromatography                           |
| 3. CENA/USP     | Radioisotopes Laboratory                      | Environmental, biological, geological, nuclear, forensics                    | Neutron Activation Analysis                  |
| 4. IPEN/LAN     | Neutron Activation Analysis Laboratory        | Environmental, biological, geological, archeological, agricultural, metallic | Neutron Activation Analysis                  |
| 5. IPEN/CNEN-SP | Laboratory of Gas Analysis                    | Analysis of gases in metals  | Infrared<br>Gas Chromatography               |
| 6. IPT          | Laboratory of Organic Chemistry Analysis      | Plastifiers in PVC base toys   | Gas Chromatography<br>Infrared               |
| 7. IPT          | Laboratory of Inorganic Chemistry Analysis    | Metals in water and soils  | Atomic Absorption<br>ICP OES<br>Fluorescence |
| 8/9. PUC-Rio    | Laboratory of ICP MS<br>Laboratory of ICP OES | Toxic chemical species in environmental and biomedical samples               | Atomic absorption<br>ICP MS<br>ICP OES       |

| Institution                                       | Laboratory   | Matrix/Analytes  | Analytical techniques  |
|---|--|--|--|
| 10. ITEP  | LabTox – Analysis of Agrotoxic Residues  | Agrotoxic Residues   | Gas Chromatography<br>Fluorescence<br>UV-Visible   |
| 11. CNEN/CDTN                                     | Analytical Chemistry Laboratory-Service of Chemistry and Mineralogy                | Ceramics, metals, special alloys, ores, biological, samples, nuclear materials               | X-Ray-Fluorescence<br>Atomic absorption<br>Gas and Liquid Chromatography<br>$\alpha$ , $\beta$ and $\gamma$ Ray spectrometry<br>Neutron Activation Analysis<br>Fluorimetry |
| 12. ITAL  | CTC-Center of P&D on Meat<br>Center of P&D on Food Chemistry and Applied Nutrition | Foodstuffs and Meat Products   | ICP OES<br>UV VIS<br>Gas and Liquid Chromatography   |
| 13. TECPAR  | Brazilian Reference Center on Biofuels-CERBIO                                      | Biofuels   | Gas and Liquid Chromatography  |
| 14. CNEN/Poços de Caldas                          | COLAB  | Uranium in soil, sediments, vegetables, waters   | ICP<br>UV-Visible,<br>$\alpha$ , $\beta$ and $\gamma$ Ray spectrometry   |
| 15. IPEN/<br>CNEN-SP                              | Department of Environmental Radioactivity  | Radioactivity in environmental samples   | $\alpha$ , $\beta$ and $\gamma$ Ray spectrometry   |
| 16. CETESB  | Division of Physical-Chemical Analysis   | Environmental Samples  | Atomic Absorption ICP MS<br>Fluorescence<br>Chromatography   |
| 17. Analytical Solutions                          |  | Metals in waters, soil, sediment (lab in São Paulo)  | Several analytical techniques  |
| 18. IPEN/<br>CNEN-SP                              | X-Ray Fluorescence Laboratory  | Elements in rocks, soils, steels, and alloys   | X-Ray Fluorescence   |
| 19. Ministry of Agriculture, MAPA (Reference Lab) | Laboratory for Analysis of Mycotoxins  | Aflatoxins B <sub>1</sub> , B <sub>2</sub> , G <sub>1</sub> , G <sub>2</sub> and ocratoxin A | Gas and Liquid Chromatography  |
| 20. IPEN/<br>CNEN-SP                              | Center on Chemistry and Environment  | Trace Elements in Water  | Atomic Absorption<br>ICP OES<br>Fluorescence<br>Ion Chromatography   |
| 21. OXITENIO                                      | Center for Analytical Research   | Surfactants, break fluids  | Thermo analysis<br>Wet Chemistry<br>GC MS<br>LC MS   |

## Metrological Demands

A recent survey conducted with the participating laboratories revealed the following main metrological demands, as pointed out by their managements:

- Support for the participation in inter-comparisons and proficiency tests;
- Availability of national reference materials for metals in blood serum and in water;

- Diffusion of the concept of contamination control in chemical analysis;
- Support for the acquisition of reference materials of different kinds of matrixes, such as: biological, geological, environmental and others;
- Support for participation in courses related to the field of Chemical Metrology;
- Availability of reference materials for the area of petroleum and derivatives;
- Availability of reference materials for chemical species, such as Se, As and Hg;
- Alternatives for accreditation of laboratories working in an R & D environment;
- Accreditation of the analysis of agrotoxics acquired by Brazilian laboratories;
- Quantification of elements of the uranium natural decay series, in order to calculate doses received by the population under influence of nuclear installations or uranium/thorium mines;
- Improvement in the quality of soil and sediment analysis;
- Improvement of awareness on the importance of Chemical Metrology;
- Support for the learning of statistical test application and uncertainty evaluation; and
- Support for participation in Chemical Metrology-related courses.

## **Situation of the laboratories with respect to Quality Systems and Accreditation**

The surveys that have been carried out by the coordinators of the RBMQ network and of the LACM sub-network have shown the following situation of the laboratories, with respect to the implantation of Quality Systems, participation in inter-comparisons and proficiency tests and accreditation by the Brazilian Metrological Institute, INMETRO or qualification by the metrological networks of the Brazilian states (such as REMESP) or evaluation by the European Community:

100% of the laboratories participate in inter-comparisons and/or proficiency tests;

76% of the laboratories have implanted Quality Systems and are submitted to internal audits;

52% of the laboratories have received accreditation by INMETRO, qualification by the metrological networks of their states or by REBLAS or ANVISA, including one laboratory audited by the European Community; and

29% of the laboratories have the intention of being submitted to accreditation in the near future.

## **Conclusions**

In the framework of the Brazilian Network for Chemical Measurements (RBMQ) a sub-network of 21 laboratories was formed (LACM) to provide the country's chemical community with reliable references for specific analytical techniques, analytes and matrixes.

The support of the RBQM to the sub-network was very positive and consisted mainly of acquisition of reference materials, participation in inter-comparisons and in courses related to the metrological area.

The laboratories of the sub-network pointed out to the coordinators several metrological demands, which are important information to the management of the

network and to Brazilian institutions for research support, providing them with data for better decisions in establishing their priorities.

All the laboratories at the moment participate in inter-comparisons and/or proficiency tests, the great majority has implanted Quality Systems and either already has accreditation or qualification by Brazilian institutions or intends to do so in the near future.

## **Acknowledgements**

The authors would like to acknowledge the financial support of CNPQ and FAPESP.

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