

08 October 2012

Peer Review of INTI

**“Research and Development of Special Organic
Contaminants”**

National Institute of Industrial Technology - INTI

Date: 4 - 5 June 2012

Reviewer: Dr. Mariana Arce Osuna, Materials Metrology, CENAM, Mexico

Buenos Aires, Argentina.

1. Scope of the peer review

The peer review is to evaluate INTI's capability and competence in establishing traceability in chemical and material properties measurements and dissemination of traceability to its customers on the basis of the criteria mentioned in ISO/IEC 17025:2005 and ISO Guide 34:2000. In addition the criteria agreed by the Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB) and the criteria on CRM's, formulated by the CCQM and approved by the CIPM, will be taken into account.

Further, the peer review team has taken into account:

- The Calibration and Measurement Capabilities (CMCs) as claimed by INTI and published in the Appendix C of the CIPM MRA, as well as will be formulated and claimed in the near future in the fields of organic analytical measurements.

During the peer review particular attention was devoted to:

- Traceability
- Measurement uncertainty calculations
- Measurement methods and method validation
- Reporting and records of measurement
- Qualifications of the scientific and technical staff

2. Evaluation process

The review process started on Tuesday morning 4 /June/ 2012 with an introduction describing INTI's legal status, organization, finances, marketing, staff, aims and activities carried out.

- Héctor Laiz, director responsible for INTI's program on Quality and Environmental gave a general introduction;
- Lic. Adriana Rosso, director of the Center for Research and Development of Special Organic Contaminants, in charge of organic analytical measurements in the scope of the CIPM MRA;
- Dra. Celia Puglisi, Manager INTI Chemical Metrology program
- Lic. Mariana Ruiz de Arechaveleta, Technical Unit Coordinator
- Lic. Gládys Mastromónaco
- Lic. Silvina Aued, Responsible for Quality Program

The information included a description of the quality system, actual Calibration and Measurement Capabilities (CMCs) in the scope of the CIPM MRA, participation in and results of SIM, CCQM and relevant comparisons, laboratories, measuring equipment and staff involved in metrology in organic chemistry in the Organic Contaminant Center.

The assessment of the laboratories started on Tuesday afternoon and continued until Wednesday afternoon.

During most of the time the peer review focused their review on organic analytical measurements, specifically for volatile organic contaminants in water and methanol, and ethanol in water.

The review process generally consisted of:

- A review of the quality system and manual

- Review of procedures in the laboratory
- Understanding of traceability and the realization of traceability
- Calibration of laboratory equipment
- Examination of measurement methods
- Validation of methods
- Examination of uncertainty calculations
- Laboratory visits (equipment and environmental conditions)
- Discussions with staff members (qualifications and understanding of specific metrological issues)
- Discussions on the assignment of values and on issues such measurement uncertainty
- Consideration of the results of key comparisons, pilot studies and other relevant results of comparisons and traceability dissemination in INTI measurements services
- Review of (samples) of the Calibration and Measurement Capabilities of INTI, planned to be published in the Appendix C of the CIPM MRA
- Examination of selected measurement and certification reports
- A review of INTI's measurement and value assignment services in relation to the requirements of ISO/IEC 17025.

During the assessments some so-called "vertical" audits have also been undertaken, checking the whole process of value assignment of a sample offered to a customer. The Organic Contaminant Center had been selected specific services where the traceability to the SI is intended to disseminate to their customers, such as aromatic hydrocarbons in water and in methanol, and water in ethanol, therefore particular attention was devoted to both services process.

It has to be remarked here that the peer review is of course just a snapshot taken at a certain moment and that it is recommended that such a review be repeated from time to time. The last peer review of INTI's programme on Metrology in Chemistry took place in August 2007.

3. Executive Summary

The Peer Reviewer wishes to acknowledge the hospitality, assistance, the open atmosphere and full cooperation of all the INTI staff members throughout the review process, especially the guidance of Lic. Adriana Rosso.

The staffs of the Research and Development of Special Organic Contaminants of INTI consist of a group of enthusiastic, technically skilled and competent co-operators. It is noted that the staff becomes regularly trained and they have the possibility to complete a MSc or PhD degree. Two students have joined INTI recently and are under training.

It is also noted that the number of staff in Research and Development of Special Organic Contaminants of INTI has practically not been increased over the last 5 years. This sustains the quality of Research and Development of Special Organic Contaminants of INTI measurements and deliverables to the customers

The results generated by INTI in key comparisons and pilot studies are in general satisfactory, although there are examples in which it became clear that more experience is still needed. However, since the activities in chemistry in INTI still in development and moreover the metrology concepts in measurements in chemistry are not well understood for all staffs, still necessary to include within INTI training program all base chemical metrology concepts. It is observed that Research and Development of Special Organic Contaminants of INTI is making very good progress in the right direction.

The Research and Development of Special Organic Contaminants have a large amount of chemical analysis equipment available. The facilities are similar to those in other national metrology institutes. They are suitable for the current activities.

In general, the measurement methods applied are adequate. Measurement uncertainty evaluation is based on the GUM and related EURACHEM documents. In some cases the claimed uncertainty seems to be on the optimistic side and should be reconsidered, of course taking into account the results of pilot studies and other bilateral comparisons and in particular of key comparisons. Thorough method validation is still necessary addressing all potential uncertainty components. Taking into account some reconsideration as mentioned above, the claimed calibration and measurement capabilities as proposed to become published in the Appendix C of the CIPM MRA are acceptable and seem to be reliable.

Several INTI laboratories have been accredited for a number of test methods on the basis of ISO 17025 by UKAS, UK and by the OAA in Argentina.

In the scope of the CIPM MRA, INTI has to have a quality system in accordance with ISO 17025. Therefore the Quality Manual has to be completed with chapters and procedures addressing the typical metrological and chemical calibration issues and the traceability services to the INTI customers, based on ISO/IEC 17025.

The detailed observations in the laboratory visited are detailed in this report.

Concluding and summarizing we can state that INTI has in principle the capabilities and competences to deliver organic services to its customers, now claimed in the CMCs that have been tabled for review by SIM and the other RMOs.

However, INTI should continuously review, improve, and update its quality manual in order to have it in full compliance with ISO/IEC 17025 with respect to the metrological measurement services.

INTI should also reconsider a number of its claimed measurement uncertainty statements. Further, in order to improve the obtainable measurement accuracy and realize a more sustainable metrology in chemistry activity it is highly recommendable to locate the metrological activities in separate laboratory rooms with better environmental control and own dedicated equipment. The metrological activities should be carried out by metrological trained staff, preferably having had the opportunity to work for some time as a guest worker in one of the matured NMIs having long standing experience in metrology in chemistry.

The peer review is only a snapshot taken during a few days. Therefore it is recommended that the peer review be repeated after a few years in order to build up a long term picture of the quality, capabilities and competence of the Institute.

The peer reviewer will appreciate INTI informing her after a period of about 6 months what corrective actions have been taken by the Institute on the basis of the recommendations.

4. Overview of INTI Program in Testing Contaminants Organic

The Organic Contaminants had a temporary Center set up in July 1987 with the aim of developing a research project related to sterilization of thermo sensitive biomedical products. In addition to the issues related to the sterilization by ethylene oxide, the Center studies other compounds of toxicological interest. The Research and Development Center on Special Organic Contaminants was set up by agreement as a permanent center within INTI on May 9th of 1997. The staff consists of 10 personnel.

The focus of this Center is on the establishment of high quality laboratory facilities for analytical services and methods evaluation for the determination of organic contaminants in solutions and natural matrices as related to products, packaging, materials, environment, biomedical products, and food. In addition, they investigate and incorporate new analytical determinations of organic compounds of toxicological interest in environmental matrices. As the national primary reference the Contaminants Center also are to promote the activities in Chemical Metrology to assure the traceability and comparability of chemical measurements of the Center, and to support the quality of organic contaminant measurement of Argentina.

The long-term goal of INTI's testing and measurement program in organic contaminants is to provide measurement and testing services to facilitate the traceability to INTI for their customers in Argentina. To assure the quality of chemical measurement within the country, one of the main objectives of the center is to provide reference values regarding measurement of organic contaminants traceable to SI or to primary methods or measurements systems by using appropriate CRM and participating in SIM, CCQM Key comparison and pilot study as well as bilateral comparison. The Organic Contaminant Center responsibility is to provide services supported by their CMC and disseminate to their customers their traceability to the SI, consequently the process of organic volatile in soil and water in ethanol services were reviewed.

5. Staff

Adriana Rosso, Director of Research and Development of Special Organic Contaminants, has worked at INTI since 1992, and the current staffs responsible for organic contaminant measurement services were interviewed and are listed below.

Name	Function	Education	Year Started at INTI	Years of Experience
<i>Professionals (minimum 5 yrs college)</i>				
Lic. Adriana Rosso	Director	MSc Environmental	1992	26
Lic. Mariana Ruiz de Arecchavaleta	Technical Unit Coordination	Environmental Chemistry	1997	15

Silvina Aued	Quality Manager	BSc Chemistry	2010	1.5
Lic. Jimena Etcheverry	Laboratory Chief	BSc Chemistry	2000	12
Water Armada	Analyst	BSc Chemistry	2009	3
Julian Gigena,	Analyst	BSc Chemistry	2005	5
<i>Technicians (minimum HS Grad)</i>				
Javier Filippetto.	Test Operator	Chemistry Technician	2003	9
Damian Piuselli	Test Operator	Chemistry Technician	2011	1
Yanina Carossia	Technical Secretary	Commercial Appraiser	1999	13
Romina Albertotti	Support	Middle School Student	2007	4

Six of the ten staff members were at INTI and working in the Special Organic Contaminants area when the 2007 Peer Review was conducted, two of them had been promoted, Jimena Etcheverry from Analyst to Laboratory Chief, and Julian Gigena who was able to finish the college in BSc Chemistry was promoted from Test Operator to Analyst. Three staff had been left, a quality manager, one test operator, and one support; and four had been hired to replaced them, a quality manager, a analyst, a test operator, and a support; they had been working in the Special Organic Contaminants area at INTI for 1.5, 3, 1, and 4 years respectively. Overall, two technician levels and one MSc-level staff have left the Special Organic Contaminants since the 2007 review.

The experienced staff and technical capabilities in the organics contaminant area were maintained, and had a middle-turnover in test operator staff. As recommended by the 2007 peer reviewer a training plan had been established and staff attended metrology courses and registered the competence in their file. The competence of the new test operators was assured through their training plan to guarantee the quality of provision of services and the activities of organic contaminant area. The competence log and the applied training plan were reviewed; where it was observed that no basic metrological concepts are evaluated.

1. Recommendation

It is recommended that the organic contaminant area of INTI include within their training program an evaluation of fundamental metrological concepts such as the incorporated in the VIM and GUM.

2. Recommendation

It is recommended to ensure in internal and external teaching courses that the value and appropriate use of CRMs and traceability are included where appropriate. This will promote suitable use of CRMs with appropriate traceability.

All staff appeared to be highly motivated, show enthusiasm, and take pride in their work activities. However, more experience in chemical metrology will be supportive to achieve other NMIs the level. Therefore leadership is very important to assure continuity in the program and to pursue technical cooperation with their peers. There appears to be a commitment by INTI for opportunities in continuous learning within Argentina. A significant training in chemical metrology could be achieved by studies at other NMIs which could range from months to up to a year or more and could include studies at universities resulting in obtaining PhD's in Analytical Chemistry or related fields. Parallel is relevant to dedicate time to organic analytical metrology and research to improve their measurement procedures.

3. Recommendation

It is recommended that INTI set up a programme for studies abroad at other NMIs to assist in the development of good metrology practices and set aside time for in-house metrology research to develop better understanding of their instrumental operations and measurements.

4. Laboratory Facilities

The laboratory facilities were found to be adequate for the measurements and testing being carried out. The environmental conditions in the laboratories were found satisfactory for the intended operations, where the temperature is controlled with air conditioning units, and each laboratory has an assigned "responsible person" and a supplemental "responsible person". Laboratory temperature and pressure are recorded daily. However no temperature control had been set in the balance room. The gravimetric measurements are fundamental to establish the traceability to the SI, as a result the temperature and humidity of the balance room can have significant contributions for obtaining good, reliable gravimetric preparation such as calibration curve and sample preparation.

4. Recommendation

It is recommended that INTI investigate the possibility of providing an adequate temperature control in their balance room, and records temperature and humidity of its balance room.

5. Equipment

Equipments are suitable for the current and selected proposed activities. This instrumentation is well maintained, one staff member is responsible for assuring the maintenances and log control for each equipment. Records are maintained that indicate when needed service to an instrument is taken, and also when repairs are made to these instruments. However, there is no room for expansion without crowding, and to attend this necessitate a new facility is under construction. Laboratories are instrumentally well equipped and organized to a manner that enhances a safe work environment; except for the gas cylinders, which are not secured to wall or table.

5. Recommendation

To improve safety in working areas, it is recommended that INTI rearrange the setting of their gas cylinders, where each of the cylinders can be secured to a base, table or wall with a band or chain.

4. CCQM and SIM Studies for Organics:

The results of international comparisons studies were reviewed

Comment: The Organic Contaminant Center had been participated in P61, CCQM-K47, CCQM-K79, AFRIMETS QM-K27, CCQM-K27.2, CCQM-K55.b and in SIM coordinated by Brazil. INTI had been performed well for aromatic hydrocarbons in methanol, but for ethanol in water was poor. As recommended in the 2007 peer reviewer they had been participate AFRIMETS QM-K27 and in SIM for which the report is underway.

6. Recommendation

It is recommended that INTI's Organic Contaminant Center continues their participation in relevant SIM and CCQM OAWG studies to ensure that the measurements made using revised methods /new types of instrumental systems, ambient condition, etc. for service delivery are properly compared.

5. CIPM MRA KCDB, CMCs for Organics:

INTI's published CMCs were reviewed, and their CMC are applied, and documented. The Organic Contaminants Center has six CMC claims that have been reviewed both within in SIM and this peer review process. These claims are one each for benzene, toluene, o-xylene, m-xylene, p-xylene and ethylbenzene in methanol. The INTI claims are supported by there measurement capabilities as presented from their data.

Website of published CMCs: <http://kcdb.bipm.org/appendixC/default.asp>

Website of CIPM Comparisons: http://kcdb.bipm.org/appendixB/KCDB_ApB_search.asp

7. Recommendation

The actual INTI hydrocarbons in water CMC was claimed using methanol as a solvent, and actually N-N-Dimethylacetamide (DMA) is used, therefore it is recommended that the Organic Contaminant Center evaluate if the results of the method using DMA as a solvent are comparable with the results of the method using methanol as a solvent.

6. Method Validation in Organic:

The validation of measurements procedures used in the value-assignment of organic analytical services for volatile organic contaminants in water, methanol and soil, and ethanol in water were reviewed. A method procedure is described in INTI's QS, applied, and documented. When I requested a particular record to review, the records were given to me within a few minutes. They are well organized and readily available. In this procedure for volatile organic contaminants in methanol a ES method with a dependent calibration curve is described, this is one stock solution from where 6 diluted levels are made; while for methanol in water a IS method with a dependent calibration curve is described. Particularly for volatile organic contaminants in soil method procedure, a reproducibility of replicates samples is a restriction to accept the measurement results of a sample, since all measurands are volatiles in a complex matrix, migrating to an adequate IS method can improve the results reproducibility.

8. Recommendation

It is recommended that INTI's Organic Contaminant Center improve method validation and quality control of their volatile organic in solvent or in matrix measurements, a in house CRM or a CRM from another NMI is suggested to be used as a control sample.

9. Recommendation

It is recommended that INTI's Organic Contaminant Center improve method validation and quality control of their volatile organic contaminants measurements, a IS method is recommended to be implemented and compared with the actual ES method approach.

7. Metrological Traceability:

Balance facility, calibration records and certificates, training records and records of subsequent testing of trainees were reviewed.

10. Recommendation

See recommendation of section 3.

11. Recommendation

While there is not temperature control of the balance room, the corresponding temperature correction of the mass is recommended to apply, or the variation of room temperature should be recorded and the corresponding variation of the mass because of the room temperature variation is recommended to be included in the uncertainty budget of the affected analytical measurements.

12. Recommendation

To improve the accuracy of calibrants or samples gravimetric preparations it is recommended that the Organic Contaminant Center evaluate la possibility to acquire a microbalance.

13. Recommendation

It is recommended that INTI's Organic Contaminant Center clearly declared in each method procedure to what reference the traceability to SI is intended to be established and disseminated through a particular analytical services, therefore the control CRM and Calibrant (primary reference materials) used for this purpose should be stated in the method procedure.

14. Recommendation

It is recommended that the Organic Contaminant Center clearly declared in the Measurement Certificate the identification number of the CRM used to establish traceability to the SI

1. Non conformity

For traceable to SI measurement services such as ethanol in water and organic volatile in soil, INTI's Organic Contaminant Center, as a NMI, requires to document the reported results in a Report of Analysis, which should be readily available for internal records in QS and for customers if requested.

Purity Assessments of primary materials and CRMs:

Primary reference standards are reassessed for purity prior to each time used. The processes are described in the QS documentation, applied, and documented. INTI is obtaining pure forms of the organic species and analyzing them by GC/MS area normalization, where relative response factors of the measurand and impurities are used to assess the purity, and water or solvents, inorganic, content are not evaluated. Stability studies of the calibration solutions had been made, and the shelf –life of calibration solutions had been established and applied.

15. Recommendation

It is recommended that INTI's Organic Contaminant Center improve their purity assessment approach of primary reference standards used for calibration, by comparing, and if adequate combining at least two methods results. These methods could be based on mass balance or direct quantification of the standard, or comparison of both approaches results.

16. Recommendation

For the mass balance method, quantification of water or solvents, and identification and quantification of major impurities content when is possible is recommended.

17. Value Assignment and traceability dissemination through analytical services.

The processes are described in the QS documentation, applied, and documented. No reference material for sample preparation evaluation and control is used.

2. Non conformity

For the value assignment in a service of a volatile organic contaminants in soil analysis, a RM for method preparation evaluation and control should be used, a INTI or other NMI's certified RM will be the best option, otherwise at least a home certified reference material should be used.

17. Recommendation

To improve assessment of traceability dissemination for volatile organic measurement within Argentina is recommended that, the Organic Contaminant Center evaluate the possibility of organizing a national proficiency test for volatile organic contaminants in matrix.

18. Uncertainty Calculations/Estimations:

There is no staff in the Organic Contaminant Center specifically responsible for statistics. All staffs are trained to provide uncertainty estimations within a particular set of measurements. The involved metrologist provides the statistical and technical aspects of uncertainty estimations. However the uncertainty estimation for analytical services includes the repeatability of the measurements and calibrations curve variation within others, and not includes the contribution of calibrant dilutions. Also, the uncertainty estimation for the purity characterization of materials used as calibration materials was observed in both of cases to consist only of the repeatability of 3 characterizations and not include type B components as is usually expected at the NMI level of practice of traceability to the SI. These components may result from the assumptions regarding relative response factors of the impurities to the analyte of interest where no calibrant for these is available or where identity of impurity is not determined; a component for probably impurities not detectable under the conditions used, homogeneity of material, etc. The use of these type B components is often the result of having a relatively small amount of calibrant material that precludes use of ashing or other characterization methods that require large quantities of material. The uncertainty estimation calculations of each measurement service is recorded in a general proceeding, and are not included in the methods.

18. Recommendation

*It is recommended to convene a meeting of organic contaminant staff of presentation/discussion of expectations for critically evaluation of the results of a analytical measurements, or purity characterizations of materials used as calibrants (primary reference materials), as well as **the overall uncertainty** of the assigned value for a analytical services, or for purity of a calibrants, and the inclusion of their corresponding contributions in the uncertainty budget.*

19. Recommendation

Particularly, for the ethanol in water analytical service is recommended that the Organic Contaminant Center includes in the uncertainty estimation of the measurement result, the contribution of the primary reference material used.

20. Recommendation

For the ethanol in water and organic volatile in soil analytical services is recommended that the Organic Contaminant Center documents the uncertainty estimation steps in the method.

19. Storage of primary materials and CRMs.

Appropriate processes are described in the QS documentation, applied, and documented. There is sufficient refrigerator and freezer space. Temperatures are recorded in the morning of each work day.

21. Recommendation

To improve the preservation of a sensitive CRM is recommended that INTI's Organic Contaminant Center evaluate the possibility to acquire a -80 °C Freezer.

20. Complaints and Non-conformities.

These were reviewed for the 2007-current time period. Most of the recommendations were attended, or actions are in their ways. No valid complaints with respect to technical aspects of services in Organics Contaminant Center were in these records.

21. Measurement Records

The Organic Contaminants Center has improved their documentation of procedures and measurements. All documents and forms are secured in INTI network and have a back up copy, and are readily and quickly obtainable.
