Report, 04 November 2012

INTI – Peer review for Chemical Inorganic Analysis Methods

25-27 June 2012

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Scope of the peer review

The peer review evaluated the capabilities and competence of INTI in establishing traceability in chemical measurements and the dissemination of traceability to customers on the basis of the criteria mentioned in ISO/IEC 17025.

The peer review focused on specific measurement chemical methods and the corresponding Calibration and Measurement Capabilities (CMC's) as may be claimed by the laboratory when participating in the CIPM MRA, as well as may be formulated and claimed in the near future in the fields of inorganic analytical measurements.

During the peer review particular attention was devoted to:

- Traceability
- Qualifications of the scientific and technical staff
- Measurement methods and method validation
- Reporting and records of measurement
- Quality of results reported in CCQM intercomparisons
- Efforts for further development in support of the NMI (e.g. new CMC claims)

Evaluation process

The peer review process was carried out during the period 25-27 June 2012. It started on Monday morning (25 June 2012) with a presentation by the Lic. Liliana Valiente, technical director of INTI - Chemistry. She gave an overview of current activities of INTI, its organization, customers, finance, staff, new laboratory space and equipment and future prospects.
Dr. Héctor Laiz, director responsible for INTI's programme on Metrology, Quality and Certification gave an overview of the current metrological activities of INTI related to participation in SIM and CCQM international intercomparisons, provision of reference values and proposed calibration and measurement capabilities in the scope of the CIPM MRA.
Most of the INTI staff attended the Monday morning session.
The assessment of the different areas (trace, metal analysis and water technology) within inorganic analysis started on Monday afternoon and continued till Tuesday afternoon.

The review process included:
- Examination of selected measurement methods
- Discussions with staff with regards to validation of methods
- Examination of uncertainty calculations
- Planning and execution of CCQM comparisons
- Records of staff training and competence
- Calibration of instrumentation
- Laboratory visits (records, instrument log books, environment, key instrumentation and staff)
- Laboratory demonstrations by INTI staff
- Understanding of traceability and the realization of traceability

- Review of some of the Calibration and Measurement Capabilities of the INTI, to be claimed in the near future in the scope of the CIPM MRA.

It is important to note that insufficient time was available to conduct a detailed review of the quality manual and thus the focus was on specific measurement methods relevant to future CMC claims and on the competence of the staff delivering the inorganic analytical measurements.

Executive Summary: General feedback

I wish to thank INTI staff and, in particular, Lic. Liliana Valiente, for the hospitality and warm welcome. Also for their assistance and transparency, which facilitated the discussions throughout the entire process.

First of all, I was particularly impressed with the high calibre and enthusiasm of the staff. Their deep knowledge of metrology and high competence are undeniable. Staff at all levels have a good understanding of the work they are carrying out. A number of staff have participated in international metrology comparisons or meetings, so there is a good understanding of the requirements of CIPM MRA. They also receive training (e.g. method validation, measurement uncertainty calculation) from Dr. Pablo Alvarez.

INTI's most important mission in providing reference values to relevant intercomparisons in different industry sectors came across clearly. Of particular importance is the continuous support of the Government in providing the required resources to maintain and further develop the NMI status. A clear example is the recent investment in expanding the laboratories and the purchase of new ICP-MS instrumentation, required for implementation of isotope dilution reference methods.

It is recommended that a more clear strategy for future production of reference materials is defined. This will help focus resources and efforts and determine relevant CMCs to be claimed and supported.

In the scope of the CIPM MRA, INTI has a quality system that complies with ISO 17025. Also, the services provided to customers comply with the requirements of CIPM MRA. In relation to this, this peer review has made specific recommendations to improve the quality system and operations in line with the future claim of new calibration and measurement capabilities (CMCs). These are detailed in further sections of this report.

In general, the measurement methods applied are adequate. Measurement uncertainty evaluation is based on the GUM and related EURACHEM documents.

The documentation and data storage system should become more harmonised and transparent, as currently the different parts have to be found in different places, partly electronically, partly on paper in cupboards or personal notebooks. For electronic data recording a security system like LIMS is recommended.

It is also recommended to have a clearly defined plan before carrying out CCQM comparisons. Such plan should clearly state the actions to be undertaken, timescales and the responsibilities of each member of staff in its execution. Also, the process of planning number of replicates and measurement batches for a CCQM study should be more clearly documented.

The process of judging the competence of the staff to run the different measurement systems should become more formalized.

The increasing use of ICP-MS (more routinely) and the implementation of IDMS procedures is highly recommended since this will improve the quality of the metrological activities. To achieve this, staff training should be undertaken.

Taking into account some considerations as described above, the proposed calibration and measurement capabilities are acceptable and seem to be reliable.

Attestation

The INTI has the capabilities and competences to carry out the services it is claiming to deliver to its customers in the scope of the CIPM MRA.
The quality system, as far as it could be reviewed, is in compliance with ISO/IEC 17025:2005 and covers the near future claimed CMCs.
No Non-Compliances have been found, however a few minor non-compliances have been observed. Those are detailed in further sections of this report. A number of recommendations for further improvements are, therefore, added in the peer review report.

Note: The final approval of CMCs is obtained through the intra- and inter RMO review process.

Signed, November 19, 2012-11-19

Heidi Goenaga Infante

Report on peer assessment of technical capabilities of INTI: Measurement methods within Trace Analysis and Water Technology Units.

Reviewer: Heidi Goenaga Infante, Principal Scientist, Chemical Measurement and Calibration, LGC Limited

Overview

The National Institute of Industrial Technology – INTI and its Chemistry centre were was created in 1957. Since then, it has supported different sectors of industry. One of the responsibilities of INTI is to realize and maintain the national measurements standards of Argentina.
Most of the activities in the Chemistry Centre are dedicated to "testing". The metrological activities in the field of inorganic chemistry are carried out by the Trace Analysis laboratory, the Metal Analysis laboratory and the Water Technology unit of the Chemistry Centre.
The Chemistry Centre has 113 staff, of which 44% are professionals, 41% are technicians and the rest is administrative and maintenance personnel.
The laboratory has excellent contacts with NMI's from other countries and has been involved with the work of SIM and CCQM for a number of years.

1. Recommendation

It is recommended that a more clear strategy for future production of reference materials is defined. This will help focus resources and efforts and determine relevant CMCs to be claimed and supported.

Personnel

Personnel involved in the peer review included four staff members in the Metal Analysis group (Monica Borinski, Mabel Puelles, Nadia Hatamleh and Osvaldo Acosta), two in the Water Technology unit (Ana Hernandez and Ariel Galli) and four in the Trace Analysis group (Liliana Valiente, Margarita Piccina, María Lorena Iribarren and Christian Salamone). Staff at all levels have a good understanding of the metrology work they are carrying out. They were very enthusiastic, transparent, easy to talk to and very keen to learn new concepts.

Training records were documented. Evidence for competence was obtained by the analysis of blind samples.

2. Recommendation

INTI recently acquired ICP-MS instrumentation. It is highly recommended that ICP-MS is more routinely used and IDMS procedures get implemented. This will improve the quality of the metrological activities. To achieve this, staff training by secondment visits to other NMI's or expert laboratories should be undertaken.

3. Recommendation
A more formal procedure for staff training and approval of competence, including a record of who granted approval, what specific capability for and when, should be introduced.

Accommodation and environmental conditions

After the 2007 review, the laboratory area has been extensively modernised and extended to incorporate ICP-MS instrumentation and adequate sample preparation facilities. The area has sufficient space along with an appropriate separation of activities. It is suitable for preparation and storage of calibration solutions, reference materials and samples.

Equipment and instrumentation

The laboratories are well equipped with one ICP-OES, one ICP-MS, four AAS (equipped with graphite furnace and hydride generation), ionic chromatography, etc. The number of scientists is relatively small compare with the wide range of instrumentation. Therefore, implementing and validating the new ICP-OES and ICP-MS instruments and their associated procedures has imposed an additional workload. Ample facilities are available for preparation of calibration standards and samples with a good range of ancillary laboratory equipment. All of this has been purchased and maintained at the level expected of an NMI and all relevant equipment is well maintained.

4. Recommendation
It is recommended to provide more detailed information about specific applications, daily problems and solutions and instrument daily performance on the instrument log books.

5. Recommendation
It is recommended to assign a first and second contact staff responsible for each of the instruments and equipment. This is important to keep up instrument utilisation.

Measurement procedures

Detailed procedures have been documented for a number of analyses required for international intercomparisons, certification of reference materials or reference values for PT schemes. These are appropriate for a metrology institute but are currently mostly based on AAS. Future plans involve using AAS, ICP-OES and ICP-MS as complementary techniques, where appropriate, for material certification and provision of values to CCQM comparisons.

6. Recommendation
The ICP-MS should be increasingly used as quickly as possible so that it can be used for much of the relevant metrology work.

7. Recommendation
It would be preferable to give priority to development of IDMS procedures so that the ICP-MS can be used on its own for certification, CCQM comparisons, reference value provision, with AAS and/or ICP-OES used only for confirmation.

Method development and validation

IINTI maintains controlled records of validation based on a series of documents stored, partly electronically, partly on paper in cupboards or personal notebooks. A detailed discussion was held of selected procedures including methodology for the determination of Hg in fish using cold vapour AAS (Traces group), methodology for the determination of Pb in tin alloys using ICP-OES (Metals group) and for nitrate in calibration solutions using ion chromatography (Water Technology group). Again, method selection was adequate. All the staff involved is well aware of the advantages and drawbacks of the techniques used and the procedure operation steps. Laboratory balances were found properly calibrated and calibration certificates were up to date. In general, it was noticed the use of volumetric flasks to prepare calibration solutions although the working units are in *w/w*.

8. Recommendation
It is recommended to improve record keeping for method selection, calculations, planning, decision taking, preferably electronically.

9. *Recommendation*
It is recommended to state the provenance and purity (if available) of reagents used for all the methods under discussion.

10. Recommendation
For methods, involving use of HF (e.g. for Pb in Sn alloys), it is recommended to put in place a risk assessment procedure to be understood and followed by all staff.

11. Recommendation
For ICP-OES methods, it is recommended the more frequent use of calibration by internal standardisation to minimise influence of instrumental drifts, etc on the measurement uncertainty.

12. Recommendation
It is recommended that amount units are consistently expressed in mg kg^{-1} rather than ppm (e.g. in ion-related methods).

13. Recommendation
It is recommended to avoid use of volumetric flasks, if unnecessary, when preparing calibration solutions by weight. This will minimise risk of contamination and will help improve efficacy.

Uncertainty

The staff as a whole have a good general understanding of measurement uncertainty and those interviewed were able to explain how they had evaluated uncertainty budgets. Comprehensive records of uncertainty budgets are maintained but, again, partly are stored electronically and partly on paper in cupboards or personal notebooks.

14. Recommendation
Detailed information and instructions on the calculation of measurement uncertainty should be consistently documented (preferably, electronically) with each individual measurement procedure. It is also recommended that cross checking of measurement uncertainty calculations is undertaken.

Traceability

Results using the reviewed methods are mainly traceable to SI through NIST SRMs and this should be more clearly specified in each method description. Staff has a very good appreciation of traceability and application of the concept.

Quality assurance of results

The laboratory has tracked record of participation in several SIM and CCQM comparisons for a number of years. This is used as an important part of method validation. The results for some inorganic comparisons (e.g. CCQM-K42; CCQM-K88, CCQM-P119) were examined and were generally very good. Detailed planning of CCQM comparisons is not always properly recorded. The input of statisticians in defining experimental designs (number of independent batches, number of replicates per batch) for CCQM execution and also in data processing was also missing.

15. ***Recommendation***
Until a fully electronic system can be implemented it would be useful to either maintain a complete paper file for each method or produce a single pdf file which includes everything in a correct order.

16. Recommendation
It is recommended to put in place plans (preferably electronically) for CCQM comparisons. These should be well understood and signed by all the staff involved in CCQM execution.

17. Recommendation
It is recommended to report results to CCQM which are based on at least two independent batch analyses (analysed at least in different days).

Calibration and Measurement Capabilities (CMCs)

Overall, the inorganic team has the capability to deliver its claimed CMCs. For CMCs already claimed, comments provided by peer reviewers in 2007 were undertaken.

For CMCs to be claimed within category 10 (biological tissue) and category 9.3 (polymers and plastics), it is not clear where does the lower limit of the dissemination range comes from since limits of quantification are missing in the described procedures (e.g. Se, Hg in fish or metals in plastics). The mass fractions and its associated expanded uncertainties, as reported to CCQM comparisons under these categories (CCQM-P106 and K43.1) fall well within the window defined by the concensus value and its associated expanded uncertainty.

18. Recommendation
It is recommended that that these new claims are revised in terms of lower limit of the dissemination range. Also method LOQs should be included in the method description.

19. Recommendation
For new CMCs within Metal Analysis (category 8: metals and metal alloys), it is recommended to proceed with the claims only for Fe and Cu in Cu alloys, as supported by the good laboratory performance in the CCQM comparison K67. Also for Pb in Pb-free solders as supported by the results of CCQM-K88. It is recommended that the CMC claim for the latter is extended to other tin-based solders containing silver and copper provided that the concentration range of lead is 100 mg/kg to 2000 mg/kg and that those of silver and copper are less than 4 % (mass fraction) and less than 6 % (mass fraction), respectively.