

INTI – Peer review for Chemical Inorganic Analysis Methods Action Plan

Finding (Recommendation)	Action	Status
1. 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.	Since November 2012, the Deputy Manager of Metrology of INTI is conducting an institutional plan, tending to promote and coordinate the production of reference materials in different INTI centers, and new declaration of CMCs. INTI Química is aligned with this policy. In the first step, in 2013, targets have been established in the production of CRM in milk samples and ethanol in water solutions. A peer review for these activities is being planned for this year. Also, other activities have started, with different degrees of progress. Some of them arise INTI-Química	Cleared.
2. 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.	A member of the staff is being trained (February to May 2013) in IDMS methods at LGC (UK). Then, he will contribute to implement new methods by applying the ICP-MS instrumentation.	In progress. Responsible: L. Valiente
3. 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.	The procedure, PG-02 "Staff" was created. It includes the form F-02.2, for staff qualification in specific measurements. This form includes the name of the staff, the measurement for which has been qualified, the date and the name of who granted approval.	Cleared.
4. It is recommended to provide more detailed information about specific applications, daily problems and solutions and instrument daily performance on the instrument log books.	A detailed procedure for the operation and monitoring of performance of ICP-OES equipment has been elaborated. It has also implemented a register to keep a record of daily problems and daily instrument performance.	Cleared.
5. 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.	The form, F-11.2 "Authorized staff to use the equipment", includes a first and second staff responsible for the equipment. This record is kept along with the instrument log.	Cleared.

Finding (Recommendation)	Action	Status
<p>6. The ICP-MS should be increasingly used as quickly as possible so that it can be used for much of the relevant metrology work.</p> <p>7. 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.</p>	<p>See 2. It's a policy of INTI start using IDMS methods so that the ICP-MS can be used on its own for certification.</p> <p>In this context, a member of staff is being trained at LGC (UK). Anyway, it is relevant to say that the present CMCs are not related to this equipment. Once the IDMS methods are ready and in force, improvements in the CMCs will be declared.</p>	<p>In progress. Responsible: L.Valiente</p>
<p>8. It is recommended to improve record keeping for method selection, calculations, planning, decision taking, preferably electronically.</p>	<p>A system to store information and data in electronic way was developed, in order to keep documents related to development and validation of measurement methods.</p>	<p>In progress. Estimated to June 2013 Responsible: P. Alvarez</p>
<p>9. It is recommended to state the provenance and purity (if available) of reagents used for all the methods under discussion.</p>	<p>The General Procedure PG-04 "Purchasing services and supplies" includes a form, F-04.4 "Stock Supplies Record". This form was modified to include more complete information.</p>	<p>Cleared.</p>
<p>10. 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.</p>	<p>The measurement procedure PMTMet-03 "Determination of lead in tin alloys" was modified by incorporating recommendations about the use of HF. In addition, a leaflet with instructions is kept in the working place.</p>	<p>Cleared.</p>
<p>11. 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.</p>	<p>The material (yttrium) was purchased. Once it arrives it will be used as internal standard for ICP-OES methods. Then, the calibration by internal standardization will be incorporated in the procedure.</p>	<p>In progress. Estimated to: Apr 2013 Responsible: P. Alvarez</p>
<p>12. It is recommended that amount units are consistently expressed in mg kg⁻¹ rather than ppm (e.g. in ion-related methods).</p>	<p>All the documents including amount units expressed in "ppm" were modified indicating the right expression in "mg kg⁻¹".</p>	<p>Cleared.</p>
<p>13. 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.</p>	<p>Volumetric flasks are been discarded in the preparation of the calibration solutions. Instead, Falcon-type flasks are being used.</p>	<p>Cleared.</p>
<p>14. 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.</p>	<p>All the calculations of measurement uncertainty are being recorded electronically with detailed information. Cross checking of measurement uncertainty calculations is being undertaken when possible,</p>	<p>Cleared.</p>
<p>15. 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.</p>	<p>A general protocol was designed, including experimental design, equipment to be used, staff involved, among other fields.</p>	<p>Cleared.</p>

Finding (Recommendation)	Action	Status
16. 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.	The same protocol as for 15 is applied.	Cleared.
17. It is recommended to report results to CCQM which are based on at least two independent batch analyses (analysed at least on different days).	It was implemented.	Cleared.
18. 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.	In all measurement procedures for traces elements by atomic absorption, the lower dissemination range matches the LOQ (limit of quantification), except in the PAA Met-10: Determination of cadmium, chromium and lead in polypropylene and various polymers by atomic absorption spectrometry with electrothermal atomization, which has already been amended and approved taking the same criteria as the other procedures. The LOQs were added to all procedures.	Cleared.
19. 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 Sn-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.	Consideration is being given to expanding the scope for the determination of lead (Pb) in tin alloys containing higher amounts of silver and copper. Also, the need to expand the range of measurement of lead at 2000 mg/kg is being evaluated	In progress. Responsible: L.Valiente