Report

Technical Meeting on Quality Assurance for Nuclear Spectrometry Techniques

(G4-TM-36923)

IAEA, Vienna, Austria

12 – 16 October 2009

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Executive summary

A Technical meeting was held in the IAEA headquarters in Vienna, Austria from October 12-16, 2009. The objectives of this meeting were to review the current status in implementation and use of Quality Assurance in nuclear spectrometry techniques; to assess/demonstrate the benefits and impacts of QA on improvement of reliability of chemical measurement results in order to further extend the applicability range of the analytical techniques, and to produce a meeting report (proceedings).

Participants representing 20 Member States (MSs) and 21 organizations (see list of participants in Appendix 1) presented their approaches on quality assurance implementation and laboratory accreditation for alpha, beta and gamma-ray spectroscopy and X-ray fluorescence spectrometry, applied to a variety of fields such as environmental monitoring, food quality assessment and neutron activation analysis (see agenda in Appendix 2). The presentations resulted in technical discussions on sources of errors, interpretation of international requirements, pitfalls and developments in instrumentation, metrology and calculus.

The participants concluded that the objectives of the meeting were accomplished by the wealth of information exchanged in presentations and discussions. Summarizing, the participants concluded that

- In several laboratories technical expertise is fading.
- Accreditation of management systems is never a mandatory issue, but quality assurance (including quality control) is indispensable. Accreditation for compliance with the ISO/IEC17025 may become a pre-requisite for nuclear spectroscopy laboratories if the laboratories provide test results for e.g. environmental monitoring and global trade. The IAEA is playing an important role in stimulating MS laboratories in implementing QA/QC, and reaching formal accreditation.
- Sensitization of executive management needs planned attention for the implementation of QA/QC practices and the final step towards accreditation.
- There is, to some extent, a need to consider harmonized guidelines for QA/QC practices as examples to laboratories for supporting the process leading to accreditation. Education in the principles and practices of QA/QC was identified as one of the important issues for successful implementation and sustainability.
- There is a need for assuring the sustainability of metrological competence in MS nuclear spectroscopy laboratories, given the retirements of experienced personnel as well as the existence of single-points-of-failure in small staffed groups, the developments in instrumentation and calculus and the increasing requirements (and importance) to the quality of measurement data.
- New metrological approaches have been proposed for nuclear analytical techniques, including calibration of equipments, quantification of associated uncertainties, detection limits, etc. Development of materials for proficiency testing is needed to meet the newest metrological requirements.
- In-situ measurements have not yet reached a satisfactory level of implementation of QA/QC practices and field measurements may need to get adequate attention with respect to QA/QC.

These conclusions resulted in the following recommendations to the IAEA:
• To organize in 2010 a consultancy meeting to prepare the terms of reference of a new CRP on (some of) the below mentioned topics.
• To create a database of nuclear analytical laboratories to provide information on accredited laboratories, current status of nuclear spectrometry, etc; serving as a platform for creating a network of nuclear spectroscopy laboratories.
• To continue the development and revision of guidelines and TECDOCs concerning QA/QC as well as the metrology of NATs (Nuclear Analytical Techniques) to foster laboratory accreditation.
• To assist MS laboratories by harmonized guidelines for QA/QC practices to facilitate comparability and internal audits.
• To continue organizing workshops and technical meetings on QA/QC in specialized fields of nuclear spectroscopy to monitor the progress of implementation, to share experiences and to discuss innovations and recent developments.
• To strengthen support of nuclear analytical techniques to improve and sustain metrological competence by supporting implementation of a mechanism to assure and strengthen the metrological competence in methodologies for determining alpha, beta and gamma-ray emitting radionuclides. The mechanism should also strengthen the collaborations between research and university laboratories, as well as laboratories involved in the Agency’s ALMERAn (Analytical Laboratories for the Measurement of Environmental Radioactivity) network with national metrology institutions for measurement of radiation.
• To direct attention for QA/QC and metrology of (semi-quantitative) in-situ measurements of radionuclides and hazardous elements.

At large, the IAEA is recommended to continue the organization of coordinated research projects on subjects related to quality and metrology of nuclear spectroscopy, to organize proficiency testing schemes for nuclear spectroscopy as well as to support requests of Member States for the relevant TC projects and fellowship training.

Full Report

1. Introduction

    Quality assurance has always been important aspect to the IAEA as an indispensable tool contributing to safety as can be seen in its Code of Practice for Safety in Nuclear Power Plants and ensuing documents. The importance of quality assurance/quality management has been well recognized in areas of (globalization of) trade, health, safety, and environmental protection as an instrument to provide confidence in the product and to carry out processes in an efficient and effective manner.

    Laboratories using nuclear spectroscopy techniques (alpha, beta, gamma-ray spectroscopy; total alpha/beta counting and X-ray fluorescence spectrometry) deal with an additional driving force for ensuring the quality of their results: society’s skepticism and disbelief in almost anything labeled as ‘nuclear’ imply that results from tests in such laboratories should be beyond any doubt. The beneficiaries of these laboratories include, for example, government agencies regulating public health, safety, and the environment; private organizations seeking product certifications for export or import; and, both public and private institutions seeking to maximize agricultural safety, crop preservation, and nutritional utilization.
The degree of accuracy of results is initiated by the demands of the laboratory’s customers, and can be established by internal quality control, eventually to be confirmed by participating in interlaboratory comparison exercises. Preventing poor results to be generated requires, however, a quality assurance program that is strongly built on technical competence, viz. (i) an understanding of the physico-chemical processes and principles, including the operation of equipment; (ii) the identification of (potential) sources of errors (both organizational and technical), and (iii) the implementation of techniques for monitoring the occurrence of such errors. Nuclear spectroscopy techniques, as they are based on physical principles, are very well understood in all respects and many textbooks and publications in scientific journals may serve as a basis for competence building. However, in several laboratories such expertise is fading given the retirements of experienced personnel as well as the existence of single-points-of-failure in small staffed groups, and the lack of opportunities for familiarizing with developments in instrumentation and calculus. Thus, systems for quality assurance may have gaps with regard to potential sources of errors.

Quality control and quality assurance are therefore indispensable for ascertaining the quality of results. However, they do not provide objective evidence on the trustworthiness. This can be accomplished by accreditation of the management systems associated to quality assurance for compliance with international standards (such as the ISO/IEC:17025). It should be noted that this is not a mandatory act, and strongly depends on other strategic considerations, e.g., if laboratories get active in serving third parties with results of measurements. While these requirements provide a challenge to developed countries, for developing countries they can be an even greater challenge.

Around the turn of the century, the IAEA has started assisting Member States’ laboratories in implementing (quality) management systems, as a partner in development in guiding its Member States facilities towards self-sustainability and providing a stepping stone to accreditation.

Now, 10-15 years later after the start of these programmatic activities, time has come for evaluating the current status in implementation and use of quality assurance in nuclear spectroscopy techniques; as well as to assess/demonstrate the benefits and impacts of quality assurance on improvement of reliability of chemical measurement results in order to further extend the applicability range of the analytical techniques.

The participants selected Mr. Sheldon Landsberger, USA to chair the technical meeting.

2. Summary of individual presentations

The participants have given their individual presentations (see Appendix 3) and submitted full papers included in Appendix 4. Summary of the presentations is given in Table 1 below.

Table 1. Summary of the presentations.
<table>
<thead>
<tr>
<th>Country</th>
<th>Organization</th>
<th>QA/QC Activities</th>
<th>Accreditation Details</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>GHANA</td>
<td>School for Nuclear and Allied Sciences</td>
<td>Teaching QA/QC at the tertiary level of education. For sustainability of NAL it is important to inculcate QA/QC culture in young scientists during their formative periods.</td>
<td>Not accredited</td>
<td>Research activities and some analytical services in XRF and NAA Labs.</td>
</tr>
<tr>
<td>CUBA</td>
<td>Centre of Radiation Protection and Hygiene-Environ. Radiological Surveillance Lab. (LVRA)</td>
<td>Internal quality control and use of CRMs in gamma ray spectrometry. There is the need for harmonization of QA systems in terms of calculations (uncertainties, critical limits, reporting of results)</td>
<td>Accredited since 1997 (ISO 9000) and from 2006 according to ISO 17025 for determination of radionuclides, by gamma spectrometry, in food and environmental samples.</td>
<td>Determination of beta emitters by LS counting. Alpha-Beta counting (Gas proportional counters)</td>
</tr>
<tr>
<td>Country</td>
<td>Activity Details</td>
<td>Accreditation Details</td>
<td>Additional Notes</td>
<td></td>
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<tr>
<td>PORTUGAL, Instituto Tecnológico e Nuclear</td>
<td>Corrections for Geometry, Matrix and Coincidence Summing Effects in Gamma Spectrometry. In order to establish metrological traceability in high resolution gamma spectrometry, the above corrections must be implemented.</td>
<td>Preparations towards accreditation in gamma spectrometry of environmental samples in the energy range 46 – 2000 keV.</td>
<td>RER/0/031(2008-11)</td>
<td></td>
</tr>
<tr>
<td>TURKEY, Turkish Atomic Energy Authority</td>
<td>Experiences in QMS and implementation of QA/QC to alpha spectrometric measurements. Implementation resulted in better performance, and increased public confidence.</td>
<td>Accredited in May 4th 2009 by national accreditation body for - Tritium Analysis in water; - Radioactivity Analysis of Cs-137 and Cs-134 in Foodstuffs and Ra-226, Th-232, K-40 and Cs-137 in Soil And Construction Materials with Gamma Spectrometric Method in Food, Soil and</td>
<td>Thermo-luminescence dosimetry, ionizing radiation metrology, liquid scintillation counting and total alpha/beta with proportional counters</td>
<td></td>
</tr>
</tbody>
</table>

- Alpha Spectrometric Analyses of U-234, U-238 and Ra-226 in water
- Sr-90 analyses by LSC Spectrometer
Designated Institution for ionizing radiation metrology
<table>
<thead>
<tr>
<th>AUSTRIA, TU Wien, Atominstitut</th>
<th>Accreditation for wafer surface analysis with TXRF</th>
<th>Accreditation of University lab was established considering integration of teaching aspects.</th>
<th>TXRF for chemical analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Construction Materials;</td>
<td>- Gross Alpha/Beta Radioactivity in drinking water; Hp10 and Hp0.07 with Thermo Luminescence Dosimeter (TLD);</td>
<td>- Determination of Hp(10) and Hp (0.07) with film dosimeter; - Element (Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe, P, Sc, V, Cr, Co, Ni, Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Ba, Pb, La, Th and U) Analysis by WDXRF Spectrometry in Geological Samples Such as Soil, Sediments, Rock and Clay Samples; - Establishing the Radiation Sterilization Dose; - Determination of the Population of Microorganisms on Medical Product; - Sterility Control (for medical purposes) in medical products; - DNA Comet Assay for the Detection Foodstuffs- Screening Method; - The Detection of Irradiated Foods Containing Cellulose By ESR Spectroscopy Awaiting accreditation by national authorities for wafer surface analysis., having passed the audit tests,</td>
<td></td>
</tr>
</tbody>
</table>
| ARGENTINA, CNEA, NAT Group (NAA) | Uncertainty evaluation in RNAA of Se in biological samples | Accredited since 2001 by national accreditation body in:  
- InAA for Sm, Sc, Fe, Co, La, Th, Ce, Eu, Cs determination in geological and related matrices  
- RNAA for Se determination in biological samples | Accredited in:  
- INAA for Sm, Sc, Fe, Co, La, Th, Ce, Eu, Cs determination in geological and related matrices  
- RNAA for Se determination in biological samples  
- INT/1/054: PT organization and evaluation and RM preparation (2002-4)  
- RLA 2/014 (ARCAL): QA/QC in Analytical laboratories,  
- PT organization and evaluation and RM preparation (2009-2011) ARCAL IV | Dosimetry laboratory (CRRD):  
- Metrology Laboratory (LMR)  
- Uranium laboratory (LADCU)  
- Laboratory for nuclear fuel cycle, certified for ISO 9001 |
| ARGENTINA, Nuclear Regulatory Authority | QA in gamma and liquid scintillation spectrometry | Accredited in:  
- Tritium (2008)  
Uranium by Fluorimetric method and Kinetic Phosphorescence Analysis (2007) | Participatio n in PT:  
Cito-genetic Dosimetry, Calibration of field and source detectors  
Environmental laboratory for 90Sr and 226Ra surveillance  
Laboratory of 3H measurement in food products |
<table>
<thead>
<tr>
<th>INDONESIA, Centre of Nuclear Technology for Materials and Radiometry - BATAN</th>
<th>Accreditation in analytical laboratory: Neutron Activation Analysis laboratory</th>
<th>Sampling and Sample Pretreatment Laboratory (2006) Accredited by Technical and Professional Staff of the Nuclear Regulatory Authority</th>
</tr>
</thead>
<tbody>
<tr>
<td>INCREDIBLE, Atomic Energy Authority.</td>
<td>Accreditation of Nuclear Analytical Laboratories: Gamma Spectrometry and XRF Laboratories</td>
<td>Accredited by national body since 2006 in: gamma spectrometry for particulate matter samples (Ti, I, V, Br, Mn, Na, K, Cl, Cu, Al, Ca, As, Fe, Co, Zn, Ag, La, Cr, Sm) using NAA technique.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Participati on in PT IAEA NAT-7 on airborne particulate samples</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Trace elements in coal and coal fly ash samples, lichen, soil, sediments and sludge, geological rock samples, f uranium and thorium in zircon sands, and health-related samples such as hair and blood, analysis of food sample to determine the Daily Diet Intake (DDI)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>U analysis by gamma spectrometry Trace element analysis by TXRF</td>
</tr>
<tr>
<td>Country</td>
<td>Institution</td>
<td>Experience/Implementation</td>
</tr>
<tr>
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</tr>
<tr>
<td>TURKEY, Gazi University,</td>
<td>Physics Department</td>
<td>Experience on QA/QC of NATs. Establishment of quality assurance system for XRF, gamma and alpha/beta labs</td>
</tr>
<tr>
<td>MOROCCO, National Centre for</td>
<td>Energy.</td>
<td>Implementation of QM in nuclear spectrometry</td>
</tr>
<tr>
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<td></td>
<td></td>
</tr>
<tr>
<td>Sciences and Nuclear Techniques</td>
<td>INDIA, Board of Radiation and Isotope Technology</td>
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<tr>
<td>laboratories.</td>
<td>Measurement of Cs-137 in Food &amp; Other samples by Gamma Spectrometry. The QA/QC practices in the measurement of Cs-137 in different samples were presented. The Indian experience in controlling the disposal of radioactive waste from Nuclear Power Plants was also discussed.</td>
<td></td>
</tr>
<tr>
<td>The application of QM principles improves the quality of measurements, and is cost-effective even for small volume of work labs.</td>
<td>Accredited since 2007, by national body: measurement of radioactivity levels in food samples, and for issuance of Radioactivity Test Certificates.</td>
<td></td>
</tr>
<tr>
<td>Spectrometry in water and soil - Gross alpha/beta counting in water samples</td>
<td>PT: IAEA-CU-2007-12 IAEA-CU-2008-03 IAEA-CU-2009-03</td>
<td></td>
</tr>
<tr>
<td>Not applicable</td>
<td>(Inductive Coupled Plasma ICP-AES and High Performance Liquid Chromatography - Tritium analysis by Liquid Scintillation)</td>
<td></td>
</tr>
<tr>
<td>5 accredited laboratories, (4 testing, 1 calibration Labs). The accreditation granted by national body in 2003. scope of accreditation found at: <a href="http://www.sa.gov.si/teksti-1/doc/test/LP022.pdf">http://www.sa.gov.si/teksti-1/doc/test/LP022.pdf</a> <a href="http://www.sa.gov.si/teksti-1/doc/cal/LK017.pdf">http://www.sa.gov.si/teksti-1/doc/cal/LK017.pdf</a></td>
<td>QA/QC is implemented for the work of all methods that are conducted by the laboratories</td>
<td></td>
</tr>
<tr>
<td>QA/QC is implemented for the work of all methods that are conducted by the laboratories</td>
<td>Gamma spectrometry is being effectively employed in the measurement of radioactivity in food and other samples and radioactivity test certificates were issued which is mandatory for export-import trade practice. Steel samples were analyzed for Co-60 and certificates were issued.</td>
<td></td>
</tr>
<tr>
<td>Implementation of QA and quality audits. Some experiences on statistical analysis of nonconforming work, results of inter-comparison runs, warnings on basis of previous results of the same kind of samples on same locations were presented as well. The importance of audits (internal and external) was also addressed.</td>
<td>Member of EURAMET Designated [by Metrological Institute of Republic of Slovenia (MIRS)] Institute (DI) in the field of ionizing radiation.</td>
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</tr>
<tr>
<td>UKRAINE, Chernobyl Radio-ecological Center</td>
<td>The detection efficiency and energy calibration of low-level gamma spectrometry system. The efficiency and energy calibration were carried out using RGU-1, RGTh-1, RGK-1, RMs. The results are traceable to RMs</td>
<td>Measurements of $^{137}$Cs, $^{90}$Sr, $^{238}$Pu, $^{239/240}$Pu, $^{241}$Am in air surface and ground water, wildlife, soils and precipitation.</td>
</tr>
<tr>
<td>IRAN, Iranian Nuclear Regulatory Authority, National Protection Dept</td>
<td>Preparation for accreditation of INRA Laboratories for determination of radionuclides (3H, $^{235/238}$U, $^{226/228}$Ra, $^{210}$Po, $^{222}$Rn, $^{137}$Cs, $^{90}$Sr, $^{238/239/240}$Pu, $^{241}$Am) in food products, plants, soils, water, air filters and building materials by gamma spectrometry, LSC and total alfa/beta measurements Sampling and sample preparation laboratory</td>
<td></td>
</tr>
<tr>
<td>KENYA, Radiation Protection Board, Min. of</td>
<td>Radiation Protection Board is the Regulatory Authority;</td>
<td>Not accredited</td>
</tr>
<tr>
<td>Roads, University of Nairobi, Kenya</td>
<td>University of Nairobi leads teaching on QA/QC at the tertiary level of education; while Kenya Bureau of Standards is the custodian of standards and accreditation formalities.</td>
<td>Accredited since 1993 by national body for: - determination of elements Eu In, Ir, Au, Sc, Co, Cs, Mn, Sm, Hf, Dy, V, Ta, La, Tb, Rh, Re, Sb, Lu, As, Ga, Trm, Ho, Ag, Na, U, W, Cu, Br, Th, Yb, Se, Nb, I, Al, Pd, Er, Hg, Te, Ru, Os, Pr, Gd, Zn, Ce, Cl, Cr, Sr, Pt, Cd, Mo, Rb, F, Ba, Nd, K Ge, Ti, Sn, Mg, Zr, Ni, Ca, Fe, Y, Si, S in solids and liquids</td>
</tr>
<tr>
<td>NETHERLANDS, Delft University of Technology</td>
<td>For sustainability of NAL it is important to inculcate QA/QC culture in all their operations. Importance of technical competence in QA/QC. It is important to master the physical/chemical fundamentals of the technique, methodology and instrumentation; We need to understand potential sources of error and how to monitor them</td>
<td>ARCAL IV, ARCAL XXVI, RAF 4/012, RAF 2.008, RER 2.004, RAS 2.010, RAW 2/005, RUS 7003/01….and many, many more</td>
</tr>
<tr>
<td>UNITED STATES, University of Texas at Austin</td>
<td>Importance of non-conformance management Teaching of QA/QC in nuclear analytical techniques in undergraduate and graduate research. It is important to teach the culture of total quality</td>
<td>Not accredited</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Preparation of air filters for inter-comparison studies, hosting of numerous IAEA fellows</td>
</tr>
</tbody>
</table>

the country, radiation protection activities in radiological facilities is done by the RPB.

Research activities in Gamma and Beta Spectrometry, EDXRF and NAA Laboratories in the institutions mentioned.

- Reactor Operations
- Design and construction office
- Radiation Detection Department
- Project Management Bureau

Research activities and analytical services in NAA Labs.
3. Major ingredients of QA

Major ingredients of laboratory management focus on quality and complying with the requirements of the international standard ISO/IEC 17025. These areas are:
- Organization
- Technical competence of personnel and training
- Documentation and document control
- Equipment management, performance tests and calibration
- Method validation, including estimation and calculation of the uncertainty of measurement, equipment and traceability of the values of the measurement results
- Quality control /assuring quality of test and calibration results
- Continuous improvement by registration and analysis of nonconformities (nonconformities of the work process and of the outcome of results, remedial, corrective and preventive actions, complaint management)

Organization, personnel and training

Organizational structure (legal identity, responsibilities and structure of the (quality) management system) has to be well defined for sake of transparency. Appropriate key personnel should be identified at all levels (executive management, technical management, and quality managers) including laboratory personnel as well as their deputies. Good communication processes within the laboratory and between laboratory and executive management are important and it must be clear how this is accomplished. Commitment of executive management to the quality system implemented may serve as a stimulating example for all laboratory personnel. Commitment of personnel to contributes to the effectiveness and desired quality of the work, and requires continuous attention to sensitization and motivation of personnel. The technical competence of personnel is a vital component, and requires regular training, associated qualification on demonstrated performance, as well as regular re-qualification of personnel.
The requirements to the technical competence make necessary to expand the knowledge on:
- Physico-chemical principles of the method and equipment used
- Potential sources of error
- Method validation
- Uncertainty determination
- Correct use of (certified) reference materials, participation in intercomparison laboratory exercises and associated interpretation of the results
- Sampling and sample preparation
- Documentation and use of software.

An increase in technical knowledge also contributes to perform better internal audits on the technical aspects of the work processes.

Finally, it is important to train the trainers – to expand the knowledge – in order to avoid single-point-of-failure in the organization. This may sometimes be accomplished via regional projects.

**Documentation and document control**

Documentation (written policies, work instructions, registration of experimental conditions and observations from tests and by personnel) is important for clarifying the work process, for archiving the expertise and for reproducing the manner of work. The basic principle is “Write down what you agreed to do, do as it has been documented and if you have to differ, register a non-conformance and if necessary, update the procedure.” So, work instructions prescribe what to do and how to do it.

The laboratory itself decides on the extent of the documentation depending on the impact of the absence thereof. The documentation also contains records that provide objective evidence of the results of the test and how the processes and procedures have been done in the practice. Harmonization of procedures is often necessary for transparency and to limit the number of errors and at least these points should be addressed:
- Define the processes in the laboratory
- Avoid duplicate information within the quality system
- Consider computerized systems (like a LIMS – Laboratory Information Management System) for management of documentation
- Hardcopies and electronic copies should be properly balanced
- Do not prepare your procedures more rigorous as required by the standard (unless it is necessary because of other demands)

**Method validation**

Method validation is a basic requirement to demonstrate that the methodology is fit for the intended purpose, i.e. to serve the requirements of the client. However, in spite of large amounts of guidance documents, it is often not well understood or practiced.

If the method used is standard one (i.e., like an (inter)national norm), only verification is needed; the laboratory developed methods have to be validated. If a standard method is modified, only the modified part shall be validated.
Before starting the method validation process, the working plan should be prepared in detail:
- Define the fitness for intended purpose
- Verification of performance of norms
- Assess if there is already information in the laboratory’s archives for (some of the) performance indicators to be determined
- Do additional experiments
- Sometimes it may be needed also to validate self-made software
- Keep in mind that method validation is a balance between costs, risks and technical possibilities.

Quality control

Verification of the quality (e.g. required trueness and precision of the final results) implies the laboratory has to implement a quality control program. It must be a planned action with predefined performance indicators.

There are several ways of doing so:
- Measurements/analysis of objects of known composition (such as (certified) reference materials
- Participation in laboratory in intercomparison exercises, proficiency testing schemes or by (e.g.) bi-lateral exchange of samples
- Internal comparisons using replicates or by repeated testing.

Results have to be evaluated, discussed and final conclusions have to be stated. Processing the results of the critical parameters of the measurements via graphical control charts allows for inspection of trends. The records about quality control shall be extensive enough to reproduce the experimental conditions and have to be archived of course (including records about equipment settings, calibration etc.; see above).

Analysis of nonconforming work

Registration of non-conformities, root cause analysis, remedial and corrective actions all result in a systematic elimination of sources or error and in a continuous improvement. Systematic analysis of nonconforming work can point out weak areas of quality system and give information for improvement of working processes. Non-conformities may be identified as e.g.
- Complaints (external, internal)
- Deviations from planned work processes
- Results outside pre-defined criteria
- Nonconformities observed in (internal, external) audits
- Conclusions from the management review.

4. Establishment of QA/QC in analytical laboratories
Implementation of systematic quality assurance and quality control should be considered as a project, characterized by objectives, expected outputs, measurable performance indicators and well-defined starting and conclusion dates. Laboratories are recommended to operate such a project along the principles of project management, involving subsequently an orientation, definition, implementation and assessment phase. Some practical hints for establishing QA/QC are summarized below.

**Orientation phase.**

The following issues should be considered. The laboratory informs itself why the QA/QC is needed. Are there already problems and if so, where? For what is the QA/QC system needed: for all activities in the laboratory, or just for one technique? Is it for routine or for R&D activities? Will it also apply to, e.g., educational experiments by students? Is the ultimate goal to go for accreditation?

It is recommended to organize –if possible- a visit to a laboratory, institution or company with an operational/accredited QA system. This will help to orientate on the difference between the current situation and the system to be reached, how much time it will cost, as well as the pitfalls that can be expected.

In this phase, the laboratory also should assess where they are right now: what is already in place. And finally, it is important to discuss when the project should be completed.

The outcome of this orientation must be discussed at all levels, and result in a go/no-go decision with commitment of executive management.

**Definition**

In this phase, the laboratory refines its orientation and set the boundary conditions. Again, the why question must further be defined serving sensitization and contributing to the motivation of all involved. Commitment of executive management is needed to be communicated to all layers of the organization. A gap analysis has to be done to define precisely the current situation; here the IAEA may assist by an expert mission. The scope of the QA/QC system has to be defined (for which laboratory, which technique, type of samples, matrix, radionuclides, degree of trueness required), type of customers, and scope of the activities: only internal quality control or also external quality control.

Planning is a key element in the definition phase. Planning is based on setting priorities and involves a scheme with an answer to the question “Who is going to do what and how, how will it be assessed and when will it be ready?” It is also important at this point to define responsibilities. The answer “How it will be assessed?” implies the definition of a mechanism for monitoring the progress of implementation, and also how often a review meeting will be held, eventually with executive management.

All in the above result in the definition of the resources needed: time (man-hours), hardware including utensils and possibly also software. Time (and costs) for training and assistance by external (IAEA) experts must also be included.

The definition phase is concluded with communication of the implementation plan and the go/no-go acceptance thereof.

**Implementation**

The implementation plan is now brought into the practice.

It is generally observed that motivation will drop at the start of implementation because people may loose overview on what is required, get worried by paperwork to be completed,
fear for bureaucracy, fear for loosing identity by far-going harmonization and limitation of freedom in making decisions how work has to be done. In this phase, management should concentrate on shared values and stimulate bottom-up ownership of the QA/QC system. Teambuilding and frequent group meetings may help. It will be important to demonstrate benefits by keeping records how many things went wrong in the past and how the situation is improving. Don’t try to speed up motivation and don’t sell slogans as people don’t believe them. The laboratory manager should set the example as a leader by showing commitment. Eventually, it may be unavoidable to accept people’s nature and to design the QA around it. At large, it is recommended to implement non-conformance management at the start of implementation, so as to keep records of things going wrong, potential causes thereof and bottom-up suggestions for improvement.

Study of relevant literature and norms is important in finding approaches for improvement and QA/QC measures that can be applied. The IAEA issued an extensive document (TCS24) providing examples and guidelines for development of a quality assurance/quality management system. There are other TECDOCs as well to be consulted. Some keywords of possible QA/QC actions for specific deficiencies, derived from TCS24, are summarized below:

- **Insufficient or outdated technical competence**: get training

  *Incomplete documentation*: make forms, registers, logbooks, SOPs

  *Environmental conditions*: register temperature, humidity, background, cross contamination; decide for either centralized information control or personal archives.

  *Equipment*: implement systematic performance checks, set criteria, specify spare parts, set-up preventive maintenance, collect original manuals

  *Supply management*: establish specifications of critical supplies

  *Method validation*: study literature, identify ‘intended purpose’: ‘good’ is good enough; find what is already available: results of ILCs, PTs, reference material analysis

  *Calibration*: check your certificates for information on traceability and uncertainty; consult IAEA if not sure; if necessary: get balances calibrated, buy calibrated masses, get new sources but all with proper certificates (same for chemicals).

  *Uncertainty/traceability*: study IAEA TEDCOC 1401; be pragmatic: bottom up (GUM) helps you to identify what is important and what is not, but it takes time; top down (reproducibility) is acceptable too, but you don’t know what causes it.

  *Samples*: establish an unambiguous coding system

  *Organization*: make a good system for review of requests for analysis (who wants what, how good, how many samples, when ready, willing to pay, need for confirmation/quotation, contract, name of contact person etc.)

  *Harmonization of operations?* Discuss and be pragmatic. Ensure a bottom-up development and ownership. This usually may costs quite some time.

  *Housekeeping*: Clean up the lab and offices. Throw away all that is redundant and not working anymore

  *Poor or unacceptable analytical results*: Establish quality control either internal quality control by control samples (such as reference materials), blanks, replicates, control charts, statistical analysis; set criteria for acceptance; and by external quality control such as interlaboratory exercises, proficiency testing, exchange of samples with other labs
Monitor the implementation by recording regularly how many planned QA/QC actions have been completed and give evidence thereof in, e.g., histograms in monthly reports to all involved.

Assessment

At the end of the projected implementation period, the laboratory must assess its success by, e.g.,
- Comparing the types and number of non-conformities at the beginning and at the end of the project
- Measure the time and costs needed for QA/QC
- Gap analysis.

A management review is held, in which also a decision is taken for additional corrective actions, and if a follow-up project is needed, e.g., for extension of the QA/QC system or further improvement.

5. Benefits and impacts of QA on improvement of reliability of chemical measurements

Problem Identification, Assessment and Correction

The implementation of a total quality management (TQM) system which includes QA and QC, can lead to the improvement of methods and techniques and identification of large and small problems. Eventually, this enables the laboratory to implement preventive and corrective actions.

Demonstration of Quality

Increased efficiency and effectiveness, a lower rate of susceptible measurements and higher grade of transparency of procedures will increase the confidence and reliability of the results. This will lead to enhanced productivity and improved reputation of the laboratory.

Improved level of Customer Analytical Services

The reputation of any laboratory clearly relies on both the quality of the results as well as the agreed delivery time of analytical measurements. Once established it can bring an important increase in the number of customers resulting in extended research and financial gains.

Accreditation of Laboratory

The accreditation of a laboratory has impacts beyond improved QA and QC procedures. In particular, a reputable accredited laboratory can provide its services to a wide range of regulatory institutions which may have significant impact in the social and economic domains. Accreditation contributes to the sustainability of the quality assurance system as well.

Training and Teaching
The establishment of an accredited laboratory allows the effective teaching and training of new staff and students. Most important is the establishment of a culture to follow rigorous QA and QC procedures. It also helps in preservation of knowledge.

6. Problems in implementation of QA

Based on the experience of the participants the following major problems were identified in implementation of QA in analytical laboratories:

- Lack of adequate documentation
- Over-dependence on a few experienced scientists/technicians
- Lack of resources
- Demonstrating traceability of mathematically based calibration (Monte Carlo based methods)
- Inadequate Proficiency Test schemes (selection of PT samples, follow-up actions to improve the methods, implementation of corrective actions, duration of PT exercise)
- Implementation of proper terminology (e.g., definition of calibrant, traceability, detection limits)
- QA practices for in situ (field) measurements
- Establishment of metrological traceability
- Lack of technical auditors in some countries (e.g., Portugal, Poland, Slovenia) for selected techniques (e.g., LSC, NAA)

7. Importance of certification and accreditation

The participants identified the following benefits of certification and accreditation:

- Motivation to improve quality
- Increased number of customers
- Improved credibility and trust
- Increased discipline to follow the written rules and procedures
- Provision of strong arguments to negotiate with management.

8. Role of the IAEA in promotion and establishing QA in nuclear spectrometry laboratories in Member States

- Promotion of development and applications of NATs
- Strengthening support for implementation of QMS
- Provision of training in the field of specific aspects of quality management for NATs
- Providing technical expertise and support in metrological competence
- Organizing PT with samples with traceable metrological property values
- Expand the availability of reference materials characterized for radionuclides in different matrices.
- Provide training for auditors, and trainers. Assist in the implementation of ToT activities.
• Develop recommended methods for radioactivity measurements
• Organize technical meetings, workshops and seminars for member states in QA/QC issues.
• Promote coordination between laboratories in member states
• Increase involvement in the development and standardization of new international accepted terminologies (for example new VIM)
• Create a network of qualified expert auditors in radioactivity measurements to assist accreditation bodies of member states.
• Organize a meeting for representatives of accreditation boards in member states to better understand the nuclear analytical techniques and quality components (technical requirements, uncertainty…)
• Facilitate improvement of metrological traceability of nuclear data (e.g., half life of short-lived radionuclides) for evaluation of nuclear-related analytical results.
• Reinforce support for TC projects dedicated to QA/QC
• Monitor and disseminate new trends in calculations relevant to applications of NAT
• Production of QA/QC documentation for in-situ applications
• Strengthen activities in the provision of appropriate calibrators for QA/QC purposes.

9. Conclusions

The objectives of this Technical Meeting were:

- To review the current status in implementation and use of Quality Assurance in the nuclear spectrometry techniques.
- To assess/demonstrate the benefits and impacts of QA on improvement of reliability of chemical measurement results in order to further extend the applicability range of the analytical techniques.

Overall
The participants concluded that these objectives were accomplished by the wealth of information exchanged in presentations and discussions.

Networking
In several laboratories technical expertise is fading. The participants concluded that there is a need for exchanging technical know-how. The contacts established at this technical meeting may serve as a stepping stone for building networks between laboratories.

Accreditation
Accreditation of management systems is never a mandatory issue, but quality assurance (including quality control) is indispensable, irrespective if tests are done for internal use or for serving third parties. Accreditation for compliance with the ISO/IEC17025 may become a
pre-requisite for nuclear spectroscopy laboratories if the laboratories provide test results for e.g. environmental monitoring and global trade.

IAEA is playing an important role in stimulating Member States’ laboratories for nuclear spectroscopy laboratories in implementing QA/QC, and reaching formal accreditation.

The participants observed the need for sensitization of their executive management to ensure commitment (top-down) and resources for the implementation of QA/QC practices and the final step towards accreditation.

Quality assurance and quality control
It was concluded that the level of and approaches in QA/QC implementation varies from country to country. There is, to some extent, a need to consider harmonized guidelines for QA/QC practices as examples to laboratories for supporting the process leading to accreditation. Education in the principles and practices of QA/QC was identified as one of the important issues for successful implementation and sustainability.

Metrology
The participants identified the need for assuring the sustainability of metrological competence in MS nuclear spectroscopy laboratories, given the retirements of experienced personnel as well as the existence of single-points-of-failure in small staffed groups, the developments in instrumentation and calculus and the increasing requirements (and importance) to the quality of measurement data.

The participants observed and reported new metrological approaches for nuclear analytical techniques, including calibration of equipments, quantification of associated uncertainties, detection limits, etc. They recognized the need for provision of adequate materials for PT in order to meet the newest metrological requirements.

Gaps
Participants identified that in-situ measurements have not yet reached a satisfactory level of implementation of QA/QC practices. Field measurements may need to get adequate attention with respect to QA/QC and analytical quality (trueness, uncertainty, representativeness of the measured object) for the interpretation of the results in support of decisions.

10. Recommendations to the IAEA

General
To organize in 2010 a consultancy meeting in order to prepare the terms of reference of a new CRP on (some of) the above mentioned topics.

Networking (further expanded below under specific actions)
To create a database of nuclear analytical laboratories to provide information on accredited laboratories, current status of nuclear spectrometry, etc. This database can be a platform to create a network in order to motivate collaboration and cooperation between MS.

Accreditation
To continue the development and revision of guidelines and TECDOCs concerning QA/QC as well as the metrology of NATs to foster laboratory accreditation.
**Quality Assurance and quality control**
To assist MS laboratories by harmonized guidelines for QA/QC practices in nuclear spectroscopy laboratories so as to facilitate comparability and internal audits.

To continue organizing workshops and technical meetings on QA/QC in specialized fields of nuclear spectroscopy in order to monitor the progress of implementation, to share experiences and to suggest anticipation on innovation and recent developments in QA/QC.

**Metrology (further expanded below under specific actions)**
To strengthen support of nuclear analytical techniques to improve and sustain metrological competence.

To support implementation in MS institutions a mechanism to assure and strengthen the metrological competence in methodologies for determining alpha, beta and gamma-ray emitting radionuclides. This metrological competence should be in compliance with newest terminology (as defined by BIPM/IUPAC/ISO). The mechanism should also strengthen the collaborations between research and university laboratories, as well as laboratories involved in the Agency’s ALMERA network with national metrology institutions for measurement of radiation.

**Gaps**
To direct attention for QA/QC and metrology of (semi-quantitative) in-situ measurements of radionuclides and hazardous elements.

At large, the IAEA is recommended to continue the organization of coordinated research projects on subjects related to quality and metrology of nuclear spectroscopy, to organize proficiency testing schemes for nuclear spectroscopy as well as to enable Member States in submission of requests for TC projects (with assistance in acquisition of calibrators or equipment) and fellowship training.

**Specific actions**

*a. Networking*

To create a network of nuclear spectroscopy laboratories. Some suggestions for objectives, organization, activities and expected output are:

**Objectives**

# To serve as a tool to foster and expand knowledge, experience and competence in nuclear spectroscopy. Experts have been retiring, and new generations are not very much seduced by this field of science. The network could be a means of keeping this community “alive and kicking”.
# To support less developed countries in their efforts to improve the performance of methods and the quality of results.
# To encourage/promote/facilitate collaboration and cooperation between laboratories for solving commonplace technical problems and for endeavoring appealing new challenges.
# To easily provide training/education for laboratories in need. The Network would avoid the more time consuming process of the technical and regional cooperation projects.
# To assist Member States in the practices of quality system implementation in nuclear spectroscopy laboratories.
# To ease communication between laboratories.
# To promote guidelines for harmonization of QA/QC practices.

**Organization of the network**

The Network could be organized in Network Members/Partners and Network Participants, similar to already existing networks within the IAEA such as the Underground Research Facilities Network (URF), the Environmental Management and Remediation Network (EnviroNet) or the International Low Level Waste Disposal Network (Disponet).

**Scope of activities**

# Technical issues: detector calibration, troubleshooting, spectroscopy software, coincidence summing corrections, self-absorption corrections, statistical evaluation of data, etc.
# Modern methodologies such as Bayesian methods, Monte Carlo simulation, new detectors, new applications (e.g., quantification of actinide activities using HPGe detectors).
# Structural issues: uncertainty evaluation, detection capabilities, method validation, metrological traceability.
# Information exchange can be facilitated by a newsletter

**Expected outcome**

# Resource optimization (money, manpower)
# Sustainability of competence in nuclear spectroscopy
# Problems are solved in a time-effective manner
# Improvement of the quality of measurements in the nuclear field
# Less dependency of Member States on IAEA assistance, since the required assistance would be provided by Members of the Network.

**b. Metrology**

Specific activities to be considered are:
- Organization of specialized regional training courses for the youngest generation of staff at nuclear spectroscopy laboratories in techniques and instrumentation for alpha, beta, gamma-ray spectrometry including total alpha/beta measurements; as well as X-ray fluorescence spectrometry
- Development of self-learning tools that also serve academic education
- An update of IAEA TECDOC 1401 and IAEA TCS 24 with respect to the examples and templates provided and with guidelines for harmonized approaches
- A sustainable information mechanism on the requirements to the content of calibration certificates on statements of traceability of property values and uncertainty of measurement
- The implementation of a higher level of laboratory intercomparison exercises and proficiency tests, including comparison of software (Monte Carlo codes)
- The development of reference materials with SI traceable property values
- The development of adequate procedures for efficiency calibration, volumetric efficiency, matrix correction (gamma-ray self attenuation) and coincidence effect corrections.