Improved Performance of Neutron Activation Analysis Laboratories by Feedback workshops following Interlaboratory Comparison Rounds

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Abstract. The IAEA implemented an innovative project from 2010-2015 for assisting neutron activation analysis laboratories in improving the validity of their results by feedback workshops for discussion of results from participation in interlaboratory comparisons rounds. The participants learned during these meetings to identify the most probable sources of errors in their analysis procedures and how to implement corrective actions to prevent reoccurrence. The evaluation of the results clearly shows an improvement in the fraction of results considered as 'metrologically excellent' under the conditions applied. Moreover, there is a clear indication that improvements are consolidated in most laboratories but also stimulate laboratories to develop to a higher level of excellence.

Key Words: Neutron Activation Analysis, Interlaboratory Comparison, Feedback, Quality Improvement

1. Introduction

Over the years, the IAEA has stimulated the orientation of neutron activation analysis (NAA) laboratories worldwide on fields of application in which a large number of samples may exist for analysis. Whereas the markets for service by NAA laboratories may have been identified, demonstration of valid analytical data and organizational quality of the work process are preconditions for consolidating and expanding the stakeholder community. Eventually, laboratories and/or stakeholders may prefer that the facility's management system is accredited for compliance with the International Standard ISO/IEC17025:2005.

One of the requirements in the process towards such accreditation is that the laboratory provides evidence of the validity of its measurement results by participation in proficiency testing schemes by interlaboratory comparison. Participation in interlaboratory comparison study may reveal that some results are not satisfactory. Laboratories are then facing the problem of finding the source of such non-conformity and applying corrective actions. Obviously, providers of intercomparison rounds cannot provide such a laboratory and technique-specific after-care.

The IAEA has therefore implemented a new mechanism for supporting the NAA laboratories in its Member States in demonstrating their analytical performance by assisting them in identifying unanticipated sources of errors, to assess with them approaches for elimination thereof and to design with them a path for growing towards sustainable performance at the analytical state of the practice. This was accomplished by an evaluation and feedback meeting following the participation in proficiency testing by interlaboratory comparison.

Laboratories under the IAEA Technical Cooperation (TC) projects RAF4022, RAS1018, RER4032/RER1007 and RLA0037 participated, facilitated by the IAEA, between 2010 and 2013 in consecutive proficiency testing schemes by interlaboratory comparison of the Wageningen Evaluating Programs for Analytical Laboratories (WEPAL) to assess their analytical performances. In the year 2015, laboratories under TC projects RAF1005, RAS1019, RER1007 and RB project 1.4.2.1 participated again in these interlaboratory comparison schemes. The results have been analysed by IAEA international experts providing

first indications for potential sources of error, and further discussed by experts and participants in feedback workshops.

2. Method of implementation

WEPAL is a world-leading organiser of proficiency testing schemes in the fields of plants, soil, sediments and organic waste. WEPAL is organising this for over 50 years and currently has over 500 participants in these schemes from countries all over the world. The provider is accredited by the Dutch Council for Accreditation for compliance with the International Standard ISO17043:2010. A few of the attractive features of this provider are:

- WEPAL has a proven record of issuing the evaluation report 3 weeks after the deadline for reporting.
- Participants identify in their reports also the technique and method used. WEPAL groups the results by these identifiers. It allows for, e.g. differentiation between 'real total' amounts (e.g., resulting from NAA or X ray fluorescence spectrometry) and amounts from techniques requiring dissolution of the sample.
- The number of participants in the round on soil and plant matrices is large, typically up to one-hundred or more. This contributes to the degree of trueness of the robust median value of the results.
- In each round four samples of a specific type (e.g., soil, plant, etc.) are distributed. One sample in each round has been "blindly" distributed in previous rounds. This allows for comparison of stability and/or effectiveness of corrective actions

The IAEA facilitated participation in the WEPAL's international soil exchange (ISE) programme and in the international plant exchange (IPE) programme. Soil is considered to be an 'easy' material for neutron activation analysis, whereas analysis of plant matrices may be more difficult given the much lower induced activities and risk of contamination. An overview of the rounds implemented and the participation is given in Table 1.

TABLE 1 DATA ON WEPAL ROUNDS SUPPORTED BY THE IAEA

WEPAL round	Implemented in IAEA project	Sample dispatch by WEPAL	Laboratory reporting deadline date	Availability of WEPAL report
2010-3	RAF 4022	June 8, 2010	September 30, 2010	October 18, 2010
2010-4	RAF 4022	August 31, 2010	December 31, 2010	January 18, 2011
2011-4	RAF 4022, RLA 0037 RER 4032, RAS 1018	November 13, 2011	December 31, 2011	January 3, 2012
2012-1	RAF 4022, RLA 0037 RER 4032 , RAS 1018	January 12, 2012	March 31,2012	April 4, 2012
2013-1	RAF 4022, RLA 0037 RER 1007, RAS 1018	January 11, 2013	March 31, 2013	April 7, 2013
2015-1	RAF 1005, RAS 1019 RER 1007, RB project 1.4.2.1	December 1, 2014	March 31, 2015	April 9, 2015
2015-2	RAF 1005, RAS 1019 RER 1007, RB project 1.4.2.1	March 1, 2015	June 30, 2015	July 6, 2015

3. Participants

Both laboratories with highly experienced NAA practioners and laboratories with newcomers in the technique participated in all regional groups. In various rounds up to 36 NAA laboratories per round from all continents were participating in this initiative. There were differences in types of reactors (varying from subcritical facilities, miniature reactors to medium-size reactors) as well as different gamma-ray spectrometers and level of automation in spectrum analysis and data processing.

4. Data evaluation and Feedback workshops

WEPAL provides in these quarterly reports an overview of the results of each analyte grouped by the digestion/extraction technique as well as by 'real total' analysis. The digestion/extraction procedures and methods of detection are also indicated. WEPAL however distinguishes fewer categories of methodologies in the IPE programme than in the ISE programme. Most results of the laboratories participating in the IPE rounds were grouped in the category 'Inorganic chemical composition'.

A (normal distribution assuming) mean and standard deviation, the median and MAD as well as a z-score[1] is calculated for the mass fraction of each chemical element reported; this z-score is provided on basis of the normal distribution approximation. The participants do not report their own measurement uncertainty, so the value thereof is not accounted for by the provider.

In addition, the following criteria for evaluation, agreed by the participants, were applied in this IAEA project of proficiency testing by interlaboratory comparison:

1. A satisfactory result is defined by |z|-score ≤ 3

2. For each laboratory, the percentage fraction of the number of reported data in each round (4 samples) with $| z | \leq 3$ is determined.

3. The performance is considered
'Excellent' if this percentage is > 90 %
'Average' if the percentage if between 70 % and 90 %
'Poor' if the percentage is < 70 %

These indicators also allow for monitoring the development of the improvement of a laboratory in successive proficiency testing rounds. All performance indicators are for sake of comparison only and are not based on international conventions.

5. Feedback workshops

The WEPAL results were firstly evaluated by an IAEA international expert. The percentage of data with $|z| \leq 3$ was established and the results were inspected for gross errors and possible systematic errors that could indicate a source of error.

[1] z-score = (lab value – median value) / (standard deviation of all observations)

The feedback workshops were held rather as short as possible (with the exception for the first one) after completion of the last round in the respective periods, as can be seen from the overview in Table 2. The 2012-1 samples were re-analysed by most of the participants following the lessons learned during the related feedback workshop in Tunis. The re-analysis (denoted as 2012-1 (R) was also evaluated in the workshop in Vienna. Since these property values of the samples were known to the participants, the re-analysis is not of equivalent value to the regular intercomparisons rounds in which the participants were handling 'blind' samples.

TABLE 2 FEEDBACK WORKSHOPS HELD

IAEA project	WEPAL rounds	All results available	Date	Location
RAF 4022	2010-3 and 2010-4	January 2011	September 12-16, 2011	Antananarivo, Madagascar
RER 4032 RAS 1018	2011-4 and 2012-1	April 2012	May 22-25, 2012	Delft, The Netherlands
RAF 4022	2011-4 and 2012-1	April 2012	June 4-8, 2012	Tunis, Tunisia
RAF 4022 RER 1007 RAS 1018 RLA 0037	2012-1 (R) and 2013-1	April 2013	May 27-31, 2013	Vienna, Austria
RAF 1005 RAS 1019 RER 1007 RB project 1.4.2.1	2015-1 and 2015-2	July 2015	August 31-September 4, 2015	Delft, The Netherlands

The laboratories participating in the interlaboratory comparison rounds were encouraged to select their representative for the meeting from the person(s) that actually carried out the activation analysis. This recommendation was well followed-up, which not only eased the evaluation but also stimulated discussions and action plans for improvement to be made.

All participants in the meetings presented, using a proposed IAEA template, details of their analytical procedure applied in the interlaboratory comparison testing. Details included, e.g., information on sample masses, dry mass assessment, calibration, corrections for neutron flux gradients, internal quality control applied and status of quality assurance implementation. In addition, they provided their own view on the quality of their results and, if relevant and possible, their hypothesis on sources of error.

The results were further discussed within the broad platform of participants, moderated by the IAEA international expert and IAEA technical officer for this project. The feedback workshops were complemented with lectures on relevant aspects of the analytical procedures, such as on methodologies for internal quality control, method validation, use of control charts and sample preparation. The lessons learned during these feedback workshops were used for drafting action plans for improvements to be implemented.

6. Lessons learned

The following main sources of error were identified in the feedback workshops.

- Insufficient study of the associated documentation by the provider on the sample handling (such as estimation of the moisture fraction, and minimum sample mass to be analysed), and on reporting.
- Too tight planning of the analyses to be completed at date of deadline.
- Use of expired calibrators
- Differences in counting geometries of calibrator and standards.
- No corrections for neutron flux gradients.
- Absence on independent internal quality control such as independent reference materials and blank.
- Insufficient checking of final results upon reporting, resulting in reporting in different units than required, or exchange of samples and results.
- Insufficient trouble shooting and implementation of corrective actions.

Several participants, upon awareness of deficiencies in their analysis procedure and laboratory organization, have started implementing corrective actions. This effected improvements in the results in successive rounds which most likely also reflect the quality of other 'routine' analyses performed by these laboratories.

7. Outcome

An impression of the improvement in performance of the various (regional) participants between the first interlaboratory comparison rounds facilitated by this project, and the last ones in the year 2015, is given in Figure 1. It should be noted that the deficiencies in the results of the categories 'Average' and 'Poor' were sometimes worrying large. Often |z| values as large of 10 or higher were reported and mass fraction values differed by a factor of 100 or more from the reference values. Thus, very wrong results may have been reported in the past. These laboratories were strongly recommended to cease any other analyses until the source of the deficiencies had been found and taken away.

It can be seen from the diagrams in Figure 1 that the situation amongst the participating NAA laboratories in the year 2015 has improved significantly. The single remaining 'poor' performing laboratory in the European region is the one trying to perform NAA using a subcritical assembly at an extremely low neutron flux which may contribute to the bad results. The Asian region participated in the year 2015 for the first time in these interlaboratory comparison rounds, and could not yet demonstrate how lessons learned at the feedback meeting were brought into the practice. The differences in performance between 2015-1 and 2015-2 are entirely on participants own initiatives but with an indication that some improvement has been made.

It can also be seen from Figure 1 that laboratories perform better for soil-type materials than for plant-type materials in these interlaboratory comparison rounds, although major improvements in performance are visible too.

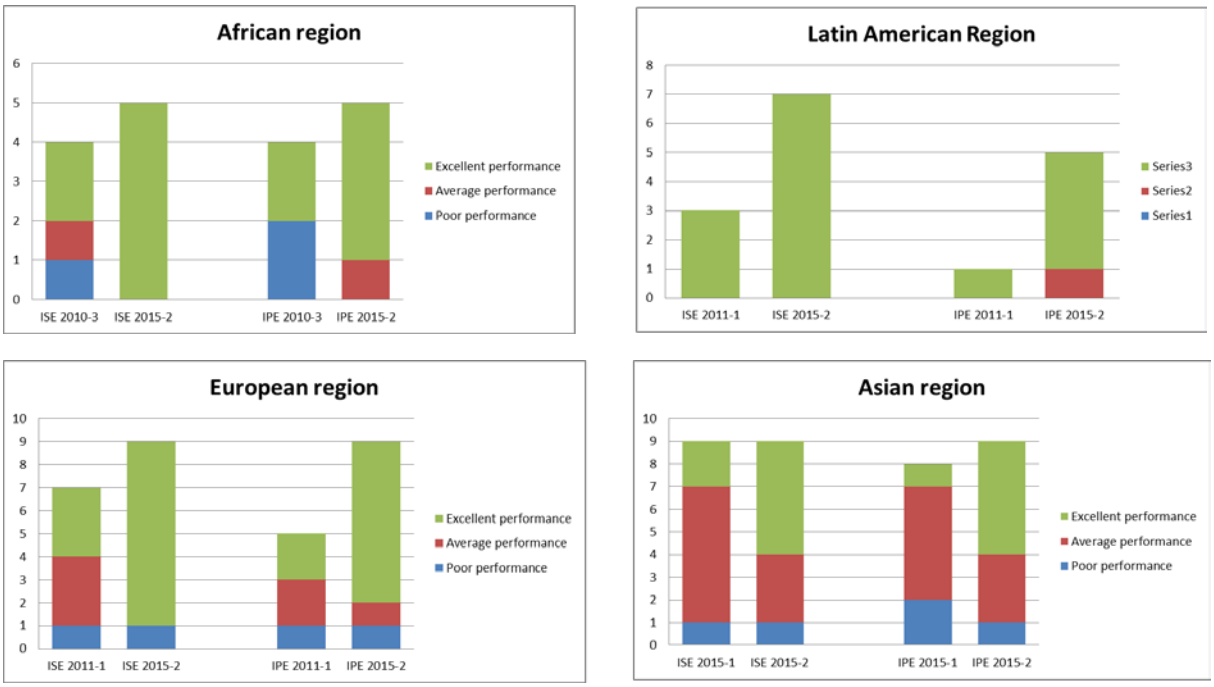

Figure 1. Performance of NAA laboratories from different regions in the first and the last interlaboratory comparison rounds participated. For performance qualification (Excellent, Average, Poor) see text.

8. Conclusions

This IAEA's initiative to facilitate laboratories participating in proficiency testing schemes complemented by the new approach of feedback workshops, some expert missions and procurement of indispensable reference materials resulted in a significant increase in the analytical and associated organizational performance of most participating laboratories. Several other laboratories demonstrated consolidation of their already satisfactory performance.

The results from the WEPAL ISE and IPE interlaboratory comparison rounds in 2015 confirmed that many laboratories operating neutron activation analysis have reached an operational level at which they periodically report excellent results for the last 5 years. Several other laboratories reported in the 2015 interlaboratory comparison campaigns a significant increase in the analytical performance compared to their performance in 2012/2013, moving from the category 'Poor' in their first participations towards 'Excellent' in their 2015 participations. A few other laboratories, representing ~10 % of all participants, did not show much improvement in their results and should continue their efforts for better performance.

Many participating laboratories have insufficient resources of their own to participate in interlaboratory comparison testing. Therefore, it has been advised to consider bi- or multi-lateral exchange of samples for analysis as an alternative. In Asia, the Forum for Nuclear Cooperation in Asia (FNCA) has an activity in which interlaboratory comparison testing is organized amongst NAA laboratories in FCNA member states. Similar activities may be initiated amongst regional research reactor networks.

The IAEA intends to continue this approach of facilitating participation in interlaboratory comparison rounds followed by feedback workshops in the year 2017.

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