PROFICIENCY TESTING BY INTERLABORATORY COMPARISON PERFORMED IN 2010-2017 FOR NEUTRON ACTIVATION ANALYSIS

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ABSTRACT

The IAEA conducts a project to evaluate the level of performance of NAA laboratories since 2010. Each test round consists of irradiation and analysis of four soil and four plant samples, prepared and distributed by an accredited company specializing in the organization of proficiency tests, Wageningen Evaluating Programs for Analytical Laboratories. To date eight rounds have been conducted, involving around 30 NAA laboratories world-wide per round. Marked increase in performance, or consolidation of excellent performance, was achieved in all regions with successive rounds. This could be identified as being the result of an increase in awareness of potential sources of error, both technical and organizational, and by the implementation of related approaches of quality control and quality assurance. We report on the results of the proficiency testing, with emphasis on the lessons learned, from which both good practices and follow-up corrective actions were derived.

1. Introduction

Enhancement of low and medium power research reactor (RR) utilization is often pursued by increasing the neutron activation analysis (NAA) activities, and this analytical technique in principle is available in more than half of 238 operating RRs world-wide [1]. Whereas the markets for NAA laboratories may have been identified, an underestimated problem remains the quality assurance and quality control, which limits tremendously the commercial routine application of this powerful technique.

In this context, a project to evaluate the level of performance of a great number of NAA laboratories has been conducted by the IAEA since 2010 [2]. Each test round consists of irradiation and analysis of standard level quality samples, four soils and four plants. An accredited company specializing in the organization of proficiency tests, Wageningen Evaluating Programs for Analytical Laboratories of Wageningen University, Netherlands, prepares and distributes samples and receives test results [3-5], while an independent expert has been contracted to evaluate laboratory performance [6-12]. After each proficiency testing round, a follow up workshop involving the participants, experts and the IAEA provides a forum for dialogue, expert advice and recommendations to all parties toward the end of improved trueness and precision in their measurements.

To date eight rounds have been conducted, involving more than 30 NAA laboratories world-wide per each round. In the initial rounds, the results from many participants were not at the level of excellence expected for an established technique that is based on sound and well-known metrological principles. In following rounds, a marked increase in performance, or consolidation of excellent performance, was achieved in all regions. This could be identified as being the result of an increase in awareness of potential sources of error, technical and/or organizational, and by the implementation of related approaches of quality control and quality assurance.

We report on the results of the proficiency testing, with emphasis on the lessons learned, from which both good practices and follow-up correcting actions were derived.

2. Proficiency testing scheme

The samples for the proficiency testing (PT) exercise have been provided, already since the beginning of this activity by the IAEA in 2010, by the Wageningen Evaluating Programmes for Analytical Laboratories (WEPAL) from The Netherlands, as part of their International Soil-analytical Exchange Programme (ISE) and International Plant-analytical Exchange Programme. WEPAL is an accredited provider of PT, and has a proven record of issuing the evaluation report 3 weeks after the deadline for reporting.

The WEPAL programme does not define predefined target criteria as it aims at interlaboratory comparison rather than on proficiency testing. Certified reference materials are not used, and the composition of the samples is not characterized a priori. The reports of the WEPAL rounds only provide indication of the deviation of the result from a given laboratory for a given element in a given sample, relative to the value of the robust mean result of all participants, taking into account the standard deviation of this mean value. This is made via calculated z-scores, defined as:

z = (lab value – median value) / (standard deviation of all observations)

Participants identify in their reports also the technique and method used. WEPAL groups the results by these identifiers. It allows for differentiation between ‘real total’ amounts (e.g. resulting from NAA or X ray fluorescence spectrometry) and amounts from techniques requiring dissolution of the sample. On the other hand, there is no mechanism for reporting uncertainties of the values reported, and therefore reporting results that have high uncertainties may lead to high z-scores.

The IAEA has facilitated the registration of a number of NAA laboratories in several of the IAEA member states (MS) for participation in WEPAL PT rounds since 2010. Initially, only participants from the Africa region were invited to join. This was followed by the Latin America and Caribbean and European regions in 2011, and Asian Pacific in 2015, although one laboratory from the latter region had already participated in 2011.

WEPAL organizes 4 rounds per year. The IAEA facilitated participation in one round per year initially, and since 2013 every two years. A total of 49 IAEA-facilitated laboratories have participated in this activity since 2010, with around 30 laboratories taking part in each round. Some labs stopped participation for different reasons, which has been compensated by newcoming labs joining. Some laboratories that participated in the past, but did not do so for a number of years, resumed participation in the 3rd quarter of 2017, the latest round sponsored by the IAEA. Some NAA laboratories received aliquots of the testing materials from registered participating laboratories.

The sample types and WEPAL samples are given in Table 1 for the different rounds. In the 2017-3 round, the samples ISE-1 (WEPAL no. 874), ISE-3 (WEPAL no. 863), IPE-2 (WEPAL no. 159) and IPE-3 (WEPAL no. 215) had already been included in previous WEPAL rounds that were facilitated by the IAEA. This is the usual practice by WEPAL, that allows to test for stability of performance in the long term.

The laboratories that participated and reported results in the run 2017-3 are listed in Table 2. A number of laboratories received samples but could not participate, for various reasons, including the reactor not being operational and difficulties with customs clearance.

TABLE 1. Sample code numbers and matrices distributed in the WEPAL ISE- and IPE-rounds 2010-3, 4; 2011-4, 2012-1, 2013-1, 2015-1 and 2015-2. In bold: Samples that have been distributed in more than one round in successive years.

Sample label	ISE rounds							
	2010-3	2010-4	2011-4	2012-1	2013-1	2015-1	2015-2	2017-3
1	861: Calcareous Clay	858: Braunerde-Pseudoclay	868: Sandy Soil	997: Sandy Soil	870: Clay from river basin	**860: Sediment**	868: Sandy soil	**874 Sandy soil**
2	**961: Clay**	998: Organic Ferrasol	900: Calcareous brown Soil	863: Clay Soil	890: Sandy soil	869: Clay	**961: Clay**	876 Clay
3	874: Sandy Soil	**872: Braunerde Clay**	952: Clay	865: Loamy Soil	919: Sandy soil	900: Calcareous brown soil	962: Sandy clay soil	**863 Clay soil**
4	**872: Braunerde Clay**	918: Sandy Soil	989: River Clay	962: Sandy Clay Soil	**961: Clay**	989: River clay	**860: Sediment**	866 Loess

Sample label	IPE rounds							
	2010-3	2010-4	2011-4	2012-1	2013-1	2015-1	2015-2	2017-3
1	198: Banana/ Musea paradisciana	133: Maize / Zea mays	169: Leek / Allium porrum	197: Maize / Zea mays	**100: Grass (gr94) / Poaceae**	**100: Grass (gr94) /Poaceae**	205: Tobacco (lea mixture) /Nicotian solanaceae	238 Banana
2	175: Tulip (tuber)/ Tulipa l.	**172: Cherry Laurel / Prunus laurocerasus**	159: Lucerne / Medicago savitum	124: Lucerne / Medicago sativum	215: Paprika / pepper (fruit + leaf) / Capsicum sp.	218: Turnip / Brassica rapa	177: poplar (leaf) populous l.	**159 Lucerne**
3	**100: Grass (gr94) / Poaceae**	180: Oil Palm (leaf)/ Elaeis guineensis	188: Oil Palm (leaves) /Elaeis guineensis	189: Banana leaves / Musa sapientum	166: Cherry Laurel /Prunus laurocerasus	171 Leylandcypress / Cupressus x leylandii	**100: Grass (gr94) / Poaceae**	**215 Paprika**
4	**172: Cherry Laurel / Prunus laurocerasus**	173: Virginia Creeper / Partenocissus quinquefolia	**100: Grass (gr94) / Poaceae**	157: Beech leaf / Fugus sylvatica l.	135: Rice (polished) / Oryza sativa l.	980: Gerbera / Gerbera cass.	224: Maize (grain) / Zea Mays	203 Cabbage

TABLE 2. NAA laboratories that participated and reported in the 2017 proficiency testing

Laboratory	Member State	Comments
Centre de recherche nucléaire de Draria (CRND)	Algeria	
Comisión Nacional de Energía Atómica (CNEA), Centro Atómico Bariloche	Argentina	
Comisión Nacional de Energía Atómica (CNEA), Centro Atómico Ezeiza	Argentina	
Institute of Nuclear Science & Technology	Bangladesh	
Centro de Desenvolvimento Tecnologia Nuclear, Comissão Nacional de Energia Nuclear (CDTN)	Brazil	
Instituto de Pesquisas Energeticas e Nucleares (IPEN); Comissão Nacional de Energia Nuclear (CNEN)	Brazil	ISE only
Comisión Chilena de Energía Nuclear (CCHEN)	Chile	
Nuclear Physics Institute (NPI); Academy of Sciences of the Czech Republic (ASCR)	Czech Republic	
Egypt Second Research Reactor ETRR-2; Atomic Energy Authority (AEA)	Egypt	
Centre for Energy Research Hungarian Academy of Sciences (KFKI)	Hungary	
National Nuclear Energy Agency (BATAN), Bandung	Indonesia	
Center for Science and Technology of Advanced Materials, BATAN, Serpong	Indonesia	Samples provided by the other NAA labs in Indonesia
National Nuclear Energy Agency (BATAN), Yogyakarta	Indonesia	
MNSR Dep.Reactor School, Esfahan	Iran	
Radiation Applications Research School, NSTRI, Tehran	Iran	Samples provided by NAA lab in Esfahan, Iran
Laboratory of Applied Nuclear Energy (LENA), University of Pavia	Italy	Participated independently
Centre of Nuclear Sciences; University of the West Indies (ICENS)	Jamaica	
Reactor Utilization Technology Department of JRTR	Jordan	Samples provided by NAA labs in Slovenia and Czech Republic
Institute of Nuclear Physics; National Nuclear Center of the Republic of Kazakhstan (NNC)	Kazakhstan	
Malaysian Nuclear Agency	Malaysia	
Centre for Energy Research and Training (CERT); Ahmadu Bello University (ABU)	Nigeria	
Directorate of Science PINSTECH	Pakistan	Two NAA labs from same institute
Directorate of Science PINSTECH	Pakistan	Samples provided by other NAA lab in Pakistan
Instituto Peruano de Energia Nuclear (IPEN)	Peru	
Joint Institute for Nuclear Research (JINR)	Russian Federation	
Jozef Stefan Institute	Slovenia	
Department of Physics; Atomic Energy Commission of Syria (AECS)	Syria	
Thailand Institute of Nuclear Technology (TINT)	Thailand	
Nuclear Research Institute	Vietnam	
University of Texas in Austin	USA	Samples provided by NAA lab in Slovenia

The performance of the laboratories in this IAEA facilitated project was evaluated on basis of the fraction of all data reported for which the absolute z-value, $|z| \leq 3$, similarly as in previous IAEA proficiency testing rounds for NAA laboratories in the years 2010, 2011, 2012, 2013 and 2015 [6-12].

Participants of the IAEA Training Workshop on Inter-Laboratory Comparison Feedback of NAA Proficiency Tests Performed in 2015 (Delft, The Netherlands, 31 August – 4 September 2015 [11]) decided to describe their performance by three categories, which will be used in this work:

1. Metrologically satisfactory performance, “excellent”, for those laboratories reporting more than 90 % of their data with $|z| \leq 3$;
2. Metrologically less satisfactory performance, “average”, for those laboratories reporting more than 70% and less than 90% of elements with $|z| \leq 3$. Minor to substantial improvements are needed to reach a higher level of performance;
3. Metrologically unsatisfactory performance, “poor”, for those laboratories reporting less than 70% of elements with $|z| \leq 3$. Major improvements are needed to reach an acceptable level of performance.

The IAEA implemented follow up feedback workshops for further discussion of the results and metrological feedback by IAEA experts on potential sources of analytical error. To this end, participants presented their activities following a strict template prepared and distributed in advance. The high level of detail in these presentations often made it possible for the experts to direct on the most probably cause of the deficiencies. Root cause analysis of deviating results was done by several laboratories.

3. Results and discussion

The results are shown in Figure 1 for each region, and in Figures 2 and 3 in aggregate, for the ISE and IPE samples, respectively. North America is not shown because it only had one participant in one round (2017-3). The evolution with time of number of laboratories in each of the performance categories (excellent-average-poor) agreed by the participants is shown. An initial marked increase in the aggregate performance was followed by some degree of stabilization, indicating consolidation of good performance by the majority of laboratories.

Considering the aggregate results for all participants, from the initial 2010-3 round to the 2015-2 round, the fraction of laboratories with excellent performance increased from 50% to 83% in the ISE rounds and from 50% to 71% in the IPE rounds. In the same period, the fraction of laboratories with poor performance decreased from 25% to 7% in the ISE rounds and from 50% to 7% in the IPE rounds.

The increase in performance, or consolidation of excellent performance, has been achieved by an increase in awareness of potential sources of error, technical and/or organizational, and related approaches of quality control and quality assurance that were implemented. In some cases assistance from well performing laboratories or international experts was the key factor for the improved analytical quality. Therefore, specific mentoring arrangements were further discussed and agreed between different laboratories and covering specific technical areas.

In the latest round (2017-3), however, a small but noticeable decrease in overall performance was observed. That result could indicate ineffectiveness of improvements on basis of lessons learned until 2015, such as calibration errors and in the quality assurance and quality control. Also, as mentioned above, gross errors continued to occur, which, had they been eliminated, would have led to significantly better performance of some of the affected laboratories.

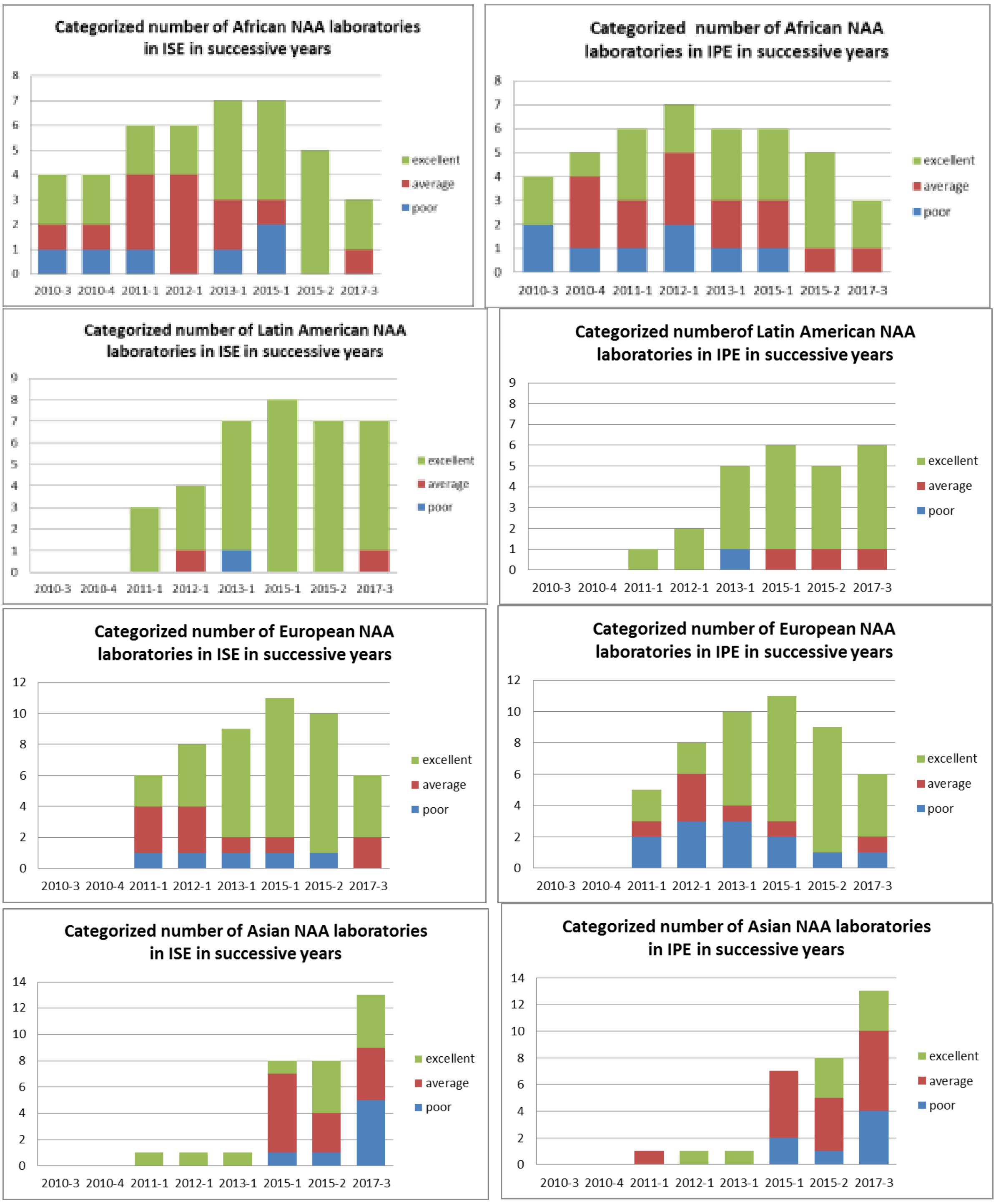

Figure 1. Number of NAA laboratories in successive WEPAL ISE and IPE rounds, categorized by fraction of reported number of data with z| ≤ 3, by region.

However, it should be noted that several of the laboratories that participated in the 2015 round but not in 2017 (6 laboratories both for ISE and IPE samples) had been in the "Excellent" category in 2015. Their absence in 2017 leads to a decrease in the number of laboratories in that category, which helps to understand the results.

It should also be noted that a few laboratories were included in the categories ‘average’ and ‘poor’ due to high |z| values resulting from human errors in the reporting stage, such as reporting in wrong units, swapping of samples, or even mixing given elements or groups of elements. We note, however, that these gross errors were only detected a posteriori. Such gross errors would be, in principle, easy to avoid by independent checking of all the steps in the calculations, and production of the final report prior to submission. These types of errors also occurred in the previously IAEA facilitated PT rounds. Their continued presence indicates that lessons from previous rounds were not fully implemented by all laboratories. In the results shown, these gross errors have not been corrected. Improvement of internal quality control according to the practice described in this report will be an important step towards improving the performance in the WEPAL ILC exercises.

4. Conclusions

The foremost outcome of this IAEA project is that, since 2010, many of the participating laboratories have expanded their knowledge of the metrology of their techniques, and have implemented or improved quality control and quality assurance procedures, thus increasing their performance in obtaining valid results of known degree of trueness.

The 2017 IAEA facilitated proficiency testing of neutron activation analysis laboratories has shown that there has not been a significant improvement in performance of laboratories that were categorized as less than 'excellent' in the 2015 PT testing. This indicates possibly insufficient follow-up to the lessons learned from the 2015 PT rounds, the related feedback

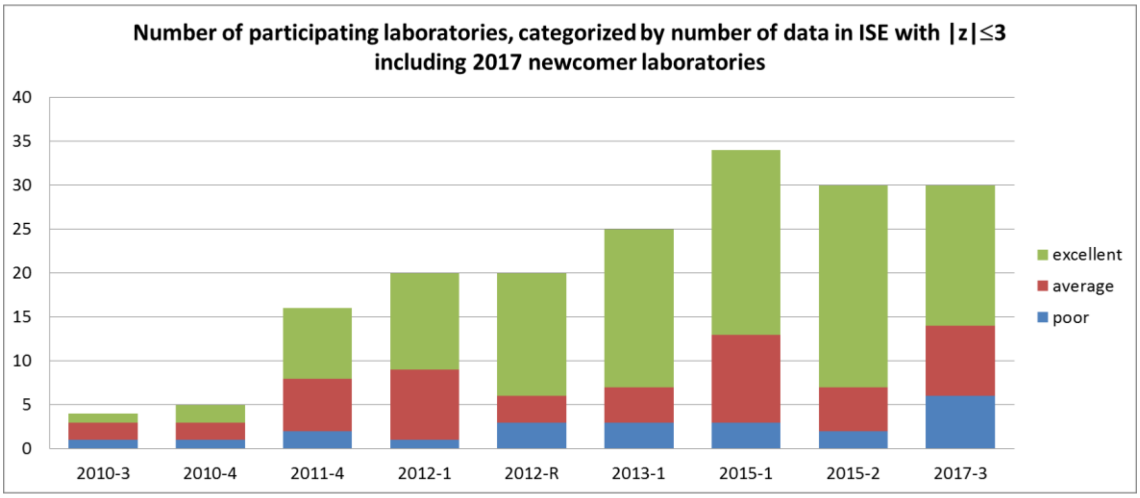

Figure 2. Total number of laboratories in the three performance categories for ISE samples, in successive years of IAEA facilitated proficiency testing exercises.

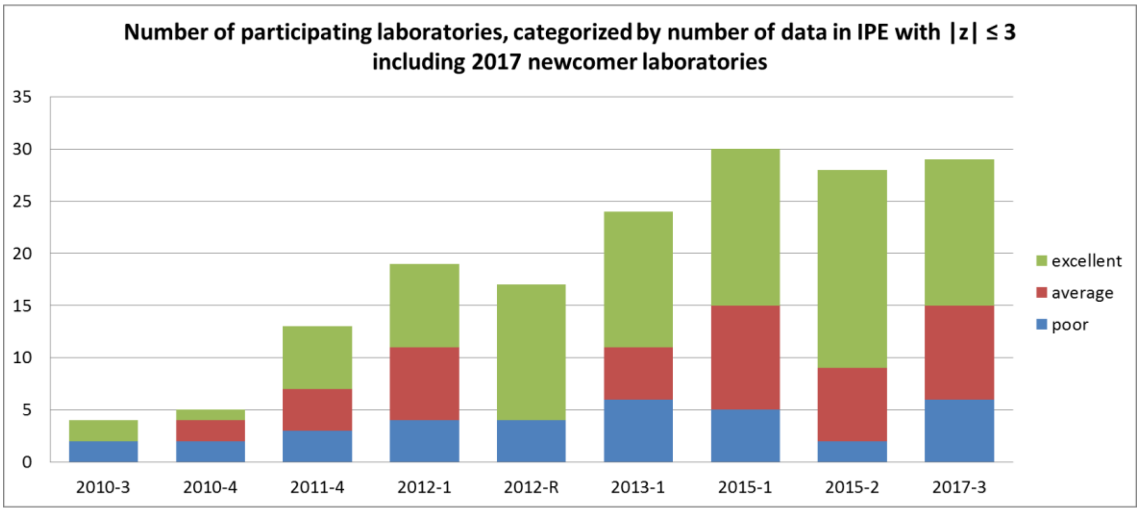

Figure 3. Total number of laboratories in the three performance categories for IPE samples, in successive years of IAEA facilitated proficiency testing exercises.

workshop and the consolidated IAEA reports of those workshops with recommendations for improvement. At the same time, some laboratories in the “excellent” category started to self-evaluate their performance in a more demanding way, by calculating how many reported results are in the $|z| \leq 2$ range.

The publication by the IAEA of a comprehensive document on the NAA proficiency testing by interlaboratory comparison, covering the 2010 to 2015 rounds [2], made available to the NAA laboratories in the IAEA Member States the results and lessons learned in this important activity. Together with the release in October 2017 of the IAEA e-learning course on NAA [13], this will contribute to the sustainability of NAA activities in research reactors, by providing an opportunity to maintain and improve the quality of NAA analyses offered by the laboratories.

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