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IAEA-TECDOC-1831

IAEA-TECDOC-1831

Proficiency Testing by Interlaboratory Comparison Performed in 2010–2015 for Neutron Activation Analysis and Other Analytical Techniques

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PROFICIENCY TESTING BY INTERLABORATORY COMPARISON PERFORMED IN 2010–2015 FOR NEUTRON ACTIVATION ANALYSIS AND OTHER ANALYTICAL TECHNIQUES

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INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA, 2017

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For further information on this publication, please contact:

Physics Section
International Atomic Energy Agency
Vienna International Centre
PO Box 100
1400 Vienna, Austria
Email: Official.Mail@iaea.org

Printed by the IAEA in Austria
December 2017

IAEA Library Cataloguing in Publication Data

Names: International Atomic Energy Agency.
Title: Proficiency testing by interlaboratory comparison performed in 2010–2015 for neutron activation analysis and other analytical techniques / International Atomic Energy Agency.
Description: Vienna : International Atomic Energy Agency, 2017. | Series: IAEA TECDOC series, ISSN 1011–4289 ; no. 1831 | Includes bibliographical references.
Identifiers: IAEAL 17-01130 | ISBN 978–92–0–108617–4 (paperback : alk. paper)
Subjects: LCSH: Nuclear activation analysis. | Laboratories — Quality control. | Nuclear reactors.

FOREWORD

The IAEA supports its Member States in increasing the utilization of their research reactors. While many research reactors were built for scientific research and training, others were intended to provide commercial products, such as radionuclides for medical and industrial applications and doped silicon for semiconductor industries. Small and medium sized reactors, representing about half of the operational facilities worldwide, are mostly used for neutron activation analysis (NAA).

Over the years, the IAEA has supported NAA groups worldwide in the shift to applications with large numbers of samples for analysis. Although markets for NAA laboratories have been identified, demonstrations of valid analytical data and organizational quality of the work process are preconditions for expanding the NAA community, particularly in routine commercial applications of this powerful technique. Over time, laboratories and stakeholders will prefer that a facility's management system is accredited for compliance with international standards for quality systems in testing and calibration laboratories.

One of the requirements in the process towards such accreditation is that the laboratory provides evidence of the validity of its measurement results by participating in proficiency testing schemes through interlaboratory comparison. However, many NAA facilities, particularly in developing countries, cannot afford the participation fees for such schemes. The IAEA through its Analytical Quality Control Services provides such interlaboratory comparison rounds at no cost. However, there are limited opportunities to assist Member State laboratories 'on demand'.

The IAEA has therefore implemented a new mechanism for supporting NAA laboratories in demonstrating their analytical performance by participating in proficiency testing through interlaboratory comparison, finding the cause of non-conformities and implementing effective approaches to eliminate them. Between 2010 and 2015, over 30 laboratories participated in proficiency testing schemes organized by the IAEA in conjunction with the Wageningen Evaluating Programmes for Analytical Laboratories (WEPAL), a provider of such schemes that is accredited by the Dutch Accreditation Council. The results were analysed by IAEA experts, who provided initial indications of potential sources of error. This publication reports the findings and lessons learned, which will be of interest to research reactor professionals involved in NAA and scientists and analysts who utilize NAA in fields as diverse as environmental studies and air quality, archaeology, materials research, cultural heritage, forensic science and health studies.

The IAEA acknowledges the valuable contributions of the participants and the support of the international experts in contributing to and reviewing this publication, in particular P. Bode (Netherlands). The IAEA officers responsible for this publication were D. Ridikas and N.P. Barradas of the Division of Physical and Chemical Sciences.

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1. INTRODUCTION

1.1. BACKGROUND

Interlaboratory comparisons are widely used for a number of purposes, and their use is increasingly internationally. Typical purposes for interlaboratory comparisons include [1]:

(a) Evaluation of the performance of laboratories for specific tests or measurements and monitoring laboratories' continuing performance;
(b) Identification of problems in laboratories and initiation of actions for improvement which, for example, may be related to inadequate test or measurement procedures, effectiveness of staff training and supervision, or calibration or equipment;
(c) Establishment of the effectiveness and comparability of test or measurement methods;
(d) Provision of additional confidence to laboratory customers;
(e) Identification of interlaboratory differences;
(f) Education of participating laboratories on the outcomes of such comparisons;
(g) Validation of uncertainty claims;
(h) Evaluation of the performance characteristics of a method (often described as collaborative trials);
(i) Assignment of values to reference materials and assessment of their suitability for use in specific test or measurement procedures; and
(j) Support for statements of the equivalence of measurements of National Metrology Institutes through 'key comparisons' and supplementary comparisons on behalf of the International Bureau of Weights and Measurement (BIPM) and associated regional metrology organizations.

Proficiency testing involves the use of interlaboratory comparisons for the determination of laboratory performance as listed in (a) to (g) above. Proficiency testing does not usually address (h), (i) and (j) because laboratory competence is assumed in these applications.

The need for ongoing confidence in laboratory performance is not only essential for laboratories and their customers, but also for other interested parties such as regulators, laboratory accreditation bodies and other organizations that specify requirements for laboratories.

Participation in interlaboratory comparison or proficiency testing schemes is listed as one of the options in Clause 5.9.1 of the International Standard ISO/IEC17025:2005 for a laboratory to monitor the validity of its tests. However, whereas the International Standard is liberal in the selection of monitoring methods ("…may include…"), National Accreditation Bodies implemented their own policies regarding the acceptable frequency of participation in Interlaboratory comparisons, following International Laboratory Accreditation Collaboration (ILAC) recommendations [2], resulting in policies that state, such as e.g. in the Netherlands, "one Interlaboratory comparison activity prior to gaining accreditation and one activity relating to each major sub-areas of major disciplines of a laboratory's scope of accreditation at least every four years; where applicable and where practical" [3].

1.2. PROFICIENCY TESTING AND QUALITY CONTROL

The validity of measurement results need to be safeguarded by internal quality control procedures to be incorporated with every batch of samples analysed, as part of a laboratory's quality assurance programme. Participation in interlaboratory comparison round cannot be used for this, as the turnaround time – the time between the deadline for analysis reporting and the issue of the

evaluation report by the provider – may vary from weeks to several months. Interlaboratory comparisons therefore cannot be considered as a direct tool to manage, guide and improve the analytical process. Secondly, one seldom finds interlaboratory comparisons with matrices, analytes and their mass fractions at the levels closely reflecting the typical day-by-day analyses. The added value of participation in interlaboratory comparison rounds is that laboratories may be faced with unanticipated sources of error, analytical and/or organizational, especially since the analyst is a-priori biased in normal internal quality control because target values are known.

The evaluation of results from interlaboratory comparisons is done by the provider by comparing the reported values with the robust median and standard deviation of all reported values, expressed (percent) difference, as *z*-scores or *zeta*- and E_n scores (the latter two requiring also an indication of the measurement uncertainty). Sometimes the provider adds a quality indicator to the value of these scores such as 'satisfactory' (e.g., for $z \leq 2$ or $E_n \leq 1$), 'questionable' (e.g., for $2 < z \leq 3$) or 'unsatisfactory' (e.g., for $z > 3$ or $E_n > 1$). Although explicitly mentioned in the ISO 17043:2010, it is in principle an inappropriate approach; only the laboratory itself can decide on its performance using the internal quality control acceptance criteria and the requirements of its customer(s).

A laboratory may face substantial problems once results from interlaboratory comparison rounds are not in agreement with its acceptance criteria. Not only the cause of the deficiency has to be found, but the deviating result(s) may set doubts to the validity of data reported to the customer(s). Such a root cause analysis requires a thorough understanding of the metrology of the analytical technique employed, and experience in troubleshooting in all steps from sample preparation to final spectrum interpretation and calibration.

1.3. PROFICIENCY TESTING AND RESEARCH REACTOR UTILIZATION

Participation was supported under the Technical Cooperation (TC) projects RAF4022/RAF1005 (Africa), RAS1018/RAS1019 (Asia-Pacific), RER4032/RER1007 (Europe), RLA0037 (Latin America) as well as under Regular Budget project 1.4.2.1 of the IAEA. A few other laboratories also decided to participate, without direct support by the IAEA. The overall objective of these projects is optimization of the effective utilization and safety of research reactors to support the socio-economic development in Member States from the Africa, Asia-Pacific, Latin America and Europe regions. The provision of services to third parties by reactor affiliated nuclear analytical laboratories, e.g. using neutron activation analysis, is one of the projected roads towards this objective. Obviously, the reported results of these laboratories need to meet or even exceed the end-users' requirements and be in compliance with international standards to ensure global acceptance.

The IAEA therefore has chosen a three-tier approach to assist the laboratories in these projects (using nuclear — mostly NAA — and nuclear-related analytical techniques) in assessing their analytical performance and effectiveness of implemented quality assurance and quality control approaches by:

- Facilitating participation in successive rounds from an accredited professional provider, with rapid issue of the evaluation report by the provider;
- IAEA expert analysis of the evaluated results providing first indications on potential sources of error;
- Follow up workshops of participants and experts with detailed discussions for identification sources of error and recommendations for actions to improvement.

1.4. PURPOSE AND STRUCTURE OF THIS PUBLICATION

The objective of this publication is to guide the analytical laboratories, mainly involved in NAA, in improving the degree of trueness and robustness of their results in a sustainable way. The outcome will be a higher degree of trustworthiness in analytical services provided by research reactor utilization, leading to an enhancement in the provision of such services.

The present report consists of six technical sections describing the main results achieved during the studies, list of references, and list of individual paper contributors together with their affiliations and individual paper titles. This publication also includes an attached CD-ROM, in which all the individual participating laboratory papers are summarized.

In addition, several Annexes have been added to this manuscript:

- Annex I: Details of WEPAL's statistical analysis;
- Annex II: Template for reporting on feedback workshops;
- Annex III: List of lectures during the feedback workshops;
- Annex IV: Country reports.

2. ORGANIZATIONAL ASPECTS OF IMPLEMENTED MECHANISM OF PROFICIENCY TESTING

2.1. PROVIDER

The Wageningen Evaluating Programmes for Analytical Laboratories (WEPAL) from Wageningen, Netherlands, was selected as provider of the proficiency testing scheme for measurement of the amounts of elements [4]. 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 considerations for selecting WEPAL were:

(a) WEPAL is accredited by the Dutch Council for Accreditation under No. R 002 for compliance with ISO 17043:2010;
(b) WEPAL has a proven record of issuing the evaluation report 3 weeks after the deadline for reporting;
(c) WEPAL provides proficiency testing schemes of soil and plant material, matrices suitable for analysis by NAA;
(d) 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;
(e) 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;
(f) In each round four samples of a specific type (soil, plant) are distributed. One sample in each round has been (blindly, i.e. not identifiable) distributed in previous rounds. This allows for comparison of stability and/or effectiveness of corrective actions.

2.2. ROUNDS AND DISPATCH SCHEME

Participants from the Africa region (RAF 4022/ RAF 1005) were invited to join rounds of WEPAL interlaboratory comparisons in 2010, 2011, 2012, 2013 and 2015 for both soil-related (WEPAL ISE) and plant-related (WEPAL IPE) materials.

Participants from the Latin America (RLA 0037/ RB 1.4.2.1) and Europe (RER 4032/ RER 1007) regions participated in the rounds in 2011, 2012, 2013 and 2015.

One laboratory from the Asia Pacific (RAS 1018) region participated in the rounds of 2011, 2012 and 2013, and was joined by another eight participants in 2015.

Further details on the participants are given in Table 1.

At the end of the year 2012, ten RAF 4022, six RLA 0037 and six RER 4032-RER1007 laboratories re-analysed samples that were distributed in the first round of their participation in the IAEA facilitated WEPAL proficiency schemes. The objective of these re-analyses was to compare the new results with those obtained before, thus obtaining insight in the degree of improvement in analysis of the same material. The results were returned to the IAEA only, and evaluated by the IAEA expert.

2.3. SAMPLES AND SAMPLE TYPES

Sample types and code numbers are given in Table 2. The samples are dispatched only with labels 1, 2, 3 and 4. Participants receive the information on the origin of the material and sample types, and their code numbers only with the WEPAL Quarterly report.

One particular sample in each round has been distributed in one of the previous WEPAL rounds (sometimes one or more years before). This provides the laboratory an opportunity to verify its medium and even long term improvement or stability of its performance. These samples are shown in bold in Table 2.

2.4. PARTICIPANTS, TECHNIQUES AND METHODOLOGIES

Participating laboratories and their techniques are listed in Table 3. The laboratory in Mexico reported that the WEPAL samples were not further dispatched to them by the local customs office because of import restrictions for agricultural products.

TABLE 1. DATA ON WEPAL ROUNDS

WEPAL round	Implemented in IAEA project	Sample dispatch by provider in the Netherlands	Laboratory reporting deadline	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
2012-R	RAF 4022 RLA 0037 RER 4032/RER 1007	Not applicable	December 31, 2012	Not applicable
2013-1	RAF 4022 RLA 0037 RER 1007 RAS 1018	January 11, 2013	March 31, 2013	April 7, 2013
2015-1	RAF 1005 RB 1.4.2.1 RER 1007 RAS 1019	December 1, 2014	March 31,2015	April 9/10 2015
2015-2	RAF 1005 RB 1.4.2.1 RER 1007 RAS 1019	March 1, 2015	June 30, 2015	July 6/7, 2015

TABLE 2. 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

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

TABLE 3. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, AND FREQUENCY OF PARTICIPATION

WEPAL Lab No.	WEPAL lab code	Lab code in this report	Laboratory	Country	Technique/ Method	ISE rounds/ IPE rounds							
						2010-3	2010-4	2011-4	2012-1	2012-R	2013-1	2015-1	2015-2
1029	MASHA	ALG-M	Centre de recherche nucléaire de Draria (CRND)	Algeria	NAA relative method	X/X	X/X	X/X	X/X		X/X	X/X	X/X
1110	NOUSSE	ALG-N	Centre de recherche nucléaire de Birine (CRNB); Commissariat à l'énergie atomique (COMENA)	Algeria	NAA k_0 method			X/X	X/X	X/X	X/X	X/X	X/X
1036	LASPEE	CAM	Institut de recherche agricole pour le développement (IRAD)	Cameroon	AAS, ICP-OES and others	X/X	X/X		X/X	X/X	X/X		
1030	CGEA	DRC	Centre d'Expertise d'Evaluation et de Certification des substances Minerales Precieuses et Semi-Precieuses	Congo	ICP-OES	X/X	X/X	X/X	X/X	X/X	X/X		
1031	ETRR	EGY	Egypt Second Research Reactor ETRR-2; Atomic Energy Authority (AEA)	Egypt	NAA, k_0 method			X/X	X/X		X/X	X/X	X/X
1032	GAEC	GHA	National Nuclear Research Institute; Ghana Atomic Energy Commission (GAEC)	Ghana	NAA, relative method and k_0 method	X/X	X/X	X/X	X/X	X/X	X/X	X/X	
1037	INSTN	MDG	Institut national des sciences et techniques nucléaires (INSTN)	Madagascar	XRF, TXRF and AAS	X/X		X/X	X/X	X/X	X/X		
1033	CNES	MOR	Centre national de l'énergie, des sciences et des techniques nucléaires (CNESTEN)	Morocco	NAA relative method and k_0 method		X/X		X/X	X/X	X/X	X/X	X/X

TABLE 3. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, AND FREQUENCY OF PARTICIPATION (cont.)

WEPAL Lab No.	WEPAL lab code	Lab code in this report	Laboratory	Country	Technique/ Method	ISE rounds/ IPE rounds							
						2010-3	2010-4	2011-4	2012-1	2012-R	2013-1	2015-1	2015-2
1034	CERT	NIG	Centre for Energy Research and Training (CERT); Ahmadu Bello University (ABU)	Nigeria	NAA, relative method	X/X	X/X	X/X	X/X	X/X	X/X	X/X	X/X
1035	NECSA	RSA	South African Nuclear Energy Corp. of South Africa NECSA	South Africa	NAA, relative method	X/X	X/X	X/X	X/X	X/X	X/X	X/X	
1038	PETRO	SUD	Sudan Atomic Energy Commission (SAEC); Ministry of Science and Technology	Sudan	ICP-OES	X/		X/		X/X	X/X		
1137	ATEC	TAN	Tanzania Atomic Energy Commission (TAEC) Nucl.Techn.	Tanzania	XRF					X/			
1089	NPIAS	CZ	Nuclear Physics Institute (NPI); Academy of Sciences of the Czech Republic (ASCR)	Czech Republic	NAA, k_0 method				X/X	X/X	X/X	X/X	X/X
1090	ARIST	GR	Aristotle University of Thessaloniki	Greece	NAA, relative method			X/X	X/X	X/X	X/X	X/X	X/X
1091	KFKI	H-K	Centre for Energy Research Hungarian Academy of Sciences	Hungary	NAA, k_0 method				X/X	X/X	X/X	X/X	X/X
1092	REAK	H-R	Institute of Nuclear Techniques, Technical University Budapest	Hungary	NAA, k_0 method			X/X	X/X	X/X	X/X	X/X	X/X
1111	LNIP	I	Laboratory Of Applied Nuclear Energy, University of Pavia	Italy	NAA, relative method			X/X	X/X				

TABLE 3. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, AND FREQUENCY OF PARTICIPATION (cont.)

WEPAL Lab No.	WEPAL lab code	Lab code in this report	Laboratory	Country	Technique/ Method	ISE rounds/ IPE rounds							
						2010-3	2010-4	2011-4	2012-1	2012-R	2013-1	2015-1	2015-2
1094	DENNAA	KAZ	Institute of Nuclear Physics; National Nuclear Center of the Republic of Kazakhstan (NNC)	Kazakhstan	NAA					X/X	X/X	X/X	X/X
1095	SACAV	PT	Instituto Superior Técnico, Instituto Tecnológico e Nuclear	Portugal	NAA, k_0 method			X/X	X/X	X/X	X/X	X/X	
1096	CAMPU	RO	Institute for Nuclear Research - Pitesti; Romanian Authority for Nuclear Activities (RAAN)	Romania	NAA, k_0 method			X/X	X/X	X/X	X/X	X/X	
1097	ROSC	R-R	Neutron Research Department, St. Petersburg Nuclear Physics Institute PNPI	Russian Federation	NAA			X/X					
1098	ATCHI	R-A	Neutron Research Department, St. Petersburg Nuclear Physics Institute PNPI	Russian Federation	NAA			X/X	X/X				
1139	NURES	R-D	Joint Institute for Nuclear Research (JINR), Dubna	Russian Federation	NAA						X/X	X/X	
1099	TEFA	SV	Jozef Stefan Institute	Slovenia	NAA, k_0 method			X/X	X/X	X/X	X/X	X/X	X/X
1109	YAZA	TR	Energy Institute Ayazaga Campus; Istanbul Technical University Maslak	Turkey	NAA, relative method			X/X	X/X	X/	X/X	X/X	X/X
1208	SAREZ	UZB	Institute of Nuclear Physics AS RUZ	Uzbekistan	NAA, relative method							X/X	X/X

TABLE 3. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, AND FREQUENCY OF PARTICIPATION (cont.)

WEPAL Lab No.	WEPAL lab code	Lab code in this report	Laboratory	Country	Technique/ Method	ISE rounds/ IPE rounds							
						2010-3	2010-4	2011-4	2012-1	2012-R	2013-1	2015-1	2015-2
1102	ACTIVA	ARG-B	Comisión Nacional de Energía Atómica (CNEA) Centro Atómico Bariloche	Argentina	NAA					X/X			
1103	TECNUC	ARG-E	Comisión Nacional de Energía Atómica (CNEA), Centro Atómico Ezeiza	Argentina	NAA, relative method			X/X	X/X		X/X	X/X	X/X
1104	IPCN	BRA-SP	Instituto de Pesquisas Energeticas e Nucleares (IPEN); Comissão Nacional de Energia Nuclear (CNEN)	Brazil	NAA			X/	X/	X/	X/	X/	X/
1136	VOLVI	BRA-BH	Centro de Desenvolvimento da Tecnologia Nuclear, Comissão Nacional de Energia Nuclear	Brazil	NAA, k_0 method						X/X	X/X	X/X
1138	CASIL	CL	Comisión Chilena de Energía Nuclear (CCHEN)	Chile	NAA, relative method					X/X	X/X	X/X	X/X
1105	CANCRA	CO	Servicio Geológico Colombiano	Colombia	NAA, relative method						X/X	X/X	X/X
1106	INDIES	JM	Centre of Nuclear Sciences; University of the West Indies	Jamaica	NAA, relative method; EDXRF				X/X	X/X	X/X	X/X	X/X
1107	CARRTOL	MEX	Instituto Nacional de Investigaciones Nucleares (ININ)	Mexico	NAA, relative method					X/		X/X	
1108	DESAR	PE	Instituto Peruano de Energia Nuclear, IPEN	Peru	NAA, k_0 method			X/X	X/X	X/X	X/X	X/X	X/X

TABLE 3. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, AND FREQUENCY OF PARTICIPATION (cont.)

WEPAL Lab No.	WEPAL lab code	Lab code in this report	Laboratory	Country	Technique/ Method	ISE rounds/ IPE rounds							
						2010-3	2010-4	2011-4	2012-1	2012-R	2013-1	2015-1	2015-2
1210	HEIGH	AUS	Australian National Science and Technology Organization (ANSTO)	Australia	NAA k_0 method							X/X	X/X
1202	NUCLT	BD	Institute of Nuclear Science & Technology	Bangladesh	NAA Relative method							X/X	X/X
1204	SARIN	IND-Y	National Nuclear Energy Agency (BATAN), Yogyakarta	Indonesia	NAA							X/X	X/X
1205	SCIENA	IND-B	National Nuclear Energy Agency (BATAN), Bandung	Indonesia	NAA Relative method							X/X	X/X
1209	SELAT	IND-S	Center for Science and Technology of Advanced, Serpong	Indonesia	NAA k_0 method							X/X	X/X
1206	AGBAN	MAY	Malaysian Nuclear Agency	Malaysia	NAA Relative method							X/X	X/X
1100	SYRAT	SY	Department of Physics; Atomic Energy Commission of Syria (AECS)	Syria	NAA, k_0 method			X/X	X/X	X/X	X/X	X/X	X/X
1194	PTNAA1	THAI	Thailand Institute of Nuclear Technology (TINT)	Thailand	NAA Relative method							X/X	X/X
1207	LUCST	VIE	Nuclear Research Institute	Vietnam	NAA k_0 method							X/X	X/X

2.5. DATA EVALUATION AND CRITERIA

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 International Plant-analytical Exchange (IPE) programme than in the International Soil-analytical Exchange (ISE) programme, see Tables 4 and 5. Most results of the laboratories participating in the IPE rounds were grouped in the category 'Inorganic chemical composition'.

For each analyte (and assuming a normal distribution), a mean and standard deviation, the median and the median absolute deviation (MAD) as well as a *z*-score[1] is calculated; this *z*-score on basis of the normal distribution approximation (see Annex I). The participants do not report their own measurement uncertainty, so its value is not accounted for by the provider.

The relative bias ({observed value minus mean value} / mean value) is sometimes easier to interpret than the *z*-score. Laboratories may have their own fitness for intended purpose criterion (e.g. maximum acceptable percentage bias, or *z*-score or *zeta* score) that can be monitored by analysis, simultaneous with the real samples, of an internal quality control material (with known property values).

It should be noted that the median value is based on the results of the participating laboratories and that it is not a certified value; and that the standard deviation is not an indication of the measurement uncertainty of the property value but rather an indication of the spread of the results reported by the various participants in a WEPAL round.

The NAA laboratory of the Delft University of Technology, accredited in 1993, has an acceptance criterion for the laboratory's internal quality control of $| zeta | < 3$ based on the analysis of (certified) reference materials. By its definition, $| zeta | < | z |$. The laboratory participates in the WEPAL schemes since the early 1980s. Analysis of its results in these schemes indicate that the percentage of outlying data reported for which $| z | > 3$ ("% $| z | > 3$|") is typically ≤ 5 %, and that the number of reported outlying data for which the relative bias was ≥ 20% ("% | relative bias| > 20 %|") is typically ≤ 10 %.

On the basis of these experiences the following criteria for evaluation were applied in this IAEA project of proficiency testing: a satisfactory result is defined by $| z |$-score ≤3 and relative bias value < 20 %. 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.

WEPAL's evaluation of the results has been made available to the participants via the quarterly report (see Table 1). WEPAL also made the results available in an electronic format to the IAEA for further evaluation. Identification of sources of error was one of the objectives of this IAEA project, and therefore the evaluations focused on the number and type(s) of outliers, i.e., those cases for which $| z | > 3$ and % | relative bias | > 20 %. The IAEA expert reported his evaluations [7-12] firstly to the IAEA, which subsequently distributed them to the participants involved.

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

TABLE 4. METHOD CATEGORIES IN THE WEPAL ISE ROUNDS [5]

ISE Group	Determinand
Real totals	Ag, Al, As, B, Ba, Be, Bi, Br, C – elementary, Ca, Cd, Ce, Co, Cr, Cs, Cu, F, Fe, Ga, Ge, Hg, I, K, La, Li, Mg, Mn, Mo, N – elementary, Na, Nb, Nd, Ni, P, Pb, Pd, Pt, Rb, Rh, S, Sb, Sc, Se, Si, Sn, Sr, Te, Th, Ti, Tl, U, V, W, Y, Zn, Zr
Acid extractable (So-called totals)	Ag, Al, As, B, Ba, Be, Bi, Br, Ca, Cd, Ce, Co, Cr, Cu, F, Fe, Ga, Hg, I, K, La, Li, Mg, Mn, Mo, N, Na, Nb, Nd, Ni, P, Pb, Pt, Rb, S, Sb, Sc, Se, Si, Sn, Sr, Te, Th, Ti, Tl, U, V, Y, Zn, Zr
Aqua Regia (ISO 11466)	Ag, Al, As, B, Ba, Be, Bi, Br, Ca, Cd, Ce, Co, Cr, Cu, F, Fe, Ga, Hg, I, K, La, Li, Mg, Mn, Mo, N, Na, Nb, Nd, Ni, P, Pb, Pt, Rb, S, Sb, Sc, Se, Si, Sn, Sr, Te, Th, Ti, Tl, U, V, Y, Zn, Zr
Extraction with boiling 2M HNO_3	Cd, Co, Cr, Cu, Hg, Mo, Ni, Pb, Tl, Zn
Extraction with 0.1M $NANO_3$	Cd, Cu, Ni, Pb, Zn
Extraction with 0.001M $CaCl_2$ 1:10	Al, B, Cd, CN, Co, Cr, Cu, Fe, K, Mg, Mn, N-NH_4, N-NO_3,N total soluble, Na, Ni, P, Pb, SO_4, Zn
Soil characteristics	C – org. others (W&B a.o.), EC-SC (ISO 11265), Fraction < 16µm, Fraction < 2µm, Fraction < 63µm, Fraction > 63µm, Org. matter (L.O.I.), pH – $CaCl_2$, pH – H_2O, pH – KCl, TC=Total C (org.+inorg.), TIC=Tot.Inorg., C ($CaCO_3$), TOC=Total Org. C
Other determinations	C^{13}, N^{15}, B – Hot water, CN – Free, CN – Total, delta 13C, delta 15N, K-HCl, Mg – NaCl, Moisture-content
Fluoride (Swiss standard procedure)	F - Total
Digestion with conc. HNO_3 + conc. HCL + H_2O_2 (UNEP-UN/EC 91075A)	Al, As, B, Ba, Be, Br, Ca, Cd, Co, Cr, Cu, F, Fe, Ga, Hg, I, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Si, Sn, Sr, Tl, V, Y, Zn, Zr
Pot. CEC using 1M NH_4 – acetate at pH=7	Al, Ca, CEC, K, Mg, Na
Pot. CEC using 1M or 0.1M $BaCl_2$-TEA at pH=8.1 (ISO 13536 OR BZE)	Al, Ca, CEC, K, Mg, Na
Pot. CEC using 1M NH_4Cl (BZE)	Al, Ca, CEC, Fe, H, K, Mg, Na
Act. CEC using 0.01M $BaCl_2$ (ISO 11260)	Al, Ca, CEC, Fe, H, K, Mg, Na
Act. CEC using 0.1M $BaCl_2$ (UNEP-UN/EC 91065A)	Al, Ca, CEC, Fe, H, K, Mg, Na
Act. CEC using cobaltihexamine (AFNOR NFX 31 130)	Al, Ca, CEC, Fe, H, K, Mg, Na
Mehlich-3	Al, AS, B, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, P, Pb, Zn
Extraction with Ca-lactate (VDLUFA)	K, P
Extraction with double lactate (VDLUFA)	K, P
Water soluble 1:10 (w/v) (EN-12457-4)	Br, Cl, F, N – NO_3
Extraction with 0.01M $CaCl_2$ + 0.005M TPA 1:10 (w/v)	Cu, Fe, Mn, Zn
Phosphorus and related analysis	Al – Ox, Fe – Ox, P – Ox, P – Al, P – Bray, P – Olsen, Pw
Extraction with 1M HCl (Polish standard)	B, Cu, Fe, Mn, Zn
Water soluble 1:10 (w/v) (NL VPR C85-06)	Br, Cl, F, SO_4

TABLE 5. METHOD CATEGORIES IN THE WEPAL IPE ROUNDS [6]

IPE Group	Determinand
Inorganic Chemical Composition	Ag, As, B, Ba, Be, Bi, Br, Ca, Cd, Cl, Co, Cr, Cu, F, Fe, Ga, Hg, I, K, Li, Mg, Mn, Mo, N - Kjeldhl, N – NH_4, N – NO_3, Na, Ni, P, Pb, Pd, Pt, Rb, Rh, S, Sb, Se, Sn, SO_4, Sr, Ti, V, Zn
Real totals	Al, C – elementary, N – elementary, Si
Acid extractable (So-called totals)	Al, Si
Other determibations	13C, 15N, delta 13C, delta 15N
Nutritional values	ADF-ash-containing, ADF-ash-free, Crude fibre, NDF- ash-containing, NDF- ash-free, Polysaccharides (starch), TDF, TDF-non-soluble, TDF-soluble, Total ash, Total Disaccharides, Total fat, Total monosaccharides

2.6. FEEDBACK WORKSHOPS

The IAEA implemented follow up feedback workshops (see Table 6 and Section 4) 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 (see Annex II). 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.

TABLE 6. OVERVIEW OF FEEDBACK WORKSHOPS HELD

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

The workshops were complemented by lectures relevant for the scope of the project (for overviews thereof, see Annex III). Participants presented their action plans for improvement and recommendations were drafted towards the laboratories themselves, the IAEA and the national governments (if applicable). In subsequent meetings, participants presented the accomplishments of their action plans. During the workshop held in Delft in 2012, the WEPAL's Manager, Mr. Bram Eijgenraam, presented WEPAL's methods for preparing test samples and receiving feedback from the participants. An overview was given of the lengthy experience, extending through some decades and numerous tests, of WEPAL in programmes for proficiency testing of laboratory analysis of soil and organic matter. The massive electronic database of test results enabled WEPAL to show a trend of lower standard deviation values since 1990, indicating, when taken as a whole, improved analytical quality by laboratories. Also WEPAL's technique for collecting, mixing and distributing samples was presented.

3. MEASUREMENT RESULTS

3.1. GENERAL OBSERVATIONS

WEPAL dispatches the samples approximately 3 months before reporting deadline (see Table 1); the only exception was in round 2011-4. Even in the latter case (dispatch ca. 6 weeks before deadline) this provides in principle still an ample timeframe for analysis by NAA (which typically requires 4 weeks at the most) or other techniques, particularly considering that the laboratories are informed and, in principle, analyses could have been planned accordingly. However, several laboratories informed the IAEA that they were not able to report in time (due, for instance, to limited reactor operation in the month December). WEPAL accepted, in this case, results delivered up to 2 weeks after the deadline and evaluated them for this Agency's project, but could not include them in the 2011-4 quarterly report.

At the start of the project, in 2010, almost all participants returned their values to WEPAL only a few days before, or on the day of the deadline. This was often due to a tight planning scheme perhaps too close towards this deadline, and sometimes by final internal checks and formal authorization prior to release of the results. This practice later improved significantly, starting in 2012 (see Table 7), after the feedback meetings in which planning, priority setting and associated organization (such as having deputies for releasing the reports and assuring reactor availability) was discussed.

TABLE 7. REPORTING DATES DURING THE ROUNDS

	Deadline	Earliest report received
2010-3	September 30, 2010	September 23, 2010
2010-4	December 31, 2010	December 27, 2010
2011-4	December 31, 2011	December 29, 2011
2012-1	March 31, 2012	March 20, 2012
2012-R	December 31, 2012	November 30, 2012
2013-1	March 31, 2013	February 13, 2013
2015-1	March 31, 2015	March 13, 2015
2015-2	June 30, 2015	May 20, 2015

The performance of participants was, during the intermediate evaluations and feedback workshops, presented with emphasis on the improvement of the number of non-satisfactory target values, that is, the percentage of reported values with $|z|>3$ or | relative bias | > 20% (the reverse of the criterion described in paragraph 2.5). In this report, the results are presented with emphasis on the ability of reporting satisfactory results as outlined in paragraph 2.5.

3.2. INTERNATIONAL SOIL-ANALYTICAL EXCHANGE (ISE)

All performance indicators of the soil analyses (WEPAL-ISE rounds) are given in Figures 1-4 for the participants from the Africa, Asia-Pacific, Latin America and Europe regions, respectively. The performance indicators of the reanalysis of the 2011-4 samples (previously denoted as '2012-reanalysis round' or '2012-R') are not shown in the figures as these analyses were not a fully 'blind' study.

3.2.1. Africa region participants

A group of seven participants used NAA (both Algerian laboratories, Egypt, Ghana, Morocco, Nigeria, and South Africa). A further six participants (Cameroon, Democratic Republic of Congo,

Madagascar, Sudan and Tanzania, which only participated in the 2012-R round) used various other techniques such as X ray fluorescence spectrometry, atomic absorption spectrometry, inductively coupled plasma emission spectrometry and others.

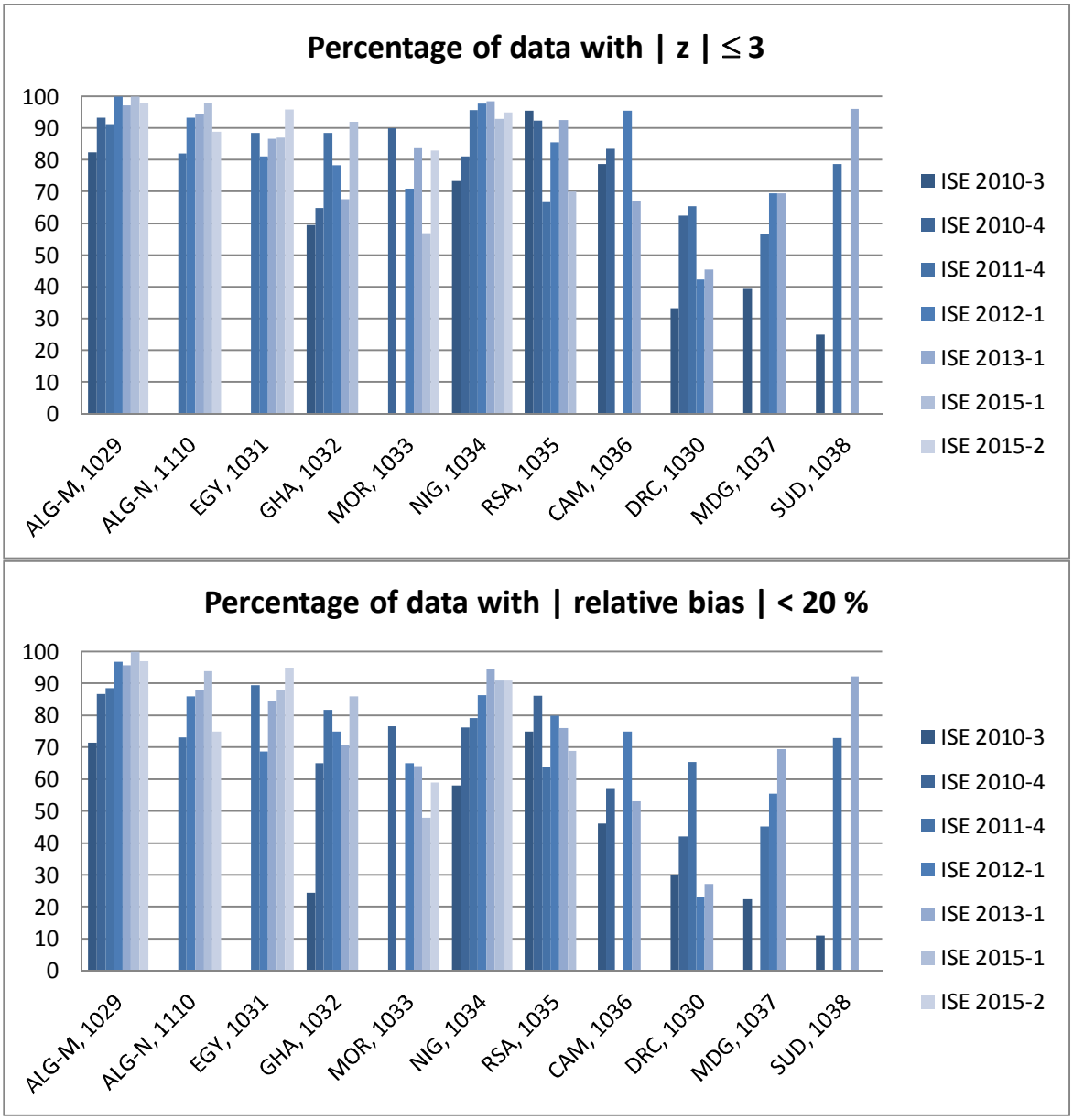

FIG. 1. Performance indicators of the Africa region participants in WEPAL-ISE.

The seven leftmost participants in Figure 1 all used NAA. Most had substantial improvement of their performance between the first intercomparison round in 2010 and the last one of 2015. The laboratories from Algeria-Masha, Egypt and Nigeria demonstrated being able to report results with the level of trueness that may be expected from the metrological status of NAA. The participants from Algeria-Nousse, Ghana, and South Africa showed some oscillation in performance; however, it may occur that the level is already acceptable for their typical type of analyses and that efforts to further improvement and consolidation of good performance are not considered worth the investment needed (e.g. in manpower). Some deficiencies in the degree of trueness of the results from the NAA laboratory in Morocco seem to continue to occur.

The four rightmost participants in Figure 1 used other techniques, as described in Table 3. None of these laboratories participated in the 2015 rounds. While the participants from Cameroon and

the Democratic Republic of Congo were not able to consolidate their performance, the laboratories from Madagascar and Sudan showed remarkable improvement between 2010 and 2013, the last round where they participated.

Half of the results reported by the laboratory from Cameroon are related to the trace element measurement using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES); the other half is related to entirely other measurands, like pH. There is some difference in performance of this laboratory for the two groups of results as can be seen in Table 8.

TABLE 8. COMPARISON OF PERFORMANCE INDICATORS IN ISE 2013-1 OF THE PARTICIPANT FROM CAMEROON FOR TRACE ELEMENTS BY ICP ONLY, AND FOR THE MEASURANDS BY OTHER TECHNIQUES

	Fraction of data with $\lvert z \rvert \leq 3$:	Fraction of data with | relative bias | ≤ 20 %
Overall in 2013	67 %	53 %
ICP-OES data only	56 %	34 %
Other measurands	78 %	72 %

The difference in performance by *z*-score and relative bias in the results of this laboratory results from the relatively high standard deviation of the median values reported for the measurands. Thus, the $\lvert z \rvert$-scores are relatively small, though still high biases may occur.

Comparing the performance indicators of the 2013-1 results with those of the re-analysis done in 2012, which were done three months before the 2013-1 exercise, renders the following observations:

(a) The laboratories in Algeria-Nousse, Nigeria, Sudan, South Africa, and Sudan continued their excellent performance from 2012 in the ISE 2013-1 round;
(b) The laboratories in Cameroon, Congo, Ghana and Morocco could not provide equally good results in 2013 as they did by the end of 2012, only 3 months earlier. In the case of Ghana, this was attributed to the k_0 method for calibration, introduced in 2013 and replacing the relative standardization used before.

3.2.2. Asia-Pacific region participants

All Asia-Pacific laboratories used NAA for their trace element measurements (Figure 2). The laboratory from Syria consolidated and improved its good level of performance from the beginning of its participation in the 2011 round to 2015.

A group of further eight laboratories participated in 2015 for the first time in the WEPAL rounds as well as in the feedback meeting. A comparison with the first-time performance of the other laboratories in the IAEA facilitated WEPAL rounds (e.g., 2010-3 for Africa and Europe) indicates almost equivalence on the median of the performance indicator: median for Africa in 2010-3:79%; for Europe in 2011-4: 86%; for Asia-Pacific in 2015-1: 85%. The relatively low percentages of results with $\lvert z \rvert \leq 3$ by the participants from Vietnam (especially in round 2015-1) and Indonesia-B are remarkable as these laboratories are both ISO/IEC17025:2005 accredited. It indicates that their internal quality control for assessment of the validity of results prior to reporting is not entirely flawless. It can also be derived from the difference in performance in 2015-1 and 2015-2 rounds that the participant from Vietnam probably has implemented corrective actions before participating in round 2015-2, being a promising sign for further improvement.

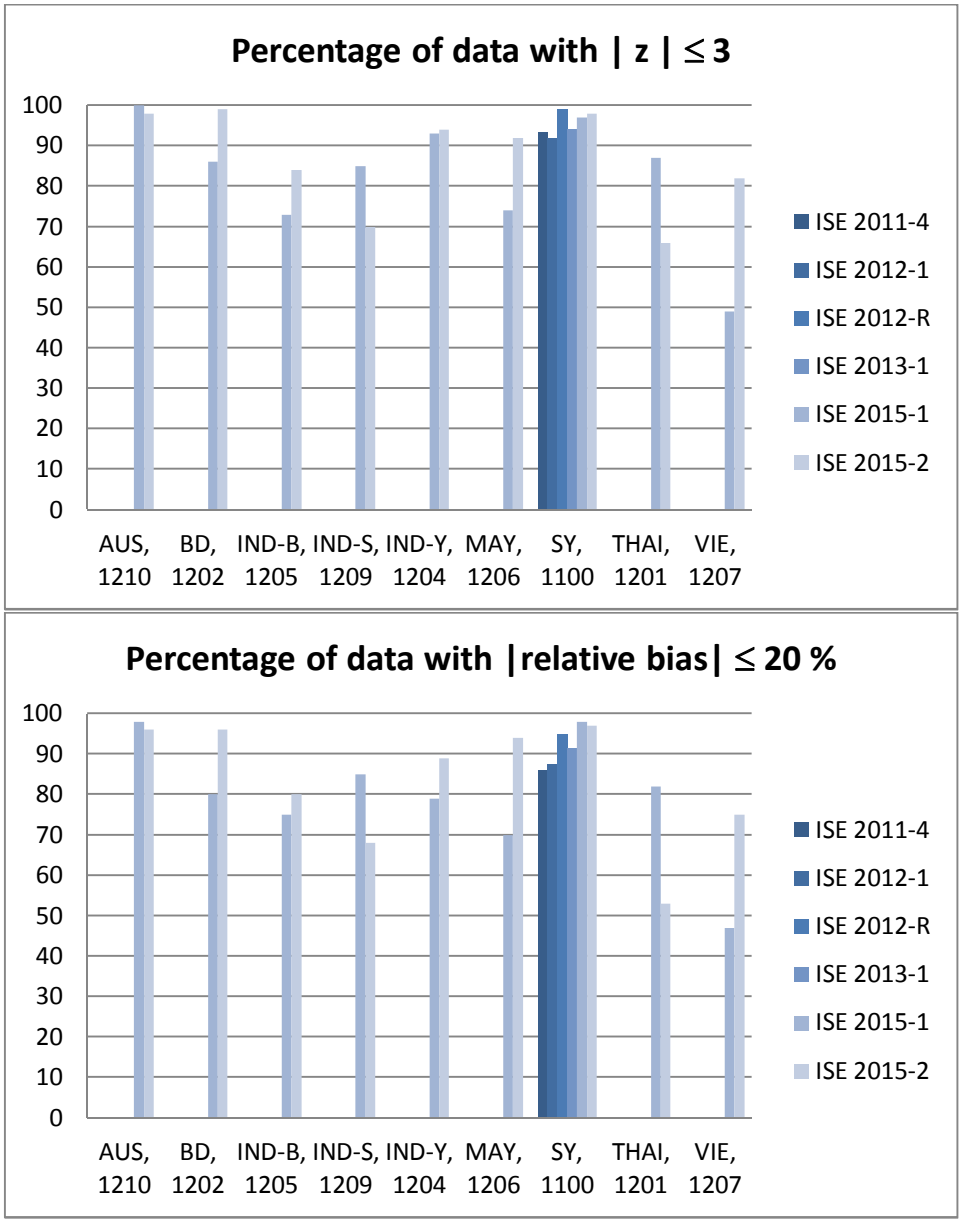

FIG. 2. Performance indicators of the Asia-Pacific region participants in WEPAL-ISE.

3.2.3. Latin America region participants

All laboratories from the Latin America region use NAA for their trace element measurements (Figure 3). The results from the Jamaican laboratory were obtained both by NAA and ED-XRF. Argentina (Bariloche) only participated in the 2012-R re-evaluation (with a good performance on basis of the z-scores but less good on the bias) and therefore is not shown in the figure.

A very high percentage of results reported by laboratories of Argentina (Ezeiza), Brazil (Both), Chile, Jamaica and Peru had a $|z| \leq 3$ (see Figure 3). Also the degree of trueness (|relative bias| ≤ 20%) is very high for most of these laboratories (Figure 3), though there was a strong oscillation in the facility of Brazil (São Paulo) in the 2015-1 round. The results from the Jamaican laboratory were obtained both by NAA and ED-XRF.

It should be noted that the participants from Colombia and Mexico reported in 2012 the results of the 2011-4 samples in the frame of the 're-analysis round'. By then, they accomplished | z | ≤ 3

for about 60 % of the reported data. The high performance of these laboratories in the 2015 rounds is remarkable, especially since the laboratory in Colombia uses a reactor that was re-licensed only as of late 2014 after a very long shut-down period.

There is no significant difference between the performance of the Latin American laboratories in the re-analysis round in 2012 (ISE 2012-R) and in the ISE round 2013-1. This emphasizes the effectiveness of the quality control and quality assurance in these laboratories.

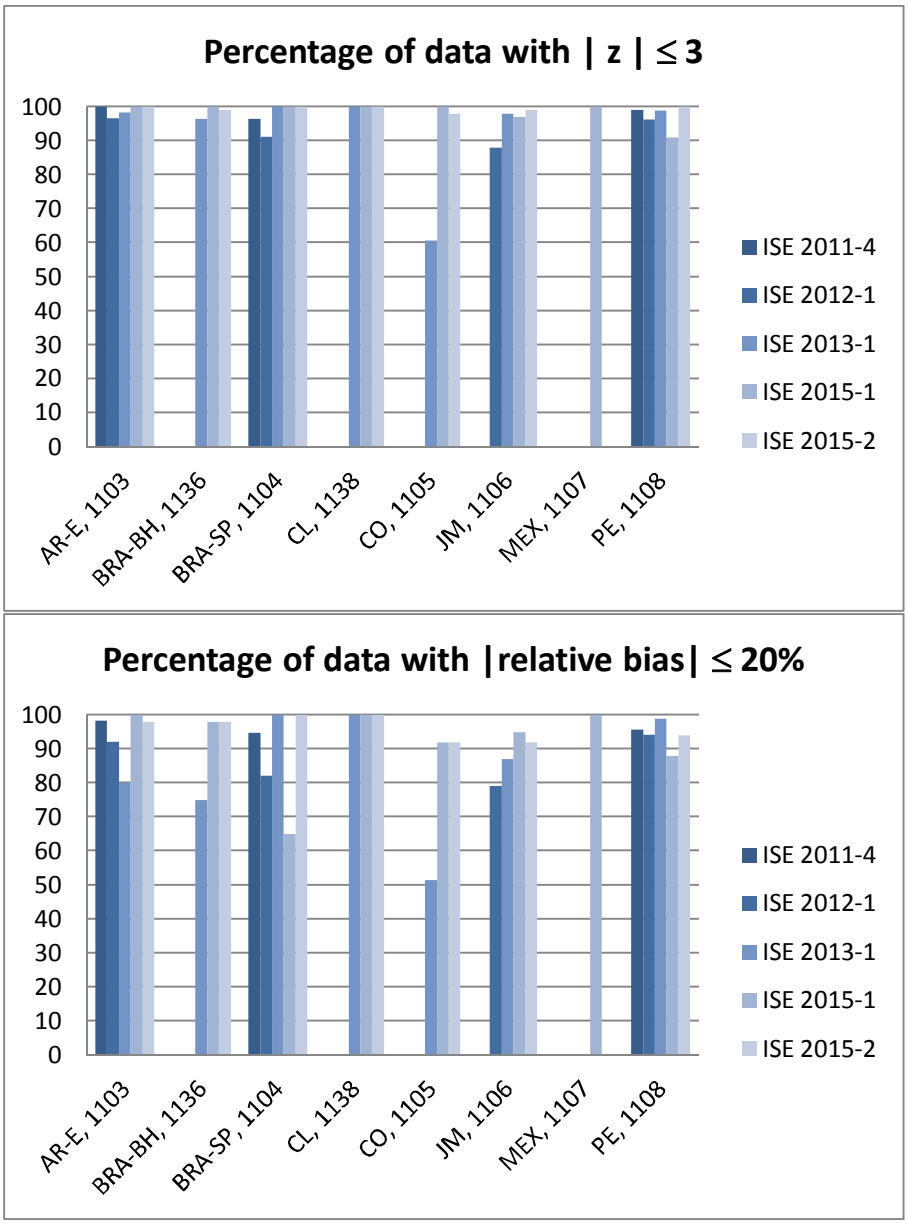

FIG. 3. Performance indications of the Latin America region participants in WEPAL-ISE.

3.2.4. Europe region participants

A very high percentage of results reported by laboratories of Czech Republic, Kazakhstan, Portugal, Slovenia and Uzbekistan had a $|z| \leq 3$ and a |relative bias| $\leq 20\%$ (see Figure 4). Both Hungarian laboratories, and also the laboratories from Romania, Russian Federation (Dubna) and Turkey had a measurable improvement of their performance during these four rounds, levelling them in the second round of 2015 with the four Europe region laboratories mentioned before.

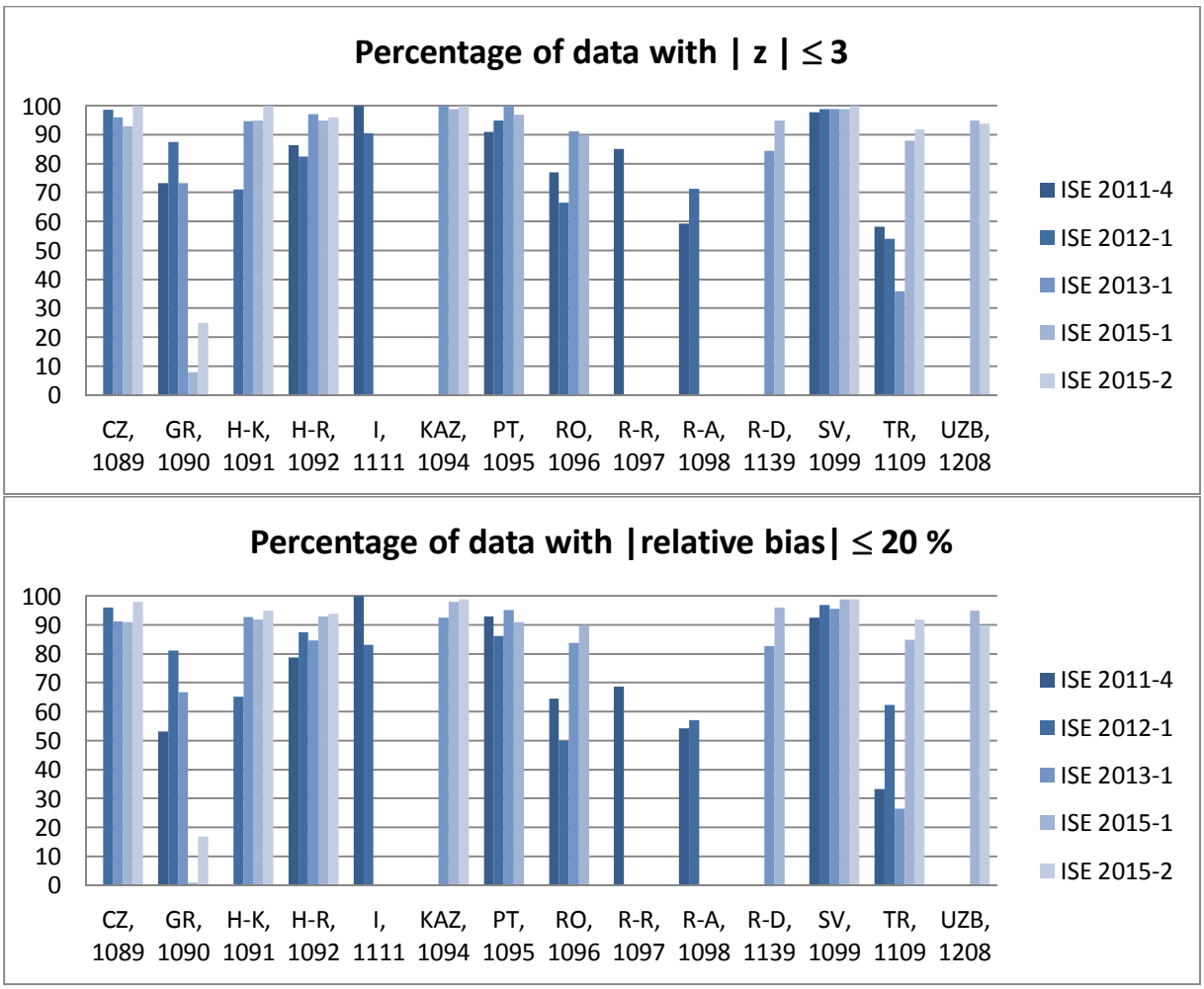

FIG. 4. Performance indications of the Europe region participants in WEPAL-ISE.

The laboratory in Greece utilizes a sub-critical assembly as neutron source with a relatively low neutron flux. Results can therefore be reported for only the major elements and the quality of the results has been degrading, with only between 0 and 25% of acceptable results in the 2015 rounds.

It can be seen from Figure 4 that the laboratories that regularly took part in the proficiency tests, with the single exception of the one from Greece, were able either to maintain a very high level of performance, or to reach it after improvements in their procedures and internal quality controls. Amongst these participants there is also no significant difference between the performance of the laboratories in the re-analysis round in 2012 (ISE 2012-R) and in the ISE round 2013-1. This emphasizes the effectiveness of the quality control and quality assurance in these laboratories.

Laboratories from other countries, such as Russia-R, Russia-A and Italy, only participated in one or two of the first rounds, which is not sufficient to ascertain reproducibility of their performance with time, either maintaining a high level of performance, or eliminating the sources of error that affect their degree of trueness.

3.3. INTERNATIONAL PLANT-ANALYTICAL EXCHANGE (ISE)

All performance indicators of the plant analyses (WEPAL-IPE rounds) are given in Figures 5-8 for the participants from the Africa, Asia-Pacific, Latin America and Europe regions, respectively. The performance indicators of the reanalysis of the 2011-4 samples (previously

denoted as '2012-reanalysis round' or '2012-R') are not shown in the figures as these analyses were not a fully 'blind' study.

As has been shown in Table 1 and 2, WEPAL grouped almost all element amount results in the 'Inorganic chemical composition' category, irrespective if the measurements were done by real-total techniques or by techniques using (incomplete) digestion. Consequently, the standard deviation of the median value of 'inorganic chemical composition' in the IPE round is often larger than in the 'real-total' category of the ISE round.

The value of the *z*-score is inversely proportional to the standard deviation, and bias values may be masked by the larger standard deviation too.

3.3.1. Africa region participants

A group of seven participants used NAA (both Algerian laboratories, Egypt, Ghana, Morocco, Nigeria, and South Africa). A further six participants (Cameroon, Democratic Republic of Congo, Madagascar, Sudan and Tanzania, which only participated in the 2012-R round) used various other techniques such as X ray fluorescence spectrometry, atomic absorption spectrometry, inductively coupled plasma emission spectrometry and others.

The seven leftmost participants in Figure 5 all used NAA. The two laboratories from Algeria consolidated the quality of their performance in the analysis of the plant samples, with relatively few results with $|z| \leq 3$. The percentage of data with | relative bias | $\leq 20\%$ by these laboratories belong also to the highest amongst this group of participants, though the percentage is smaller than for the soil samples, as could be expected (see above comment on WEPAL's methodology for the IPE rounds).

The performance of most other Africa region participants that use NAA increased significantly between WEPAL IPE 2010-3 and WEAL IPE 2013-1 as can be seen from Figure 5. In particular, the laboratories of Egypt, Morocco and Nigeria demonstrated their ability of reporting similar quality results as the laboratories from Algeria.

However, in several cases this improvement in performance was not sustained in the two 2015 rounds. The participants from Ghana and Morocco had a strong oscillation in performance in the WEPAL IPE 2015-1 round, and those from Nigeria and South Africa decreased their performance in both 2015 rounds. These findings stress the need for continuous improvement of internal quality control procedures.

The four rightmost participants in Figure 5 used other techniques, as described in Table 3. None of these laboratories participated in the 2015 rounds. A remarkable initial improvement in the performance of the participant from the Democratic Republic of Congo could not be sustained in the last round where it participated, 2013-1. Sudan participated in only one round, with promising results, but not trend can be ascertained.

Finally, remarkable differences can be observed for the performance of the laboratories from Congo, Ghana, South Africa and Cameroon in the rounds IPE 2012-R and IPE 2013-1, which were done with only three months interval. Apparently, the internal quality mechanism in these laboratories was not yet fully effective.

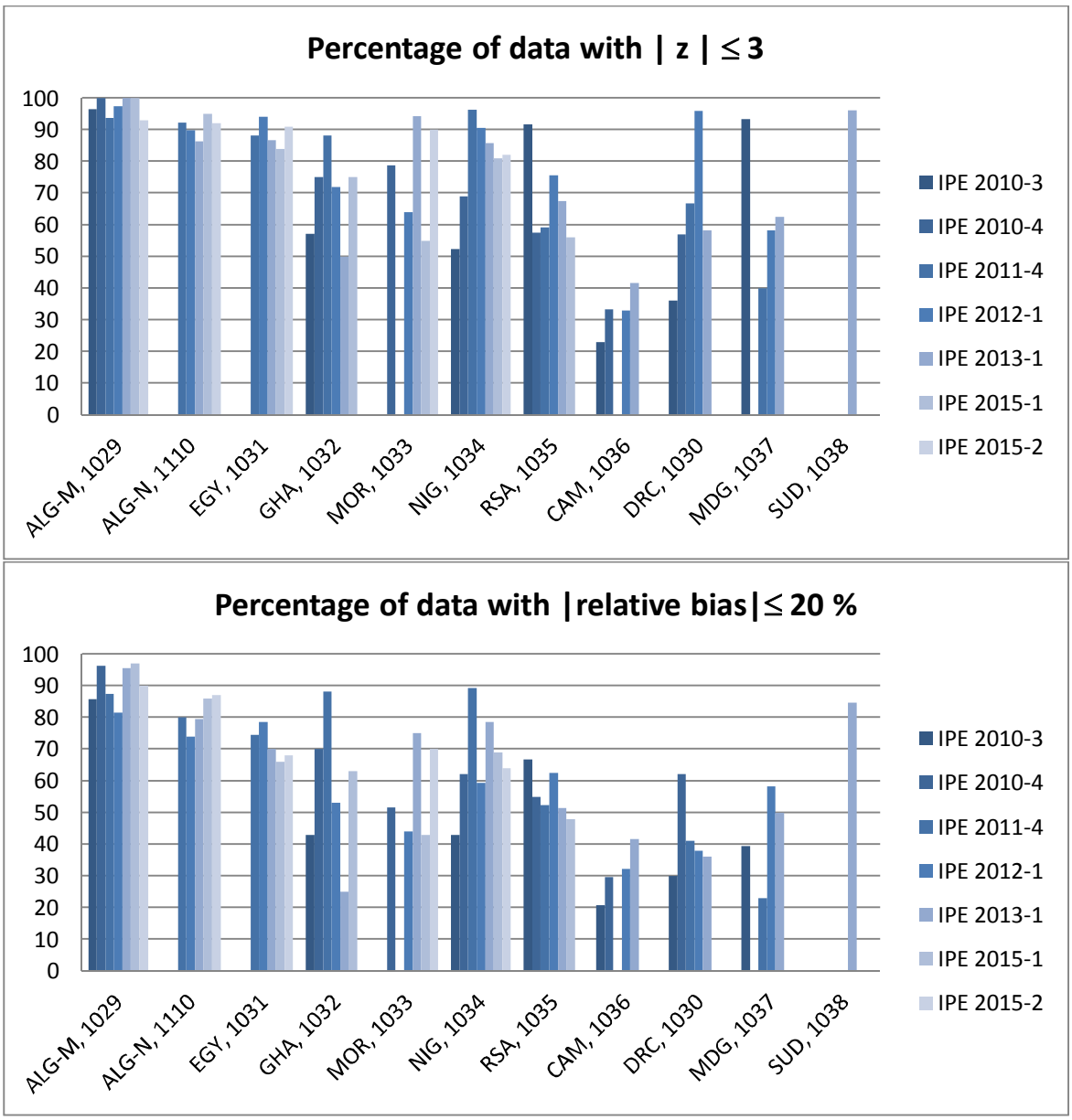

FIG. 5. Performance indications of the Africa region participants in WEPAL-IPE.

3.3.2. Asia-Pacific region participants

All Asia-Pacific laboratories used NAA for their trace element measurements (Figure 6). The laboratory from Syria consolidated and improved its good level of performance from the beginning of its participation in the 2011 round to 2013. However, it significantly decreased performance in the two 2015 rounds, particularly in the relative bias indicator.

A group of further eight laboratories participated in 2015 for the first time in the WEPAL rounds as well as in the feedback meeting. The laboratory from Vietnam only participated in the ISE rounds.

The participants from Australia, Bangladesh and Malaysia, which improved significantly between the IPE 2015-1 and 2015-2 rounds, obtained good results, with relatively few results with $|z| \leq 3$. The percentage of data with | relative bias | ≤ 20% by these laboratories belong also to the highest amongst this group of participants, though the percentage is smaller than for the soil samples, as could be expected (see above comment on WEPAL's methodology for the IPE rounds).

The three laboratories from Indonesia reached performance levels close to those of the best performing participants from the Asia-Pacific region. It can also be seen that probably corrective actions taken in Vietnam have been effective in improving the analytical quality significantly after round IPE 2015-1.

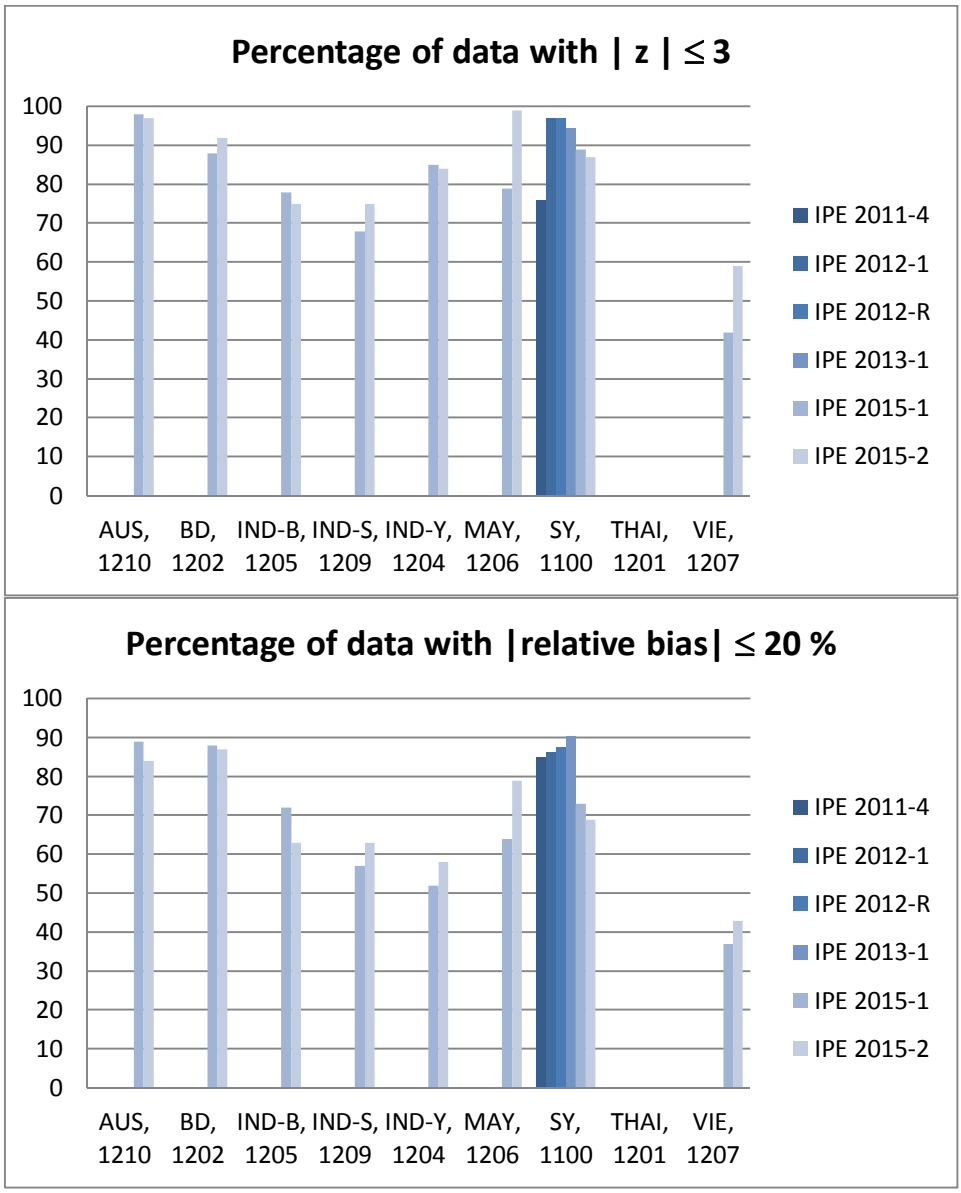

FIG. 6. Performance indications of the Asia-Pacific region participants in WEPAL-IPE.

3.3.3. Latin America region participants

All laboratories from the Latin America region use NAA for their trace element measurements (Figure 7). The results from the Jamaican laboratory were obtained both by NAA and ED-XRF. Argentina (Bariloche) only participated in the 2012-R re-evaluation and therefore is not shown in the figure. Brazil (São Paulo) did not participate in the IPE rounds.

The laboratories from Argentina (Ezeiza), Jamaica, Peru, Brazil (Belo Horizonte) and Chile demonstrated their skills of analysis plant material since a high percentage of data had $|z| \leq 3$, see Figure 7. The percentage of data with a | relative bias | ≤ 20 % was also here smaller than for the analysis of the soil samples, similar to what has been noticed in the results of the Africa and Asia-Pacific regions participants and in line with the a-priori expectations.

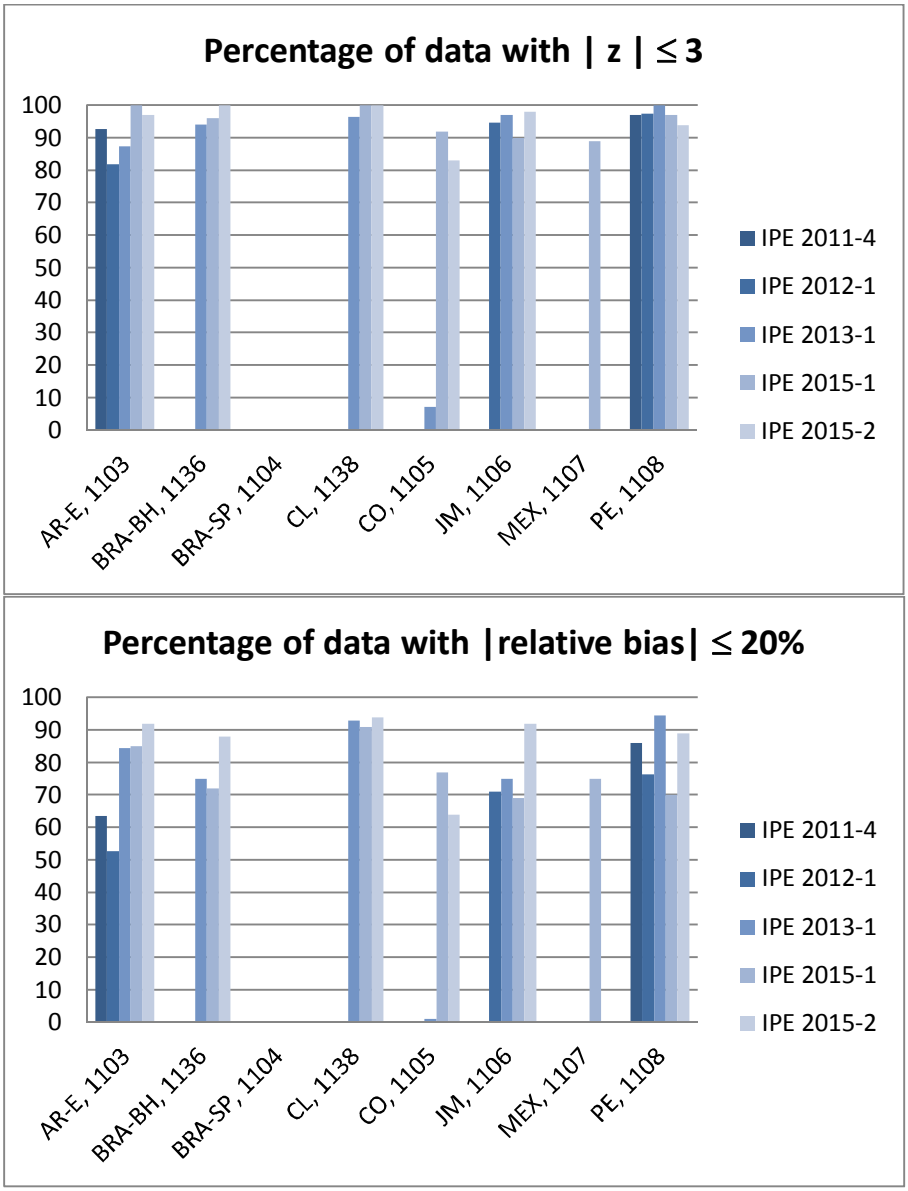

FIG. 7. Performance indications of the Latin America region participants in WEPAL-IPE.

The NAA laboratory from Colombia had initial difficulties with analysing this type of material, but showed remarkable improvement in the 2015 rounds, reaching levels close to that of the group of Latin America participants that performed best. It corroborates the performance in the ISE round and indicates that the analytical quality is well under (statistical) control.

The laboratory from Mexico had difficulties in getting the samples released by the customs and could only participate in IPE round 2015-1, showing a performance close to that of the participant from Colombia.

3.3.4. Europe region participants

The Europe region participants from Czech Republic and Slovenia and Syria reported in all rounds very large numbers of data with $|z| \leq 3$, see Figure 8. Similar good performance, but in fewer rounds, can be observed for the participant from Kazakhstan, whereas both laboratories from Hungary and those from Portugal and Uzbekistan demonstrated their improvement, reaching a good level, very close to that of the first group of laboratories mentioned above.

One participant from the Russian Federation (Dubna, R-D) had initially a similarly good performance, but could not keep it in the 2015-1 round, where it decreased to levels similar to that of the other two laboratories from the Russian Federation and the one from Romania, with percentages of results with $|z| \leq 3$ close to 60%. The improvement seen in the performance in the participant from Romania in the IPE rounds is markedly smaller than that observed in the ISE rounds. The laboratory from Italy also only participated in two rounds, showing some worsening of performance between 2011 and 2012.

This underpins the observation made for the performance in the analyses of the soil samples (Figure 4), emphasizing the presence of systematic errors in the analysis procedures. In fact, for the Russian Federation (Dubna) laboratory, there are indications that in the IPE 2015-1 round an interchange in reported results of two samples may have occurred.

As in the ISE rounds, the laboratory from Greece, which utilizes a sub-critical assembly as neutron source with a relatively low neutron flux, showed a marked degradation of performance.

The percentage of results with an | relative bias | ≤ 20 % is also for this group of participants smaller than in the WEPAL-ISE round, and the highest fractions (e.g., as obtained by the participants from Czech Republic, Kazakhstan and Slovenia in all rounds, and by the participants from Hungary, Portugal, Turkey and Uzbekistan in the more recent rounds) are of approximately the same level as the highest in the results of the Africa, Asia-Pacific and Latin America regions participants (see Figures 5 to 8).

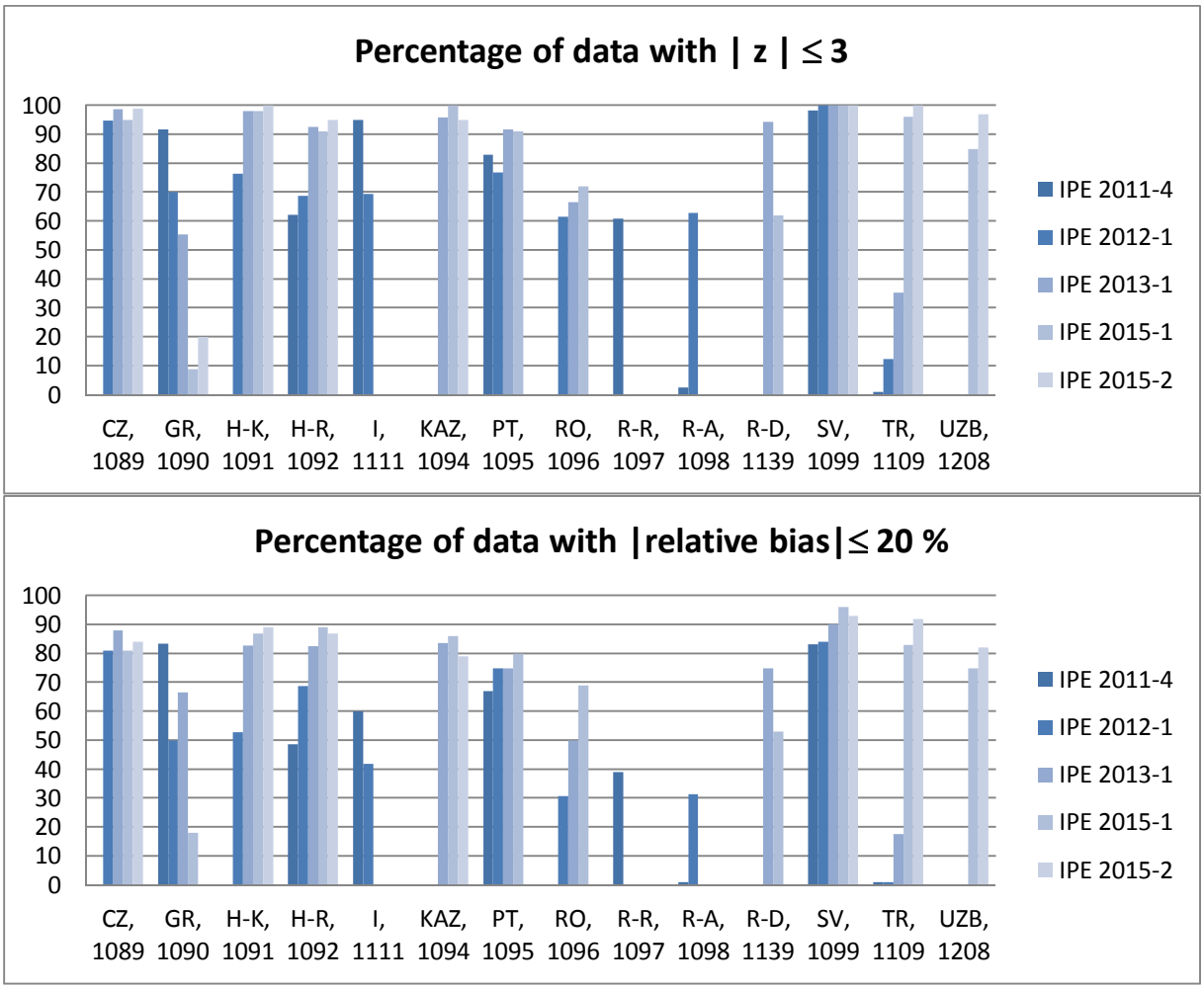

FIG.8. Performance indications of the Europe region participants in WEPAL-IPE.

4. FEEDBACK WORKSHOP EVALUATIONS

4.1. FEEDBACK WORKSHOP FOR PARTICIPANTS OF RAF 4022 PROJECTS, ANTANANARIVO, MADAGASCAR, SEPTEMBER 12–16, 2011

IAEA experts communicated intensively with the participants on pathways to analytical and even organizational improvement, the latter being mostly related to planning schedules. Participants and Experts identified several technical and organizational deficiencies that may have caused unsatisfactory performance in the Proficiency Testing rounds. These sources of error are summarized in Table 9.

Absence of internal quality control was identified as the principal deficiency as, if properly implemented, it reveals systematic errors including those from calibrations. Regarding the nuclear techniques, a major source of problems may be with insufficient validation of self-made software and underestimation of geometrical effects during irradiation and counting. It was noted that, especially in the non-nuclear techniques, big gaps exist in knowledge and experience on sample preparation, interferences and chemical matrix effects.

Several participants claimed that errors occurred due to deterioration of their reference materials used as calibrator in the relative method approach but, since internal quality control was lacking, this could not be demonstrated.

This inventory formed the basis for the assistance to the participants in setting priorities for the various steps in their action plans for improvement.

4.2. FEEDBACK MEETING FOR PARTICIPANTS OF RER 4032 PROJECTS, DELFT, NETHERLANDS, MAY 22-25, 2012

The concept of this feedback workshop was similar to the one in Antananarivo. During this workshop, the participants identified various (potential) sources of error in the analytical procedures for analysis of soil and plant material, respectively. The major ones are summarized in Table 10.

The participants in this workshop also referred to typing errors, errors in reporting such as wrong units and mix-up of data. In some laboratories deficiencies may be attributed to the renewal of staff which, having only a few years of practical experience, has still insufficient awareness on all potential sources of error in the practical execution of NAA.

Each of the participants devised an action plan for corrective actions to be taken to minimize the possibility of the type of deficiencies encountered during the proficiency testing round 2011-4 and 2012-1.

TABLE 9. MAJOR SOURCES OF DEFICIENCY WITH IMPACT TO THE ANALYTICAL PERFORMANCE OF PARTICIPANTS IN THE WEPAL ROUNDS 2010-3 AND 2010-4 UNDER RAF 4/022

Potential Source of Error	Remedy Suggested
Improper sample treatment and processing (absence of dry weight determination)	Adhere strictly to the instructions of the PT provider.
Insufficient insight in the quality of digestions methods applied.	Adhere to Experts' advice on sample digestion methods.
Inappropriate calibration of facilities.	Use of appropriate calibrators following instructions of the calibrator provider (e.g. (C)RM provider); Training of staff; Assessment of neutron fluence gradients and geometrical factors during counting; Attention to commutability of calibrators; Avoiding (certified) reference materials for calibration and preference for pure element standards, if applicable; Attention for blank and background correction; Calibration of balances, pipettes and other utilities; Introduction of performance tests.
Doubtful quality of calibrators and (certified) reference materials used; use of standards beyond expiration date.	Procurement of new calibrators, (certified) reference materials and standards; Storage and usage following the associated instructions to avoid contamination and deterioration and evaporation losses; Introduction of trend control charts to verify their stability.
Inadequate in-house training and qualification, resulting in insufficient awareness on sources of error and sources of uncertainty of measurement.	Fellowship training and expert missions.
Malfunction of instruments and inability to acquire spare parts or external maintenance.	Apply for assistance by the IAEA via regional collaboration; Expert missions.
Inadequate internal quality control throughout the entire analytical procedure, including the assessment of blank and background contributions.	Use of internal quality control materials (such as reference materials and blanks) with each batch of samples analysed; Formulation of acceptance criteria and control charts for trend analysis
Organization inadequacies, insufficient supervision and poor planning of the conduct of work, resulting in insufficient time for cross-checking of the final results and inappropriate reporting; as well as the internal distribution of the PT reports.	Implement a procedure for planning of analyses, based on reverse planning from the date of reporting; Assuring supervision and cross-checking of results prior to reporting; Adhere to PT provider's instructions for reporting; Contact the IAEA if PT reports are not received within 1 month after the provider's deadline date.
Communication gap between participants, IAEA and PT provider related to the receipt of samples and PT reports.	Contact the IAEA if samples are not received within 2 weeks after the official dispatch date and if PT reports are not received within 1 month after the provider's deadline date.

TABLE 10. POSSIBLE SOURCES OF ERRORS ENCOUNTERED IN PROFICIENCY TESTS WEPAL ISE/IPE 2011-4 AND 2012-1

Soil samples		
Sample preparation	Irradiation	Analysis
Too few reference materials Possible contamination Verification of balance functioning	Improper channel temperatures Range of elements limited by low neutron flux Interference from fast neutron reactions Inadequate accounting for axial and radial flux gradients No simultaneous irradiation of samples, calibrators and flux monitors No re-assessment of *f* and alpha values after new reactor core setup for k_0 NAA, and of their stability with reactor operation cycle/burn-up Irradiations may have been done in non-calibrated irradiation channels	Geometrical differences between test portion and calibrator in close-to-detector positioning Certain isotopes like ^{85}Sr and other well-known spectral interferences are difficult to fit during spectrum evaluation Improper or absent calibration for neutron flux, detector efficiency or transfer time Incompatibility of detector parameters if two or more Ge detectors are used Internal quality control materials (reference materials and blanks) not included in the same run as the PT samples
Plant samples		
Sample preparation	Irradiation	Analysis
Sample sizes too small Verification of balance functioning Contamination from labels External contamination of Na Inadequate homogeneity Inadequate performance of relative standardization Dry mass determination on same samples as analysed	Range of elements limited by low neutron flux Interference from nuclear reactions involving P Inadequate accounting for axial and radial flux gradients No simultaneous irradiation of samples, calibrators and flux monitors No re-assessment of *f* and alpha values after new reactor core setup for k_0 NAA, and of their stability with reactor operation cycle/burn-up Volatilization of, e.g., Br and Hg, during irradiation in high flux reactors Irradiations may have been done in non-calibrated irradiation channels	Geometrical differences between test portion and calibrator in close-to-detector positioning Improper calibration for neutron flux or k factors Incompatibility of detector parameters if two or more Ge detectors are used Internal quality control materials (reference materials and blanks) not included in the same run as the PT samples Blank not accounted for (e.g., Cr interference) Unanticipated background radiation (e.g., ^{60}Co from stainless steel)

4.3. FEEDBACK WORKSHOP FOR PARTICIPANTS OF RAF 4022 PROJECTS IN TUNIS, TUNISIA, JUNE 4-8, 2012

Participants presented the status and outcome of the action plans, drawn during the first feedback workshop in Antananarivo (see above, paragraph 4.1). The results of the PT rounds 2011-4 and 2012-1 were discussed in view of these action plans. The main conclusions of this workshop were:

- The concept of quality control/quality assurance has improved significantly compared to the level at the 2011 Feedback Meeting in Madagascar.
- Most participants have become aware of the necessity of further improvement of their analytical performance is (with respect to degree of trueness) in view of the internationally accepted state of the practice of their analytical techniques.
- Acceptance criteria in internal quality control were not always unambiguously defined, and even so, in most cases results are still released if these criteria are not met.
- Several participants argued again that their deficiencies are due to a deterioration of the quality of their calibrators (CRM's, pure element standards); however still without a solid ground for this hypothesis and without excluding other potential (internal) source of error.

Table 11 contains the main sources of error that might explain the deficiencies shown in Figures 1 and 5 and the recommendations for improvement, as discussed during this meeting.

The workshop was completed with new action plans taking into account the relevant expert's recommendations given during the meeting and summarized above in Table 11.

TABLE 11. POTENTIAL ANALYTICAL SOURCES OF ERROR AND REMEDIES

Potential Source of Error	Remedy Suggested
Use of small sample masses	Use minimum sample mass at the same level as the minimum mass recommended in the certificate of the RM applied for internal quality control
No correction for moisture content	Adhere strictly to the instructions of the PT provider
Insufficient insight in the quality of digestion methods applied.	Adhere to IAEA Expert's advice in the 2011 Feedback Meeting on sample digestion methods; if necessary contact this IAEA expert for help
Inappropriate calibration of facilities	Use calibrators following instructions of the calibrator provider (e.g. (C)RM provider) Assessment of neutron fluence gradients and geometrical factors during counting; Attention to commutability of calibrators (specially for XRF and techniques needing digestion); Attention for blank and background correction; Calibration of balances, pipettes and other utilities; Introduction of performance tests.
Missing suitable calibrators and (certified) reference materials used	Ask IAEA's assistance in procurement of new calibrators, (certified) reference materials and standards; Storage and usage following the associated instructions to avoid contamination and deterioration and evaporation losses; Introduction of trend control charts to verify their stability
Inadequate in-house training and qualification, resulting in insufficient awareness on sources of error and sources of uncertainty of measurement	Expert missions and topical workshops/conferences
Inadequate internal quality control throughout the entire analytical procedure, including the assessment of blank and background contributions.	Formulation of acceptance criteria and control charts for trend analysis, and adhere to these criteria
Insufficient time for cross-checking of the final results and inappropriate reporting; as well as the internal distribution of the PT reports. Wrong units for reporting data, exchange of results and sample codes.	Assuring supervision and cross-checking of results prior to reporting

4.4. FEEDBACK MEETING FOR PARTICIPANTS OF THE RAF 4022, RLA 0037, RAS 1018 AND RER 1007 PROJECTS, VIENNA, AUSTRIA, MAY 27–31, 2013

Participants discussed – but now in a broader context - the technical conduct of their techniques, their own evaluations of the PT results and their own assessment of the effectiveness of their various action plans over the period 2010/2011-2013. Metrological, analytical and organizational aspects were discussed. It all resulted in the recommendations summarized in Table 12.

Participants presented new action plans for further improvement. They recommended the IAEA to facilitate their participation in a new series of WEPAL ISE and IPE round(s) to be conducted in the first quarter of the year 2015 (i.e., WEPAL ISE/IPE 2015-1).

TABLE 12. RECOMMENDATIONS TO PARTICIPANTS FOR FURTHER IMPROVEMENT

Concept	Recommendations
Metrology	To get more familiar with the definitions of trueness, accuracy, precision, and related terms in International Vocabulary of Metrology – Basic and General Concepts and Associated Terms, 3rd edition (VIM3). Only SI units should be used, and IUPAC definitions should be followed for quantities, units and symbols. Results from the IAEA PT project cannot be used for method validation since the property values of the measurands are not known. k_0-NAA users should use the data from the official IUPAC database (IUPAC Technical Report published in *Pure & Appl. Chem.*76(10), 1921-1925, 2004). Synthetic multielement standards (SMELS) materials should be analysed for validation. Non-nuclear techniques should assess the degree of commutability of reference materials for calibration and the real samples.
Analytical	It is the laboratory's responsibility to assure that the test portion is representative for the PT sample, and to apply, if relevant, additional homogenization thereof. Non-nuclear techniques using digestion should consider using HF for more complete dissolution of all elements. Internal quality control can be done with any type of material of adequate homogeneity and stability. Laboratories must be aware of neutron flux gradients and energy distribution differences. Shewhart control charts presume a normal distribution of the data. Commercial control charts often produces misleading results. Trend charts should be used for variables that are not statistically correlated, like temperature, humidity, or instrument resolution.

TABLE 12. RECOMMENDATIONS TO PARTICIPANTS FOR FURTHER IMPROVEMENT (cont.)

Concept	Recommendations
Organizational	Documentation provided by the PT or (C)RM provider should be studied and followed without deviating from the instructions for e.g., drying and use of the minimum test portion mass. Moisture content should always be determined on the day of preparation of the samples, preferably by drying to constant mass. A criterion should be applied, e.g., 1 % variability is acceptable. A safety component for reporting time should be included in the planning to avoid reporting on the final day**.** Final reports should be well and independently checked (i.e., by a second person) for e.g. typing errors before submission to the client (including the PT provider). Reporting should be done in the units, requested by the provider to facilitate the analysis of the data in a timely manner The value behind the '+-' sign should be specified, e.g., (standard deviation of a single result, standard deviation of the mean, confidence interval (CI) and its confidence level – usually 95 %, combined uncertainty (coverage factor k=1), expanded uncertainty (coverage factor should be specified, usually k=2. For a large series of data, it should be checked that the data follow a normal (Gaussian) distribution before calculation the arithmetic mean and standard deviation. The Kolmogorov-Smirnov test is suitable for this purpose. A comparison of the arithmetic mean and the median provides the first estimate whether the data follow a normal distribution or whether they deviate from it.
General	Full commitment is needed from the reactor management for running ILC smoothly (four WEPAL rounds per year), and dates of the programme (irradiation-measurements-reporting) should be well defined and agreed on to avoid any surprises. The effectiveness of corrective actions should be tested by re-analysis of samples. If major deficiencies are found in the laboratories' results, it is important that clients are informed.

4.5. FEEDBACK MEETING FOR PARTICIPANTS OF THE RAF 4022, RLA 0037, RAS 1018 AND RER 1007 PROJECTS, DELFT, NETHERLANDS, AUGUST 3 – SEPTEMBER 4, 2015

4.5.1. Discussion of the results in the 2015-1 and 2015-2 rounds

A representative from WEPAL, Ms. Van Vark, presented a comparison between the mean value reported by the NAA laboratories, and the mean values of other participants in the WEPAL rounds. There is high degree of equivalence for many elements, both in the mean values as well as in the coefficients of variation. It was also noticed that the between laboratory variability of the

IAEA participants was for some elements significantly larger than the between laboratory variability of all participants in the ISE respectively IPE programmes (see the data in bold in Tables 13 and 14). This is, at a first sight, is remarkable as one would expect the opposite for a group of laboratories applying the same analytical technique. Also large differences were observed for the mean values of Ca, Cr and Sr in IPE results (bold in Table 14).

The relative large variations in Al and Mg may be attributed to differences in corrections for the interferences resulting from the well-known nuclear reactions with thermal and epithermal/fast neutrons in the P-Si-Al-Mg cluster. The result variations between NAA laboratories for Sr values in plant matrices are not surprising either, and may result from difficulties in the fitting of the 511-514 keV doublet in the gamma-ray spectrum. All participants reported Cr values in the IPE materials that are at least 15 % higher than the mean values. In several cases, the z-scores remain in the acceptable range because of the high standard deviation of the consensus value. The cause of this deficiency may either to be found in insufficient correction for blank values (Cr is a notorious impurity in plastic vials) and, most likely, in a mean value that is lower biased due to contributions from techniques using digestions methods, incomplete for Cr.

TABLE 13. COMPARISON OF THE RATIO OF THE MEAN VALUES AND THE RATIO OF THE COEFFICIENTS OF VARIATION FOR ELEMENTS REPORTED IN ISE 2015-1 AND ISE 2015-2 FOR IAEA FACILITATED PARTICIPANTS AND ALL OTHER PARTICIPANTS; THE NUMBERS OF PARTICIPANTS IS INDICATIVE [13].

Element	Number of participants		IAEA/WEPAL	
	N-IAEA	N-WEPAL	Mean	CV
Al	15	38	1,003	**1,738**
As	28	47	1,006	0,992
Ba	20	42	0,989	**1,599**
Br	21	31	0,977	0,936
Ca	18	41	0,990	**1,597**
Ce	27	38	1,010	1,007
Co	30	46	1,002	0,790
Cr	28	51	1,024	0,888
Cs	28	33	1,001	0,878
Ga	5	17	1,048	0,941
Fe	30	56	1,002	1,229
K	27	52	1,002	1,448
La	30	42	1,009	0,932
Mg	10	33	**1,421**	**2,307**
Mn	18	44	0,991	**1,338**
Na	31	51	1,005	0,958
Nd	16	23	1,025	1,048
Rb	27	42	1,014	**1,420**
Sb	26	34	0,987	0,926
Sc	31	37	0,996	0,969
Se	3	5	**1,712**	0,621
Sr	7	26	1,050	**1,460**
Th	29	37	0,995	0,915
Ti	14	36	0,980	1,267
U	21	28	0,988	0,897
V	15	34	1,002	1,070
W	6	10	1,036	0,530
Zn	25	50	1,010	**1,493**
Zr	9	23	1,030	**1,458**

TABLE 14. COMPARISON OF THE RATIO OF THE MEAN VALUES AND THE RATIO OF THE COEFFICIENTS OF VARIATION FOR ELEMENTS REPORTED IN IPE 2015-1 AND IPE 2015-2 FOR IAEA FACILITATED PARTICIPANTS AND ALL OTHER PARTICIPANTS; THE NUMBER OF PARTICIPANTS IS INDICATIVE [13].

Element	Number of participants		IAEA/WEPAL	
	N-IAEA	N-WEPAL	Mean	CV
Al	8	23	1,252	0,393
As	12	31	1,063	0,923
Ba	10	24	**2,073**	**1,350**
Br	24	24	1,000	1,000
Ca	20	128	1,023	**1,337**
Cl (as Cl)	13	33	0,975	1,157
Co	22	46	1,109	0,989
Cr	18	44	**4,279**	0,995
Cs	15	15	1,012	0,936
Fe	26	129	1,043	1,097
K	29	142	0,998	1,252
Mg	15	129	1,022	1,968
Mn	17	124	1,009	0,942
Na	25	89	0,952	0,889
Rb	24	27	1,003	0,963
Sb	14	23	1,162	0,704
Sr	11	24	**11,538**	1,199
V	8	20	0,951	0,991
Zn	28	133	0,993	**1,348**

4.5.2. General comments from the participants

The participants made several observations regarding the steps taken since the previous proficiency testing rounds and the changes introduced in the laboratories. The implementation of the action plans drafted in 2013 was discussed and their effectiveness was evaluated. Many of the facilities with lower performance in 2010-2013 brought several of the lessons previously learned into daily practice. Some of the most important points discussed were:

- Several laboratories that participated in the previous IAEA facilitated ILC rounds (2010-2013) explained their significant improvements from the implementation of a quality system in the laboratory. Reference materials and blanks are now routinely processed and the documentation of work has been improved. It all also contributes to the improvement in performance of less experienced analysts;
- All laboratories estimated the moisture content of the materials and applied corrections to the mass analysed;
- Root cause analysis of deviating results was done by several laboratories (see below, country specific comments);
- This need for simultaneous analysis of internal quality control samples, and the verification of compliance with own acceptance criteria was mentioned again as a *conditio sine qua non* for an analytical laboratory;
- Some laboratories reported results that are close to the detection limit, and may effect to high |z| scores. It has been suggested to refrain from reporting results for which the relative uncertainty of measurement – the counting statistics may serve as first estimate - exceeds 20 %. This is, e.g. often the case in the results of Sr on basis of the radionuclide 85Sr;

- Similarly, if results of replicates differ more than 25 %, no mean value should be reported;
- The minimum mass to be used in ILC studies should not be underestimated. ILC samples are probably less well homogenized than certified reference materials which often are recommend to be used in masses > 200 mg;
- Participants agreed that a rigid final control on results reported is very important. Several laboratories attributed their less good performance to errors not observed when the results were submitted. An obvious one is the reporting in dimensional units other than requested by WEPAL. Also the interchange of reported results occurred;
- Element calibration is sometimes done using single element standards (sometimes as -solutions) or mixes of elements. Preparation of such calibration test portions of adequate degree of accuracy is not trivial; besides, sometimes single element standard solutions have expired. It cannot be excluded that this is one of the sources of deviating results;
- There is still need for information on how to deal with nuclear reaction interferences (e.g., Si-Al-Mg) and spectral interferences (e.g., Nd-Cr);
- Pelletized plant samples may break during the irradiation and transport procedures. It may cause unanticipated geometrical errors during irradiation and counting. It may be better not to pelletize plant samples;
- Automatic peak search/fitting can sometimes provide inaccurate peak areas. It is also recommended to check peaks manually and not rely entirely on the software, although this can be very time consuming;
- Having a good knowledge of all the elements that may be in the blank can improve the results;
- WEPAL cannot report zeta-scores or En-scores because participants are not asked reporting uncertainties of measurement. However, laboratories should convert the WEPAL z-scores into zeta scores as part of the internal evaluation of their performance. In this respect, graphical presentation would be helpful;
- Another option for self-assessment is the calculation of the relative difference of the measured value and the reference value, i.e., the mean value as calculated by WEPAL from all results reported. Although such differences may follow the z-score, the value of systematic differences may ease the search for the cause of deviation, especially if this value is more-or-less the same for more than one element;
- Each laboratory can conclude on its performance on the basis of its own pre-defined acceptance criteria for z-scores and derived En scores or zeta scores.

4.5.3. Feedback from individual participating laboratories

In addition, the following country-specific feedback was given on the performance in the 2015 rounds and the lessons learned:

- Argentina: The laboratory obtained better results, especially for the plant materials after changing its procedure into making the dry mass determination on the same day as weighing out the samples for measurement;
- Slovenia: The laboratory obtained better results by increasing the sample mass for the ISE samples from 150 to 220 mg and for the plant samples from 200 to 250 mg;
- Morocco explained that the results until 2013 were obtained by standardization using the relative method and that the 2015 results were obtained after implementing the k_0 method (k0-IAEA software). The laboratory is aware that the calibrations have to be improved, such as for f and alpha, and for the peak-to-total curve of the detector. In addition, the peak fitting has to be further optimized. It all explains the weaker performance in the

2015-1 rounds compared to the 2013-1 rounds, but improvement is again visible in the 2015-2 rounds;

- Bangladesh: The laboratory realized that there is a need to increase the number of elements that can be reported in plants. There was an increase from round 1 and 2 but there is still a need to improve. The small number of certified element mass fractions in plant certified reference materials (CRMs) is a limiting factor therein. The laboratory therefore studies the re-introduction and routine use of the k_0 method;
- Thailand: The laboratory intends to move from relative method to the k_0 method as the consumption rate of reference materials is too costly;
- Uzbekistan: The laboratory has joined the programme recently. Although the performance was acceptable, especially for soil samples, the laboratory still sees need and room for improvement. As an example, a more rigid final control before release of reports has to be implemented since almost all of the outliers were the result of simple mistakes in recording data. Currently the relative method is used but the k_0 method may be introduced within the next 1-2 years, also to reduce the consumption of expensive CRMs. Eventually the laboratory wants to be ISO/IEC 17025 accredited;
- Hungary-K: The laboratory identified problems due to cross contamination (Zn), Kayzero for Windows software flaws (wrong lines assigned for 140La) and a contribution from the blank (Al);
- Indonesia-S: The laboratory main problems deal with the measurement of short half-life radionuclides where multi-comparators are used. Also the quality of the element standards may be a reason for deviating results. The laboratory intends repeating the analyses at a lower reactor power and to apply longer counting times. The laboratory has tried to use k_0 in the past but found that k0-IAEA software operates as a 'black box' and that the Kayzero for Windows software is limited by the number of sources available for the peak-to-total curve determination. The laboratory also needs an oven with calibrated thermometer for better moisture determination;
- Indonesia-B: The laboratory reported a thorough root cause investigation to the source(s) of results with a high |z| value, especially since results of CRM analyses did not indicate major problems. The WEPAL samples used were probably too small (25 mg); the reason for the deviating potassium values is still under investigation. This laboratory also hopes to be able to use its own reactor as it has fast pneumatic rabbit systems enabling measurement of short half-life radionuclides. They will apply independent quality control methods to check any questionable elements before reporting. This laboratory is planning to develop their own k_0 software as part of a national programme;
- Mexico: The laboratory needs to pay attention to analysing short lived radionuclides. They have pneumatic facilities but due to pressure of time they were not able to use them. They plan to start using the k_0 method. The good performance in the 2015 rounds was largely due to the experience of the analysts but it is recognized that there is a need to document the procedures so that new analysts can also perform well. This is a component of the accreditation for ISO/IEC 17025. Improvements are being made in the irradiation timing system to provide more accurate timing data for NAA;
- Malaysia: The laboratory had a big improvement in the 2015-2 round compared to the 2015-1 round. It was recognized that the Si-Al-Mg inference correction needs more attention. For other elements in plants it was found that the counting time had to be increased to improve detection limits. It was also realized that if there is a question about some elements, they should not be reported. The lab is involved in a national food programme so it is important to maximize the number of elements that can be reported accurately. They have installed k0-DALAT software in the past but the person who was

the local expert left the organization and it will need to be re-initiated. Their objective is to increase the throughput of the lab to meet customer needs;

- Vietnam: The laboratory has indicated that the many results with high |z| values probably are related to insufficient attention before the release of the data. As such, the laboratory wants to improve four things: (i) Designing a procedure for checking the results including uncertainty, and establishing criteria for the reporting or rejection of results; (ii) Paying more attention to background determination, (iii) Checking the compatibility of the results obtained on their two detectors and (iv) More attention to the procedure for moisture determination. Staff will be retrained so that they can operate independently. Once all actions have been implemented, they will reanalyse the previously collected data.

Some laboratories value their participation in the IAEA facilitated ILC programme as an indication that they are performing at a peer international level. This participation can also be seen as an advantage in marketing NAA services to potential clients. The participation can be used a means to publicize the work of the NAA lab, both internally within an organization and externally. Such exposure may also attract young scientists.

4.5.4. Presentation of the outcome of ILC testing

The IAEA's objective for implementing this project of ILC testing is to demonstrate the analytical performance of NAA laboratories in Agency's Member States and to provide a tool for continuous improvement by finding the cause of non-conformities and implementing effective approaches to eliminate them. It has been accepted by participants to express the performance by the percentage of data reported in the analysis of ILC samples for which the absolute value of the z-score is less than 3, $| z | \leq 3$.

Participants revisited the objectives of this project. Many laboratories obtained better results after bringing into the practice the lessons learned during the feedback meetings. The question 'when is good good enough' remains to be answered by the laboratories themselves in view of their mission and end-user requirements. Results with z-scores $|z| > 3$ will always occur for statistical reasons, irrespective if a laboratory has implemented a quality management system and is accredited. Nonetheless, reporting such results may have dramatic economic and social consequences as it may lead to wrong conclusions and related decisions. The participants therefore decided to describe their performances by three categories:

1. Metrologically satisfactory performance, 'excellent' for those laboratories reporting > 90 % of their data with $|z| \leq 3$;
2. Metrologically less satisfactory performance, 'average', for those laboratories reporting >70% and ≤ 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 ≤ 70% of elements with $|z| \leq 3$. Major improvements are needed to reach an acceptable level of performance.

All laboratories need to have mechanisms in place for checking the validity of the reported results, but laboratories in the second ('average') and third category ('poor') need to be restrictive in providing analytical services as long as they did not eliminate the cause of the deficiencies and provided independent evidence of a performance equivalent to as described above in category 1.

5. LESSONS LEARNED

Participants and IAEA were confronted during this proficiency testing project with the laboratory organization, calibration, quality control and quality assurance of chemical analysis. The individual lessons learned can be found in the country reports in Annex IV. A summary of the most common ones is given below.

5.1. LABORATORY ORGANIZATION

The importance of a safe planning of the analyses to ensure that reporting date is done well on time before the requested deadline. This may also require a timely communication with reactor management in view of the reactor's availability and possibly even a priority setting policy at the executive management level.

The documentation provided by the PT provider needs to be carefully read, and practitioners need to adhere to recommendations/requirements on test portion preparation. The same applies to the final reporting, such as the requested format. Independent double checking of (intermediate) results is indispensable as (human) typing, transposing errors and wrong dimensional units cannot always be avoided.

Laboratories could implement procedures for trial analysis in cases where materials of unknown or unfamiliar matrix composition have to be analysed. Thus, problems that were sometimes reported such as too short irradiation or counting times, or too small sample masses, may be avoided.

Laboratories also have to decide on the modus operandi of the participation in PT testing. Will the analyses be done under everyday conditions, in multi-fold and/or under best measurement conditions?

Laboratories also learned the importance of having a policy and procedures how to act if results from PT testing indicate major deficiencies, especially on the possible recall of results previously reported to others, e.g. customers. Non-conformance management including (root) cause analysis, remedial and corrective actions and verification of their effectiveness is still absent of at best in its infanthood in many laboratories. This may be partly due to the inability of systematic trouble shooting (see also below).

5.2. SAMPLE AND CALIBRATOR PREPARATION

Contamination control received ample attention in the discussions. It was outlined that it is essential to pay strict attention to blank measurements, including all parts of the analysis process, vials, pressed pellets, etc., and perform background correction. Not all participants have separate laboratories, weighing rooms and balances for soils, environmental and biological samples. It was recommended to have laboratories divided by levels of elements to be determined. If not possible, then it is advisable to keep records of the last time a given sample type was treated in the lab.

The non-nuclear techniques face the importance - and difficulty - of commutability of sample and calibrator. New calibrators such as (certified) reference materials were made available through the IAEA, and participants have applied better calibrations. It was emphasized that masses are to be used in agreement with the specifications in the certificates, and not to take smaller amounts for economic reasons.

Some participants were not aware at the start of the project of the importance of moisture correction and learned how to dry samples and apply this routinely.

Several recommendations have been given during the feedback workshops for attaining complete digestion of materials for analysis by non-nuclear techniques such as AAS and ICP-OES, and associated dilution and contamination control. For soil and similar samples it may be necessary to use HF in the mixture of acids for decomposition to be able to arrive at the total contents of elements. Some samples may contain significant quantities of Fe, Mn, Cr, etc. in the silicate fraction. Alternatively, the results may be reported as a fraction extractable with HNO_3, HCl, etc. Boric acid is used after the HF treatment to get rid of HF to prevent damage to glass ware, instrument, etc.

Very few participants had procedures implemented how to act if the homogeneity of the samples is not specified at the level of the test portion. Practical approaches for testing the performance of balances in-between calibrations have been demonstrated and were implemented by participants.

5.3. INTERNAL QUALITY CONTROL

The need for internal quality control in every batch of samples analysed was one of the most valuable lessons learned for many participants, as stressed in the feedback meetings. Test portions of materials with known property values should be used, as well as blanks; acceptance criteria need to be specified and trend analysis may be considered.

Participants learned about commutability between reference materials and real samples, though less important for NAA but quite relevant for XRF and techniques requiring digestion. The need for neutron flux gradient assessment by sandwiching samples and flux monitors in every batch of samples to be irradiated, the risk of errors during counting close to the detector's end-cap and (for techniques with a digestion step) the assessment of recovery were other new lessons for many. Neutron flux parameters for use with the k_0 method of standardization have to be determined in situ. The parameters f and alpha require carefully examination, especially in multipurpose experimental reactors. This may not be necessary for reactors with stable parameters, such as SLOWPOKE and MNSR type reactors. The most common neutron flux monitors in the k_0 method, Zr+Au, may be replaced by other monitors if this appears more suitable in a particular reactor and/or irradiation conditions.

Still several steps may have to be made. Commutability also requires attention in quality control. Trend analyses can be done using control charts, but require a realistic assessment of the measurement uncertainty. Measurement uncertainty and standard deviation – i.e., an estimate of precision – are different metrological concepts but are very often commonly confused, thus resulting in erroneous interpretations and conclusions.

Recoveries of digestion methods are an important factor to be determined in 'wet-chemistry' analytical methods, such as AAS, ICP-MS, (TR)XRF and also in RNAA.

5.4. METROLOGY AND QUALITY ASSURANCE

The personnel's metrological know-how of the analytical technique and method procedures require careful attention, especially in groups with a regular turnover and/or rejuvenation of staff. It is essential that practitioners are fully familiar with their measurement equations, including all correction factors. This is critical both for implementing quality assurance measures to minimize and to monitor the possible occurrence of errors (and to have acceptance criteria), as well as for

systematic trouble-shooting once deficiencies are observed during the quality control or in, e.g., PT testing. Moreover, it contributes to a realistic estimate of the measurement uncertainty.

It became evident during the feedback workshops that many participants were not familiar with the methods required to perform systematic trouble shooting in case of results outside their own acceptance criteria. Self-validation opportunities using multiple gamma-rays and multiple radionuclides formed from the same chemical element have been explained.

Many participants gained from discussions during the feedback workshops on sources of contamination, element loss (e.g. during digestion), spectral interferences and matrix interferences. Errors resulting from extrapolating point-source based photopeak efficiencies toward voluminous source efficiencies have been explained. The k_0 method of standardization gained much attention during the discussions. Participants embarking on this method learned that errors may occur if measurements close to the detector's end cap are done without appropriate coincidence summing corrections.

Control charts for inspection of trends have been implemented by several participants following the Expert presentation(s) in the workshops. Basic principles of statistical evaluation have been discussed, including the conversion from *z*-scores into *zeta*-scores in order to account also for the laboratory's measurement uncertainty, and the use of median values from replicate measurements rather than the arithmetic mean value.

Participants were introduced to the latest internationally accepted metrological concepts such as trueness, accuracy, uncertainty of measurement, precision, sensitivity and limit of detection; but also with concepts calibrators, standards, comparators, (certified) reference materials and Standard reference materials.

5.5. SUSTAINABILITY OF EXTERNAL QUALITY CONTROL

The participants were questioned during the feedback workshop that took place in Vienna in May 2013 as to how many would continue the ILC without IAEA funding (in the year 2013: 625 euros for 4 rounds/year with 4 soil samples/round and 625 euros for 4 rounds/year with 4 plant samples/round). Only 5 participating laboratories could commit at that date; some participants reported that would consult their management to determine whether continued participation would be possible. This evidenced the importance of continued support by the IAEA, provided funding is available, in Member States with reactor facilities and in Member States with the intention to build a research reactor. All participants confirmed that they were interested in participation in future PT rounds upon invitation by the IAEA.

Participants were also advised to consider organizing bi-or multilateral regular exchange of samples amongst themselves, thus serving as a laboratory intercomparison. Samples used in current analyses could be used, avoiding multiplication of efforts.

5.6. SUSTAINABILITY OF KNOW-HOW

Retirement and/or leave of experienced staff often results in gaps in the methodological principles and metrological aspects of the techniques employed with the newly entered staff, with associated consequences for the identification of sources of error and the validity of the results. In this regard, the IAEA initiative to develop an e-learning tool for NAA, directed at young specialists or beginners without sufficient experience of conducting NAA independently, was highly acknowledged.

6. ACTION PLANS

Participants of feedback workshops drafted action plans following the evaluations, discussions and lessons learned during the feedback workshops. If action plans are to be useful, they describe the activity and objective, what has to be done, by whom, how (if relevant), deadlines for completion and, if possible, how the effectiveness would be assessed. Not all participants followed this guidance strictly, but there was agreement on the importance and usefulness thereof. The action plans following the feedback workshop in May 2013 in Vienna have been integrated in the country reports (Annex IV).

The most common actions are summarized below, following the same categorization as in Section 5. In the following feedback workshop in September 2015, it was evident that adherence to the action plans often resulted in improvement or consolidation of performance. In fact, many of the facilities that had a weaker performance in 2010-2013 brought several of the lessons learned into the daily practice, which is reflected by their better performance in the 2015 rounds.

6.1. LABORATORY ORGANIZATION

More emphasis on double checking of intermediate and final results, as well as on the implementation of non-conformance management has been mentioned as important actions to reach continuous improvement (e.g. Chile, Portugal). Several participants indicate to implement a management system compliant with the requirements of ISO/IEC 17025:2005, and to apply for accreditation. (e.g. Algeria (both labs), Czech Republic, Morocco, Portugal, Romania, Cameroon). However, it was emphasized that accreditation does not make a laboratory 'error proof'.

6.2. SAMPLE AND CALIBRATOR PREPARATION

Some participants consider important to invest in chemicals of higher purity, reference materials for neutron flux monitoring (such as the Au-in-Al wires/foils), and (certified) reference materials for either calibration (relative method) or internal quality control (e.g., Hungary-K, Romania, Syria, Cameroon, Turkey, Sudan). Participants using ICP-OES indicate that more advanced microwave ovens will be procured to improve their digestion method (Cameroon, Sudan).

The k_0 method of standardization for NAA will be implemented (or completed in its implementation) by several participants. This introduces these facilities into the k_0 users' society, providing them an opportunity for many interactions and feedback on the practical aspects of the various calibration steps, as well as for having a forum in case of problems. (Argentina-E, Ghana, Morocco, Romania, Turkey).

6.3. INTERNAL QUALITY CONTROL

There is always room to improve and expand on internal quality control. More control charts but also more replicates have been mentioned as additional activities in this field. (Madagascar, Hungary-K, Brazil-BH).

Many participants outlined that their budgets are insufficient for consolidation, improvement and expansion of quality control, and that the support by the IAEA for procurement of e.g. calibrators and reference materials is indispensable. An indicative inventory of needs was made during the 2013 workshop in Vienna; results are shown in Table 15. In many cases, the need is permanent, since the materials become exhausted by utilization.

TABLE 15. INDICATIVE INVENTORY OF NEEDS FOR QUALITY CONTROL/QUALITY ASSURANCE

Participants	New (C)RMs	Single Element Standards	Synthetic multielement standards	Calibration Sources	Quartz vials	Flux monitors
ALG-M & N, 1029 & 1110	x	x		x		x
DRC, 1030	x	x		x		
EGY, 1031	x		x	x	x	x
GHA, 1032	x	x	x	x	x	x
MOR, 1033	x	-	x	x	-	x
NIG, 1034	x	x	x	x	x	x
RSA, 1035	x	x		x		x
CAM 1036	x	x	-	x	-	-
MDG, 1037	x	x	-	x	-	-
SUD, 1038				-		
TAN, 1137	x	x		x		
AR-B & E, 1102 & 1103	x	x		x	x	x
CO, 1105	x	x	x	x	x	x
JM, 1106	x	x	x	x		
PE, 1108	x	x		x	x	x
BRA-BH, 1136	x			x	x	x
CL, 1138	x				x	
CZ, 1089	-	-	-	-	-	-
GR, 1090	x	-	-	x	-	-
H-K, 1091	x	-	-	-	x	-
KAZ, 1094	-	-	-	x	-	-
PT, 1095	x	-	x	x	-	x
RO, 1096	x	-	-	-	x	x
SV, 1099	-	-	-	x	-	-
SY, 1100	x	x	-	x	-	x
TR, 1109	x	x	x	x	x	x
R-D, 1193.	-	x	-	-	x	x

6.4. METROLOGY AND QUALITY ASSURANCE

Ample attention to training programmes of existing and new staff into the metrology and execution of NAA was mentioned by several participants (Chile, Romania, Turkey). The need for method validation and more study on interferences was also seen as important steps for further improvement of the analytical quality.

Several participants planned to expand their analytical capabilities in NAA, either by developing facilities for measurement of short half-life radionuclides, the use of larger sample masses for compensating low neutron fluxes, and the use of a Compton suppression spectrometer for improving detection limits (e.g., Algeria-N, Argentina-E, Morocco, Portugal and Greece).

6.5. SUSTAINABILITY OF EXTERNAL QUALITY CONTROL

A few participants (Czech Republic, Syria, and Turkey) described explicitly in their action plans that participation in proficiency testing schemes will be a continuous activity.

The participant from Sudan explained during the 2013 feedback workshop in Vienna that the organization of a national PT scheme is being considered, following the lessons learned during this IAEA project. This plan is, however, not included in the Sudan country report (see Annex IV).

7. OUTCOME AND IMPACT

The foremost outcome of this IAEA project is that 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.

Increased and demonstrated technical competence contributes to a laboratory's reputation and its credibility as a trustworthy partner for element measurements. It is also an indispensable asset within the scope of eventual applications for accreditation of implemented quality management systems.

Increased technical competence and valid results are equally important to the IAEA as stakeholder in this important utilization of research reactors, especially if the participating laboratories apply for further IAEA support such as TC projects or for involvement in CRPs.

Demonstration of these outcomes can be ascertained by revisiting the performance of all participants in view of the three categories recommended in the feedback workshop held August 31 – September 4 in Delft, Netherlands:

1. Metrologically satisfactory performance, 'excellent' for those laboratories reporting > 90 % of their data with $|z| \leq 3$.
2. Metrologically less satisfactory performance, 'average', for those laboratories reporting >70% and ≤ 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 ≤ 70% of elements with $|z| \leq 3$. Major improvements are needed to reach an acceptable level of performance.

The results are shown in Figures 9-16 for the various regions (as not all regions participated simultaneously in all ILC rounds). The aggregate results for all participants are shown in Figures 17 and 18.

The evolution of the indicators clearly demonstrates the continuous growth in improvement of the performance of NAA laboratories in all four regions for analysing the samples from WEPAL's ISE and IPE rounds.

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.

It should be noted that a few laboratories were included in the categories ‘average’ and ‘poor’ in Figures 1-18 due to high |z| values resulting from human errors in the reporting stage, such as interchange of results of 2 samples and/or reporting in wrong dimensional units. 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.

Continued participation of NAA laboratories in ILC rounds will therefore continue to contribute to their sustainability and further improvement of their performance. To this end, the majority of participating laboratories expressed their concern that this will be not possible without external funding, and therefore the support by the IAEA was crucial.

In this respect, the IAEA’s initiative to facilitate laboratories participating in ILC’s by WEPAL, together with the feedback meetings, was highly acknowledged as having played a fundamental role in the results already achieved.

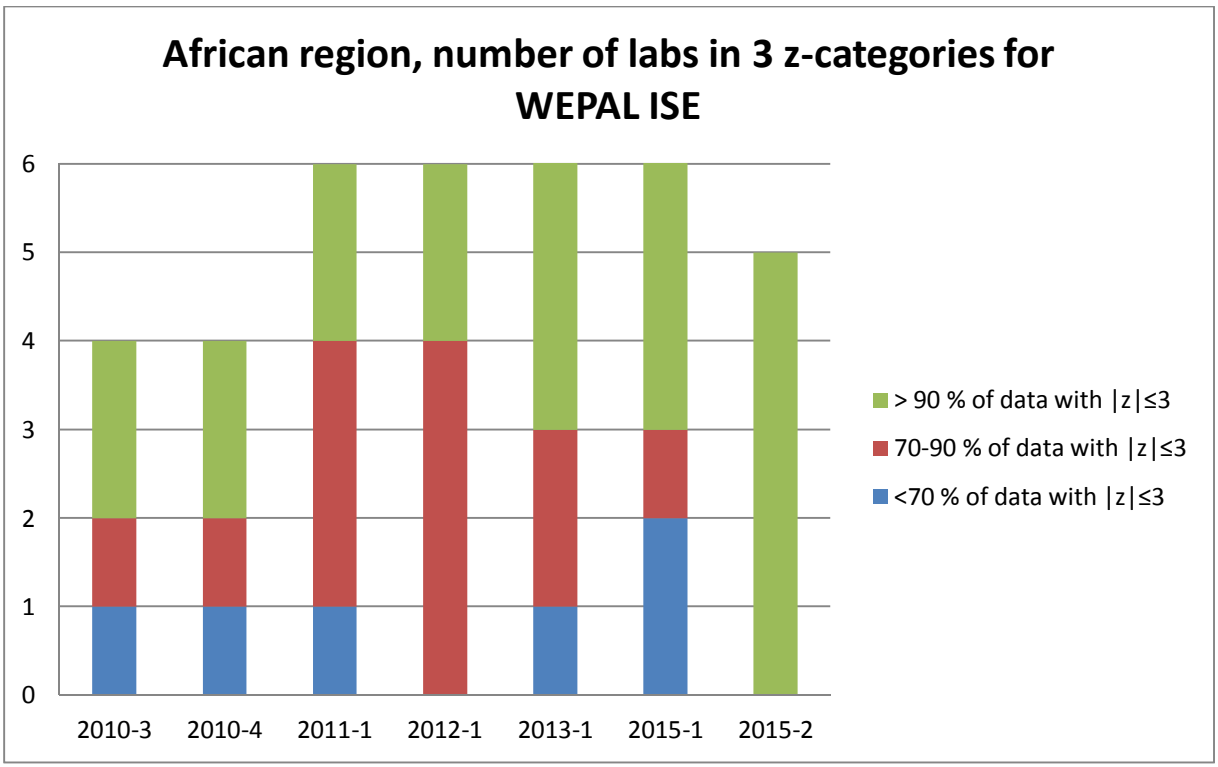

FIG. 9. Number of African laboratories in different performance categories for WEPAL ISE.

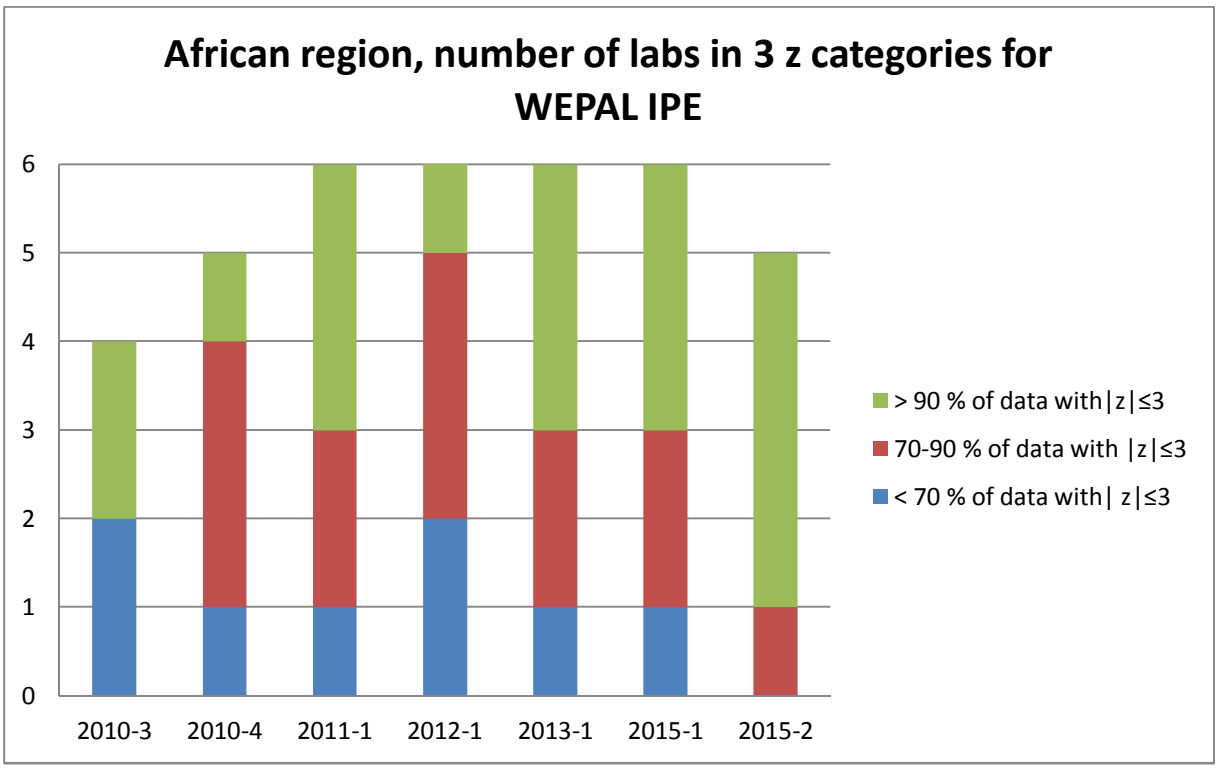

FIG. 10. Number of African laboratories in different performance categories for WEPAL IPE.

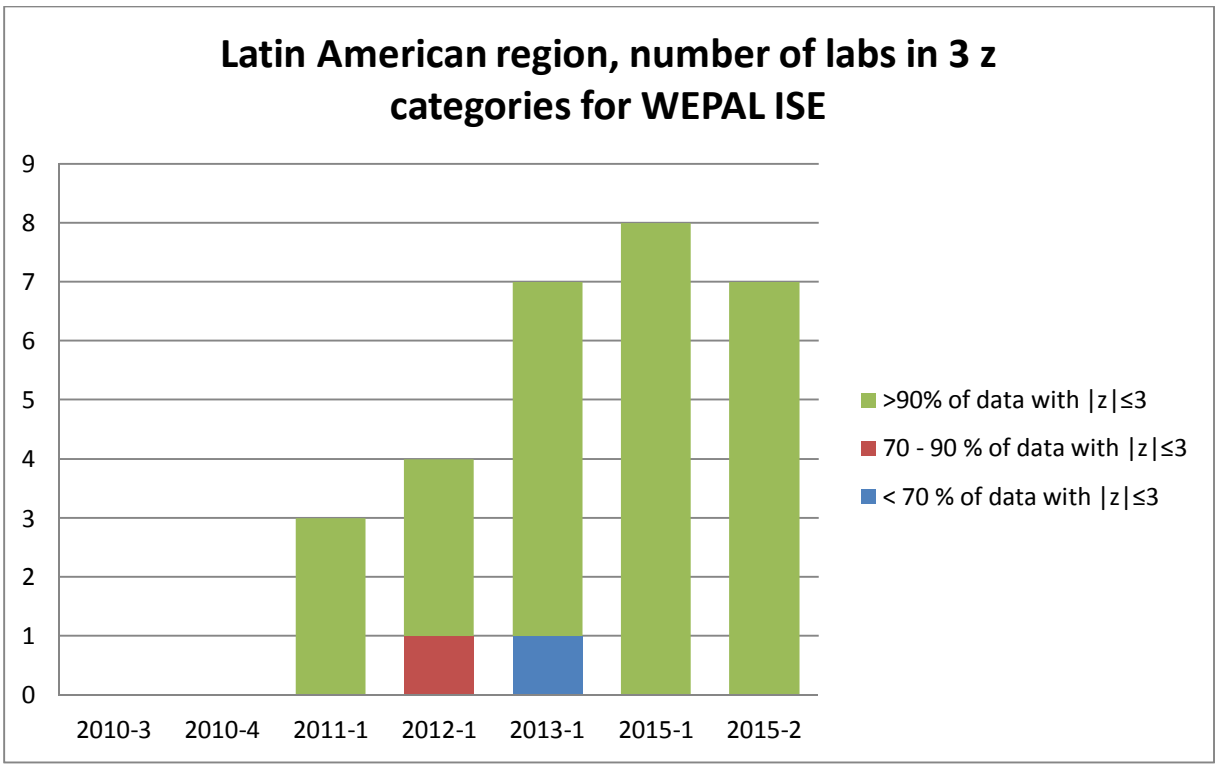

FIG. 11. Number of Latin American laboratories in different performance categories for WEPAL ISE.

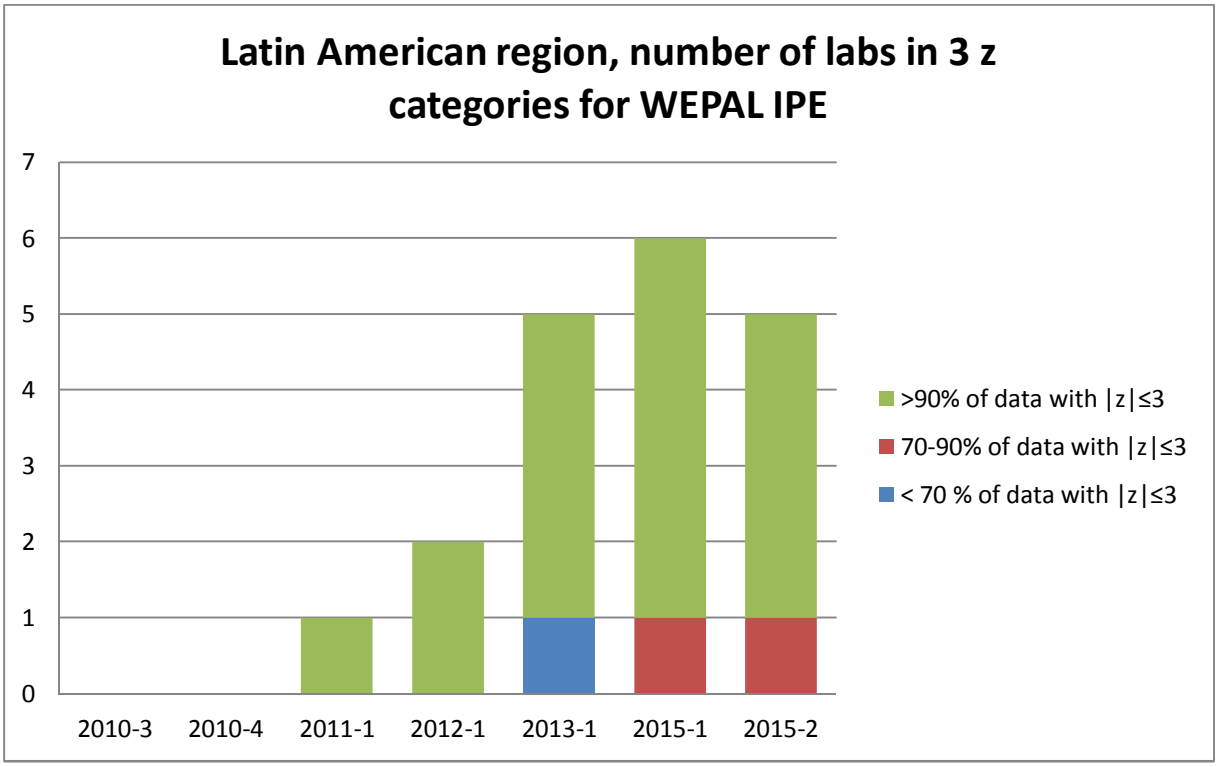

FIG. 12. Number of Latin American laboratories in different performance categories for WEPAL IPE.

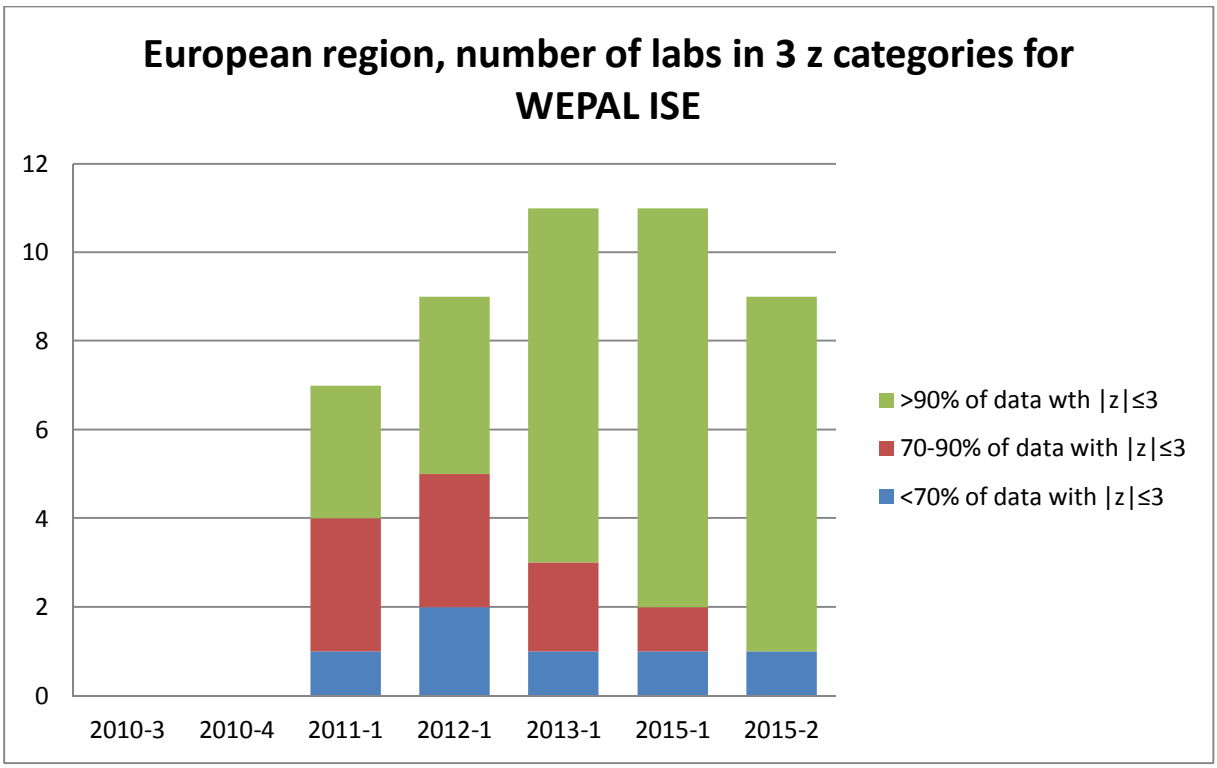

FIG. 13. Number of Latin American laboratories in different performance categories for WEPAL ISE.

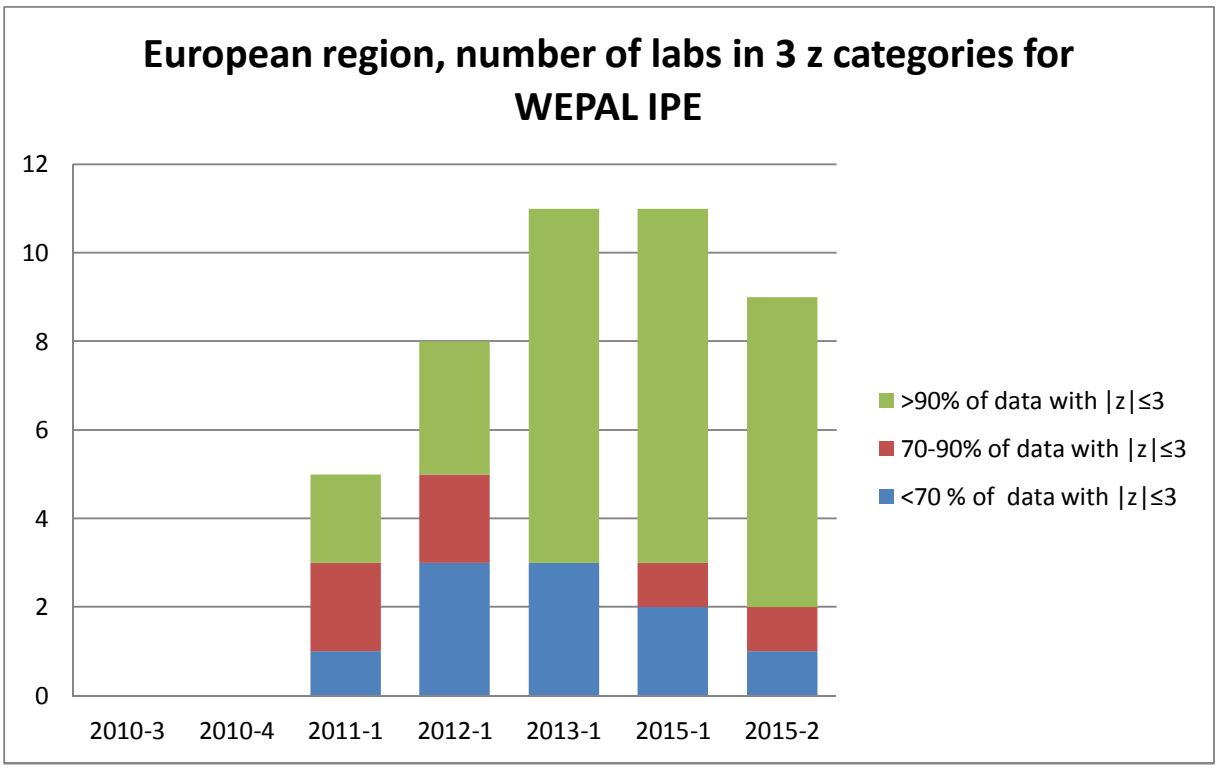

FIG. 14. Number of European laboratories in different performance categories for WEPAL IPE.

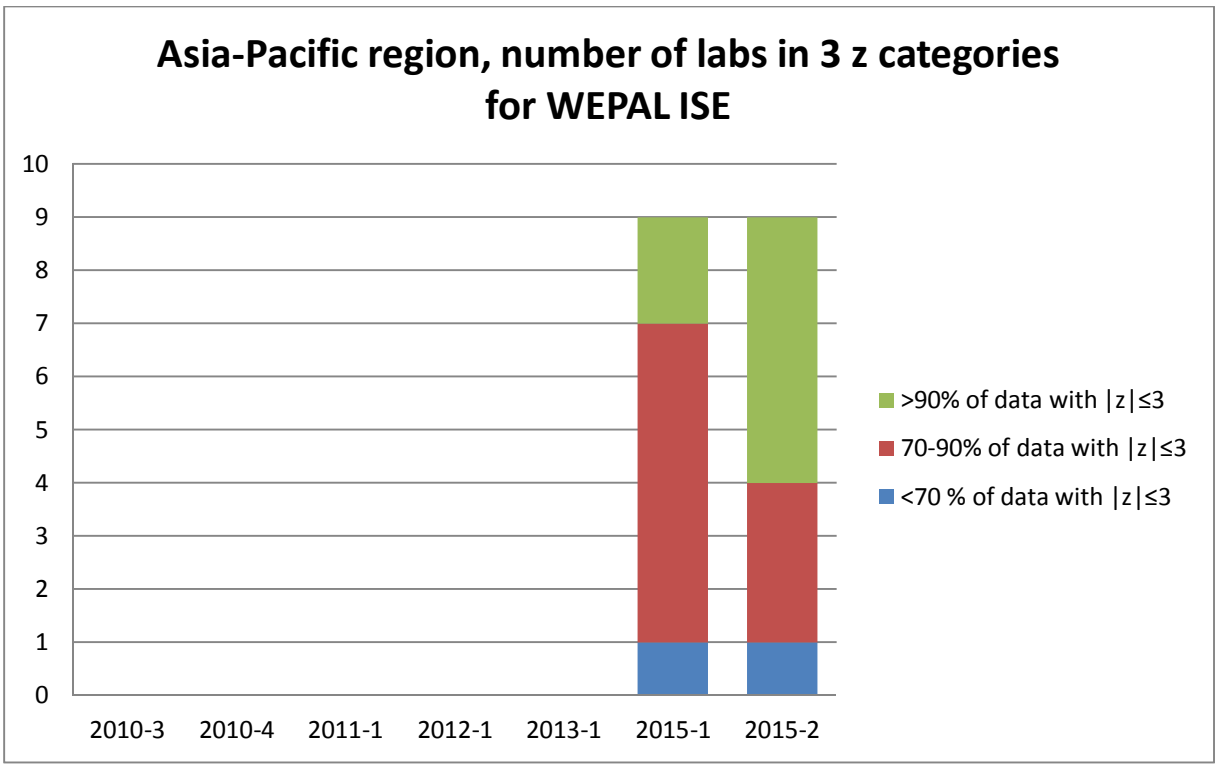

FIG. 15. Number of Asian laboratories in different performance categories for WEPAL ISE.

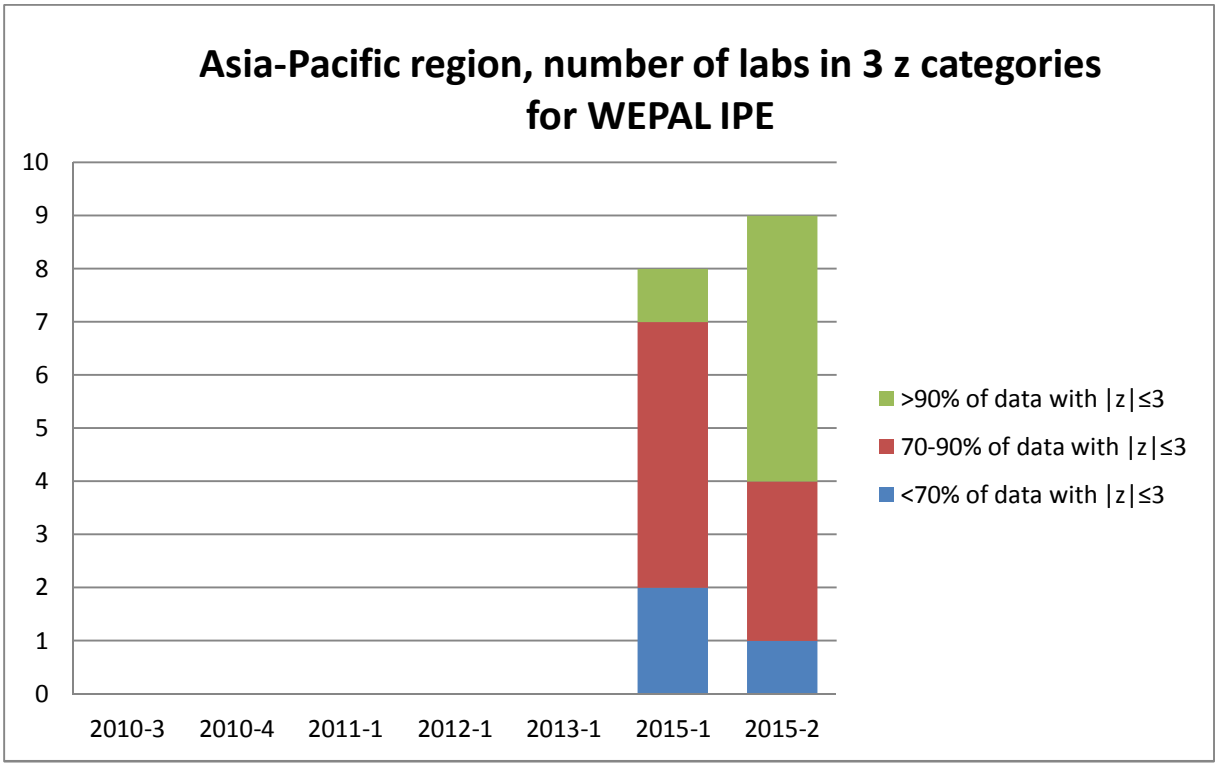

FIG. 16. Number of Asian laboratories in different performance categories for WEPAL IPE.

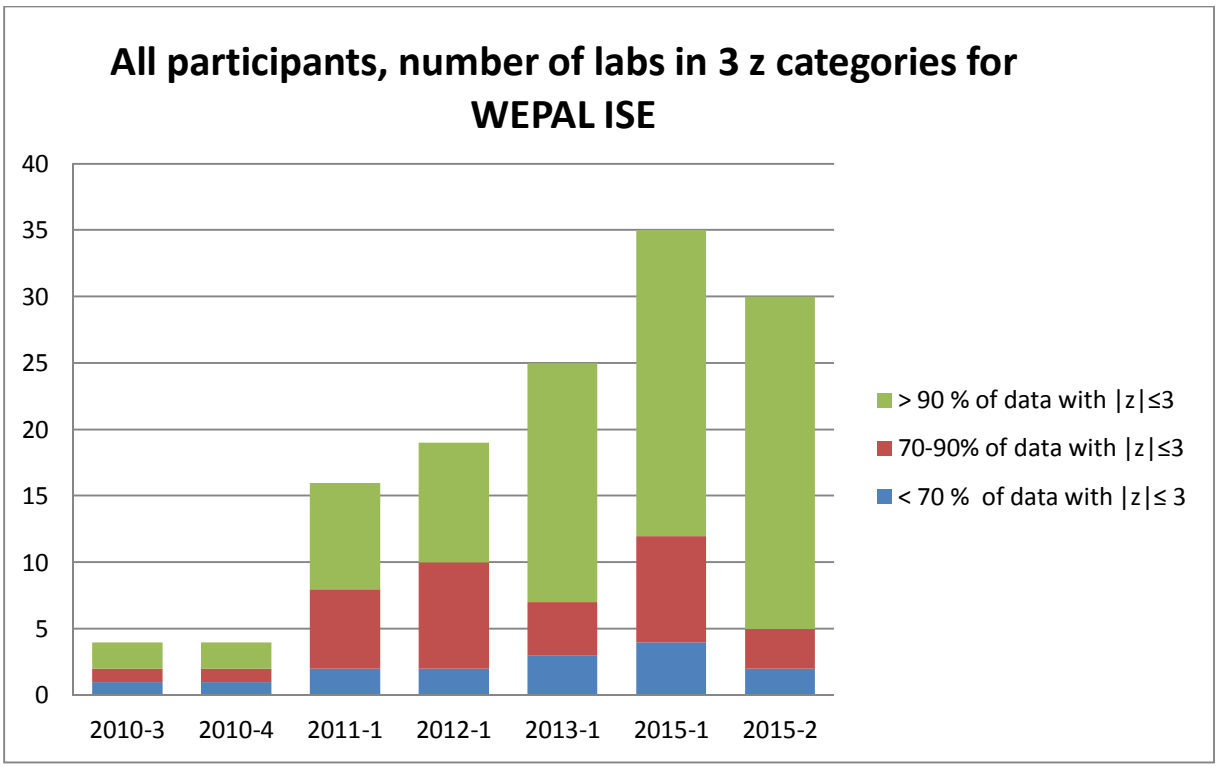

FIG. 17. All participating NAA laboratories in different performance categories for WEPAL ISE.

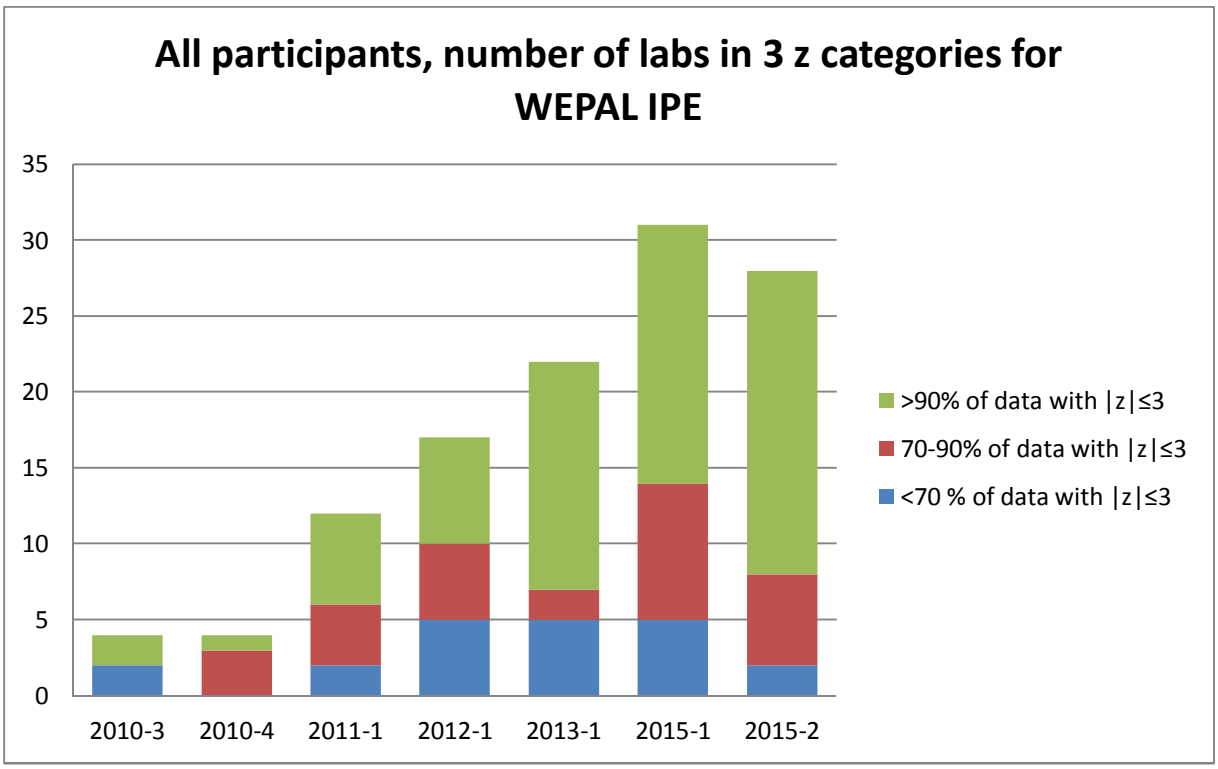

FIG. 18. All participating NAA laboratories in different performance categories for WEPAL IPE.

8. CONCLUSIONS

The IAEA's initiative to facilitate laboratories participating in proficiency testing schemes by interlaboratory comparison, together with the feedback meetings, resulted in a significant increase in the analytical and associated organizational performance of most of the participating laboratories. Several other laboratories demonstrated the consolidation of their already satisfactory performance.

Through this activity, the IAEA assisted the participants in identifying unanticipated sources of error, assessing with them approaches for their elimination and designing with them a path for growing towards sustainable performance at the analytical state of the practice.

The implemented mechanism for participation in proficiency testing schemes from a professional (accredited) provider, WEPAL, has two important advantages:

- The fast data processing and reporting by the provider after the deadline for submission of results. Participants already had insight in the quality of their performance three weeks after deadline, so that implementation of corrective actions, if needed, could rapidly be started;
- The feedback meetings provided a unique platform for evaluating the results and discussing the metrological aspects of the analytical methods used. This formed the onset for identifying sources of error and for providing expert advice to improve.

The increase in performance (or consolidation of excellent performance) was achieved by 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. In some cases assistance from well performing laboratories or international experts was the key factor. Therefore, specific mentoring arrangements were further discussed and agreed between different laboratories and covering specific technical areas.

Continuation in proficiency testing schemes will contribute to sustainability and further improvement of the performance of NAA laboratories. To this end, several participating laboratories expressed their concern that this will be not possible without external funding, and therefore the IAEA's support is deemed to be crucial.

It has been observed that retirement and/or leave of experienced staff often results in gaps in the metrological principles of the techniques employed with the newly entered staff, with associated consequences for the identification of sources of error and the validity of the results. The IAEA addressed this issue by developing an e-learning tool to increase human capacity building in NAA, which took into consideration the lessons learned in this proficiency testing project. The e-learning tool is directed at young specialists or beginners without sufficient experience of conducting NAA independently, and it covers all aspects of NAA.

Analyses for proficiency testing may often be carried out, intentionally or unintentionally, with more care than routine analyses. It all depends on a laboratory's objective in participation which may be either the assessment of its best measurement capability relative to the performance of others operating the same technique or other techniques, or alternatively, the assessment of unanticipated sources of error that may occur under routine conditions, optimized to satisfy the laboratory's regular end users. The latter approach is deemed the most useful.

Results and indications of performance are therefore not a means to make a definitive categorization of a laboratory in terms of 'good' or 'bad'. At the end, it is up to the laboratory itself to conclude about it after projecting its performance onto its own acceptance criteria for the validity of its results.

8.1. FROM LESSONS LEARNED TO FOLLOW UP ACTIONS

Participants in the feedback workshops in Vienna, 27-31 May 2013, and Delft, 31 August - 4 September 2015 suggested that the IAEA:

- Continue interregional facilitation in PTs by interlaboratory comparisons organized by WEPAL on bi-annual basis with evaluation thereof in feedback meetings and support by international experts;
- Continue development of the e-Learning tool for NAA, which will help to foster and increase human capacity building in the area of NAA as well as contribute to the overall sustainability of the technique;
- Enlarge the already existing IAEA web-based portal, presently dedicated to the IAEA Coordinated Research Project (CRP) on Development of an Integrated Approach to Routine Automation of Neutron Activation Analysis, by allowing its restricted access to all interested NAA laboratories world-wide;
- Foster and increase human capacity building in the metrology of nuclear analytical measurements by organizing education and training courses and publishing lecture notes, facilitating expert missions and fellowship training in related national IAEA TC projects;
- Assist laboratories operating techniques other than NAA in finding support from other bodies or networks for participation in PT's and evaluation of their results.

Participants in the feedback workshops in Vienna, 27-31 May 2013, and Delft, 31 August - 4 September 2015 further made the following observations concerning the laboratories:

- Laboratories are encouraged to analyse WEPAL samples in the same way that routine samples are handled, e.g., if only one aliquot is usually analysed then the same is expected to apply to the WEPAL samples;
- Laboratories are encouraged to keep trend charts for their internal quality control sample results and present them at the following feedback meeting. Trend charts can be, for example, zeta scores or measured value against assigned value for reference materials;
- Laboratories classified in the third category (less than 70 % of their results with a z-score ≤ 3) can consider to stop providing services until they have objective evidence that their corrective actions have been consistently effective in obtaining more than 70 % of their results with a z-score ≤ 3, for instance by reanalysis of WEPAL samples or other means;
- Bilateral or multilateral inter-comparisons are encouraged within regions or between national laboratories. Such inter-comparisons could target particular weaknesses that have been identified through the WEPAL programme, with the objective of improving overall performance.

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ANNEX I: DETAILS OF WEPAL'S STATISTICAL ANALYSIS

Interlaboratory studies like the WEPAL proficiency testing ring tests frequently give rise to datasets that have complex distributions including excessive tailing and multiple modes. Consequently, sophisticated statistical methods are required to obtain meaningful assessments. The strategy that was used until now makes use of an outlier test followed by straightforward statistics. Problem with this strategy is that removal of outliers causes an underestimation of variance of the dataset. Therefore a methodology is needed that does not rely on arbitrary outlier removal or subjective manual interpretations. Ideally the new methodology must provide the characteristics of the highest mode of the dataset.

A new model is chosen to calculate population characteristics (mean and standard deviation) from experimental datasets [I-1]. The model uses an estimate for the probability density function (pdf) of the measurement process and calculates a best fit based on all observed values. The implementation of the model that is used does not require uncertainty estimates for all data points. Instead it uses a normal distribution approximation (NDA) for the pdf of the individual data points. In essence, the pdf's of the individual data points are superposed on each other to create a continuous pdf representing the entire distribution (all data points).
With the mathematical model coefficients can be obtained by looking for the combination of data points that has the highest probability in the basis set. This maximization amounts to the identification of the first mode of the dataset. The coefficients can be used to calculate the weighted mean and standard deviation.

Subsequent calculations give additional modes of the distribution and for each mode the expectation value (mean), the standard deviation and a percentage indicating the fraction of observations encompassed. In this report only mean and standard deviation for the first mode (combination with the highest probability in the dataset) are given.

The model is tested on simulated data sets and datasets of several interlaboratory studies. It is demonstrated that the model is robust and insensitive to outliers. It can cope with asymmetric, strongly tailing and multimodal distributions. Publications describing the procedure in more detail and results of the tests are in preparation.

With the NDA model mean and standard deviation are calculated using all reported data when at least 8 results are left after removal of reported 'lower than' (<) and 0 (= zero) values. No outliers are removed.

Starting with the first proficiency tests in 2009 a new statically method was chosen. For reasons of continuity the statistical results of the old method will be reported in 2009. The old statistical method was preferred because strange values had less influence on the estimated central value (location) and the spread of this value (scale). Therefore estimators for location and scale were used which give less weight to observations in the tails [I-2]. For each element a median value (μ_1) and a median of absolute deviations (MAD, σ_1) are calculated using all reported data except the reported '<' and 0 (= zero) values. The median is the middle observation of the sorted array of observations in the case of an odd sample size. Otherwise it is the mean of the two middle observations. Using the median instead of mean, extreme data are of less influence. MAD is the median of the absolute values of the observations minus their median.

In case more than 7 data are reported, the values with $\left|(x - \mu_1)\right| / (f*\sigma_1) > 2$ are marked with a double asterisk (**). The factor f, aiming at 5% (singly or doubly) asterisked data in a sample of size n ($n > 7$) from a Gaussian distribution, is approximated by $(0.7722 + 1.604 / n) * t$, where t is

the 2½ percent point of Student's t with (n - 1) degrees of freedom. A second median (μ_2) and a second MAD (σ_2) are computed then leaving out the items labelled **; included values with $|(x - \mu 2)| / (f*\sigma 2) > 2$ are marked with a single asterisk (*). Finally a third median and MAD are calculated, discarding both * and **.

Rounding interval is based on the first decimal value lower than *sd / 2* (standard deviation divided by 2). If no standard deviation is available (less than 8 results) the MAD is used. At least three significant digits are shown as a minimum. If no standard deviation and MAD are available rounding is also based on three (most) significant digits. For the statistical results (mean, standard deviation, median and MAD) one extra digit is shown.

Note that larger results are also rounded (e.g. 1809 may be rounded as 1810).

For all analytical data a *Z*-score is calculated according to the formula (1):

$$Z\text{–}score = (X - X_{mean})/S_d \quad (1)$$

in which:

X is the reported value, X_{mean} is the mean of all values calculated with the NDA model, and *Sd* is standard deviation calculated with the NDA model.

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ANNEX II: TEMPLATE FOR REPORTING ON FEEDBACK WORKSHOPS

The template for reporting on feedback workshops is available on the attached CD-ROM.

ANNEX III: LIST OF LECTURES DURING FEEDBACK WORKSHOPS

Antananarivo, 2011	• Lessons learned from this proficiency testing (P. Bode); • How to participate in proficiency testing (P. Bode); • How to follow-up after participation (P. Bode); • Terminology and use of calibrators (P. Bode); • Sources of error, quality control and –assurance in nuclear techniques (P. Bode); • Calibrations, preparation, errors and uncertainties – related to non- Nuclear Analytical Techniques (E. Zeiller); • Sample dissolution in non-NAT and its possible influence on the elemental recovery (E. Zeiller); • Sources of error and interferences in non-nuclear techniques (E. Zeiller); • Avoiding contamination, think clean (E. Zeiller); • Planning, organizational aspects (P. Bode); • Planning, technical aspects (P. Bode); • Non-conformance management (P. Bode).
Tunis, 2012	• Group exercise on evaluation of PT results (P. Bode); • Implementation of quality management activities like internal quality control (P. Bode); • Use of controls charts (P. Bode); • Metrological aspects of (nuclear) analytical techniques and different methodologies for the evaluation of the uncertainty of measurement (P. Bode); • Method validation (P. Bode); • Steps to be taken towards laboratory accreditation (P. Bode).
Delft, 2012	• Lessons to be learned from PT reports; • WEPAL: organization, approaches and results (B. Eijgenraam); • Terminology and use of calibrators (reference materials and standards) (P. Bode); • The role of the neutron activation laboratory in an integrated management system (M. Cagnazzo); • 20 Years' of experience with quality management systems in an NAA laboratory (P. Bode).
Vienna, 2013	• Lessons to be learned from PT reports (P. Bode); • (Modern) trends in activation analysis (P. Bode).
Delft, 2015	• Resume of lessons learned, conclusions and recommendations after PTs 2010-2013 (P. Bode).

ANNEX IV: COUNTRY REPORTS

All reports are available on the attached CD-ROM.

Author	Affiliation	Title of paper
Hamidatou, L.	Neutron Activation Analysis Department, Nuclear Research Centre of Birine, Algeria	Algerian neutron activation analysis laboratory of CRNB: recent and future action plan
Mouzai, M.	Centre de Recherche Nucleaire de Draria, Algeria	Performance evaluation of the LNAA-CRND Laboratory for five proficiency tests for plant and soil materials
Jasan, R.C.	Comisión Nacional de Energía Atómica (CNEA), Ezeiza Atomic Centre, Argentina	Analysis of the participation of Argentinian nuclear analytical technique laboratory, of the Ezeiza Atomic Centre, in WEPAL Rounds
Menezes, M.A.B.C.	Nuclear Technology Development Centre, Brazilian Commission for Nuclear Energy, CDTN/CNEN, Brazil	Participation of the laboratory of Neutron Activation Analysis, CDTN/CNEN, Brazil, in the proficiency testing round WEPAL ISE/IPE-2013-1
Mfopou Mewouo, Y.C.	Laboratory of Soil, Plant, Water and Fertilizer (LASPEE), Institute of Agricultural Research for Development (IRAD), Cameron	Interlaboratory comparison rounds performed by the soil, plant, water fertilizer Laboratory (LASPEE) of Cameroon Institute of Agricultural Research for Development (IRAD) from 2010 to 2013 under the IAEA Technical Cooperation (TC) Projects RAF4022
Munoz, L.	Comisión Chilena de Energía Nuclear, Chile	Evaluation of the performance of the neutron activation analysis laboratory of the Chilean Nuclear Energy Commission in WEPAL proficiency testing
Pena, M.L.	Servicio Geologico Colombiano, Colombia	Colombia individual report for participation in intercomparison IPE and ISE ringtest Wageningen evaluating programmes for analytical laboratories - WEPAL

Kucera, J.	Nuclear Physics Institute, The Academic of Sciences of the Czech Republic, Czech Republic	Recent applications and developments of Neutron Activation Analysis at the Nuclear Institute as CR, Czech Republic
Mohamed, N.	Atomic Energy Authority, ETRR-2, Egypt	Interlaboratory results evaluation of the NAA Laboratory at ETRR-2
Baidoo, I.K.	Nuclear Reactor Research Centre, NNRI, Ghana Atomic Energy Commission, Ghana	IAEA-WEPAL interlaboratory comparison testing of plant and soil samples at Ghana Research Reactor-1 NAA Laboratory
Manolopoulou, M.	Atomic and Nuclear Physics Laboratory, Aristotle University of Thessaloniki, Greece	A subcritical nuclear reactor as neutron source for applying INAA in Environmental samples
Szinklai Laszlo, I.	Centre for Energy Research, Hungarian Academy of Sciences, Nuclear Analysis and Radiography Department, Hungary	Results of the k_0-based instrumental neutron activation analysis (k0-INAA) for WEPAL plant and soil IPE/ISE in Hungary
Antoine, J.M.R.	International Centre for Environmental and Nuclear Sciences, University of the WEST Indies, Jamaica	Proficiency testing at the international Centre for Environmental and Nuclear Sciences: Evaluation of the performance of the Nuclear Analytical laboratory
Randriamanivo, L.V.	Institut National des Sciences et Techniques Nucléaires-Madagascar, Madagascar	Proficiency assessment through interlaboratory comparison: Case of soil and plant testing and calibration at INSTN-Madagascar
Embarch, K.	Centre National de l'Energie, des Sciences et des Techniiques Nucleaires (CNESTEN), Morocco	Morocco: Participation of CNESTEN NAA Laboratory in PT-mechanism under RAF4022
Ahmed, Y.A.	Centre for Energy Research and Training, Ahmadu Bello University, Nigeria	Enhancement of NIRR-1 utilization and quality control through participation in IAEA interlaboratory comparison and proficiency test

Bedregal, P.S.	Instituto Peruano de Energia Nuclear (IPEN), Peru	The WEPAL proficiency testing to evaluatie the performance of the nuclear analytical techniques laboratory at Peru
Almeida, S.M.	C2TN, Instituto Superior Técnico, Universidade Técnica de Lisboa, Portugal	Performance of the Portuguese k_0 - Neutron Activation Analysis laboratory during four intercomparison exercises
Barbos, D.	Institute for Nuclear Research, Pitesti, Romania	Romania country report on interlaboratory comparison in IAEA/AFRA Project RAF 4/022, IAEA ARCLAL RLA 0037, IAEA RER 4/032 RER 1/007 and IAEA ARASIA RAS 1/018
Jacimovic, R.	Department of Environmental Sciences, Jozef Stefan Institute, Slovenia	Determination of minor and trace elements in the WEPAL IPE/ISE proficiency tests in the years 2011, 2012 and 2013 using k_0 INAA
Ebrahim, A.M.	Sudan Atomic Energy Commission, Ministry of Science and Technology, Khartoum, Sudan	Impact of proficiency testing mechanism on the improvement of results quality of Central Petroleum Laboratory (CPL), Ministry of Energy, Sudan
Sarheel, A.	Atomic Energy Commission of Syria, Damascus, Syrian Arab Republic	Participation of an interlaboratory comparison programme with WEPAL (Netherlands) under IAEA TC project RER1/007-2013
Erenturk, S.	Energy Institute, Istanbul Technical University, Turkey	Overview of Neutron Activation Analysis at ITU TRIGA Mark II Reactor

ABBREVIATIONS

AAS	Atomic absorption spectroscopy
BIPM	International Bureau of Weights and Measurement
CRM	Certified reference material
ED-XRF	Energy dispersive X ray fluorescence
ICP-MS	Inductively coupled plasma mass spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
ILAC	International laboratory accreditation collaboration
ILC	Inter laboratory comparison
IPE	International plant-analytical exchange
ISE	International soil-analytical exchange
IUPAC	International Union of Pure and Applied Chemistry
k_0-NAA	Nuclear activation analysis with the k_0 method
MAD	Median absolute deviation
NAA	Nuclear activation analysis
PT	Proficiency testing
RM	Reference material
RNAA	Radiochemical nuclear activation analysis
SMELS	Synthetic multielement standards
TC	Technical cooperation
TXRF	Total reflection X ray fluorescence
WEPAL	Wageningen evaluating programmes for analytical laboratories
XRF	X ray fluorescence

CONTRIBUTORS TO DRAFTING AND REVIEW

Barradas, N.P.	International Atomic Energy Agency
Bennett, J.	Australian Nuclear Science and Technology Organisation, Australia
Bode, P.	NUQAM Consultancy, Zuid-Beijerland, Netherlands
Kučera, J.	Nuclear Physics Institute, Řež, Czech Republic
Ridikas, D.	International Atomic Energy Agency
Yunikova, A.	ROSATOM CICE&T, Obninsk, Russian Federation

Feedback Workshops and Meetings
Antananarivo, Madagascar: 12–16 September 2011
Delft, Netherlands: 22–25 May 2012
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17-50681

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ISBN 978–92–0–108617–4
ISSN 1011–4289