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Laboratory Intercomparison Exercises Performed in 2010–2022 for Neutron Activation Analysis Laboratories

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International Atomic Energy Agency

LABORATORY INTERCOMPARISON EXERCISES PERFORMED IN 2010–2022 FOR NEUTRON ACTIVATION ANALYSIS LABORATORIES

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INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

The IAEA supports its Member States in increasing their use of research reactors. These reactors can be used for scientific research and training and for provision of commercial products, such as radionuclides for medical and industrial applications or analytical services of elemental analysis of various samples and objects. The IAEA's Research Reactor Database (RRDB) indicates that the neutrons from miniature, small and medium size reactors are mostly used for neutron activation analysis (NAA). Over the years, the IAEA has stimulated NAA groups to focus on applications in which a large number of samples may be available for analysis. The socioeconomic impact of decisions taken on the basis of the analysis results may be significant, including those related to industrial processes, food and nutrition, human and animal health, and forensic cases. Where markets for NAA laboratories have been identified, objective and impartial evidence of the validity of the results, together with organizational quality, are a precondition for expanding the stakeholder community and the services provided. Eventually, laboratories and/or stakeholders may arrange for the facility's management system be accredited for compliance with ISO/IEC 17025:2017, the international standard for testing and calibration laboratories.

Laboratories need to validate their methods and continuously monitor and verify the validity of their results. One of the requirements of ISO/IEC 17025:2017 is that laboratories also have to monitor their performance by comparison with results of other laboratories. Method validation and performance evaluation may be achieved by participation in proficiency testing and by interlaboratory comparison. The IAEA, through its Analytical Quality Control Services, provides such interlaboratory comparisons at no cost. Since 2010, these services have been complemented by support for NAA laboratories in identifying the cause of potentially unacceptable results in the interlaboratory comparison and by providing recommendations for implementing effective approaches to eliminate the related sources of error.

This publication provides a review of the performance of numerous NAA laboratories participating in interlaboratory comparisons from 2010 to 2022.

The IAEA thanks the experts who contributed to this publication, in particular P. Bode (Kingdom of the Netherlands). The IAEA officers responsible for this publication were N. Pessoa Barradas and A. Migliori of the Division of Physical and Chemical Sciences.

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1. INTRODUCTION

1.1 BACKGROUND

Participation in interlaboratory comparisons is required by the International Standard Organization (ISO) standard ISO/IEC 17025:2017 [1] for monitoring the performance by comparison with results of other laboratories, where available and appropriate (Clause 7.7.2). It is also stated in Clause 7.7.3 that "If the results of the analysis of data from monitoring activities are found to be outside pre-defined criteria appropriate action shall be taken to prevent incorrect results from being reported". In principle, the last requirement might be considered as a universal good practice, irrespective of whether a laboratory has to comply with ISO/IEC 17025 [1].

ISO also published ISO/IEC 17043:2010 "Conformity assessment — General requirements for proficiency testing" [2], and ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparison" [3][1]. Proficiency testing providers that meet the requirements of the ISO/IEC 17043 [2] are considered to be competent. Interlaboratory comparisons fulfil a number of other purposes:

- Impartial evaluation of the validity of measurement results and for monitoring laboratories' capabilities for specific tests;
- Method validation;
- Characterization of candidate reference materials;
- "Support for statements of the equivalence of measurements of National Metrology Institutes through key comparisons and supplementary comparisons conducted on behalf of the International Bureau of Weights and Measurement and associated regional metrology organization" [2].

Laboratories may derive other benefits from participation in interlaboratory comparison:

- Results may indicate unanticipated analytical or even organizational shortcomings in a laboratory's performance that are not detected by the routine monitoring of the validity of measurement results;
- Provision of impartial confidence to laboratory customers on the effectiveness and equivalence of test results or measurement method;
- Complementary evidence of analytical performance on matrices and measurands for which suitable reference materials for routine validity control are not available.

Neutron activation analysis (NAA) laboratories often encounter difficulties in finding laboratory intercomparisons for proficiency testing that are appropriate for their method and are affordable given the limited resources available. Often a (very) small number of NAA laboratories participate in a scheme, which makes it difficult to assess if the NAA results outside acceptance criteria might be due to technique-specific problems or by assigned values based on consensus values, biased by the other techniques contributing. Consequently, several NAA laboratories do not and/or cannot participate regularly in proficiency testing, which makes their analytical performance self-assessment and accreditation difficult.

[1] In addition to ISO13528:2022, ISO/IEC 17043:2023 is now also published with a revised title 'General requirements for the competence of proficiency testing providers'.

Since 2010, the IAEA has assisted NAA laboratories in assessing their analytical performance and effectiveness of quality assurance (QA) and quality control (QC) approaches, by facilitating participation in interlaboratory comparisons of the analysis of materials by NAA from a competent provider of such materials [4]. An ISO/IEC 17043 [2] accredited provider was selected for this role from 2010 to 2017. From 2018, the NAA laboratories joined the proficiency testing organized by the IAEA's Nuclear Science and Instrumentation Laboratory (IAEA-NSIL). This organization is in compliance with ISO/IEC 17043 [2] and, since 2021, with ISO 13528 [3][2]. For each proficiency testing exercise, IAEA analysis of the results has been carried out by an international expert, providing indications on potential sources of error and for assessing trends in performance following the lessons learned. Also, feedback workshops of participants and experts have been organized for detailed discussions on the experimental conditions used by the participants, identification of sources of error and recommendations for actions to implement improvements. Since 2010, the proficiency testing exercises have been organized with support from IAEA technical cooperation projects, with the latest exercises supported by IAEA's regional project RER1022 'Enhancing Utilization and Safety of Research Reactors'.

1.2 OBJECTIVE

The objective of this publication is to provide up to date technical information on the proficiency testing exercises organized by the IAEA for NAA laboratories from 2010 to 2022, to guide the NAA analytical laboratories in improving the degree of trueness and robustness of their results in a sustainable way. The expected outcome is improved quality, reliability and trustworthiness of analytical work provided by NAA laboratories, leading to increase utilization by customers and users of such services.

1.3 SCOPE

This publication provides an overview of the IAEA programme to facilitate proficiency testing exercises by interlaboratory comparison from 2010 to 2022. The exercises performed from 2010 to 2015 were previously reported [4], and the emphasis is on the methodological developments that have taken place since then, on the evolution of laboratory performance in the period covered and on the lessons learned in over one decade of this activity. Additionally, the performance of NAA laboratories is compared to the performance of laboratories using other nuclear and nuclear related analytical techniques, mostly based on X ray fluorescence (XRF) spectrometry, which participated in the same laboratory comparison exercises from 2018 to 2022.

1.4 STRUCTURE

The present publication consists of this introduction, seven technical sections describing the approach, organization, main results, feedback, evaluation, lessons learned and outcome of the intercomparison exercises performed, followed by conclusions and a list of references. One annex contains the details of participants in the IAEA facilitated interlaboratory comparisons for proficiency testing from 2017 to 2022.

[2] The ISO 13528:2022 was introduced in August 2022. The use of ISO 13528:2015 was announced in the call for participation in the laboratory comparison organized in 2022. Reference to this standard was maintained in the evaluation and reporting as the major changes in the revised version of the standard do not apply to the statistical evaluation selected from the standard by IAEA-NSIL. IAEA-NSIL continues compliance to the latest valid version of the appropriate standards for its laboratory intercomparisons.

2. VALIDITY OF MEASUREMENT RESULTS BY NEUTRON ACTIVATION ANALYSIS

This section provides an overview of validity of measurement results by the NAA technique.

2.1 MONITORING THE VALIDITY BY PROFICIENCY TESTING

The results of NAA measurements need to be trustworthy on the basis of their known and impartial established validity, and need to be traceable to relevant standards.

Analytical laboratories, including those conducting NAA, need to give objective evidence of their technical competence and of the validity of results and reliability and consistency of their performance. Laboratories may provide the requested evidence by accreditation of their management system for compliance with ISO/IEC 17025 [1] as it includes independent assessment of the competence of the laboratory in performing tests and in the validity of the measurement results.

The validity of measurement results needs to be first assessed by method validation prior to the conduction of regular analysis, then verified by planned validity monitoring procedures ("quality control") as part of a laboratory's QA programme. Participation in interlaboratory comparisons provide an additional impartial verification of validity, as the target values are already known. Results from interlaboratory comparison may reveal unanticipated deviations of the validity of results.

Validity monitoring may be achieved by analysis of materials of known composition and property values, closely matching the type of samples to be analysed. This is preferably done with every batch of samples to be analysed. (Certified) reference materials are commonly used for this, but also materials of formulated composition, replicate testing or comparison by measurement with other techniques may be used. This approach is preferred as it provides indications of validity simultaneously with the analysis results, whereas it may take several months before results of interlaboratory comparison are available. In addition, the materials made available by the provider of interlaboratory comparisons might not be representative for the matrices and measurands (and their mass fractions) routinely analysed.

The results from interlaboratory comparison are evaluated by comparing the reported values with the assigned values and the standard deviation for proficiency testing. This is further described in Section 3. This produces a categorization of submitted results (as 'satisfactory', 'questionable' or 'unsatisfactory'), commonly on the basis of statistical considerations. This is, in principle, an inappropriate appraisal as also mentioned in ISO/IEC 17043 [2], since only the laboratory itself can decide on its performance given its own validity acceptance criteria and the requirements of its customer(s).

Results from interlaboratory comparisons may be 'unsatisfactory', irrespective if this is according to the categorization of the provider or on basis of the laboratory's own criteria. A laboratory needs[3] to find the cause of the deficiency and consider the need for implementing actions to prevent recurrence. A cause analysis and eventual corrective action involves a thorough understanding of the metrology of the analytical technique, and experience in trouble shooting in all steps from sample preparation and calibration to final spectrum interpretation and reporting. This follow-up on a nonconforming result is a necessary procedure in

[3] In the terminology used in ISO/IEC 17025 [1], the laboratory "shall" do this.

laboratories with a management system complying with ISO/IEC 17025 [1] as it forms the basis of continuous and sustainable improvement. There are, however, NAA laboratories in which such an approach is not yet implemented and/or which are less experienced in finding the cause of problems and selecting effective actions towards improvement. Providers of proficiency testing exercises do not usually make available reports on potential sources of error for each participating technique, nor do they undertake root cause analysis. Since 2010, the IAEA has therefore implemented a programme for monitoring the validity of measurement results of Member States' NAA laboratories by interlaboratory comparison, supplemented by feedback support in identifying potential sources of nonconformities and recommendations for implementing an effective QA and QC system.

2.2 THE IAEA APPROACH

This section describes the approach taken by the IAEA in supporting nuclear analytical laboratories in implementing QA/QC, and management systems.

2.2.1 Quality assurance and quality management implementation

The IAEA has been supporting nuclear analytical laboratories, including NAA laboratories, for many years in implementing QA/QC, and (quality) management systems. These activities include:

- Development of certified reference materials (CRM) by the IAEA Marine Environment Laboratories and the Terrestrial Environmental Radiochemistry Laboratory [5] and laboratory intercomparisons by the IAEA Nuclear Science and Instrumentation Laboratory [6], thereby continuing the Analytical Quality Control Services [7];
- Model projects on QA and QC with associated training courses in all regions up to the early 2000s;
- Development of guidance publications [8–13];
- Development of the IAEA e-learning NAA course, and in particular the development of modules on QA and QC, quality management, method validation and sources of error and troubleshooting [14];
- Provision of international experts engaged by the IAEA's technical cooperation programme, providing on-site training workshops and reviewing laboratories and providing recommendations for further improvement;
- An interlaboratory comparison exercise of software relevant for the k_0 method in NAA [15];
- Providing support to individual NAA laboratories through national and regional technical cooperation projects, including procurement of equipment, CRMs, flux monitors and other materials, expert missions and fellowship trainings.

2.2.2 Proficiency testing and validity monitoring by interlaboratory comparison

The IAEA programme for monitoring the validity of measurement results by NAA laboratories as an indicator for their analytical performance in the period 2010 –2022 included:

- Facilitation of participation in successive interlaboratory comparisons. This was realized in the years 2010–2013, 2015 and 2017–2022. The Wageningen Evaluating Programmes for Analytical Laboratories (WEPAL, [16]) from Wageningen, Netherlands, was selected for this in 2010, 2011, 2012, 2013, 2015 and 2017. IAEA-NSIL implemented the

interlaboratory comparisons from 2018 onwards. Both providers made materials available suitable for (trace) element measurement by NAA and were able to issue the evaluation report very shortly after due date for submission, typically within a few weeks;

— Definition of performance indicators for evaluation and monitoring trends in performance in interlaboratory comparisons;
— A technical and analytical analysis of the provider's evaluation of laboratory intercomparison and of the submitted results by an IAEA expert for first indications on potential sources of error and initial feedback;
— Feedback workshops and workshops on QA/QC with selected participants and IAEA experts shortly after availability of the reports by the proficiency test provider and the analysis by the IAEA expert. These workshops included discussions for identifying unanticipated sources of errors (both technical and managerial), appropriate and pragmatic QA/QC procedures for mitigation, monitoring and effective prevention of recurrence and discussions on other practical aspects for QA and QC. The workshops were held in 2011, 2012, 2013, 2015, 2017, 2018, 2020 (virtual) and, due to the COVID-19 pandemic, again in 2022;
— Questionnaires for providing information on the technical conduct of NAA analysis, calibration procedures and applied validity monitoring were sent to the participants in 2018 and 2021.

3. ORGANIZATIONAL ASPECTS

This section discusses organizational aspects of the implemented mechanism of proficiency testing.

3.1 PROVIDERS

The IAEA used WEPAL for the initial proficiency testing exercises between the beginning of the activity in 2010 and until 2017. From 2018, in-house organization of proficiency testing was done by taking advantage of the experience of IAEA-NSIL in organizing proficiency testing exercises.

WEPAL organizes proficiency testing schemes in the fields of plants, soil, sediments and organic waste. Both the international soil-analytical exchange (ISE) and international plant-analytical exchange (IPE) programmes were considered suitable for participation by NAA laboratories. WEPAL has organised these programmes for over 50 years and has over 500 participants in the schemes from countries all over the world. The considerations for first selecting WEPAL were [4]:

- WEPAL is accredited by the Dutch Council for Accreditation under No. R 002 for compliance with ISO 17043:2010 [2];
- WEPAL has a proven record of issuing the evaluation report three weeks after the deadline for reporting;
- WEPAL provides proficiency testing schemes for analysis of soil and plant material, matrices suitable for analysis by NAA;
- Participants identify in their reports 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 XRF) and amounts from techniques requiring dissolution of the sample;
- The number of participants in intercomparisons on soil and plant matrices is large, typically up to one hundred or more. This contributes to the degree of trueness of the robust median value of the results;
- In each round four samples of a specific type (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.
- The materials used by WEPAL for its interlaboratory comparison exercises are produced within its own facilities. Several tests are done to assess the within and between bottle homogeneity although this is done on a limited number of measurands. Nonetheless, WEPAL verifies the degree of homogeneity by statistical evaluation of the results of the participants analysing the samples.

IAEA-NSIL continued the IAEA Analytical Quality Control Services by which reference materials and interlaboratory comparisons have been organized for several decades. IAEA-NSIL has been organizing laboratory intercomparisons for proficiency testing since the year 2001 for laboratories utilizing XRF based analytical techniques.

Using the in-house resources by NSIL, all facilities with NAA capabilities (as known through the IAEA Research Reactor Database [18]) were invited to participate. IAEA-NSIL conducts its interlaboratory comparisons in compliance with the intention of ISO/IEC 17043 [2] although it is not formally accredited as such. In addition, in 2021, IAEA-NSIL implemented the

statistical methods for use in proficiency testing by interlaboratory comparison as described in ISO 13528 [3]. This also facilitated the availability of the report of the interlaboratory test within a month.

The IAEA-NSIL materials for interlaboratory comparison were selected either from IAEA's own repository of reference materials or procured from reference material providers, preferably operating in agreement with ISO17034:2016 [19], or from reference materials issued by WEPAL, appropriately characterized on the basis of statistical evaluation of results from their repeated use in its exercises.

3.2 INTERLABORATORY COMPARISON DISPATCH AND REPORTING SCHEMES

An overview of the laboratory interlaboratory comparisons between 2010 and 2022, the providers and the IAEA projects by which these tests have been facilitated is given in Table 1. Participants had to analyse the samples with a typical timeframe of 4-5 months, which was considered sufficient for planning and conducting a NAA procedure for multi-element assessments.

At the end of 2012, NAA laboratories from the European, Latin American and African regions re-analysed samples that were distributed in the first round of their participation in the IAEA facilitated WEPAL proficiency schemes (referred to as 2012-R in Table 1). This exercise was organised by the IAEA and not by WEPAL. The reanalysis results were evaluated by the IAEA, assessing the progress made by each laboratory, eliminating any possible influence that might have been caused if similar, but different, materials had been used.

3.3 SAMPLES AND SAMPLE TYPES

Sample types and code numbers are given in Table 2. WEPAL provided four soil and four plant samples (typically coded as 1, 2, 3 and 4) in each round of its ISE and IPE programmes, respectively. In the interlaboratory comparison rounds organized by IAEA-NSIL, one or two samples were made available for testing. In all cases, participants received information on the type of the material, which allowed them to plan for a safe and optimal execution of the analysis.

3.4 PARTICIPANTS, TECHNIQUES AND METHODOLOGIES

Neutron activation analysis laboratories participating in the IAEA facilitated laboratory intercomparisons from 2010–2022 are summarized in Table 3. Details on their analysis of soil type material and biological type material, e.g. plant-animal tissue are given in Annex 1 for the period 2017–2022, supplementing the details on the period 2010–2015 in [4]. It is noted that several NAA laboratories, in all regions, could not participate in all interlaboratory comparison rounds due to reactor shutdown periods, e.g. for maintenance, refurbishment or fuel modification, and/or in view of the COVID-19 pandemic. Some NAA laboratories could only participate in the analysis of the soil type material because biological type material was held at customs for agricultural border protection purposes.

TABLE 1. DATA ON THE INTERLABORATORY COMPARISONS FACILITATED BY THE IAEA FROM 2010 TO 2022

WEPAL round	Regions in which the comparison was implemented	Sample dispatch by provider in Netherlands	Laboratory reporting deadline date	Availability of WEPAL report
2010-3	Africa	June 8, 2010	September 30, 2010	October 18, 2010
2010-4	Africa	August 31, 2010	December 31, 2010	January 18, 2011
2011-4	Africa, Latin America and the Caribbean, Europe, Asia and the Pacific	November 13, 2011	December 31, 2011	January 3, 2012
2012-1	Africa, Latin America and the Caribbean, Europe, Asia and the Pacific	January 12, 2012	March 31,2012	April 4, 2012
2012-R	Africa, Latin America and the Caribbean, Europe	Not applicable	December 31, 2012	Not applicable
2013-1	Africa, Latin America and the Caribbean, Europe, Asia and the Pacific	January 11, 2013	March 31, 2013	April 7, 2013
2015-1	Africa, Latin America and the Caribbean, Europe, Asia and the Pacific, North America	December 1, 2014	March 31,2015	April 9/10 2015
2015-2	Africa, Latin America and the Caribbean, Asia and the Pacific, Europe,	March 1, 2015	June 30, 2015	July 6/7, 2015
2017-3	Africa, Latin America, Europe, Asia and the Pacific, North America	June 1, 2017	September 30, 2017	October 6, 2017
IAEA-NSIL round	Regions in which the comparison was implemented	Sample dispatch by IAEA-NSIL	Laboratory reporting deadline date	Availability of IAEA report
2018	Africa, Latin America, Europe, Asia and the Pacific, North America	July/August 2018	November 23, 2018	January 30, 2019
2019	Africa, Latin America, Europe, Asia and the Pacific, North America	August 2019	December 15, 2019	March 11, 2020
2020	Africa, Latin America, Europe, Asia and the Pacific, North America	August 5, 2020	February 28, 2021	May 17, 2021
2021	Africa, Latin America, Europe, Asia and the Pacific, North America	August 2021	January 31, 2022 (Extended to February 8, 2021)	March 3, 2022
2022	Africa, Latin America, Europe, Asia and the Pacific, North America	August 2022	November 25, 2022 (Extended to December 9, 2022)	December 23, 2022

TABLE 2. SAMPLE CODE NUMBERS AND MATRICES DISTRIBUTED IN THE WEPAL ISE AND IPE-INTERLABORATORY COMPARISON ROUNDS 2010–2017, AND IAEA-NSIL INTERLABORATORY COMPARISONS ROUND 2018–2022

		WEPAL Sample bottle label and material type			
Year	Interlaboratory comparison code	Bottle 1	Bottle 2	Bottle 3	Bottle 4
2010	ISE 2010-3	861: Calcareous Clay	**961: Clay**	874: Sandy Soil	**872: Braunerde Clay**
	IPE 2010-3	198: Banana/ Musea paradisciana	175: Tulip (tuber)/ Tulipa I.	**100: Grass (gr94)/ Poaceae**	**172: Cherry Laurel/Prunus laurocerasus**
	ISE 2010-4	858: Braunerde-Pseudoclay	998: Organic Ferrasol	**872: Braunerde Clay**	918; Sandy Soil
	IPE 2010-4	133: Maize / Zea mays	**172: Cherry Laurel/Prunus laurocerasus**	180: Oil Palm (leaf)/ Elaeis guineensis	173: Virginia Creeper / Partenocissus quinquefolia
2011	ISE 2011-4	868: Sandy Soil	900: Calcareous brown Soil	952; Clay	989: River Clay
	IPE 2011-4	169: Leek / Allium porrum	159: Lucerne / Medicago savitum	188: Oil Palm (Leaves) / Elaeis guineensis	**100: Grass (gr94) / Poaceae**
2012	ISE 2012-1	997: Sandy Soils	863: Clay Soil	865: Loamy Soil	982: River Clay Soil
	IPE 2012-1	197: Maize / Zea mays	124: Lucerne / Medicago sativum	189: Banana leaves / M United States of America sapientum	157: Beech leaf / Fugus sylvatica l.
2013	ISE 2013-1	870:Clay from River Basin	890: Sandy Soil	919: Sandy Soil	**971: Clay**
	IPE 2013-1	**100: Grass (gr94) / Poaceae**	215: Paprika / pepper (fruit + leaf) / Capsicum sp.	166: Cherry Laurel /Prunus laurocerasus	135: Rice (polished) / Oryza sativa l.
2015	ISE 2015-1	**860: Sediment**	869: Clay	900: Calcareous brown soil	989: River Clay
2015	IPE 2015-1	**100: Grass (gr94) / Poaceae**	218: Turnip/Brassica rapa	171: Leylandcypress/Cypressus x leylandi	980: Gerbera/Gerbera cass
	ISE 2015-2	868: Sandy Soil	961: Clay	962: Sandy Clay Soil	**860: Sediment**
	IPE 2015-2	205 Tobacco leaf-mixture/nicoyian solanaceae	177 Poplar leaf Populis l.	**100: Grass (gr94) / Poaceae**	224 Maize (grain) Zea Mays
2017	ISE 2017-3	**874 Sandy Soil**	876 Clay	**863 Clay Soil**	866 Loess
	IPE 2017-3	238 Banana	**159 Lucerne**	**215 Paprika**	203 Cabbage

TABLE 2. SAMPLE CODE NUMBERS AND MATRICES DISTRIBUTED IN THE WEPAL ISE AND IPE-INTERLABORATORY COMPARISON ROUNDS 2010–2017, AND IAEA-NSIL INTERLABORATORY COMPARISONS ROUND 2018–2022 **(**Cont.)

		IAEA Sample type	
Year	Interlaboratory comparison code	Bottle 1	Bottle 2
2018	PTNAAIAEA15	IAEA RM 456, Marine sediment	IAEA RM 452, Scallop (pecten maximus) Animal Tissue
2019	PTNATIAEA17	IAEA RM 157 Marine sediment, (Soil type material)	IAEA RM V-9, Cotton Cellulose (Land plant material)
2020	PTNATIAEA18	WEPAL RM 867 Sandy Soil	
2021	PTNATIAEA19	**WEPAL RM ISE 861, Calcareous Clay**	**WEPAL RM IPE 166 Cherry Laurel /Prunus laurocerasus (Plant)**
2022	PTNATIAEA20	WEPAL RM 970 Riverclay	**WEPAL RM Plant 224 Maize/Zea mays (Plant)**

In **bold**: the samples that have been distributed in more than one round.

TABLE 3. MEMBER STATES WITH PARTICIPATING NAA LABORATORIES IN ONE OR MORE OF THE IAEA FACILITATED INTERLABORATORY COMPARISONS 2010–2022

Region	Member States; and number of NAA laboratories between brackets
Africa	Algeria (2), Egypt, Ghana, Morocco, Nigeria, Rep. of South Africa
Europe	Austria, Belgium, Czech Republic (2), Germany (2), Greece (2), Hungary (2), Italy, Kazakhstan, Netherlands, Poland, Portugal, Russian Federation (2), Romania, Slovenia, Türkiye, Uzbekistan
Latin America and the Caribbean	Argentina (2), Brazil (3), Chile, Colombia, Jamaica, Mexico, Peru
Asia and the Pacific	Bangladesh, China, Indonesia (3), Isl. Rep. of Islamic Republic of Iran (3), Jordan, Rep. of Korea, Malaysia, Pakistan (2), Syria, Thailand, Vietnam, Australia
North America	Canada (3), United States of America (5)

See Annex 1 for details.

3.5 SUBMISSION OF MEASUREMENT RESULTS

Participants could submit their measurement results (mass fractions) online, both for WEPAL and IAEA-NSIL. The mass fractions need to be based on dry mass, and instructions for dry mass assessment are given by the provider upon the dispatch of the samples. Both providers define the units in which the results have to be given, such as $mg \cdot kg^{-1}$or $\mu g \cdot kg^{-1}$. WEPAL uses $g \cdot kg^{-1}$ for results of major components whereas IAEA-NSIL asks to report such data in percentage. The uncertainty of measurement is not reported in the proficiency testing schemes of WEPAL whereas the uncertainty has to be submitted in the schemes of IAEA-NSIL. No limits were set to the number of significant figures of the data reported in the WEPAL

intercomparison rounds, whereas for IAEA-NSIL a maximum of three digits after the comma can be inserted, for which the user needs to select the proper units for each element.

Participants also have to provide information on the technique used. In the schemes operated by WEPAL, NAA is categorized as a 'real total' analysis technique without any further detailing toward, e.g. method of calibration. More detailed information on the analytical procedures has to be submitted in the schemes of IAEA-NSIL [20], such as on the method of calibration, type of software and sample preparation, if applicable (Table 4).

TABLE 4. CODES FOR SAMPLE PREPARATION, GAMMA-RAY SPECTRUM ANALYSIS AND INTERPRETATION RELATE TO MEASUREMENT RESULTS SUBMITTED BY LABORATORIES USING NAA IN PROFICIENCY TESTING ORGANIZED BY IAEA-NSIL

Sample preparation	
Code	**Description**
2.0	Pelletizing without binder no info on thickness pellet
3.0	No sample preparation applied, sample analysed as loose powder
7.0	Sample analysed directly without any preparation
9.0	Sample preparation technique developed by the user
10.0	Other sample preparation

Quantification algorithm	
Code	**Description**
8.1	Free software from IAEA for k_0-NAA
8.2	Software for k_0-NAA
9.0	Algorithm developed by the user
10.0	Other quantification algorithm

Software for spectrum evaluation	
Code	**Description**
3.1	DOS software developed by IAEA for gamma spectrum evaluation
3.2	Software for NAA spectrum evaluation
3.3	AMETEK/ORTEC software for gamma spectra evaluation
3.4	Canberra software for gamma spectra evaluation
4.0	Provided by instrument manufacturer
5.0	Software developed by the user
10.0	Other fitting software

3.6 DATA EVALUATION AND CRITERIA

This section reports on the data evaluation performed and on the criteria used.

3.6.1 WEPAL evaluation (2010–2017)

The WEPAL reports summarise the results for each measurand by grouping them by technique used for digestion or extraction and by method used, such as 'real totals', 'acid extractable (so-called totals', extraction by different procedures, and others. The ISE programme [21] has more categories of methodologies than the IPE programme [22], see Tables 5 and 6. Most results of the laboratories participating in the IPE rounds were grouped in the category 'Inorganic chemical composition'.

For each measurand a mean and standard deviation are calculated using robust statistics by a normal distribution approximation, in which outliers are not removed (for details, see Annex 1 in Ref. [19]). The mean value and standard deviation are used for calculating the *z*-score. The

participants do not report their own measurement uncertainty, so the value thereof is not accounted for by the provider. In addition, the median of all results has been calculated for comparison with the robust mean value.

It is stressed that the WEPAL samples are not CRMs. As a consequence, the mean value provided by WEPAL for each measurand is not a certified value. It is the mean of the results reported by the laboratories involved in the exercise. In the same way, the standard deviation provided by WEPAL for each measurand is not the uncertainty with which the measurand was determined. Instead, it reflects the spread of the results reported by the laboratories involved in the exercise.

TABLE 5. METHOD CATEGORIES IN THE WEPAL ISE ROUND

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 0.01M $CaCl_2$ 1:10	Al, B, Cd, CN, Co, Cr, Cu, Fe, **K**, **Mg**, Mn, **N - NH4**, **N - NO3**, N total soluble, Na, Ni, P, Pb, SO_4, Zn
Soil characteristics	**C - org others (W&B a.o.)**, **EC-SC (ISO 11265)**, **Fraction < 16 µm**,**Fraction < 2 µm**, **Fraction < 63 µm**, **Fraction > 63 µm**, **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	B - Hot water, CN - Free, CN - Total, delta ^{13}C, delta ^{15}N,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 $BaCl_2$-TEAat 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, Mn, Na
Act. CEC using 0.01M $BaCl_2$ (ISO 11260)	Al, Ca, CEC, Fe, H, K, Mg, Mn, Na

TABLE 5. METHOD CATEGORIES IN THE WEPAL ISE ROUND (Cont.)

ISE Group	Determinand
Act. CEC using 0.1M $BaCl_2$ (UNEP-UN/EC 91065A)	Al, **Ca**, CEC, Fe, H, K, Mg, Mn, Na
Act. CEC using cobaltihexamine (AFNOR NFX 31 130)	Al, Ca, CEC, Fe, H, K, Mg, Mn, 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 DTPA 1:10 (w/v)	**Cu, Fe, Mn, Zn**
Extraction with 1M KCl 1:10 (w/v)	N - NH_4, N - NO_3
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
UK Soil Methods	**K - NH_4NO_3 (1/5), Mg - NH_4NO_3 (1/5), P – $NaHCO_3$ (1/20),pH - H_2O (2/5)**

Measurands in **bold** are in the scope of WEPAL's accreditation.

3.6.2 IAEA evaluation between 2018 and 2020

Some elements did not have certified or indicative values reported by the producer of the material. In those cases, elements where at least five laboratories reported valid results, and following a normal distribution were included in the evaluation, using the mean or mode values as assigned values. Otherwise, the elements were not included in the evaluation.

For each analyte, the reproducibility standard deviation has been assigned using a modified Horwitz function [23] demonstrated in Eq. (1).

$$H_A = \begin{cases} 0.22X_A & when \quad X_A < 1.2 \cdot 10^{-7} \\ 0.02(X_A)^{0.8495} & when \quad 1.2 \cdot 10^{-7} \leq X_A \leq 0.138 \\ 0.01\sqrt{X_A} & when \quad X_A > 0.138 \end{cases} \qquad (1)$$

In which the assigned value of analyte, X_A, is expressed as a mass fraction. This reproducibility standard deviation is used to calculate the target value of the standard deviation for proficiency testing (σ_A) given in Eq. (2).

TABLE 6. METHOD CATEGORIES IN THE WEPAL IPE ROUND
MEASURANDS IN BOLD ARE IN THE SCOPE OF WEPAL'S ACCREDITATION

IPE Group	Determinand
Inorganic Chemical Composition	Ag, **As**, **B**, **Ba**, Be, Bi, Br, **Ca**, **Cd**, **Cl**, **Co**, **Cr**, Cs, **Cu**, F, **Fe**, Ga, **Hg**, I, **K**, Li, **Mg**, **Mn**, **Mo**, **N - Kjeldahl**, N - NH4 (as N), **N - NO3**, **Na**, **Ni**, **P**, **Pb**, Pd,Pt, Rb, Rh, **S**, Sb, **Se**, Sn, SO4, **Sr**, Ti, **V**, **Zn**
Real totals	**Al**, **C - elementary**, **N - elementary**, Si
Acid extractable (So-called totals)	**Al**, Si
Other determinations	**delta ^{13}C, delta ^{15}N**
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

Measurands in **bold** are in the scope of WEPAL's accreditation.

$$\sigma_A = kH_A\,, \quad k = 0.5,\ 1.0,\ 1.5 \tag{2}$$

Depending on the value of the factor *k,* the target value of the standard deviation for proficiency testing is recognized as fit-for-purpose at one of three levels of uncertainty: $k = 0.5$ - appropriate for high precision analysis; $k = 1.0$ - appropriate for well-established routine analysis; $k = 1.5$ - satisfactory for common analytical tasks.

The *z*-score is calculated, for each value of k, as in Eq. (3).

$$z = \frac{x_i - X_A}{\sigma_A} \tag{3}$$

The *z*-scores calculated for k=1.0 are statistically equivalent to those calculated in the WEPAL programme.

A set of seven different outlier rejection tests were used to test for the presence of outliers and reject any outliers found. The results that passed the outlier rejection procedures were used to calculate the consensus mean value of analyte and corresponding consensus value of its standard deviation.

3.6.3 IAEA evaluation from 2021

The evaluation method introduced in 2021 is in compliance with ISO 13528 [3]. Assigned values were available as the ones certified by the external producer of the test material. For elements without certified mass fractions, robust statistics (Algorithm A in, Annex C3 of ISO 13528 [3]) has been used for estimating the robust average value (x_i*) as assigned value and the robust standard deviation (s_i*).

The uncertainty of the assigned value $u(x_{pt})$ is the combined standard uncertainty of the assigned value. If the property values have been declared as "certified" by the producer of the material,

e.g., obtained through an independent inter-laboratory survey, $u(x_{pt})$ is determined as the standard deviation of the mean property value: $u(x_{pt}) = SD/\sqrt{N}$. When certified values are not available, $u(x_{pt})$ can be obtained from the results of the participants of the proficiency test, through the application of robust statistic methods. It is shown in Eq. (4).

$$u(x_{\mathrm{pt}}) = 1.25 \cdot \frac{s^*}{\sqrt{p}} \tag{4}$$

where s^* = standard deviation of the consensus value and p = number of results for that element in this test.

The standard deviation for proficiency assessment σ_{pt} is determined from the assigned values using the modified Horwitz function (σ_{R}) as in Eq. (5).

$$\sigma_{\mathrm{pt}} = \sigma_R = \begin{cases} 0.22x_{pt} & when \quad x_{\mathrm{pt}} < 1.2 \cdot 10^{-7} \\ 0.02(x_{pt})^{0.8495} & when \quad 1.2 \cdot 10^{-7} \le x_{pt} \le 0.138 \\ 0.01\sqrt{x_{pt}} & when \quad x_{pt} > 0.138 \end{cases} \tag{5}$$

Both blunders and outliers have been defined. Blunders are values differing more than an order of magnitude from the median of all submitted results; they are not further considered in the application of robust statistics. Outliers are values differing more than 4.5 standard deviations from the assigned value. Outliers are taken into account for the determination of the consensus values and their standard deviations through robust statistics if there are at least five valid results. It is noted that outlier values do not affect the evaluation of submitted results for the elements with assigned values but may affect the evaluation of submitted results for the other elements as they contribute to the estimation of x_i^* and s_i^*.

In case $u(x_{\mathrm{pt}}) \le 0.3\sigma_{pt}$, for every result (including those identified as blunders and outliers) a z-score was calculated, defined as Eq. (6).

$$z_i = \frac{(x_i - x_{pt})}{\sigma_{pt}} \tag{6}$$

where x_i is the reported mass fraction of the measurand.
If $u(x_{pt}) > 0.3\sigma_{pt}$, for every result a z'-score was calculated, defined as Eq. (7).

$$z_i' = \frac{(x_i - x_{pt})}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}} \tag{7}$$

The z and z' -scores are calculated even if there are only a few results and if it is an element with a (certified) assigned value.

The submitted results were accompanied by the estimate made by the participant of the combined standard uncertainty $u_{x,i}$. These values were used to calculate the zeta scores illustrated in Eq. (8).

$$\zeta_i = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \quad (8)$$

In order to provide a performance indicator having an easier and more intuitive interpretation than z- and z'-scores, the values of the ratios R_i were also reported as Eq. (9).

$$R_i = \frac{x_i}{x_{pt}} \quad (9)$$

Although this parameter is not included in ISO 13528 [3], its values can provide more direct feedback on the data submitted to the participant.

The equation for the z-score can also be expressed as a function of the laboratory mass fraction, x_i and assigned value, x_{pt} only, resulting in a relationship of the z-score with the relative bias of the laboratory result and the assigned value. This results inEqs (10–12) denoting the standard deviation for proficiency assessment, σ_{pt}, is differently related to the assigned value by the modified Horwitz expression:

For $x_{pt} \leq 1.2.10^{-7}$	$R_i = 0.22 * z + 1$	(10)
$1.2.10^{-7} < x_{pt} < 0138$	$R_i = z * 0.22*(x_{pt})^{0.1505} + 1$	(11)
$x_{pt} \geq 0.138$	$R_i = z * 0.01/\sqrt{(x_{pt})} + 1$	(12)

The R_i values calculated with these equations are graphically shown in Figure 1.

It can thus be seen that a value of $|z| = 3$ indicates that the result can deviate by about 10% to the assigned value for mass fractions of several percent to up to a factor of 2 from the assigned value for mass fractions of about 0.1 mg·kg^{-1}.

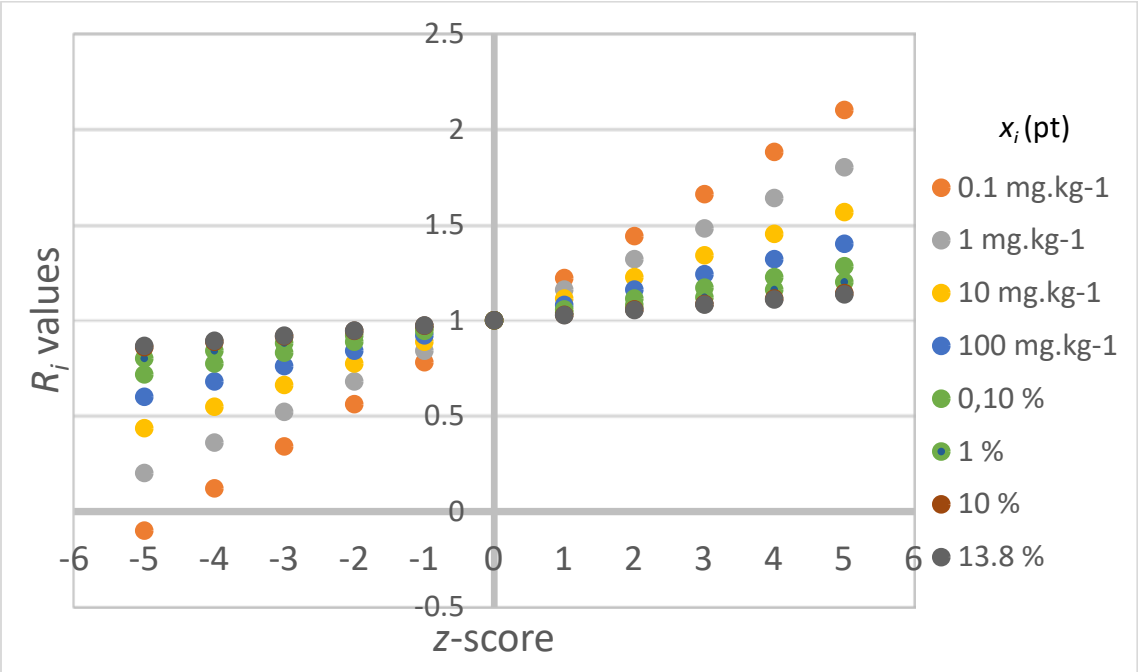


FIG. 1. Relation between the ratio R_i of a measurement result, x_i, and the assigned value, x_{pt}, and the z-score for various values of the mass fraction of the assigned value, x_{pt}.

3.6.4 Additional performance criterion

Participants of the IAEA Feedback Training Workshop in 2015 in Delft, The Netherlands, recommended the IAEA describe their performance with an additional metric [24], derived from the z-scores. Typically, participating NAA laboratories reported mass fractions for up to 25 elements in each sample of the soil type materials, and up to 20 elements in each sample of the biological type material. The previous performance of some NAA laboratories in proficiency testing exercises showed that they could routinely report at least 90% of data with $|z|< 3$. As such, this 90% percentage was recommended as an indicator for "excellent" performance of the NAA laboratories. The recommendation resulted further into three categories of performance:

1. Metrological satisfactory, "**Excellent**", if ≥ 90% of the submitted data has $|z| < 3$.
2. Metrological less satisfactory, "**Satisfactory**"[4], if ≥70% and < 90% of the submitted data has $|z| < 3$. Minor to substantial improvements are needed to reach a higher level of performance.
3. Metrological unsatisfactory, "**In development**", if < 70% of the submitted results has $|z| < 3$. Major improvements are needed to reach an acceptable level of performance.

This evaluation is in agreement with clause 9.9.4 of ISO 13528 [3] in which it is stated that 'In proficiency testing schemes that involve a large number of measurands, a count or proportion of the numbers of action and warning signs can be used to evaluate performance'.

The (z, z')-scores do not take into account the uncertainty of measurement of the laboratory itself. As a result, laboratories often use the zeta score, which can lead to a smaller number of unsatisfactory data since the denominator in the equation of the zeta score might be larger than the standard deviation for proficiency testing, the denominator in the equation of the z or z'-score is illustrated in Eqs (6 –8).

The performance indicator (percentage of the arithmetic sum of submitted data with $|z|$ resp. $|z, z'| < 3$) was used to follow the evolution of the performance of the participating laboratories over the years, in successive proficiency testing rounds. It is noted that the performance indicator was developed for this purpose and is not based on international conventions.

3.6.5 Degree of equivalence of WEPAL and IAEA evaluation

A comparison of the data evaluations by WEPAL and the IAEA is given in Table 7. The main difference in the calculation of the z-scores is with the value of the denominator in the equation of the z-score, i.e. the standard deviation for proficiency assessment, σ_{pt}. WEPAL uses the standard deviation of the mean of the robust consensus value for all measurands (at least 8 valid results) whereas the IAEA uses the modified Horwitz standard deviation in both their approaches until and after 2021. In addition, the IAEA started to apply the z' score for cases in which the uncertainty of the assigned value is more than 30% higher than the standard deviation for proficiency assessment, which is not done by WEPAL.

[4] Participants from the 2017 IAEA Feedback workshop in Ljubljana recommended to replace the original annotations 'excellent', 'average' and 'poor', to keep the categorization on basis of the percentage of submitted data but also to refer to the number of elements reported [25]. The IAEA has accepted to change the annotations but did not follow-up on the second recommendation.

TABLE 7. COMPARISON OF PARAMETERS USED IN THE EVALUATION OF THE SUBMITTED RESULTS INTERLABORATORY COMPARISONS BY WEPAL, IAEA UNTIL 2021 AND IAEA FROM 2021 ONWARDS

	WEPAL (2010–2017)	IAEA (2018–2020)	IAEA (from 2021)
Assigned value, x_{pt}	Not defined. Information derived from equation for the *z*-score: Mean of all values calculated by the normal distribution approximation (NDA). At least 8 results. Outliers are not removed.	Reference values provided by producer of the material. Consensus value of basis of at least 5 valid results (outliers removed) , provided data has normal distribution.	Certified values provided by producer of material. Non-certified values: Consensus value on basis of at least 5 valid results (blunders removed) following algorithm A of ISO 13528 [3].
Standard deviation of assigned value, *SD*	Not defined	Not defined	Standard deviation of the certified property values, as declared by the producer of the material. Standard deviation *s** on basis of consensus value used as assigned value.
Uncertainty of assigned value, $u(x_{pt})$	Robust standard deviation divided by S QRT of number of results $u(x_p = SD_{robust}/\text{sqrt}(N)$ *N*= number of results	Not defined	For certified values: $u(x_{pt}) = SD/\text{SQRT}(N)$ (*N*= number of results contributing to the certified value) For non-certified values: $u(x_{pt})=1.25 \cdot s*/\sqrt{p}$
Standard deviation for proficiency assessment, σ_{pt}	Derived from equation for the *z*-score: Standard deviation calculated with the NDA model	Based on Horwitz-Thompson standard deviation	Based on Horwitz-Thompson standard deviation
Consensus value	Not defined as such but the same as x_{pt} i.e., the mean as calculated using the NDA model. At least 8 results.	Mean of reported values, outliers removed.	Algorithm A from ISO 13528 [3]; at least 5 valid results, blunders removed.
Straggler	$2 < \|z\| \leq 3$	Not defined	Not defined
Outlier	$\|z\| > 3$	Based on 7 outlier tests.	$\|z\| > 4.5$
Blunders	Not defined	Not defined	$x > 10 * x_{median}$
ISO/IEC 17043 [2]	Yes, accredited	Yes	Yes
ISO 13528 [3]	No	No	Yes
z-score	$(x\text{-}x_{pt})/\sigma_{pt}$	$(x\text{-}x_{pt})/\sigma_{pt}$	$(x\text{-}x_{pt})/\sigma_{pt}$
Action if $u(x_{pt}) > 0.3\sigma_{pt}$	No action	No action	$z' = (x\text{-}x_{pt})/\sqrt{((x_{pt})^2+(\sigma_{pt})^2)}$

The data provided by the producer of the test materials in the 2021 exercise (PTNATIAEA19) was used to compare the modified Horwitz standard deviation, σ_R, with the standard deviation of the certified values calculated with the NDA model by WEPAL, SD(NDA). It can be seen from Figs 2 and 3 that σ_R is equivalent within +/- 20% to the SD(NDA) in the range of 1 mg·kg^{-}

[1] to about 100 mg·kg^{-1}, up to a factor of 2 smaller than the SD(NDA) for mass fractions > 100 mg·kg^{-1}, and up to a factor of 2 lower for mass fractions < 1 mg·kg^{-1}.

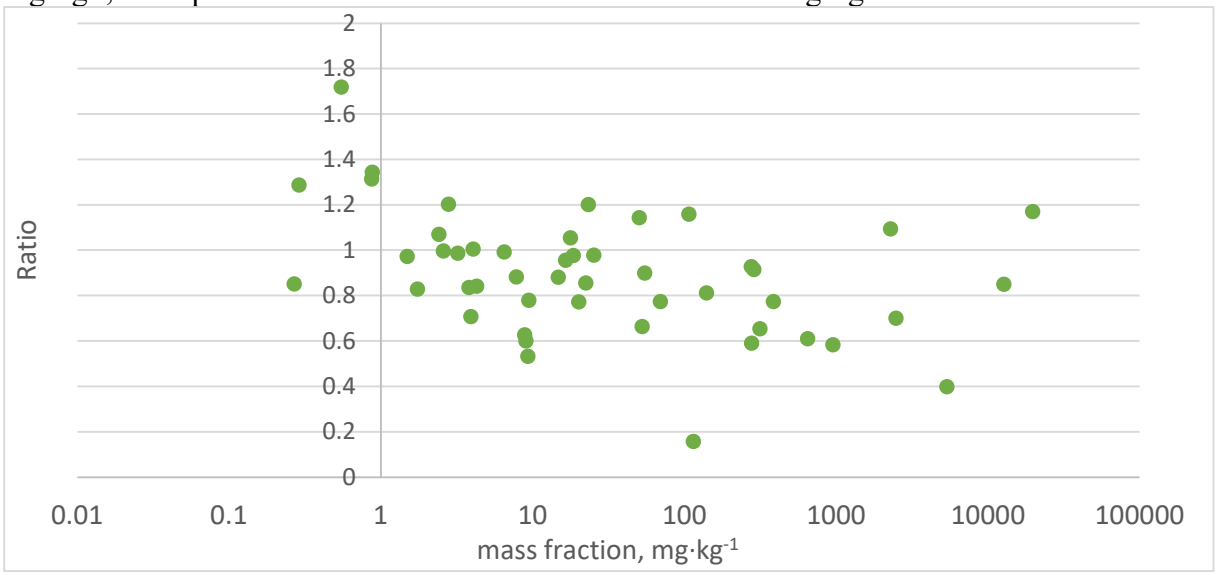


FIG. 2. Ratio between the modified Horwitz standard deviation σ_R and the standard deviation of the assigned value on basis of the NDA model, SD(NDA) for the soil type material sample, used in PTNATIAEA19.

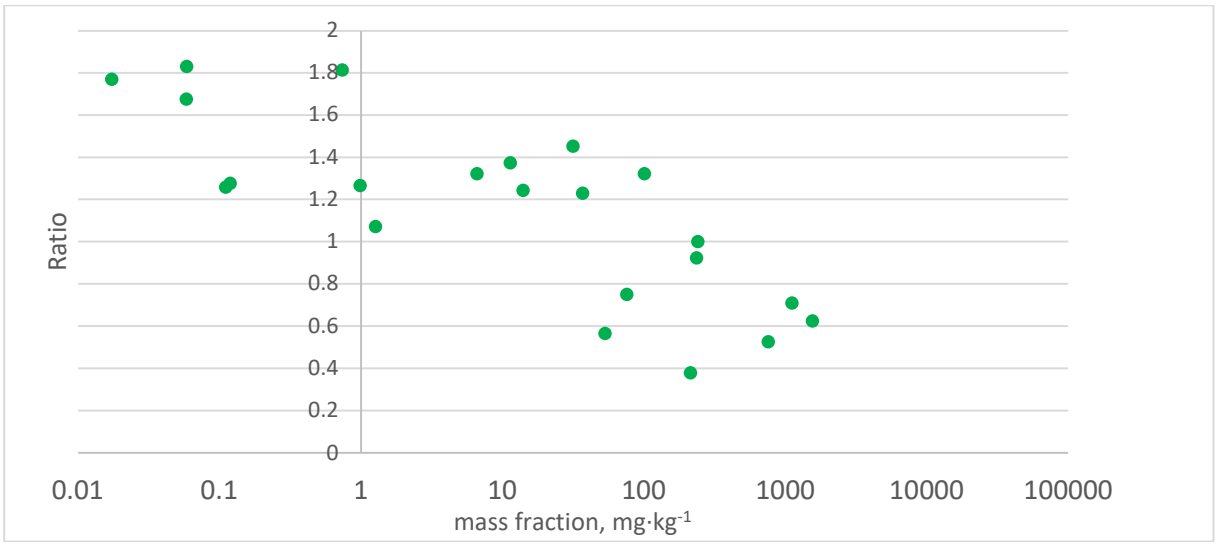


FIG. 3. Ratio between the modified Horwitz standard deviation σ_R and the standard deviation of the assigned value on basis of the NDA model, SD(NDA) for the biological type materials used in PTNATIAEA19.

As such, *z*-scores calculated in IAEA interlaboratory comparisons would be equivalent to those calculated by WEPAL for mass fractions in the range of 1 mg·kg^{-1} to 100 mg·kg^{-1}, but IAEA *z*-scores could be up to a factor 2 larger than the *z*-scores calculated by the WEPAL for elements with mass fractions > 100 mg·kg^{-1}, and up to a factor 2 smaller for mass fractions < 1 mg·kg^{-1}.

The degree of equivalence of the *z*-score calculation by the software used by the IAEA until 2021 and from 2021 onwards was evaluated by reprocessing the submitted results by NAA laboratories in PTNATIAEA16 (2017) and PTNATAIAEA17 (2018) with the new IAEA software. The percentage of submitted results with $|z, z'| < 3$ was compared with the original percentage of $|z|<3$ and is shown in Table 8.

There are some differences in the percentage of submitted results but, with a few exceptions, it does not affect much the interpretation of the performance in terms of the classification as

'excellent', 'satisfactory' and 'in development'. The major differences (the scores from laboratories 139 and 195 for the results of the biological type material, in **bold** in Table 8) are due to the small number of reported results and the fact that the old software did not calculate any score for outliers, whereas in the new software, the *z*' score is calculated for such results.

TABLE 8. COMPARISON OF THE PERCENTAGE OF SUBMITTED RESULTS WITH $|z| < 3$ AND $|z, z'| < 3$ IF THE SAME SUBMITTED DATA ARE EVALUATED USING THE IAEA SOFTWARE IN USE UNTIL 2021 ('OLD') AND FROM 2021 ONWARDS ('NEW')

Percentage of data with |z, z'|<3 (New) and |z|<3 (Old)						
	PT16, biological type		PT17, soil type		PT17, biological type	
NAA Lab code	New	Old	New	Old	New	Old
40	90	91	85	85	89	87
55			95	95	64	44
61	89	88	96	96	100	93
76			**71**	**60**	29	25
139	67	56	100	100	**100**	**50**
149	69	57	50	50	40	38
152	78	84	78	70	64	56
167	86	75				
169	**71**	**67**	100	100	100	100
170	86	83	100	93	71	73
171	92	92				
172	74	79	100	100	83	81
175	83	83	100	97	67	67
176	89	84	100	97	100	92
178	57	56	85	82	38	21
182			100	100		
183	69	67	**90**	**77**	100	82
184	63	63	96	96	**100**	**63**
186	64	60	81	73	50	50
191	100	100				
192			93	93	85	83
193	74	75	100	96	45	35
194			88	81	67	67
195	82	82	67	67	**100**	**40**
196	100	100				
198			100	100	100	92
199	86	86	91	82	83	88
202	60	45				
203	67	62	82	82	86	70
211			35	35	0	0
213			62	69	0	14
214			100	100	**86**	**90**
215			100	97	93	95
222			100	100	90	93
223			**80**	**64**		

Percentage of data with |z, z'|<3 (New) and |z|<3 (Old)					
	PT16, biological type	PT17, soil type		PT17, biological type	
224		100	97	92	93

In **bold:** see text.

The degree of equivalence of the $|z,z'|$ calculation with the IAEA software from 2021 onwards with the z-score calculation on basis of WEPAL's NDA model was also verified using the submitted results by NAA laboratories in PTNATIAEA19. The materials used for testing in PTNATIAEA19, WEPAL RM ISE 861 (Calcareous Clay, here denoted as 'Clay') and RM IPE 166 (Cherry Laurel / Prunes laurocerasus, here denoted as 'Plant'), have both been characterized by WEPAL on the basis of multiple interlaboratory comparison data and application of the NDA software, resulting in certified values and robust standard deviations. The results of this evaluation are shown in Table 9.

The differences in performance on basis of the $|z, z'|$ score or on the $|z|$ score, and resulting in a different categorization, are also relatively small here (marked in **bold** in both tables); a few large differences are again related to a relatively small number of results submitted.

It can be concluded that differences in the degree of equivalence between the data evaluation by WEPAL, IAEA between 2018 and 2020 and IAEA from 2021 do not seem to contribute to significant differences in the interpretation of the trend in performance of the NAA laboratories.

3.7 FEEDBACK AND QA/QC TRAINING WORKSHOPS

The IAEA implemented feedback and QA/QC workshops (see Table 10) for further discussion of the results, metrological feedback by IAEA experts on potential sources of analytical error, and recommendations for associated QA/QC practices. It is noted that the feedback workshops for the evaluation of the results from the PTs organized in 2019 and 2020 did not take place due to the COVID-19 pandemic. However, some aspects of ensuring the validity of results and QA/QC in NAA were discussed in 2018 during an IAEA Training Workshop on the IAEA e-learning course on NAA. Similarly, QA/QC aspects of NAA were discussed in an IAEA Training Workshop on Optimization of performance and processes in NAA in 2020.

Participating laboratories were encouraged to select the person(s) that actually carried out the analyses as their representative for the feedback and QA/QC workshops. Each laboratory provided details of the analytical procedure followed by them in the interlaboratory comparison testing, using a pre-established reporting template. The details presented included, for instance, the sample masses, dry mass assessment, calibration procedure, irradiation and measurement geometries, corrections for neutron flux gradients, internal QC applied and status of QA implementation.

The results were discussed within the broad platform of participants, moderated by the IAEA experts and the IAEA officer for these projects. Participants provided their own view on their performance and, if relevant and possible, their hypothesis on sources of error. The level of detail in these presentations often made it possible for the IAEA experts to point out the most probable cause of the deficiencies. A number of participants, in most cases either newcomer laboratories or established laboratories with inexperienced staff, did not have sufficient theoretical and practical knowledge to perform root cause analysis and implement adequate corrective actions. To address this issue, the feedback and QA/QC workshops also included lectures on the analytical and QA/QC procedures. Participants and experts discussed QA/QC

approaches for mitigation of sources of error and prevention of recurrence, and several best practices in the different types of calibration in NAA and other aspects of the analytical procedure.

TABLE 9. COMPARISON OF THE PERCENTAGE OF SUBMITTED DATA WITH $|z, z'|<3$ AND $|z|<3$, RESPECTIVELY OF THE SAME RESULTS ARE EVALUATED WITH THE IAEA SOFTWARE IN USE FROM 2021 ONWARDS, AND USING THE APPROACH USED BY WEPAL

	% data in PTNATIAEA19 Clay		% data in PTNATIAEA19 Plant	
NAA Lab code	IAEA approach $\|z,z'\|< 3$	WEPAL approach $\|z\|<3$	IAEA approach $\|z,z'\|< 3$	WEPAL approach $\|z\|<3$
55	100	100	92	92
61	100	100	92	92
84	83	83	50	50
99	80	80	**75**	**50**
149	78	88	25	25
152	85	88	**86**	**69**
166	93	95	100	100
167	83	82	78	78
169	100	100	100	100
170	100	100	100	100
171	93	92	100	100
172	94	92	93	92
176	100	100	100	100
181	100	100	**67**	**100**
182	**84**	**92**	36	40
183	94	96	63	67
192	100	100	100	94
194	77	84	33	45
195	50	63	78	78
197	33	40	75	67
199	100	100	100	100
202	93	95	67	64
203	86	93	80	87
205	82	88		
215	100	100	100	100
217	88	82	58	64
219	67	85	80	89
221	93	96	19	7
232	100	100	100	100
237	97	100	100	100
240	46	55	75	83
245	81	81	83	80
247	100	100	100	100
248	95	93	100	94

In **bold**: see text.

The lessons learned during the workshops from 2010–2015, and the associated recommendations have been summarized in a previous IAEA publication [4].

The participants in the 2017 training workshop emphasized, in addition to the need to follow-up on the recommendations from previous workshops, the importance of careful independent and documented verification of the validity of measurement results prior to reporting, the need

for protocols to prevent transposing and reporting errors, and the importance of having succession and knowledge management plans.

TABLE 10. OVERVIEW OF FEEDBACK AND QA/QC TRAINING WORKSHOPS

Applicable rounds	QA/QC workshop	Date	Location
2010-3 and 2010-4		12–16 September 2011	Antananarivo, Madagascar
2011-4 and 2012-1		22–25 May 2012	Delft, Netherlands
2011-4 and 2012-1		4–8 June 2012	Tunis, Tunisia
2012-1 (R) and 2013-1		27–31 May 2013	Vienna, Austria
2015-1 and 2015-2		31August – 4 September 2015	Delft, Netherlands
2017-3		6–10 November 2017	Ljubljana, Slovenia
2010–2017	E-learning in NAA and QA/QC aspects	3–7 September 2018	Virtual, Vienna, Austria
2018, 2019	Optimization of performance and processes in NAA	30 November – 4 December 2020	Virtual, Vienna, Austria
2020, 2021	QA and QC in Proficiency Testing	22–26 August 2022	Delft, Netherlands

During the 2020 training workshop, participants concluded on the importance of a well-designed, optimised and documented QC programme and ensuring the validation of the NAA method. They also agreed that more insight is needed on how method validation in NAA can be done in a practicable and economical way. Risk based thinking needs to be considered for the different steps of the analytical method, thereby optimising some variables in the process (sample size, detector type and efficiency, decay and counting time, calculation steps). It was recommended to maintain control charts with, e.g. the results of analyses of reference materials to demonstrate the consolidation of the method's validation status and the validity of the measurement results.

The participants of the training workshop in 2022 repeated the recommendation from the training workshop in 2017 that laboratories at the lowest category of performance ("in development") need to consider stopping reporting results to external partners or customers if the risk of recurrence of unsatisfactory results is unacceptable prior to effectively mitigating its root cause by QA/QC actions. In addition, the participants expressed their concern that the current metric for performance evaluation does not account for the number of elements reported (see also Section 8).

Several of the recommendations on the conduct of NAA have been compiled in new modules on the concepts and practice of method validation, and a review of the modules related to QA/QC in the IAEA e-learning course on NAA [14] and in a 2022 IAEA publication Technical Reports Series No. 487 'Quality Assurance and Quality Control in Neutron Activation Analysis: A Guide to Practical Approaches' [13]. The workshops concluded with action plans for sustainable improvement of the analytical and organizational performance of the NAA laboratories and with recommendations towards the laboratories themselves, the IAEA and the national governments (if applicable).

4. RESULTS

This section provides the results obtained.

4.1 GENERAL OBSERVATIONS

WEPAL dispatches their samples approximately 3 months before the reporting deadline; the IAEA provided in the period 2018–2022 an approximately similar timeframe of 4-5 months for reporting. An NAA procedure requires typically measurements (i) within seconds to minutes after irradiation, (ii) 3-6 days after (a different second) irradiation and (iii) 3-4 weeks after the second irradiation during a few hours. There is only marginal added value of measurements made after an even longer decay period. The time between receipt of materials and reporting deadline is therefore, in principle, sufficiently long for conducting an NAA procedure on the two samples from the laboratory intercomparison exercise. NAA laboratories confirmed in advance that they would be able to report in time. However, in almost every proficiency testing round, several laboratories informed the IAEA that they were not able to report in time. Sometimes this information was sent in a timely manner, e.g. several weeks in advance of the deadline, but some NAA laboratories asked for late submission close to, or even after the date of the deadline. In some cases, this was related to planned or unplanned reactor maintenance activities.

Nonetheless, it has been repeatedly advised in the feedback training workshops to plan any analysis in such a way that ample time remains for final internal checks and formal authorization prior to release within the agreed time frame, and to ensure the availability of deputies for authorization of analysis reports and submission of results. Still, in the most recent interlaboratory comparison (PTNATIAEA20), with sample dispatch in August 2022, twenty (20) out of 34 NAA laboratories submitted their results in the last 4 days prior to the original deadline of November 25, 2022, and 8 NAA laboratories submitted their results up to December 10, 2022, only one thereof having informed the IAEA in advance. It is to be noted that reporting beyond the deadline without notifying the sample originator is not compatible with a service oriented laboratory practice, and it also violates clause 7.1.5 of ISO/IEC 17025 [1].

The performance indicators of analysis of soil type materials and biological type materials in successive interlaboratory comparison rounds by NAA laboratories from IAEA Member States in Africa, Europe, Latin America and the Caribbean, Asia and the Pacific, and North America are described below.

4.2 NAA LABORATORIES FROM EUROPE

Several NAA laboratories in Europe have the ability of achieving a performance categorization of ‘excellent’ both for the soil type material and the biological type material, as can be derived from Figs 4 and 5. The percentage of results with $|z|<3$ is lower for the biological type material. Several laboratories participated in 2021 for the first time and their performances vary from ‘excellent’ to ‘in development’. No conclusions can be drawn if an excellent performance of such a first-time participant reflects a sustainable and effective QA/QC or, a less optimal performance might indicate a systematic lack of QC, or incidental analytical and/or organizational errors (such as transposing and/or reporting errors).

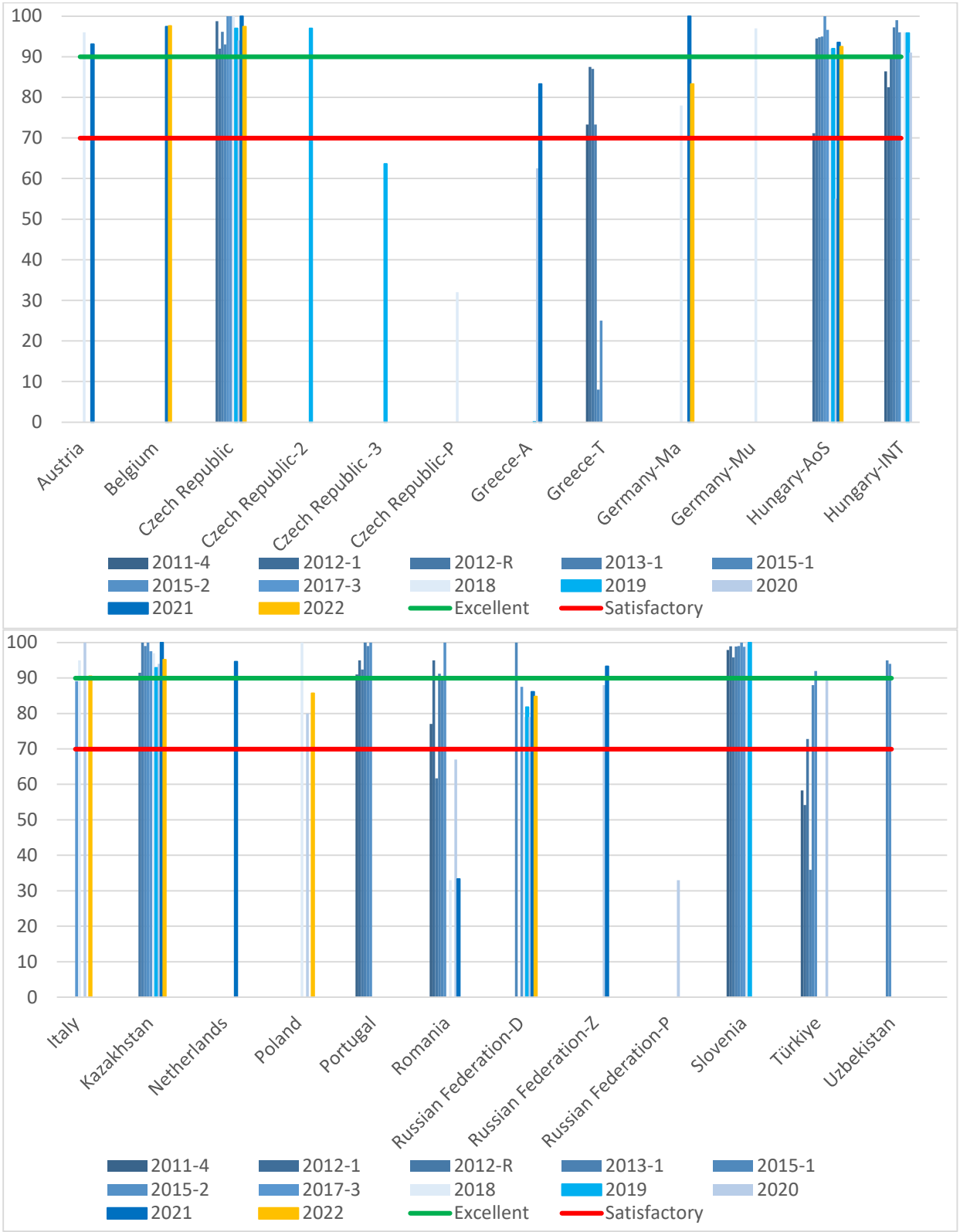


FIG.4. Percentage of data, submitted by NAA laboratories (see Table 3) from Europe in successive interlaboratory comparisons of soil type material from 2011 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

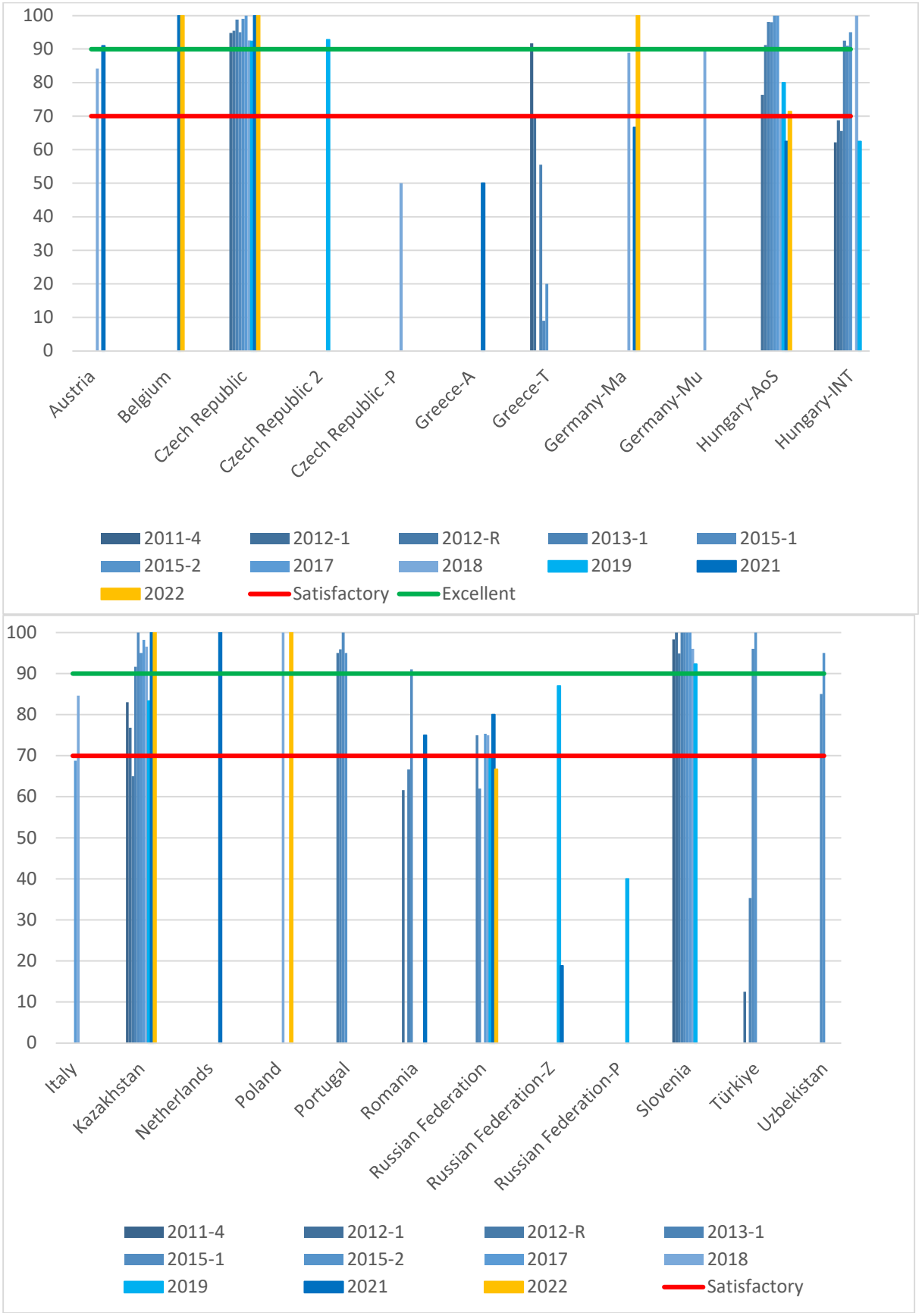


FIG.5. Percentage of data, submitted by NAA laboratories from Europe in successive interlaboratory comparisons of biological type material from 2011 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

There are a few laboratories that participated in successive years with consolidated excellent performance in both types of sample, such as the laboratories from Czech Republic, Kazakhstan and Slovenia. Others are having difficulties in improving their performance and remain categorized as 'satisfactory' and a few are facing a declining performance. It is noted that the NAA laboratory from Greece uses a low-flux ^{241}Am-Be isotopic neutron source resulting in measurement capability of only a few elements; any result with $|z| \geq 3$ has therefore a more significant impact of the performance categorization than for laboratories submitting many more results. As mentioned before, the laboratory from Hungary-K is reporting results obtained both by NAA and prompt gamma activation analysis (PGAA), and the decline in performance seen since 2018 seems to be due to a combination of reporting a relatively small number of results for the biological type material and within these, some unsatisfactory results obtained by PGAA. As an example, in the PTNATIAEA19 round of 2021, the results based only on NAA would render this laboratory a 100% percentage for soil type sample analysis (aggregate 93%) and an 83% score for the biological type sample analysis (aggregate 62%).

A summary of the performance by the NAA laboratories from Europe is shown in Figure 6. It is noted that not always the same laboratories participated in each of the interlaboratory comparisons and that the 2020 round included only a soil type material and not a biological type material (see also Table 2 in Section 3.4).

4.3 NAA LABORATORIES FROM AFRICA

Neutron activation analysis laboratories from all operational research reactors in Africa participated in all interlaboratory comparison rounds facilitated since 2010 by the IAEA for both types of material. The performance indicator, percentage of submitted results with $|z|<3$ is shown in Figures 7 and 8 for soil type material and biological type material, respectively.

Most laboratories have demonstrated their ability to analyse soil type material at the level leading to results categorized as 'excellent' in some of the intercomparison rounds (Figure 7). However, a decline in performance of these laboratories and inability in successive years to return and consolidate to the previous excellent performance level can be noticed.

Almost all laboratories have more difficulties with achieving a similar high fraction of accurate results in analysing the biological type material. Almost all laboratories reported in one or more proficiency testing rounds a high fraction of unsatisfactory results. A similar indication of decline in performance is visible for the analysis of biological type material (plant or animal tissue) can be recognized in Figure 8. The performance for the biological type material of the 2022 exercise needs to be evaluated with some care as all laboratories submitted results on only few elements — sometime less than five — and one or more unsatisfactory results has a significant impact on the performance indicator.

The reasons for a decline in performance and/or inability to improve are not clear. However, it indicates that not all laboratories are fully successful in bringing the recommendations from the feedback workshops for improving QA and QC in practice.

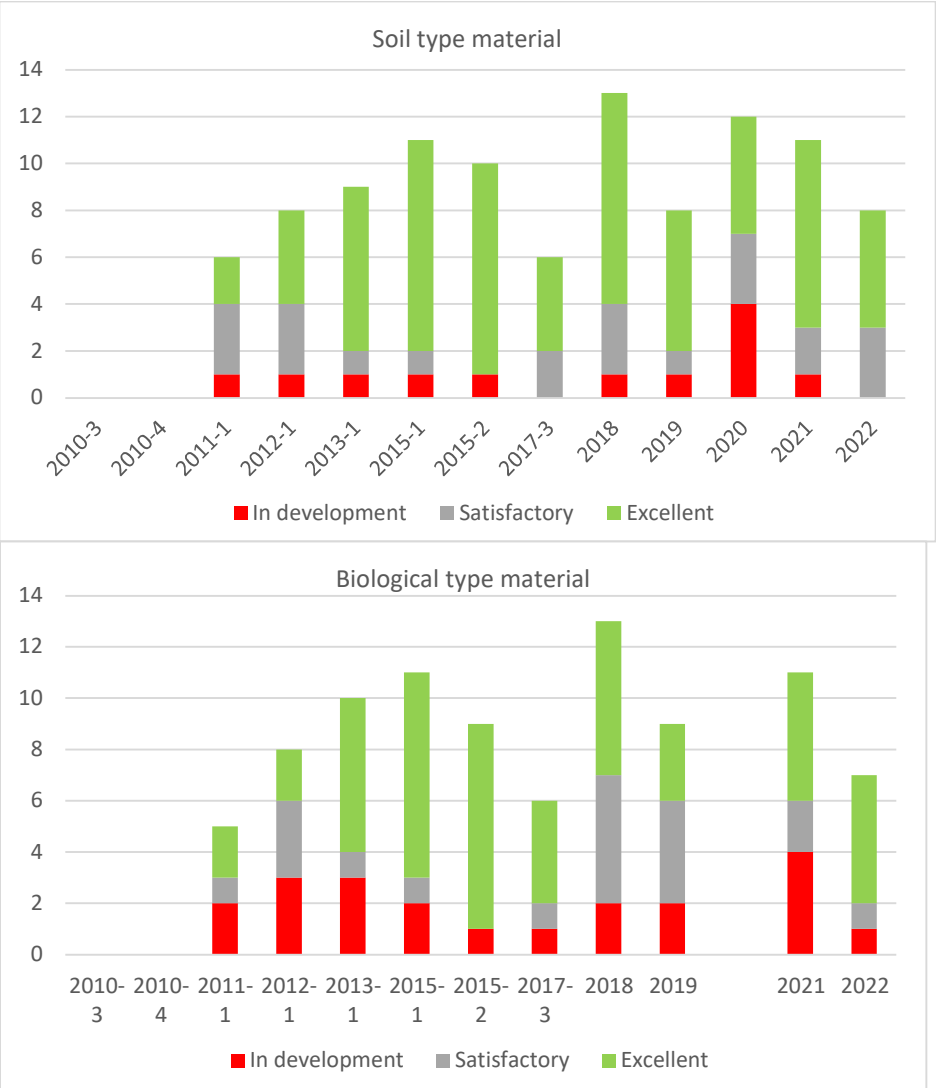


FIG. 6. Aggregate trends in performance by NAA laboratories from Europe in IAEA facilitated proficiency testing by laboratory intercomparisons from 2010–2022 on soil type and biological type materials.

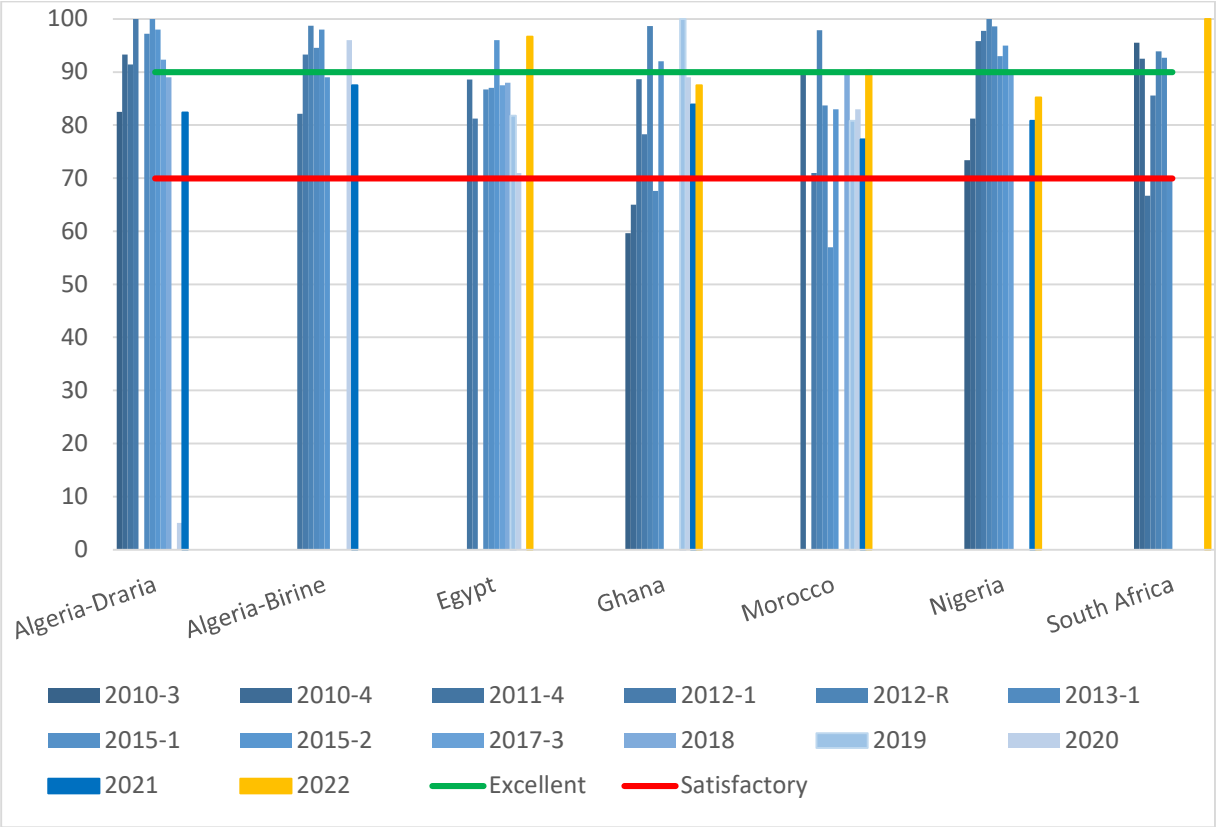


FIG. 7. Percentage of data, submitted by NAA laboratories (see Table 3) from Africa in successive interlaboratory comparisons of soil type material from 2010 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

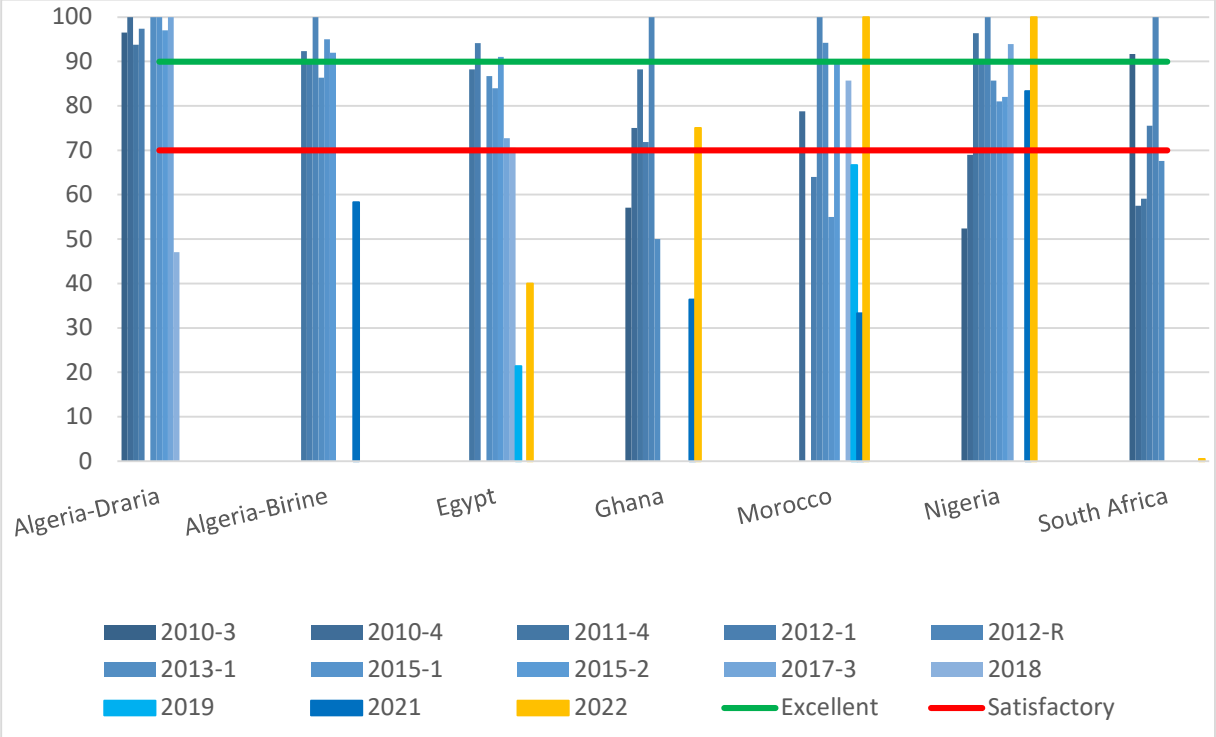


FIG. 8. Percentage of data, submitted by NAA laboratories from Africa in successive interlaboratory comparisons of biological type material from 2010 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

A summary of the performance by the NAA laboratories from Africa is shown in Figure 9. Not all the same laboratories participated in each of the interlaboratory comparison and the 2020 round included only a soil type material and not a biological type material (see also Table 2 in Section 3.4).

4.4 NAA LABORATORIES FROM LATIN AMERICA AND THE CARIBBEAN

Almost all NAA laboratories in this region demonstrate, consistently since 2011, the sustainability of their effective QA/QC systems, resulting in consolidated excellent performance in the interlaboratory comparisons, as is shown in Figs 10 and 11. With the exception of the results for the biological type material by the NAA laboratory from Mexico, there are no signs of systematic decline in performance as is observed amongst some of the NAA laboratories in e.g. Africa and Europe. Moreover, the differences in performance in the analysis of soil type and biological type material are small, which also confirms the effectiveness of the QA systems even if, as in some of the laboratories in this region, less experienced staff has continued the participation in the proficiency testing rounds.

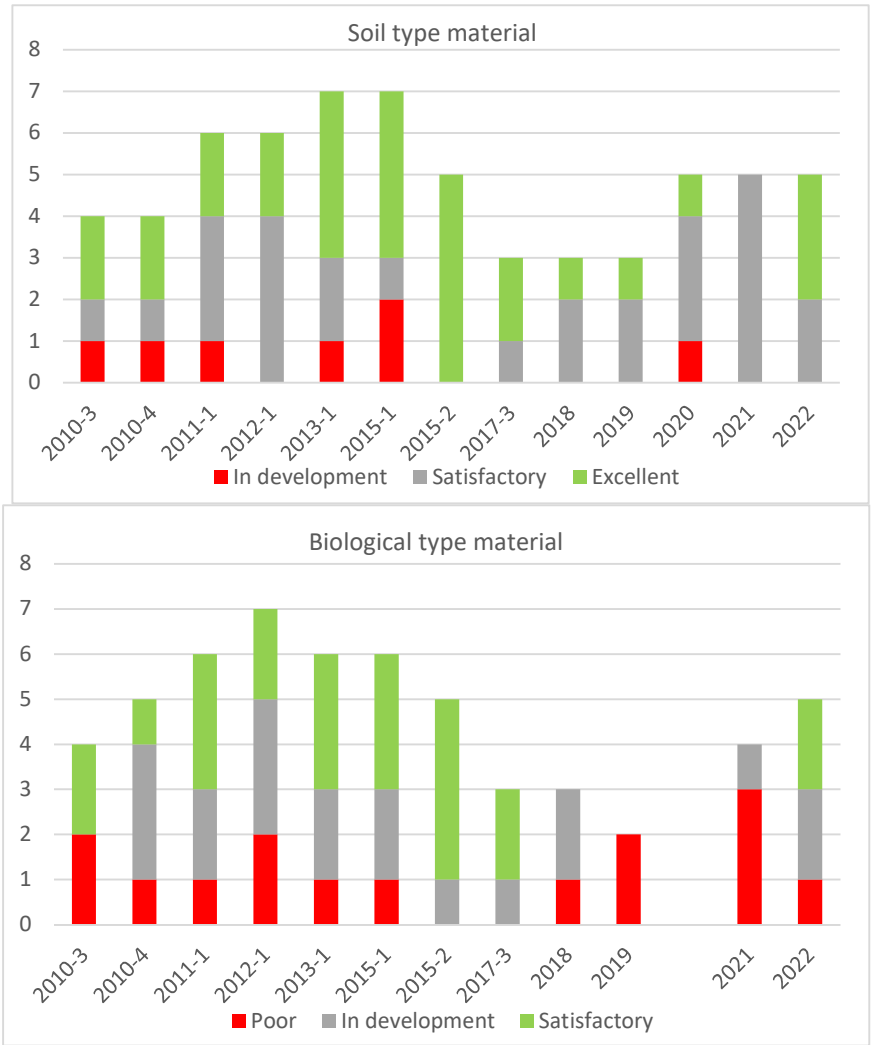


FIG. 9. Aggregate trends in performance by NAA laboratories from Africa in IAEA facilitated proficiency testing by laboratory intercomparisons from 2010–2022 on soil type and biological type materials.

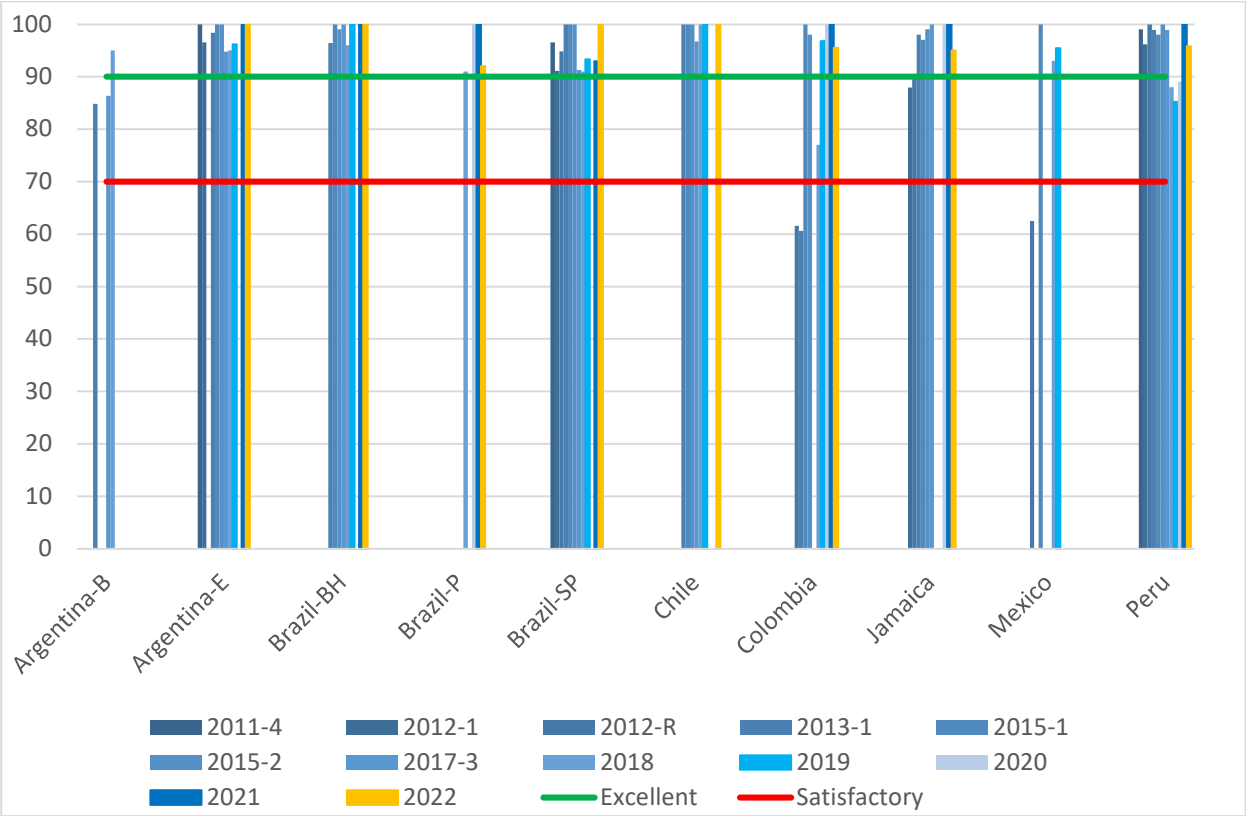


FIG. 10. Percentage of data, submitted by NAA laboratories (see Table 3) from Latin America and the Caribbean in successive interlaboratory comparisons of soil type material from 2011 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

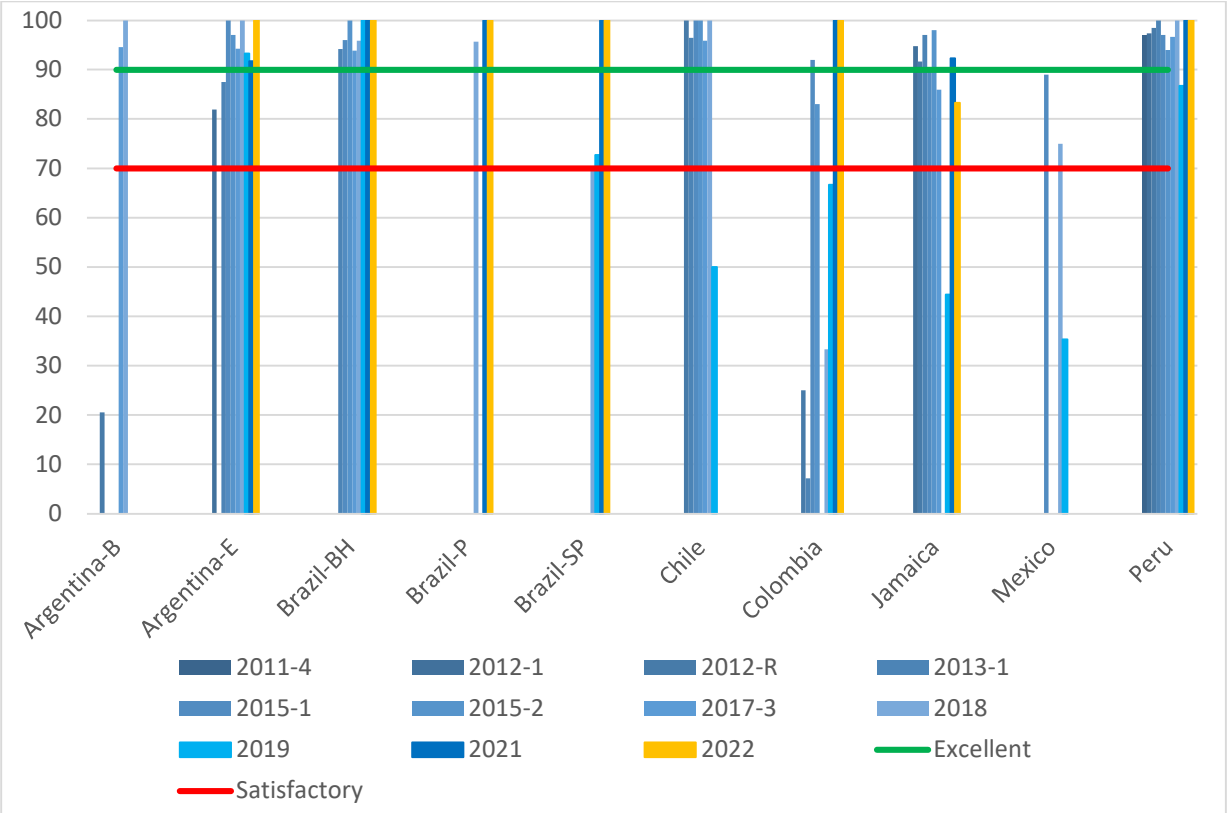


FIG. 11. Percentage of data, submitted by NAA laboratories from Latin America and the Caribbean in successive interlaboratory comparisons of biological -type material from 2011 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

A summary of the performance by the NAA laboratories from Latin America and the Caribbean is shown in Fig. 12. Often, the same laboratories participated from 2013 onwards in each of the interlaboratory comparisons and the 2020 round included only a soil type material and not a biological type material (see also Table 2 in Section 3.4).

4.5 NAA LABORATORIES FROM ASIA AND THE PACIFIC

The laboratory from Syria participated in all interlaboratory comparisons since 2011 with regularly (almost) continuous excellent performance as is shown in Figs 13 and 14 although some lesser results since 2018 can be noticed.

There are several NAA laboratories in this region that achieved excellent performance (such as from Australia, Syria and Viet Nam) but some of them participated only once or twice. Many other laboratories, including those that took part in several interlaboratory comparisons, do not seem to have found an effective approach to improve and sustain their QA/QC system and remain in the lower categories, a few of them even below the 'satisfactory' category or oscillating between 'excellent', 'satisfactory' and lower performance. The laboratories in this region also have more difficulties with attaining acceptable results for the biological type material. Some small steps towards improvement can be noticed but there are no indications of trends of systematic decline of performance. The performance by the NAA laboratory from Jordan is encouraging as it started its operations in 2016 only by relatively unexperienced staff.

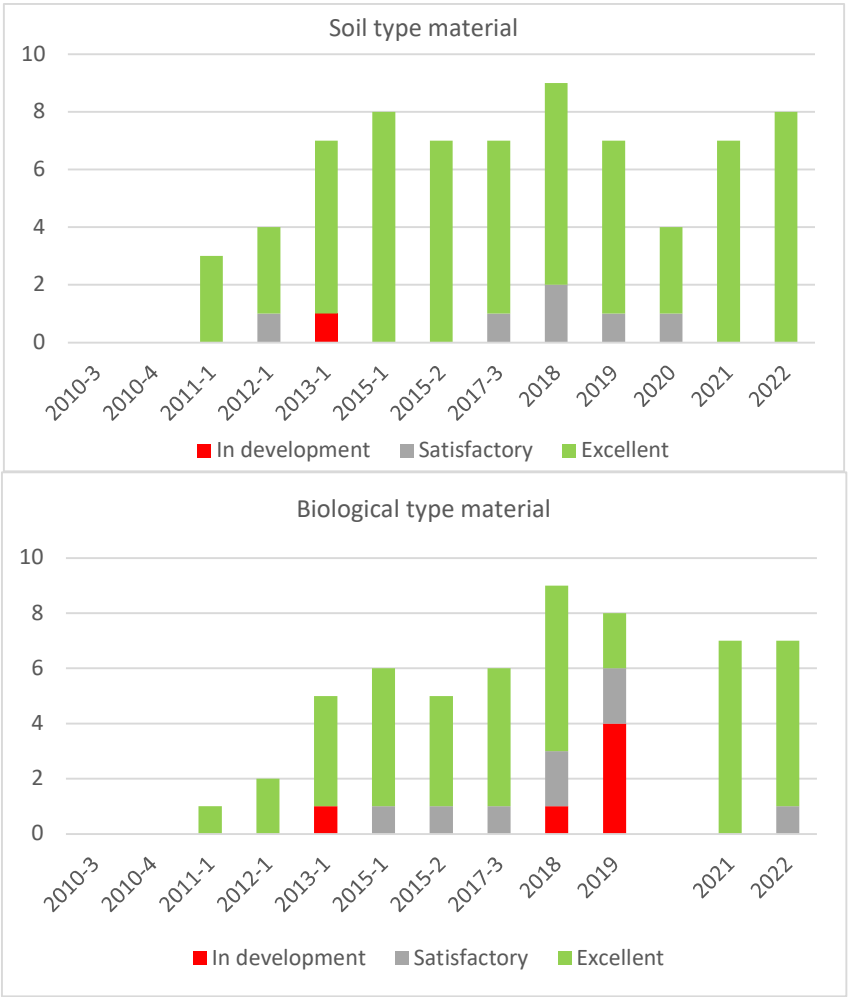


FIG. 12. Aggregate trends in performance by NAA laboratories from Latin America and the Caribbean in IAEA facilitated proficiency testing by laboratory intercomparisons from 2010–2022 on soil type and biological type materials.

FIG. 13. Percentage of data, submitted by NAA laboratories (See Table 3) from Asia and the Pacific in successive interlaboratory comparisons of soil type material from 2011 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

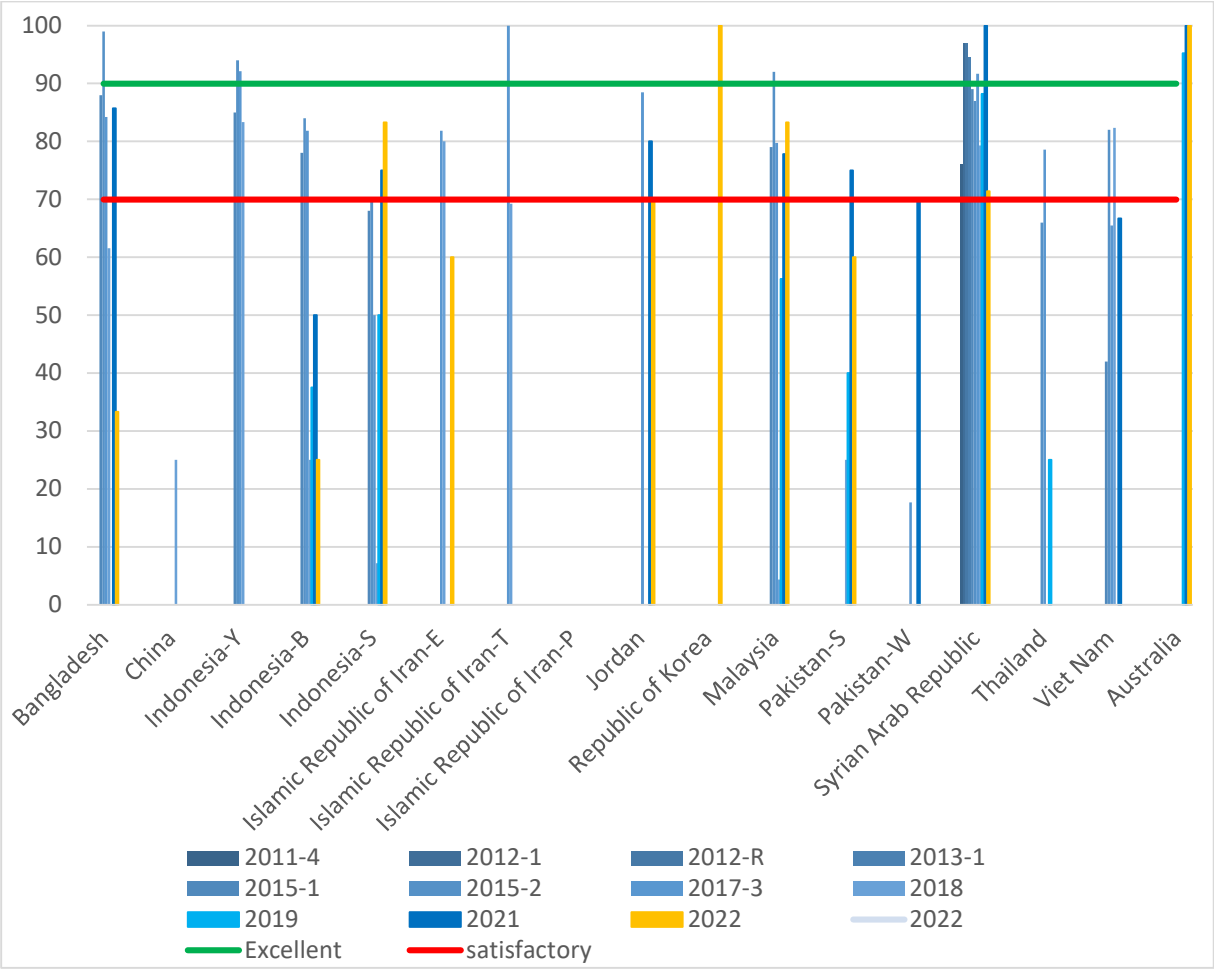


FIG. 14. Percentage of data, submitted by NAA laboratories from Asia and the Pacific in successive interlaboratory comparisons of biological type material from 2011 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

A summary of the performance by the NAA laboratories from Asia and the Pacific is shown in Fig. 15. Not all the same laboratories participated in each of the interlaboratory comparisons and the 2020 round included only a soil type material and not a biological type material (see also Table 2 in Section 3.4).

4.6 NAA LABORATORIES FROM NORTH AMERICA

Very few NAA laboratories from the North America region participated regularly in the interlaboratory comparison rounds, see Figs 16 and 17. The laboratories from Canada and the United States of America (United States of America-N) have excellent performance in analysing the soil type material, and the one from Canada also for the biological type samples; United States of America-N did not analyse this type of sample. The performance by the other NAA laboratories from the United States of America is below satisfactory but the limited participation does not allow to conclude whether this is due to lack of effectiveness of the QA implemented.

A summary of the performance by the NAA laboratories from North America is shown in Fig. 18. It is noted that, as explained and shown in the above, the number of participating NAA laboratories from North America is small, and only one laboratory participated in more than two interlaboratory comparisons. Moreover, the 2020 round included only a soil type material and not a biological type material (see also Table 2 in Section 3.4)

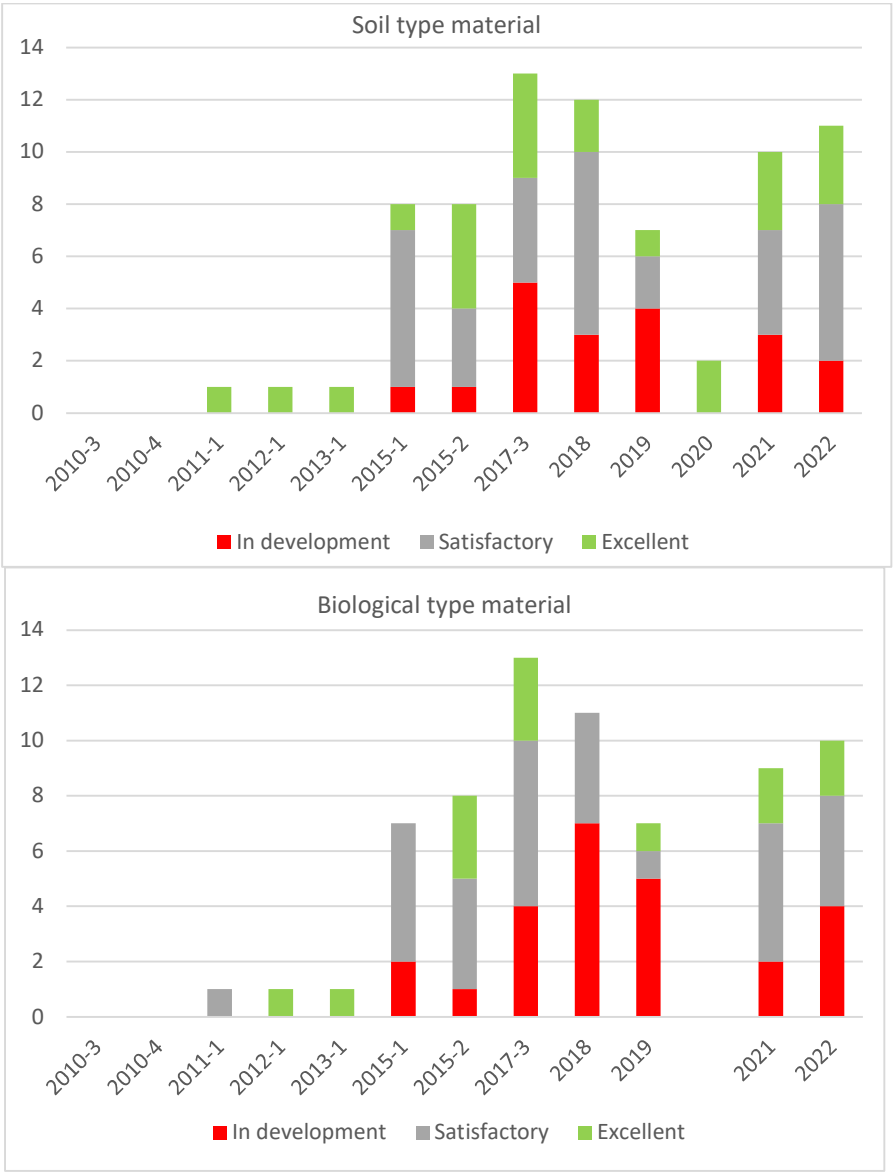


FIG. 15. Aggregate trends in performance by NAA laboratories from Asia and the Pacific in IAEA facilitated proficiency testing by laboratory intercomparisons from 2010–2022 on soil type and biological type materials.

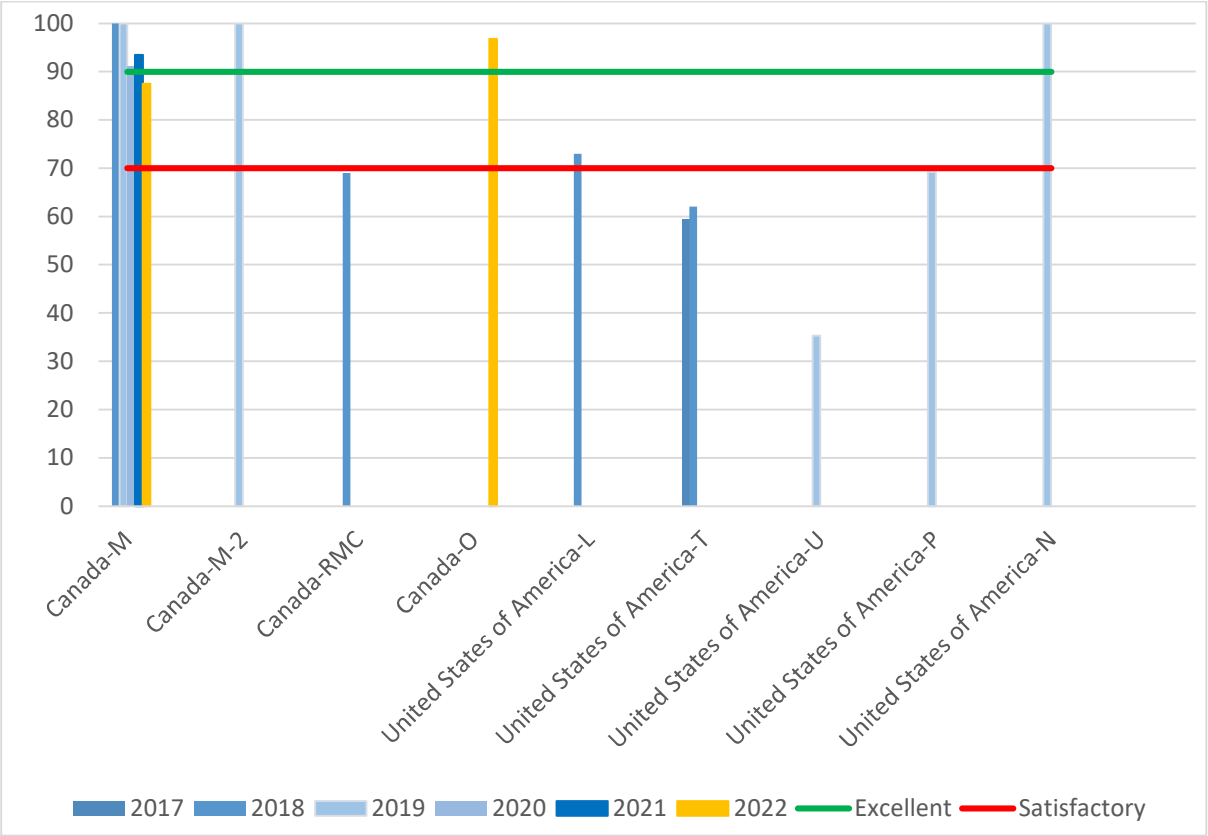


FIG. 16. Percentage of data, submitted by NAA laboratories (see Table 3) from North America in successive interlaboratory comparisons of soil type material from 2017 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

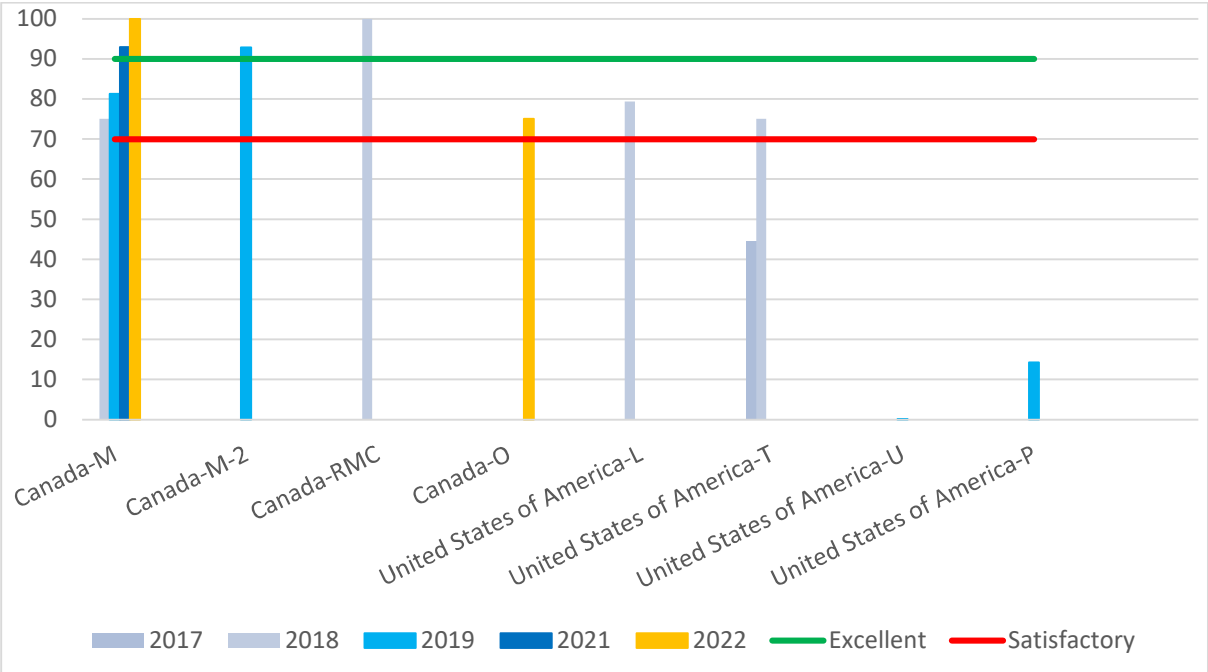


FIG. 17. Percentage of data, submitted by NAA laboratories from North America in successive interlaboratory comparisons of biological type material from 2017 until 2022, with indication of the minimum performance levels for laboratories to be categorized as of 'excellent' and 'satisfactory' performance.

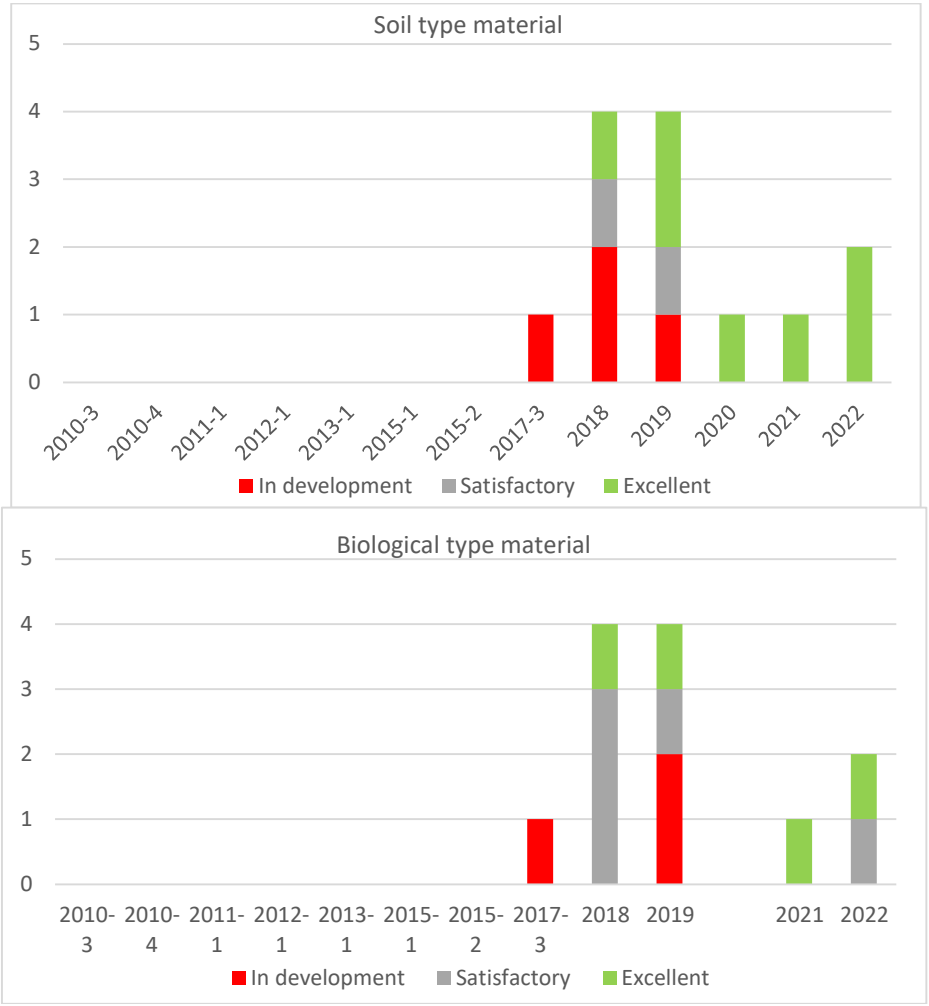


FIG. 18. Aggregate trends in performance by NAA laboratories from North America in IAEA facilitated proficiency testing by laboratory intercomparisons from 2010–2022 on soil type and biological type materials.

4.7 PERFORMANCE SUMMARIZED BY ALL REGIONS

A summary of the performance by the NAA laboratories from all regions is shown in Fig. 19 related to the number of participating laboratories, and to the percentage of qualification categories.

On the average, about 50–60% of the NAA laboratories have demonstrated controlling the validity of measurement results for both soil type and biological type material, resulting in the performance characterization ‘excellent’, in accordance with IAEA’s criteria. Only 5–10% of the NAA laboratories have significant difficulties with achieving satisfactory results in the analysis of soil type material, and up to 40% of the laboratories with the analysis of biological type material. The latter is most likely related to the relatively low trace element content resulting in enhanced risk of contamination during sample preparation and the associated low induced activity resulting in less favourable measurement conditions.

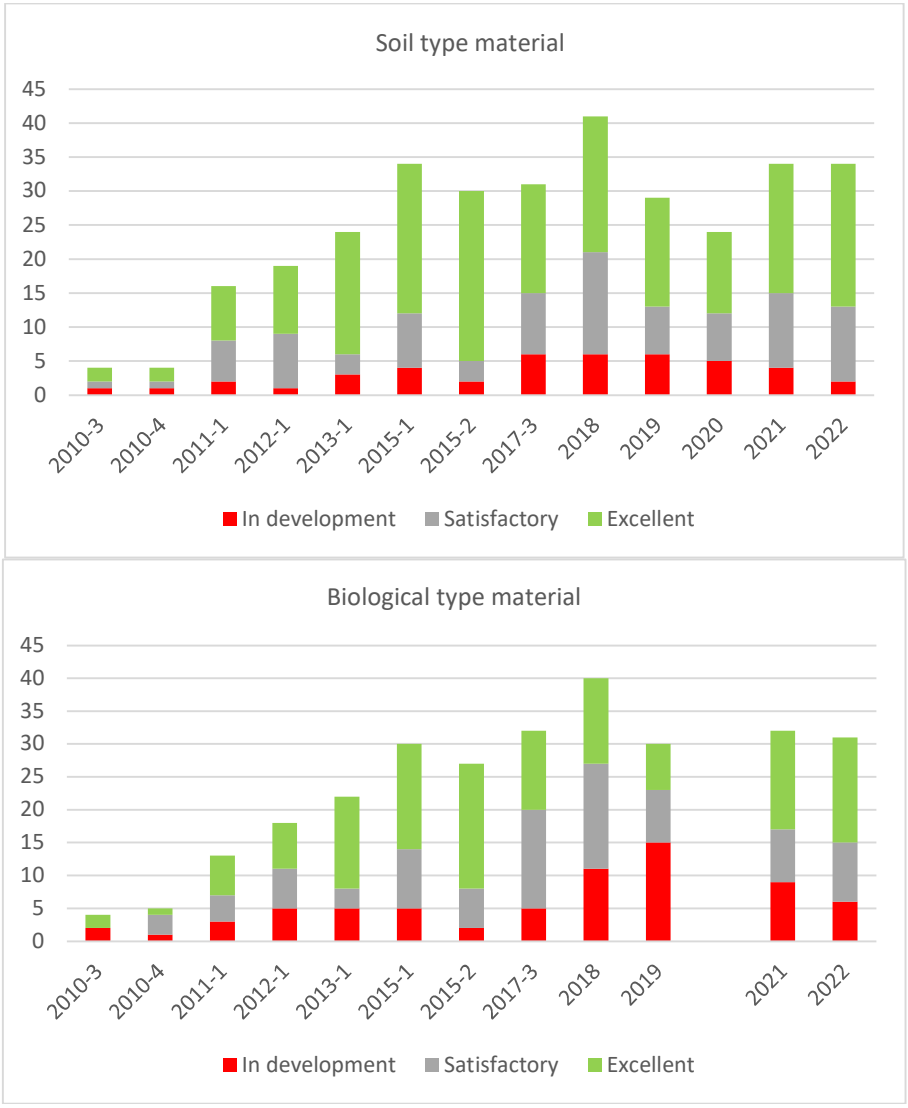


FIG.19. Aggregate trends in performance by NAA laboratories from all regions in IAEA facilitated proficiency testing by laboratory intercomparisons from 2010–2022 on soil type and biological type materials.

4.8 PERFORMANCE IN RELATION TO NAA METHODOLOGY

This section discusses the performance of laboratories with respect to the NAA methodology used.

4.8.1 Calibration method

Conversion of the measured gamma ray spectrum towards elements and their mass fractions can be done by three approaches; an absolute method, in which tabulated physical constants are used, the relative method in which the measurement data of an unknown sample is compared to those of a simultaneously processed sample of known composition, and the k_0 method of calibration [26] in which empirical calibrations are combined with tabulated physical constants. The latter two methods are the most commonly applied. The relative method is relatively easy to apply, as it does not require empirical calibrations (such as for the neutronic irradiation conditions and detector efficiency) and corrections (such as for the coincidence summing effects). The application of the method has limitations since mass fractions might not be known for all measurands in the reference sample. The k_0 method can provide mass fractions on any

element measured but requires extensive calibrations of reactor parameters and detector efficiency.

The relationship of performance of the NAA laboratories in PTNATIAEA20, conducted in 2022 and the calibration method are shown in Figs 20 and 21 for the soil type material (clay), and the biological type material (a land-based plant). Participants are grouped by their region.

It can be seen that the majority of the participants have applied the relative method in this interlaboratory comparison. Overall, there are no indications for a systematic correlation between the performance and the type of calibration (k_0 method or relative method) implemented.

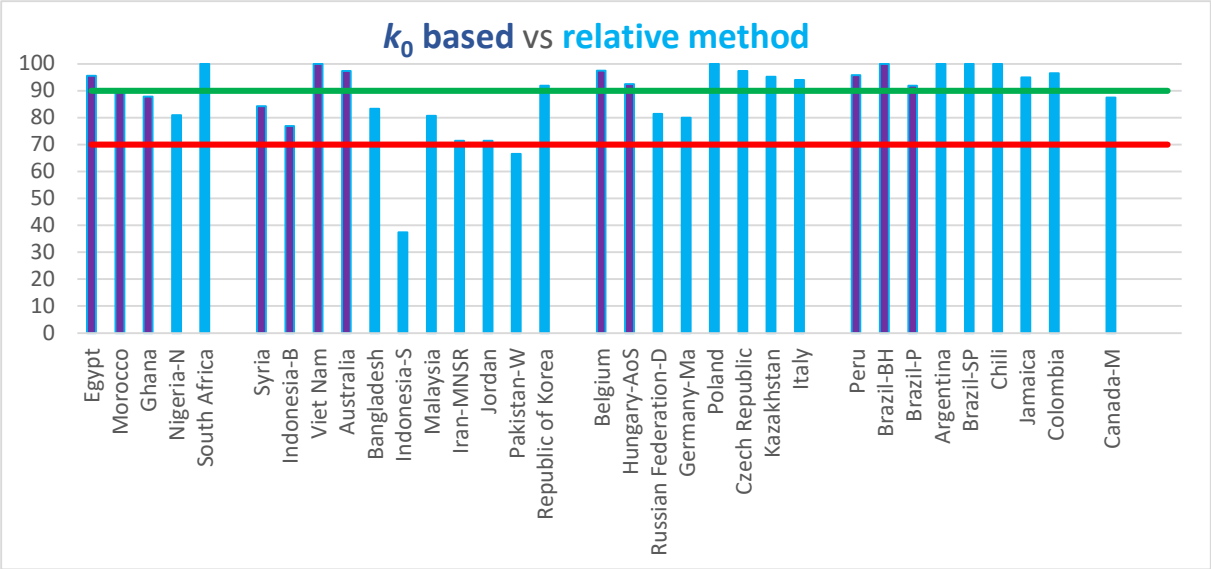


FIG. 20. Performance of NAA laboratories for analysing a soil type material in PTNATIAEA20 with reference to the calibration method: k_0 based or relative method based, and indication of the minimum levels for being categorized as 'excellent' or 'satisfactory'.

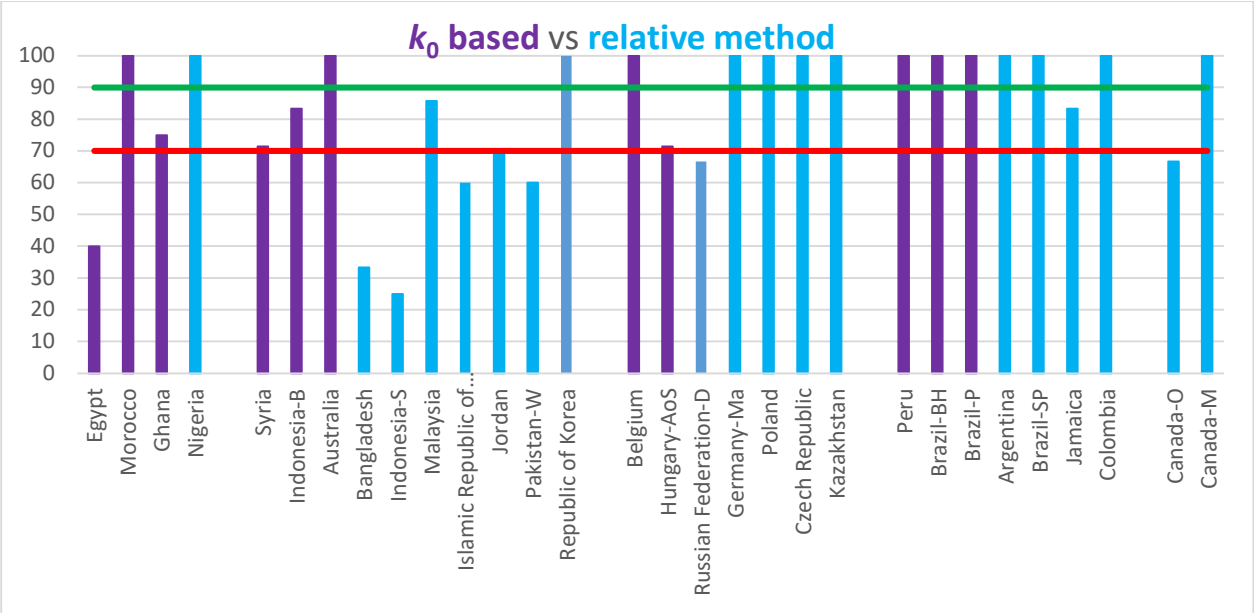


FIG. 21. Performance of NAA laboratories for analysing a biological type material in PTNATIAEA20 with reference to the calibration method: k_0 based or relative method based, and indication of the minimum levels for being categorized as 'excellent' or 'satisfactory'.

4.8.2 Software used

NAA laboratories are using mostly commercial software for gamma ray spectrum acquisition and analysis resulting in the number of counts of the detected gamma ray energies and the emitting radionuclides. The interpretation thereof, i.e., the assignment of the radionuclides to their parent chemical elements and conversion of the number of net counts of the peak areas towards mass (fractions) can be done using self-made software, commercial software specific for the k_0 method (e.g. Ref. [27]) , software provided by the IAEA for the k_0 technique [28] and/or other software, e.g. developed by other NAA laboratories. The potential relation between the performance indicator and the type of software used was evaluated on the basis of the information submitted by the participating NAA laboratories in PTNATIAEA20 and is shown in Figs 22 and 23.

There are no apparent indications for a systematic significant effect to the performance of the NAA laboratories by the type of software used for interpretation of the gamma ray spectrum analysis. This conclusion corroborates with the outcome of the IAEA laboratory intercomparison of software used in the k_0 method for interpretation of gamma ray spectra which also indicated that any differences are hardly significant compared to the combined uncertainty of measurement [15].

4.8.3 Elements reported

The NAA laboratories that implemented the k_0 method are reporting, as expected, more results in the soil type in PTNATIAEA 20 than most laboratories using the relative method, but there is no significant difference for the number of elements reported for the analysis of the biological type, as can be derived from Figs 24 and 25. It can also be derived that several laboratories are able to report on the same number of elements using the relative method as by the k_0 technique operating laboratories.

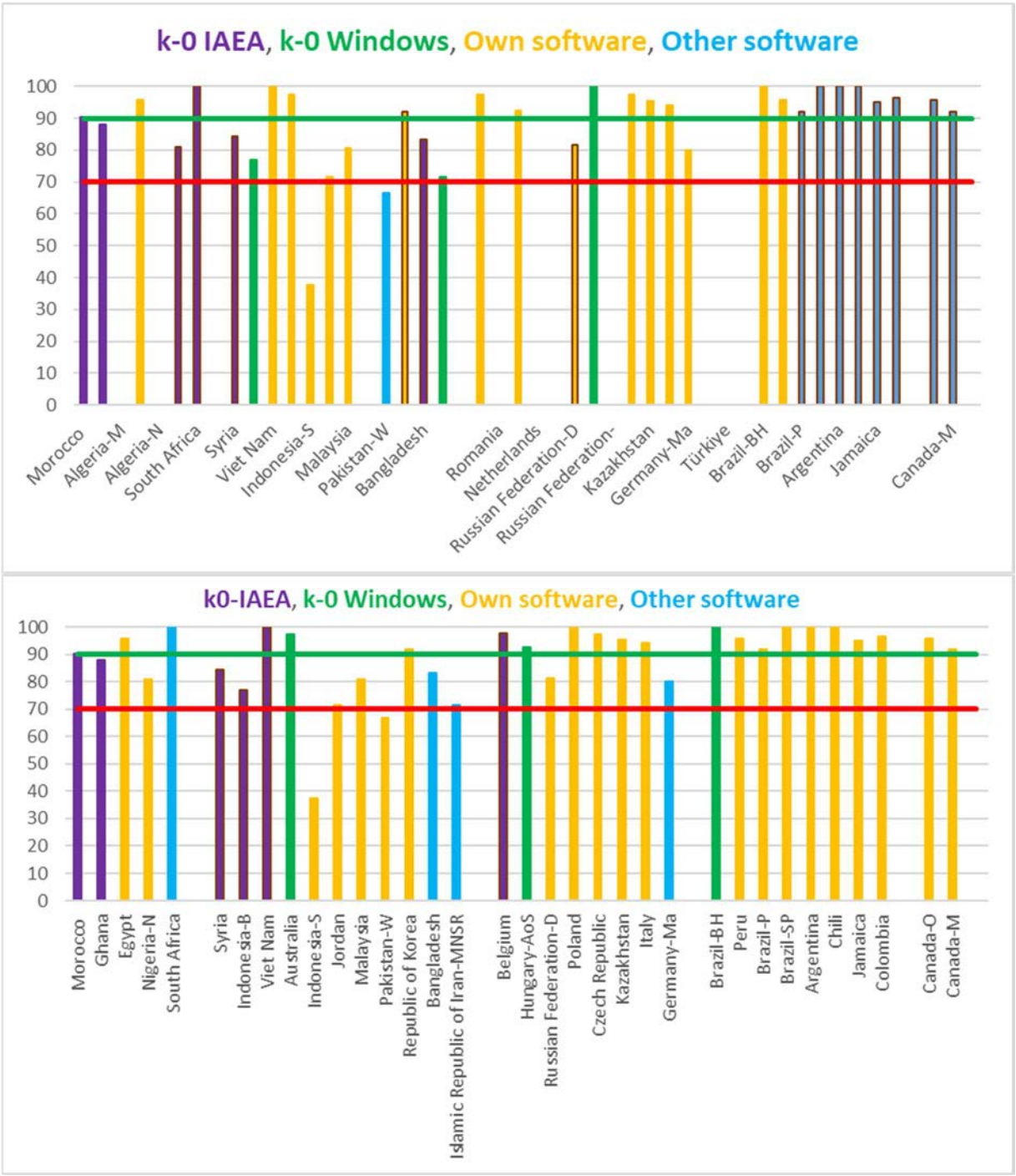


FIG. 22. Performance of NAA laboratories for analysing a soil type sample in PTNATIAEA20 with reference to the software used for spectrum analysis result interpretation (K0-IAEA, Kayzero for Windows, own software or other software) and indication of the minimum levels for being categorized as 'excellent' or 'satisfactory'.

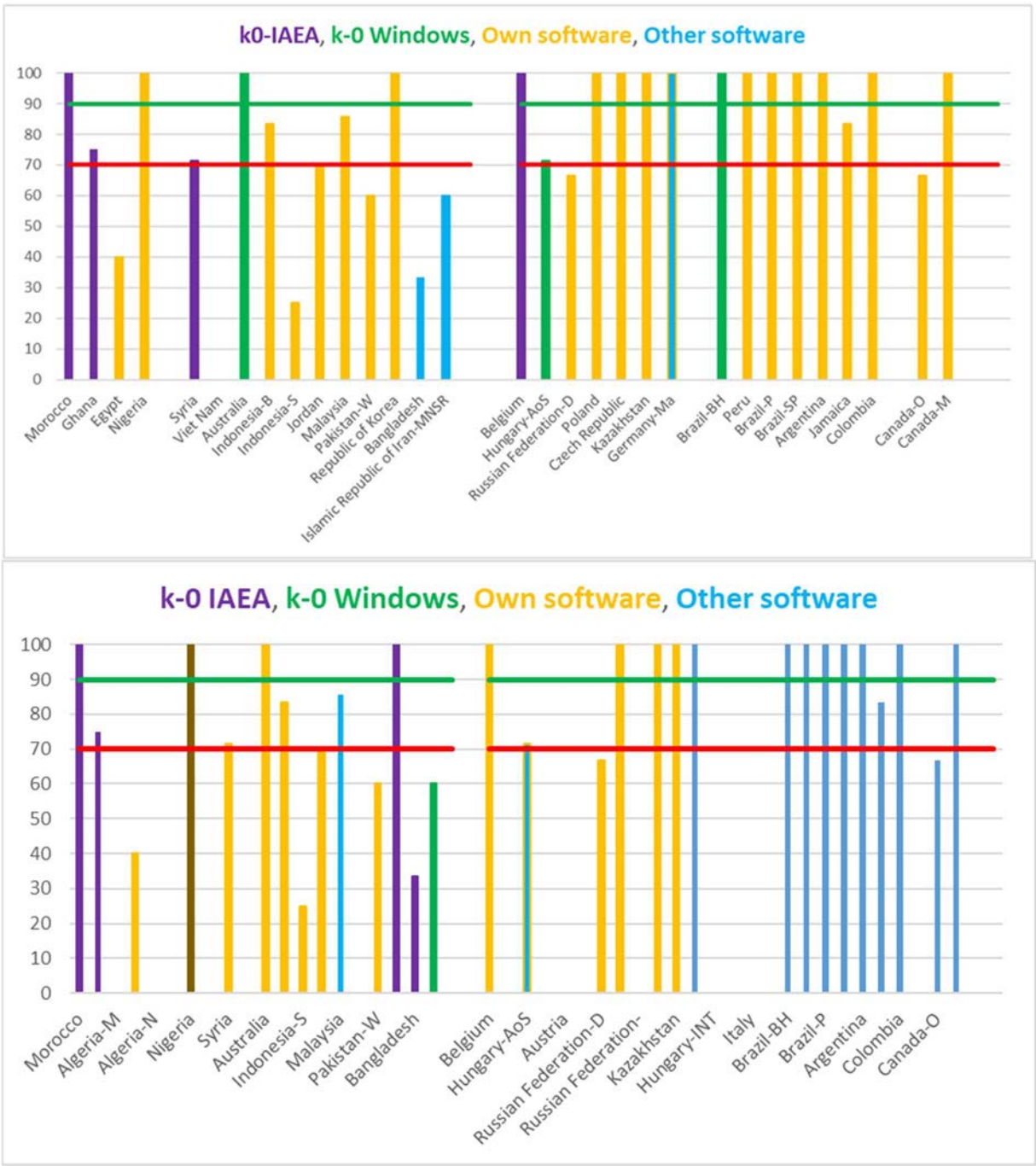


FIG. 23. Performance of NAA laboratories for analysing a biological type in PTNATIAEA20 with reference to the software used for spectrum analysis result interpretation (K0-IAEA, Kayzero-Windows, own software or other software) and indication of the minimum levels for being categorized as 'excellent' or 'satisfactory'.

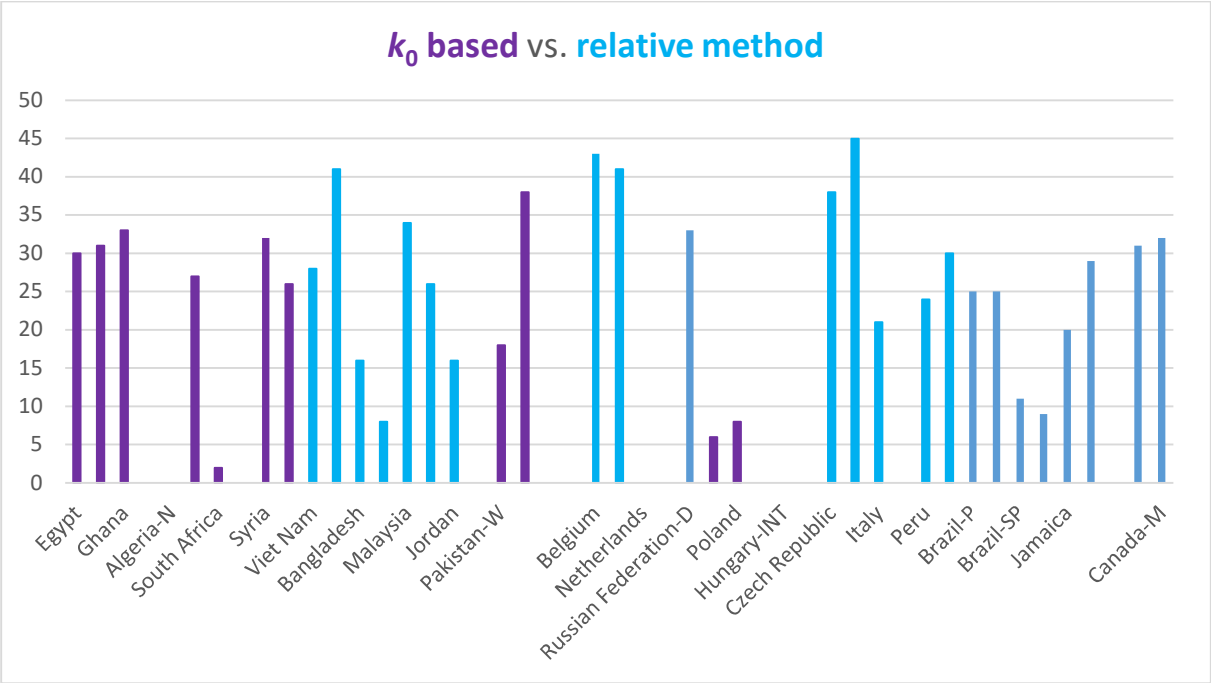


FIG. 24. Number of elements reported by NAA laboratories in analysing a soil type material sample from PTNATIAEA20 with reference to the calibration method, k_0 based or relative method.

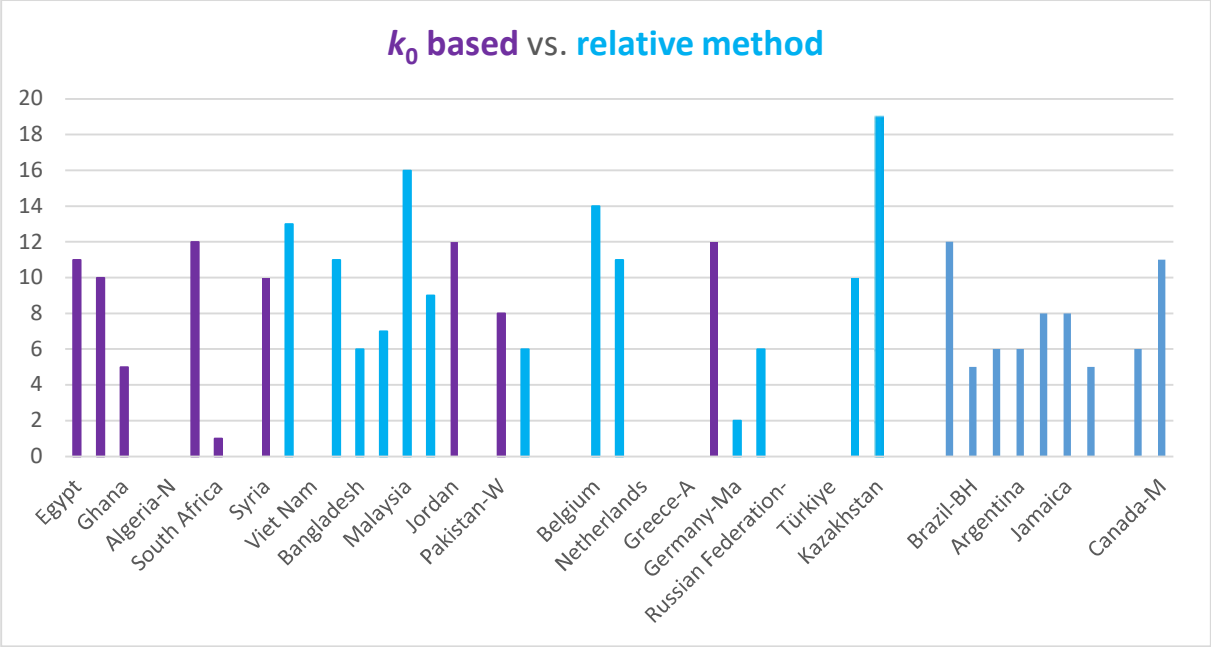


FIG. 25. Number of elements reported by NAA laboratories in analysing a biological type material from PTNATIAEA20 with reference to the calibration method, k_0 based or relative method.

5. EVALUATION

This section provides an evaluation of the results obtained.

5.1 THE IAEA EVALUATION CRITERION

Several NAA laboratories in all regions have demonstrated, often consistently, 90 – 100% of their results with a high degree of accuracy, expressed as $|z|<3$ both for soil type and biological type material. Such a performance can therefore be considered as a minimum benchmark for other NAA laboratories. One needs to keep in mind that the classification on basis of the fraction of data with $|z|$ resp. $|z, z'| <3$ may provide a too optimistic view on the quality of the results. In the ISO/IEC 17043 [2] and ISO 13528 [3], it is described that data with $|z|$ resp. $|z, z'| \leq 2$ are considered as 'satisfactory', those with $2< \{|z|$ resp. $|z, z'|\} <3$ as 'questionable' and with $|z|$ resp. $|z, z'|\geq 3$ as 'unsatisfactory'. This implies that laboratories with 90% of their results with $|z|$ resp. $|z, z'||< 3$ still could have a large fraction of (or even all) 'questionable' results with $2<\{|z|$ resp. $|z, z'|\}<3$.

However, several NAA laboratories demonstrate their ability to achieve results for both types of material with the majority of the values in the range of $|z,z'| \leq 2$. Such laboratories might prefer using this as a more common criterion (see above) for acceptance as their state-of-the-practice.

The performance of NAA laboratories in PTNATIAEA19 on basis of $|z|$ resp. $|z, z'| \leq 2$ has been compared with the performance on basis of $|z|$ resp. $|z, z'| <3$, and is shown in Figs 26–35 for NAA laboratories in all regions and for soil type material and biological type materials. The differences are none to marginal for most laboratories, but a major difference can be seen for the NAA laboratories from Bangladesh and Indonesia for the soil type analysis, and for Nigeria, Malaysia and Russian Federation for the biological type analysis.

It can be also concluded that the fraction of data with $|z,z'| \leq 2$ could be used as a performance indicator hardly affecting the categorization of laboratories under the benchmarks as ≥ 90% for 'excellent' classification and ≥ 70% for 'satisfactory". The choice for the use of $|z,z'| \leq 2$ would bring the performance indicator in better agreement with international accepted interpretations as described in ISO/IEC 17043 [2] and ISO 13528 [3].

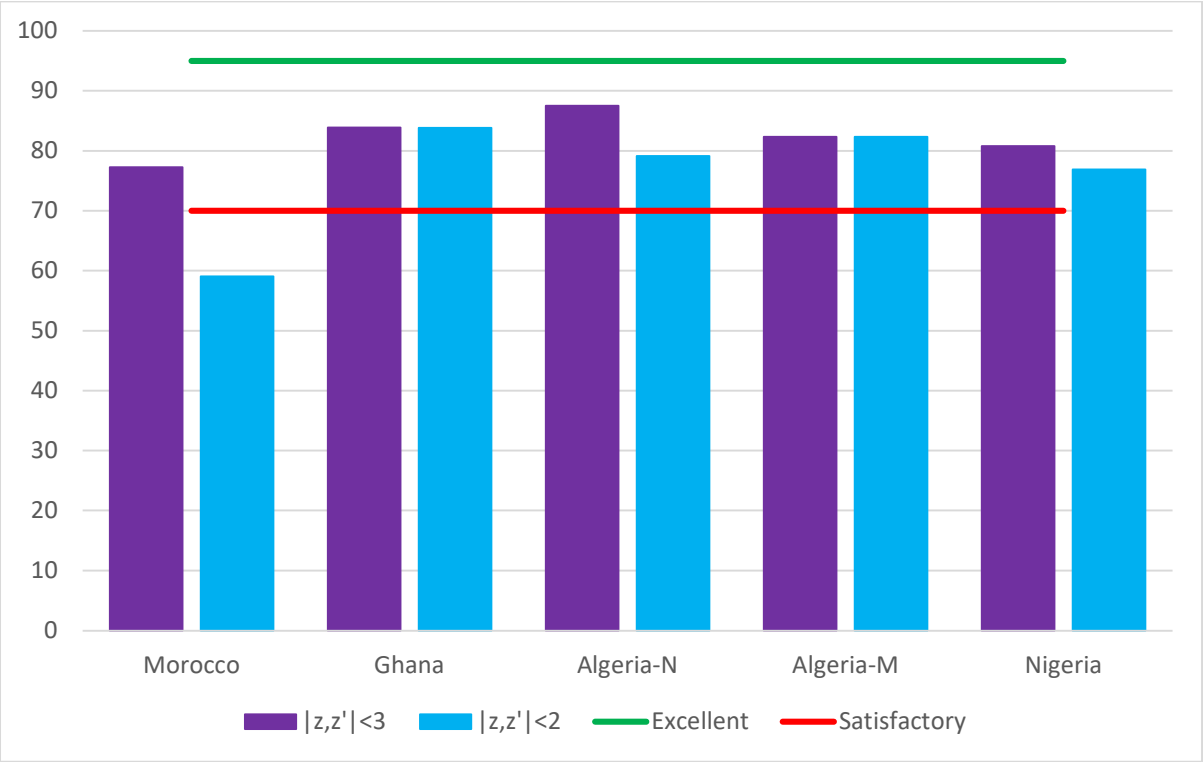


FIG. 26. Performance of NAA laboratories from Africa for analysis a soil type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

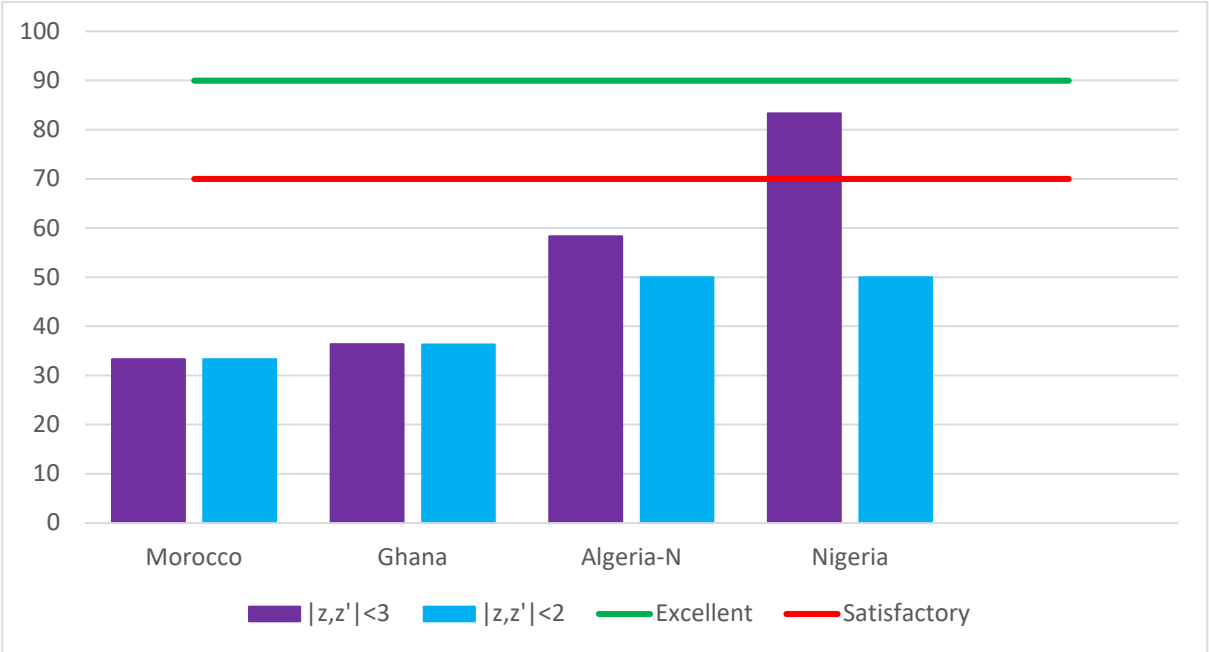


FIG. 27. Performance of NAA laboratories from Africa for analysis a biological type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

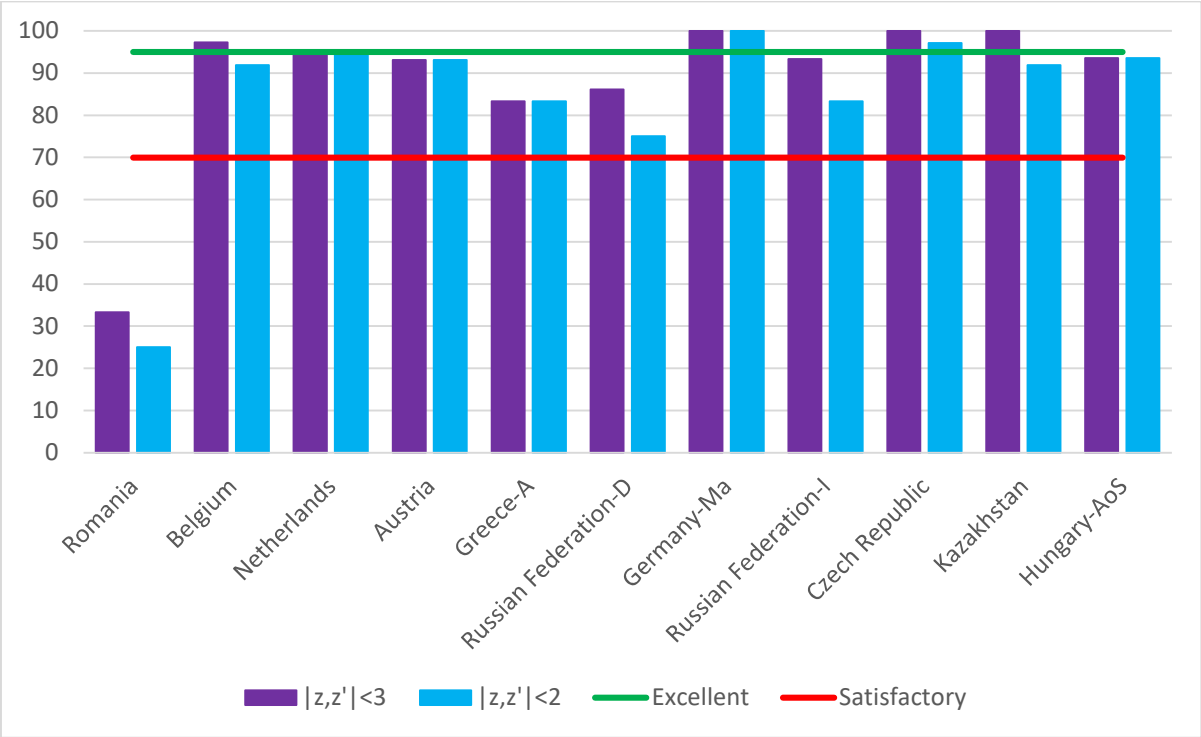


FIG. 28. Performance of NAA laboratories from Europe for analysis a soil type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

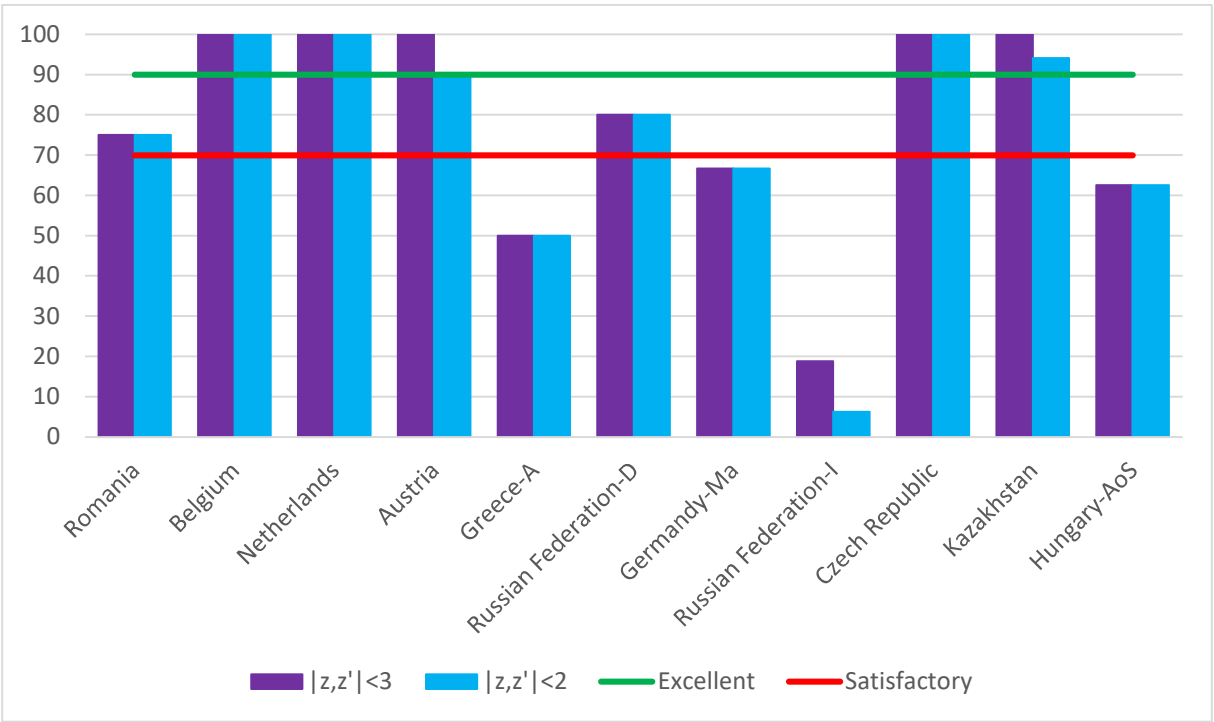


FIG. 29. Performance of NAA laboratories from Europe for analysis a biological type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

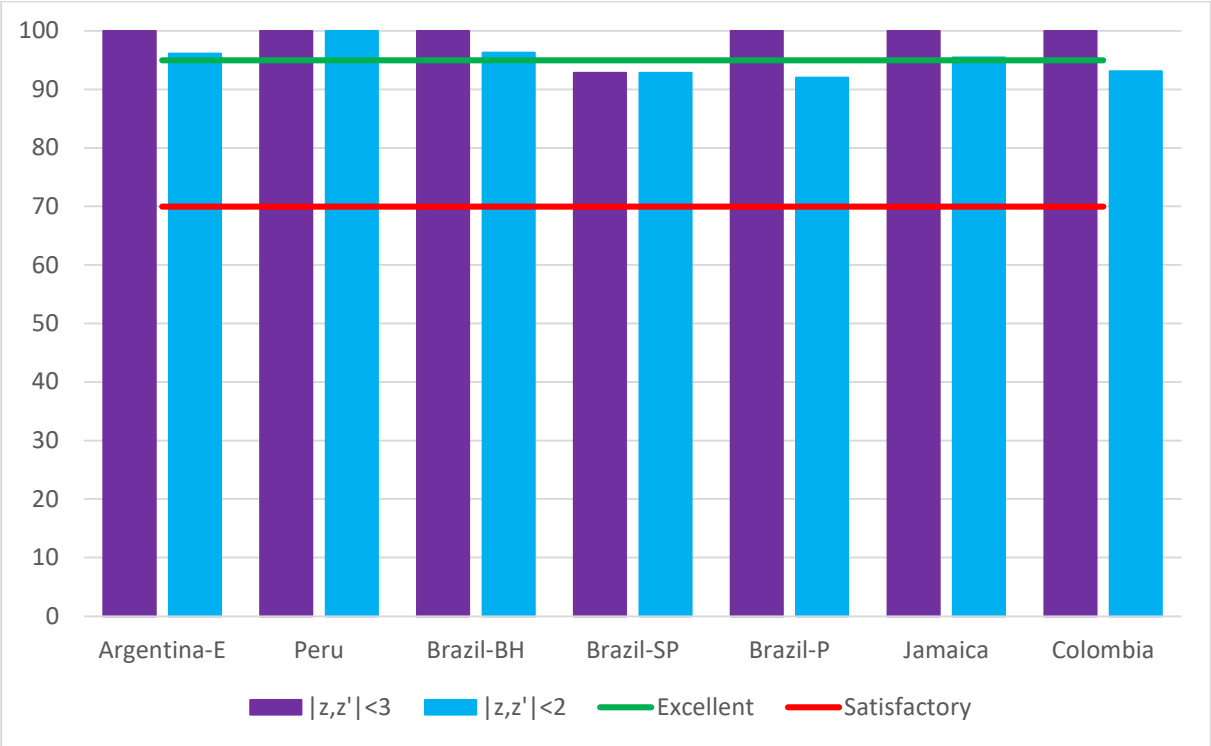


FIG. 30. Performance of NAA laboratories from Latin America and the Caribbean for analysis a soil type in PTNATIAEA19 on basis of |z| resp. |z,z'| ≤2 is compared with the performance on basis of |z| resp. |z,z'| <3.

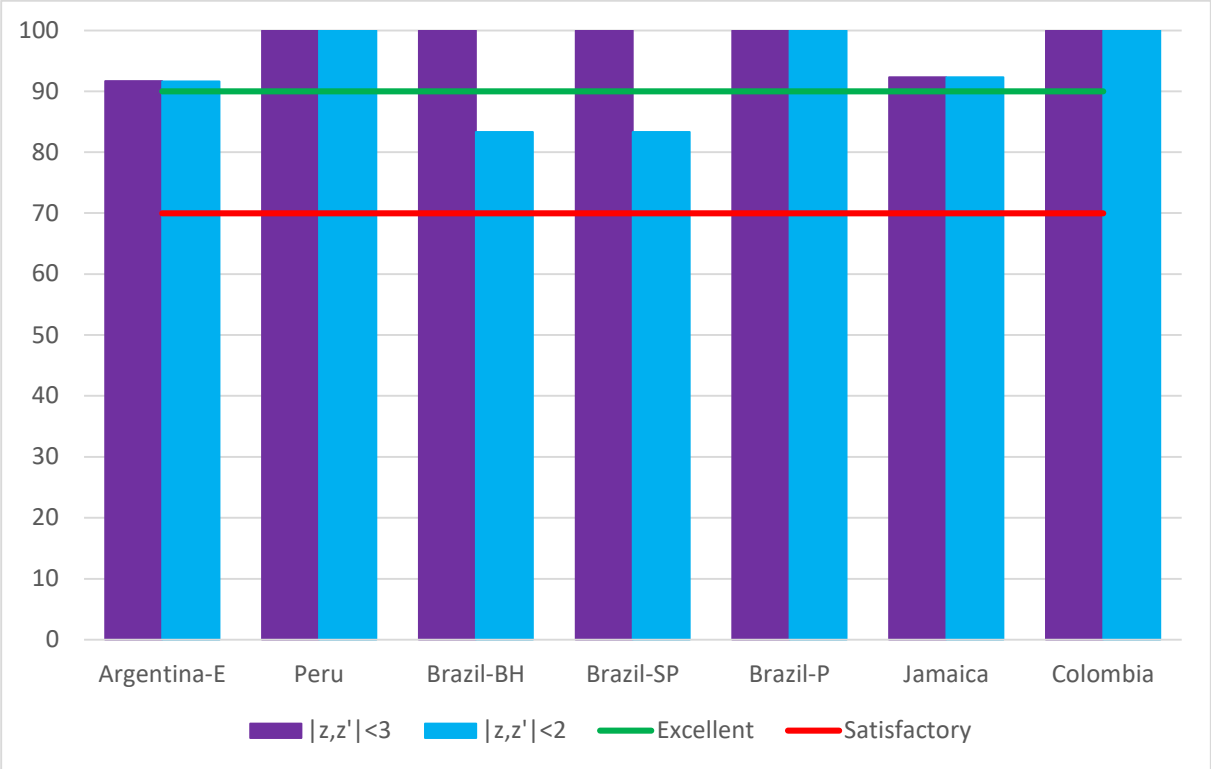


FIG. 31. Performance of NAA laboratories from Latin America and the Caribbean for analysis a biological type in PTNATIAEA19 on basis of |z| resp. |z,z'| ≤2 is compared with the performance on basis of |z| resp. |z,z'| <3.

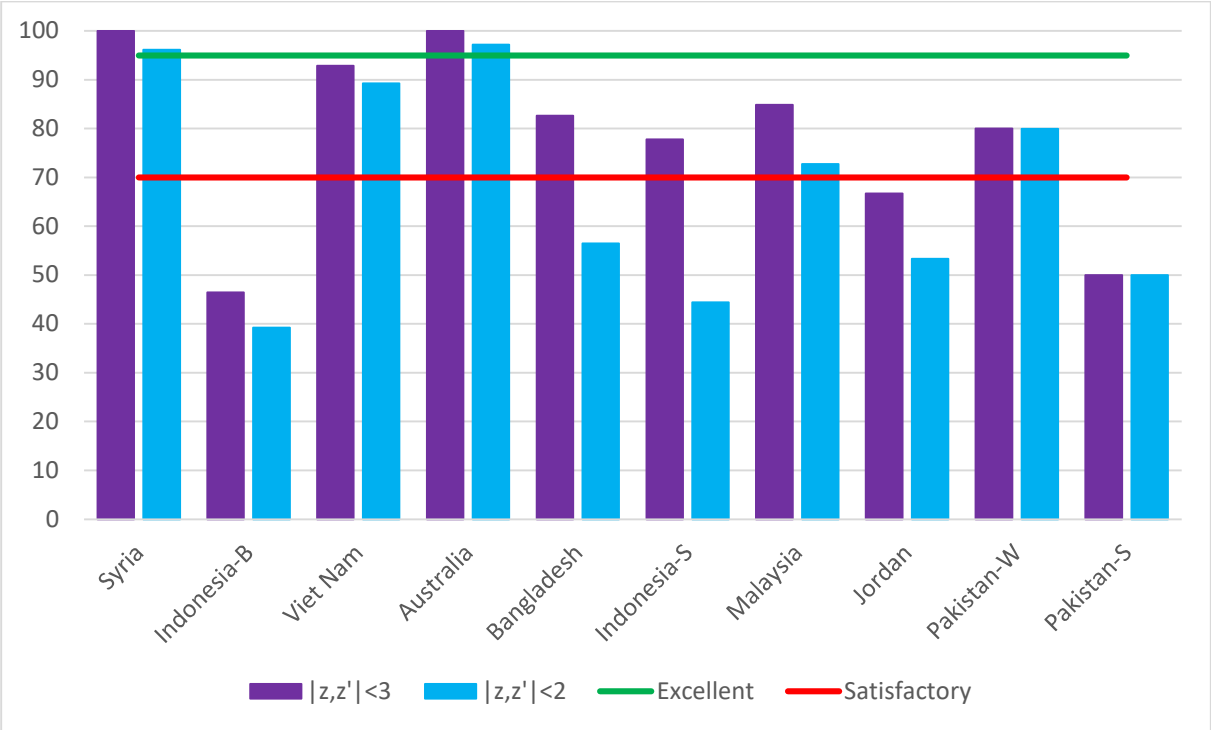


FIG. 32. Performance of NAA laboratories from Asia and the Pacific for analysis a soil type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

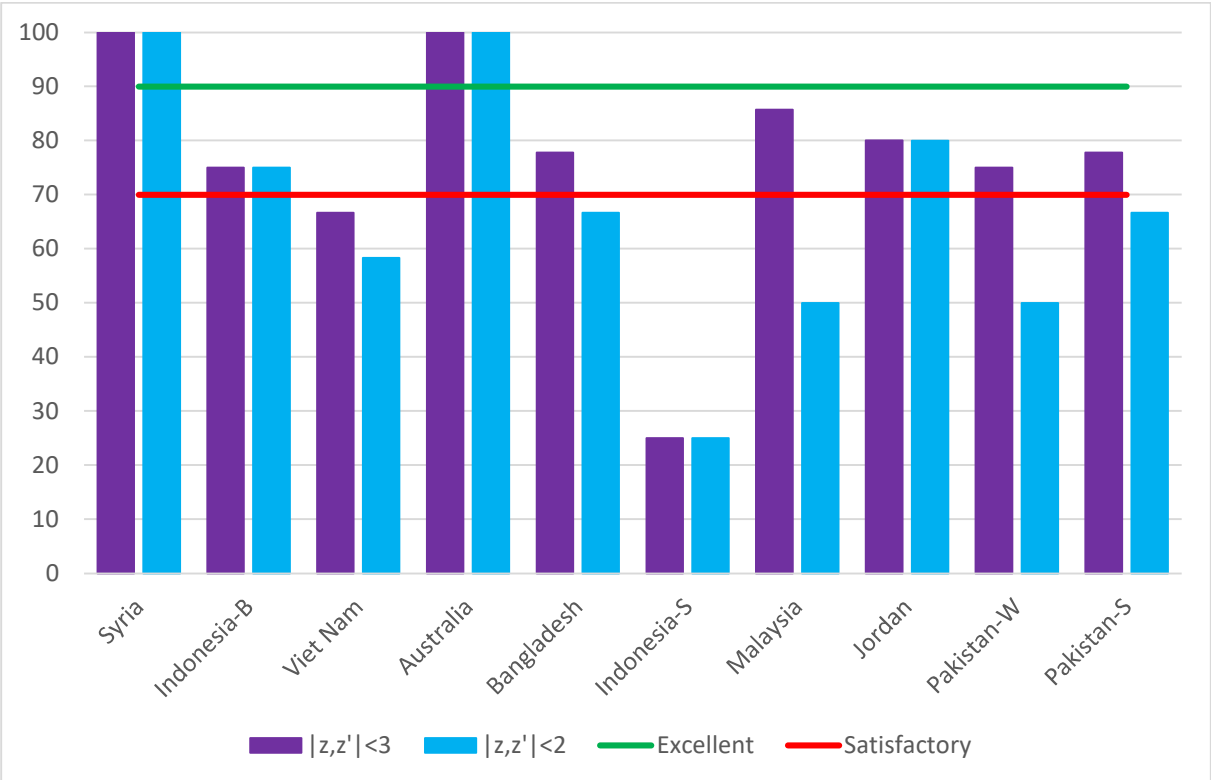
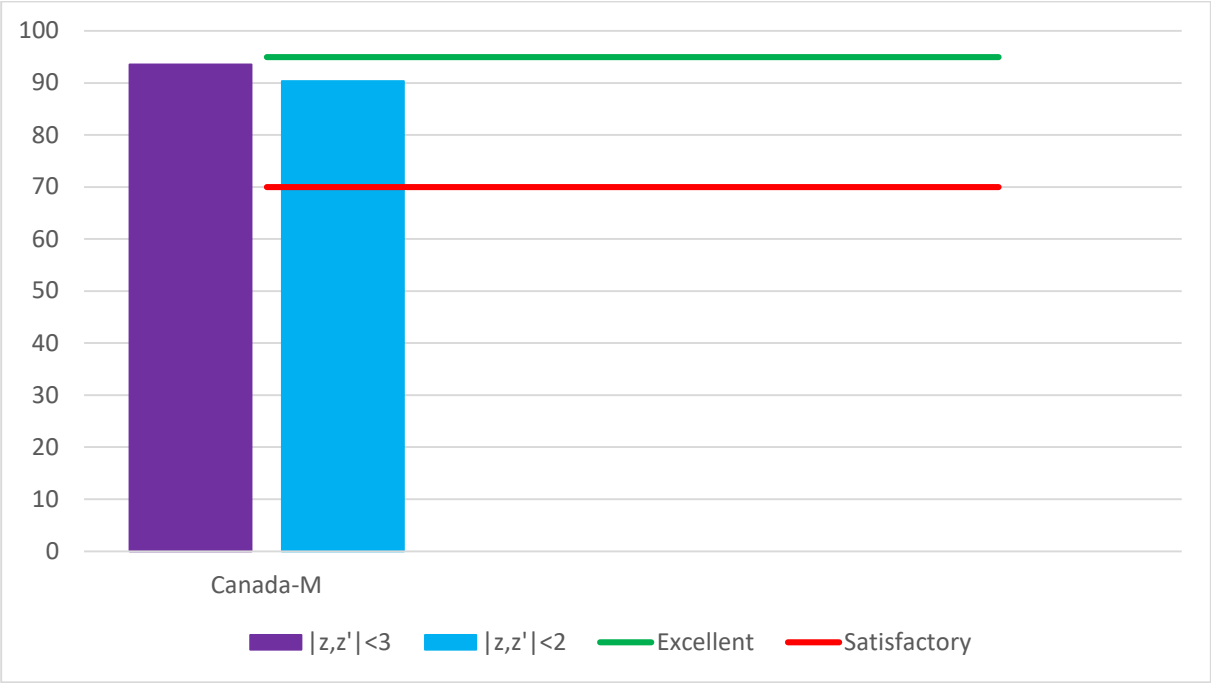


FIG. 33. Performance of NAA laboratories from Asia and the Pacific for analysis a soil type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.



FIG, 34. Performance of NAA laboratories from North America for analysis a soil type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

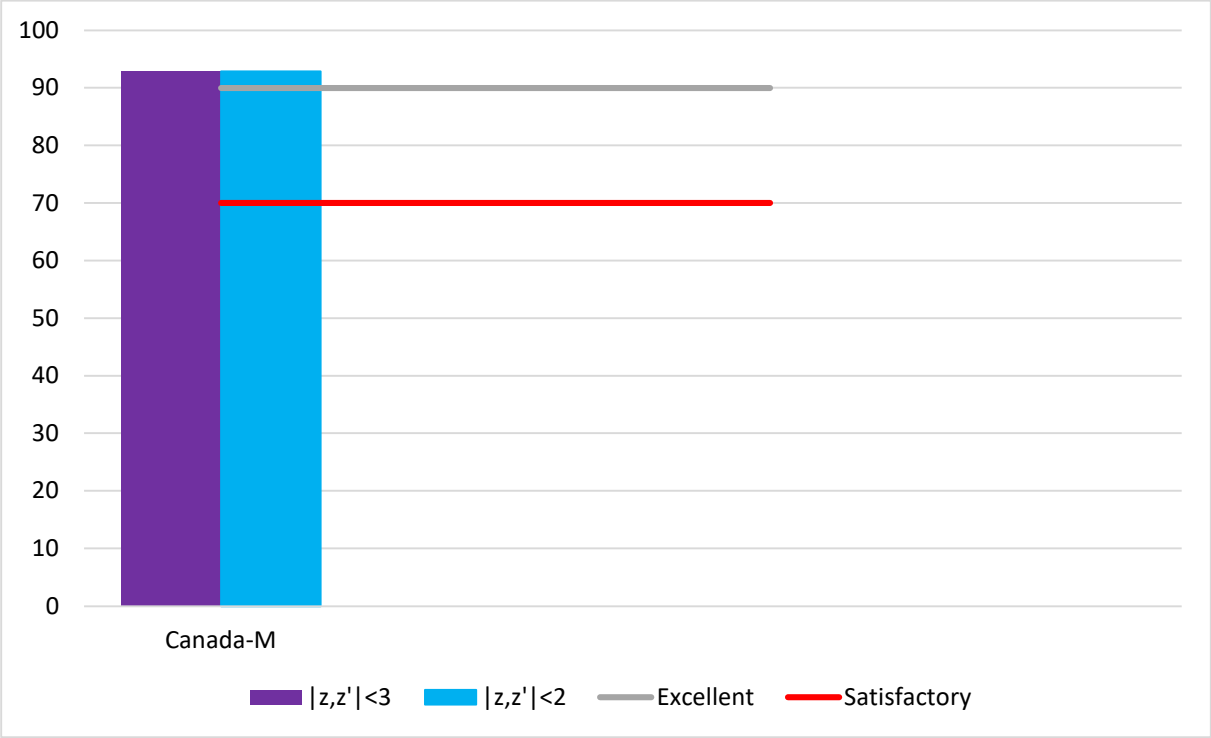


FIG. 35. Performance of NAA laboratories from North America for analysis a biological type in PTNATIAEA19 on basis of |z| resp. |z, z'| ≤2 is compared with the performance on basis of |z| resp. |z, z'| <3.

5.2 PERFORMANCE INDICATOR AND NUMBER OF ELEMENTS REPORTED

The categorization of laboratories on the basis of the performance indicator — the percentage of data meeting a defined criterion — depends on the number of results of elements reported for which assigned values are available. This might be one of the reasons that the performance for biological type material is often less good than for soil type material, for which often more results are reported.

Participants in the feedback and QA workshops discussed an unwanted consequence of the chosen indicator and questioned whether it is a correct judgement that a laboratory with e.g. 3 unsatisfactory results out of 25 reported values can be categorized lower than a laboratory reporting only 3–4 satisfactory results, as sometimes happened.

Rather than weighting the performance on the number of elements to be reported, it might be considered to establish in advance the elements to which each participant needs to report data in the interlaboratory comparison on basis of expert judgement on NAA's capabilities for the material to be analysed. This would probably be a smaller number of elements than some laboratories now report, but it may lead to a uniform comparison of the performance of the laboratories. The proficiency testing exercises implemented by IAEA-NSIL up to 2022 have not done this.

5.3 TRENDS IN PERFORMANCE

The absence of improvement in performance in some NAA laboratories was partly attributed to retirement and/or departure of experienced staff resulting in insufficient transfer of practical guidance to new staff. However, it was also acknowledged at the 2017 feedback workshop by some laboratories that insufficient attention had been given to the lessons learned from the (reports of the) previous workshops and the related recommendations for the practice of performing NAA, and/or that these were insufficiently or not transferred to successors. The inconsistency (or even sometimes, a decline) in performance observed in some laboratories was the main reason to develop extra guidance such as: the 2022 IAEA publication Technical Reports Series No. 487 [13] and modules on QA/QC, trouble shooting and method validation in the IAEA e-learning course [14]. Both have been promoted at the 2022 QA workshop in Delft and participants have been urged to consider the publication as a reference laboratory guide [29].

5.4 COEFFICIENT OF VARIATION AMONGST NAA LABORATORIES

A representative of WEPAL showed at the 2015 feedback workshop in Delft, Netherlands, the comparison between the results of the NAA laboratories in interlaboratory comparison 2015-2, and those of all other laboratories using the coefficients of variation and mean values of the mass fraction of the elements [24]. The outcome is shown in Figs 36 and 37. It is noted that in these WEPAL proficiency tests, the coefficient of variation of all participants serves as the standard deviation for proficiency assessment and is used for estimating the z-scores (see Table 7).

The mean values for Mg and Se by all participating NAA laboratories in WEPAL's ISE 2015-2 (soil type material) are the only ones that differ up to 50% of the mean values by all participants in this laboratory intercomparison. In case of the biological type materials, the elements Cr and Sr differ significantly from the mean value of all participants. The relatively large coefficients of variation in the results for Al and Mg in the analysis of the soil type

material (Fig. 36) can be assigned to differences in the corrections performed for the interferences in the cluster of P, Si, Al and Mg due to different reactions with thermal, epithermal and fast neutrons. The variation in the results for Sr values in biological type materials (Fig. 37) is possibly due to the fitting of the 511–514 keV doublet in the gamma ray spectrum, which is difficult to resolve. All participants reported Cr values in the biological type materials at least 15% higher than the mean values reported by WEPAL. As the WEPAL reported variance is high, the z-scores were nevertheless acceptable in several cases. The cause of this discrepancy could be due to lack of correct evaluation of the Cr content in the blank samples, given that Cr is a known impurity in plastic vials. However, it is more likely due to the WEPAL mean value for Cr being biased, since most results in the WEPAL rounds come from techniques that require digestion, which is known to be incomplete for Cr.

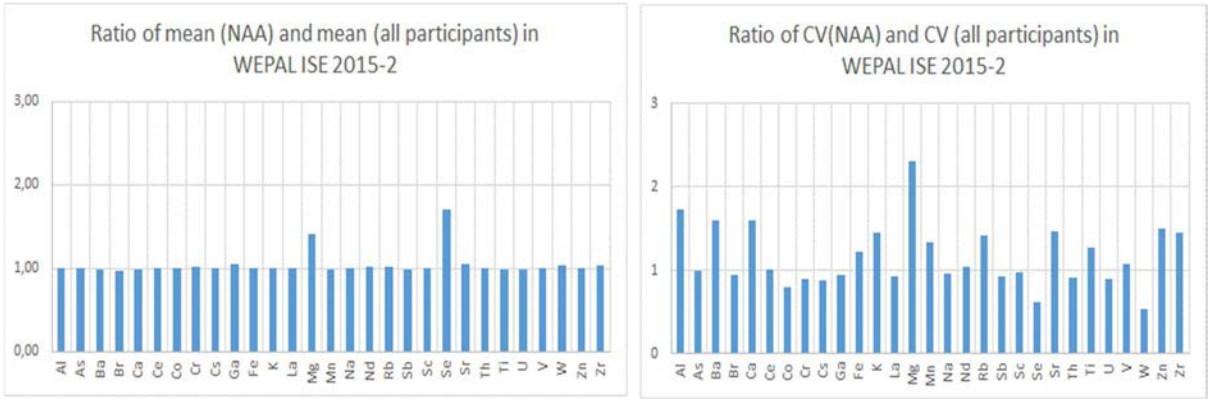


FIG. 36. Comparison of the ratio of the mean values and the ratio of the coefficients of variation for elements reported in WEPAL ISE 2015-1 (Soil type material) for IAEA facilitated participants and all other participants.

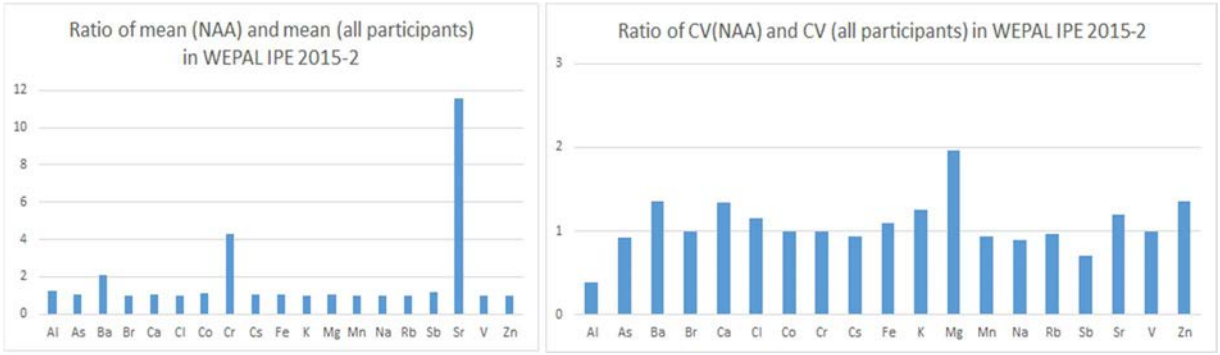


FIG. 37. Comparison of the ratio of the mean values and the ratio of the coefficients of variation for elements reported in WEPAL IPE 2015-2 (biological type material) for IAEA facilitated participants and all other participants.

A similar comparison was made on basis of the evaluation of the results submitted for PTNATIAEA20 [30]. To this end, the consensus value of the NAA results, x^* and the standard deviation of the consensus value of the NAA results only, s^*, have been compared with the assigned value and the standard deviation for proficiency testing, σ_{pt}, respectively. The results are shown in Figs 38 to 41 for the soil type material and biological type material used in PTNATIAEA20.

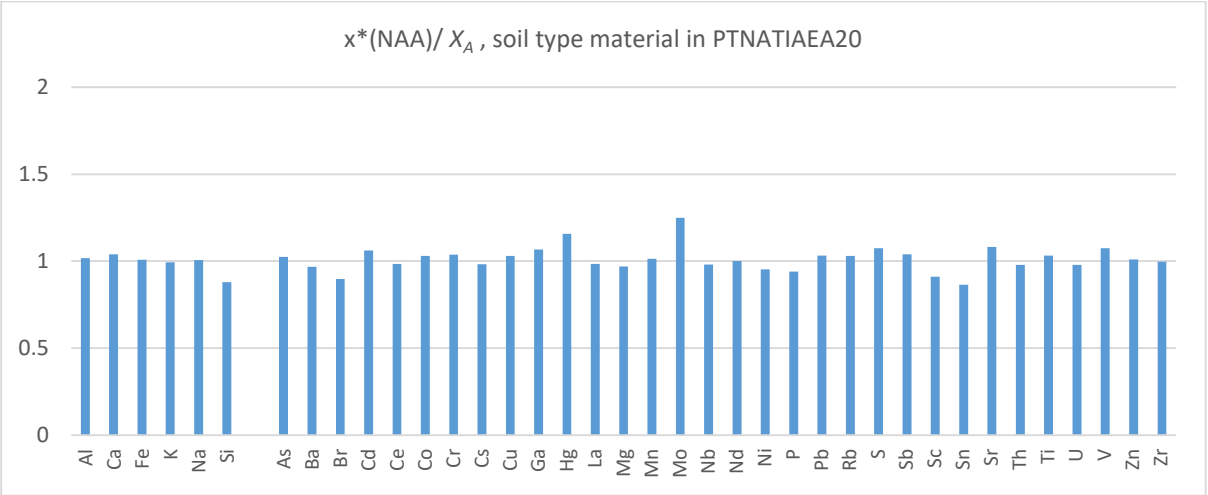


FIG. 38. Ratio of mean value (x^*) and assigned value X_A for NAA results only of soil type material of PTNATIAEA20.

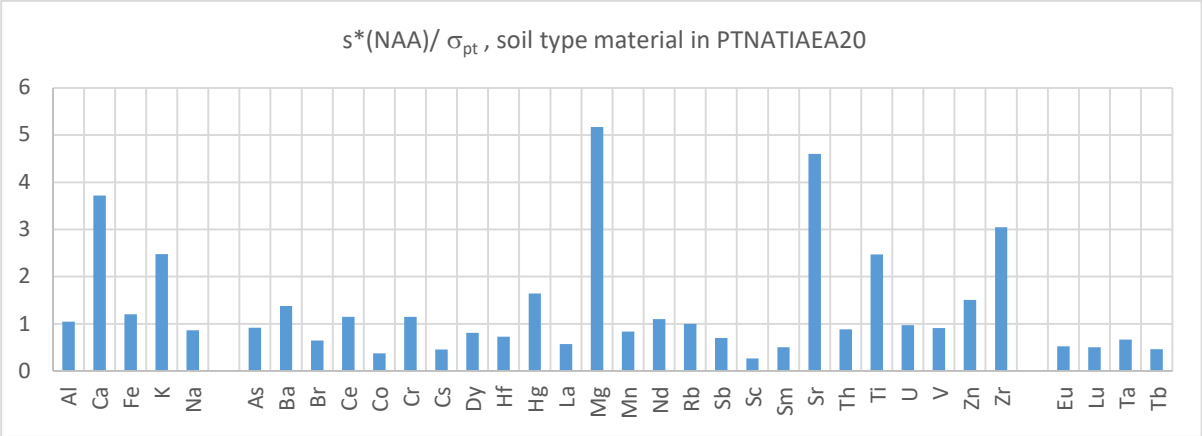


FIG. 39. Ratio of standard deviation s^* and standard deviation for proficiency assessment σ_{pt} for NAA results only of soil type material of PTNATIAEA20.

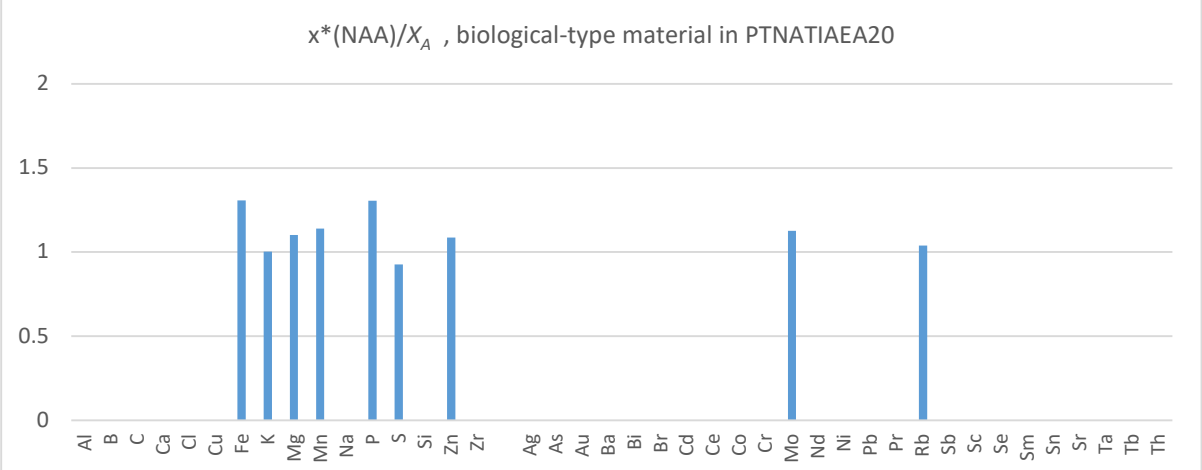


FIG. 40. Ratio of mean value (x^*) and assigned value X_A for NAA results only of biological type material of PTNATIAEA20.

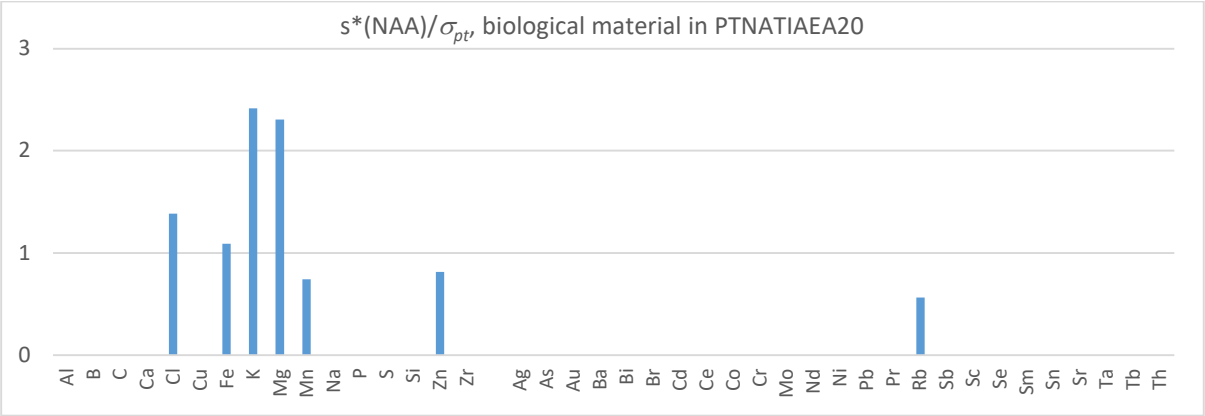


FIG. 41. Ratio of standard deviation ($s^{*)}$ and standard deviation for proficiency assessment σ_{pt} for NAA results only of biological type material of PTNATIAEA20.

Ratios much larger than unity indicate variations between results of the various laboratories that cannot be explained by e.g., the sampling error resulting from inhomogeneity — which is more dominant than the uncertainty of measurement and covered by the modified Horwitz equations as used in the IAEA software — and therefore indicate technique-specific difficulties in achieving agreement on the degree of trueness. It can be seen that there is a high degree of agreement between the results of the NAA laboratories for PTNATIAEA20 with the assigned values for many elements in case of the soil type material and that the elements Ca, K, Mg, Sr, Ti and Zr seem to be the most troublesome although the mean values are close to the assigned values. The elements Mg and Sr are giving recurrent problems for the reasons explained above. Spectral interferences and calibration problems may explain the difficulties with the elements Ca, Ti and Zr. For the biological type material, K and Mg are the problematic elements in PTNATIAEA20. There is no straightforward explanation for the relatively large variation in the results for the element K in both types of material since this element can be measured interference free by NAA.

5.5 PERSISTING SOURCES OF ERROR IN NAA

Wrong results are still regularly produced by NAA laboratories in spite of the recommendations on QA/QC. This is partly related to statistical considerations resulting from acceptance criteria for results from analysis of materials for validity control. But there are also random errors resulting from mistakes in the organization, preparation and conduct of NAA. Many of these deficiencies have been presented and discussed during the feedback and QA/QC workshops but there are, unfortunately, indications that they still persist, such as:

— Transposing errors in home-made spreadsheet calculations.
— Human errors in spectrum analysis and interpretation, such as use of the wrong efficiency curve.
— Errors resulting from measurements close to the detectors endcap, such as differences between sizes of sample and calibrator, and coincidence summing effects not accounted for.
— Wrong corrections for interfering nuclear reactions, especially for the cluster of the elements Si, P, Al, Mg, and Na if using the relative method since the contributions of the interferences are different in the comparator material and the unknown sample analysed.
— No application of validity control by simultaneous processing of a sample of an adequate reference material. This seems to be partly resulting from lack of resources to obtain such

materials and it is insufficiently recognized that even remaining material from previous interlaboratory comparisons may equally well serve for such validity control.
— Use of smaller masses than recommended in certificates of reference materials.
— No corrections for neutron flux gradients between sample and standard.
— Ineffective follow-up on deficiencies in results from participation in intercomparisons.

5.6 BLUNDERS

There are many steps in the NAA procedure and calculus in which data have to be transposed from hand-written registration forms into computerized systems, and from one software package into another. Mistakes may happen anytime, irrespective if QA procedures have been implemented. Some of these mistakes might not even be revealed by the laboratory's validity control. In all IAEA facilitated laboratory intercomparisons, results have been submitted that deviate by a factor of 10 or more from the assigned value (both higher and lower). Up to 2021, such values were included as outliers and therefore could have an effect to the rejection of data not passing the outlier tests as such and thus to the consensus value. After the alignment of the data processing with ISO 13528 [3], the values differing by more than an order of magnitude from the median of the results' distribution are now separately categorized in the IAEA proficiency testing rounds as 'blunders' and excluded from further processing. Blunders are mostly due to submission in wrong dimensional units than requested by the provider. Examples of other types of error resulting in data that can be categorized as blunders are the interchange of samples, wrongly combining data from different measurements, measurement of samples and calibrators under different geometries or at different detectors. An overview of the percentage of data now categorized as blunders on basis of the criterion introduced in 2021 within the total number of submitted results by all NAA laboratories in the interlaboratory comparisons from 2010–2022 on soil type and biological type material is shown in Fig. 42. It is noted that blunders are still accounted for in the calculation of the percentage of submitted results for which $|z|$ or $|z, z'| < 3$.

It can be seen that NAA laboratories took effective actions after the feedback workshops in 2011 and 2012 by minimizing the probability of the major error, reporting in wrong dimensional units. Occasionally, the percentage of blunders increases sometimes resulting from first-time participation of less experienced NAA laboratories, like in the interlaboratory comparison 2017-3. The increase in the percentage of blunders in the 2021 and 2022 interlaboratory comparisons is partly due to the much smaller total number of results reported.

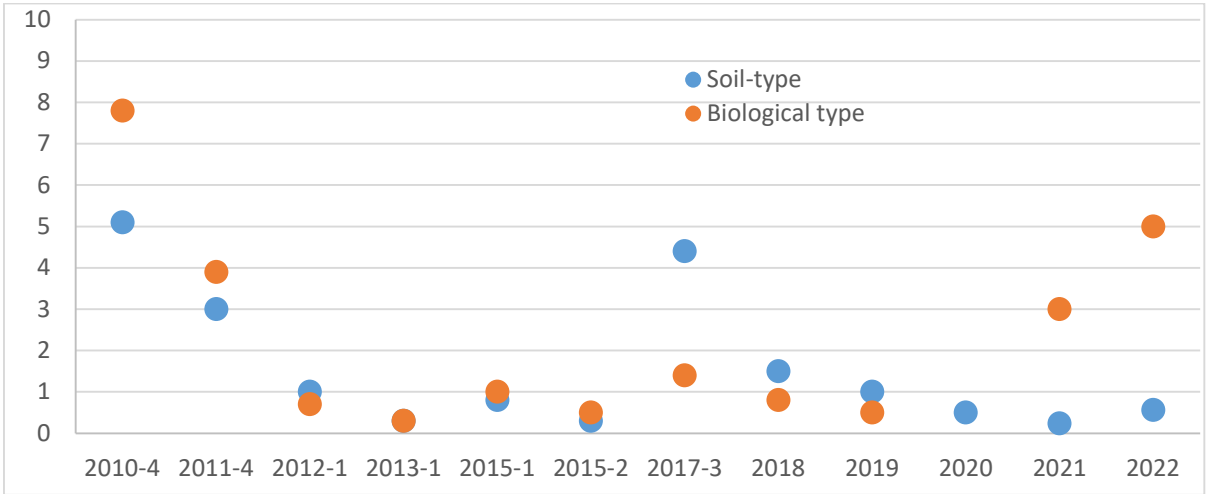


FIG.42. Percentage of submitted data categorized as 'blunders' in successive interlaboratory comparisons from 2010 until 2022 of soil type and biological type material.

5.7 FEEDBACK AND QA/QC WORKSHOPS

The feedback and QA/QC workshops are of value to the participants, the IAEA and the IAEA experts. Participants can be guided towards potential sources of error and pragmatic approaches of QA to minimize the probability of occurrence and recurrence, and validity (quality) control to monitor the effectiveness of the QA. The IAEA and IAEA experts learn about unexpected wrong practices in NAA that may explain unacceptable results. They thus can improve and focus their guidance on the practice in QA, QC, calibration and other analytical and organizational aspects of NAA.

Nonetheless, several laboratories are not able to improve their performance to the highest level as can be seen from the results of successive participation in interlaboratory comparisons, and even several cases of decline have been noticed. This may be also due to insufficient (opportunities for) expertise transfer upon experienced staff leaving, especially on assessing the root cause of unacceptable results and design and implementation of corrective actions including the verification of their effectiveness. The IAEA may provide guidance and facilitate NAA laboratories to get in contact with experts on NAA for discussion of practical problems, but it may remain difficult for experts to identify the cause of the deficiencies without witnessing the operations in person.

6. LESSONS LEARNED

This section discusses the lessons learned from the proficiency testing exercises organized.

6.1 LABORATORY ORGANIZATION

Interlaboratory comparisons are typically announced in a timely manner before shipment of samples, and typically, the deadline for reporting is several months after the projected date of receipt of the sample by the laboratory. The acceptance of an invitation for participation in an interlaboratory comparison requires advance assessment of the availability of the required resources in the months needed for the conduct of the analysis:

- Human resources: technically competent and skilled people, and supervisors.
- Operational laboratory tools (e.g. balances, ovens, freeze driers) and gamma ray spectrometers.
- Consumables: liquid nitrogen, vials or other encapsulation materials, reference materials for calibration, and different ones for validity control.
- Reactor operation: advance communication with the reactor manager on the availability of reactor and possibly even a priority setting policy by the executive management level may be necessary. An NAA procedure for the analysis of one or two samples involves typically not more than 4 hours reactor operating time.

It is in the laboratory's interest to include a safe margin in the planning for ample checking of the validity of the results prior to submission thereof. As mentioned previously, many NAA laboratories still submit their results in the final days before deadline although 3 to 4 months are available for conducting the analysis. Such laboratories do not appreciate that there might remain insufficient time for corrective actions if the laboratory's validity control indicates unsatisfactory results or mistakes.

The strong reduction in the percentage of blunders resulting from reporting in wrong dimensional units, and the questions from participants on guidance in e.g. moisture content determination and required minimum test portion mass indicates that the interlaboratory comparison provider's accompanying documentation is studied more carefully than at the start of the project in 2010.

The root cause of the remaining gross errors (e.g., factors 2 to 10 or more) is not clear; it can be partly calculation errors, transposing errors or typographical errors during submission. Such errors may indicate that independent control of calculations, transposing and final reporting is ineffective or possibly missing, although such a QA/QC has been repeatedly mentioned during all feedback workshops.

Laboratories also report in the IAEA interlaboratory comparisons their uncertainty of measurement. It can be derived from sometimes large differences that some laboratories report the uncertainty of measurement of only one sample, whereas others report the standard deviation of the mean value of replicates. Participants also mentioned the analysis of replicates in interlaboratory comparisons during the workshops. Participation in interlaboratory comparison might be of more value to laboratories if it is done under everyday conditions and it is not clear if the use of three or more replicates, as is often the practice in interlaboratory comparisons, is a representative reflection thereof.

6.2 SAMPLE AND CALIBRATOR PREPARATION

Ample attention was given in the feedback and QA/QC workshops on potential sources of contamination and approaches for monitoring and control thereof. This is especially relevant in the analysis of biological type materials because of the relatively low trace element mass fractions. It has been strongly recommended to pay ample attention to measurements of blanks by using e.g. empty vials passing simultaneously with the samples all parts of the analysis process, and to perform regular background verification to inspect for radioactive contamination at the counting position. Not all participants have effective separation of areas with incompatible laboratory activities, like separated balances and sample preparation benches for soil, environmental and biological samples.

New calibrators such as CRMs were made available through the IAEA, and participants have the opportunity to improve their calibrations. It could be derived from participants' presentations at the feedback workshops that often rather small amounts of the interlaboratory comparison materials are used, sometimes as little as 30 mg. This needs to be seen in the light of the recommended test portion by producers of CRMs, which is typically in the order of 200–300 mg for materials of similar composition as the interlaboratory comparison. WEPAL does not provide recommendations for the minimum sample mass for analysis. The. IAEA recommends using at least the minimum sample mass as published in the certificates of the reference materials selected for the interlaboratory comparison. There was no information from WEPAL on the mass used for testing the homogeneity of the materials used in their interlaboratory comparisons; nor is information provided on the minimum recommended sample mass in the certificates of their reference materials. It can be argued that a laboratory itself has to verify the homogeneity of the materials to be tested but as this requires at least tenfold analysis [31], it is an expensive, time-consuming procedure, which cannot always be done as 'routine'. Very few participants had such procedures implemented. The IAEA experts have therefore repeatedly emphasized and recommended that sample masses — also those of the interlaboratory comparison materials — needs to be in agreement with the recommended minimum amounts in the certificates of similar type (certified) reference materials used for calibration or validity control, and that smaller amounts are not to be taken.

When the IAEA initiated the NAA intercomparison exercises in 2010, some laboratories did not routinely perform moisture correction. As a result of this activity, those laboratories became aware of the importance and started to apply the correction in their day-to-day practice. The performance of the balances used is also an important factor, and practical methods to perform their calibration were shared and implemented by the laboratories.

6.3 VALIDITY (QUALITY) CONTROL

The importance of validity (quality) control for every batch of samples analysed was one of the revelations for many participants, especially newcomers in NAA. It may reveal systematic errors, and corrections may be made prior to reporting. Such a validity or QC has been recommended in all feedback and QA/QC workshops. It was strongly recommended to include test portions of a material with known property values, and blanks with every batch of samples to be analysed, even if the batch consists of only one sample, and this seems now to be implemented by all NAA laboratories as could be derived from the presentations at the recent workshops in 2017–2022. Acceptance criteria needs to be specified, and control charts may be considered as a tool for trend analysis and require a realistic estimate of the measurement uncertainty. Nonetheless, many NAA laboratories reported at the workshops of having doubt about the validity of reference materials, purchased many years ago, and of lack of resources to

replace them or getting more appropriate ones with respect to matrix and/or number of elements certified. It has been recommended at the 2022 QA workshop to consider the material from past IAEA facilitated interlaboratory comparisons as reference material. Still, many NAA laboratories depend on the IAEA for obtaining new CRMs.

Many laboratories also became aware that the neutron flux gradient needs to be determined and considered in the analysis, one method being by introducing flux monitors on the top and bottom of samples, in every batch of samples to be irradiated. Another lesson learned was that counting with the sample close to the detector end-cap entails the risk of errors. Ample attention has been given in several of the workshops on practical aspects thereof in view of the k_0 method of standardization, such as the measurement of the neutron flux parameters f and alpha, and their regular verification in multipurpose research reactors. It is noted that some reactors, such as SLOWPOKE and MNSR type reactors, have stable parameters, and this regular verification might not be necessary. It was also noted that in some cases different neutron flux monitors, other than the Zr+Au neutron flux monitors often used in k_0-NAA, may be preferable, depending on neutron flux spectrum and/or irradiation conditions [32].

The importance of having policies and procedures for acting on deficiencies in the results of validity control were explained, especially in view of the possible correct erroneous results reported to third parties. A quality management system with management of non-conformities, including (root) cause analysis, remedial and corrective actions and verification of their effectiveness, does not yet seem to be a routine approach in many NAA laboratories, and is often entirely absent or, at best, in its infanthood. This deficiency, together with lack of expertise in systematic troubleshooting, may explain that some NAA laboratories are not able to improve systematically their performance.

6.4 METROLOGY AND QUALITY ASSURANCE

Introductions have been given at the feedback and QA/QC workshops on the internationally accepted metrological terms and concepts such as trueness, accuracy, uncertainty of measurement, precision, sensitivity and limit of detection; and on mass fraction and concentration, calibrators, standards, comparators, and (certified) reference materials. Relevant documents, issued by the International Bureau of Weights and Measurement and the International Union of Pure and Applied Chemistry were made available.

Quality assurance and quality control were the recurrent themes in all workshops. The presentations and discussions often resulted in tailored recommendations for participant-specific experimental conditions. The QC upon the final analysis results, also known as ‘validity control’, received ample attention. IAEA experts explained how to take advantage of the self-validation opportunities of NAA by using multiple gamma rays and multiple radionuclides from the same chemical element, especially to identify and/or circumvent problems due to spectral and matrix interferences, and coincidence summing effects. Repeated attention was given, as explained before, on preventing and identifying contamination and/or element losses.

As a result of the lessons learned during the workshops, several laboratories started to use Shewhart control charts for visualization of data trends. Basic principles of statistical evaluation such as the use of the zeta score instead of the z-score as an indicator of the results from interlaboratory comparison testing, and the use of median values from replicate measurements rather than the arithmetic mean value, were also covered in the workshops.

Special modules on troubleshooting have also been added to the IAEA e-learning course on NAA [14] to serve as a stepping stone for building up experience in the daily practice of finding potential sources of error. Also, the modules on good practice in NAA, and on sample preparation, provide information on minimizing the risk of errors. Moreover, the 2022 IAEA guide on QA/QC in NAA [13] may serve for easy daily reference.

Method validation remains to be the missing metrological component in many NAA laboratories. It is a requirement for those laboratories intending to apply for ISO/IEC 17025 [1] accreditation of their management system. Method validation provides insight in, amongst others, the degree of trueness of the results but also in the limitations such as due to spectral or matrix interferences. Only a few NAA laboratories achieved such accreditation and not many papers have been published describing method validation in NAA and associated performance indicators such as robustness [33–39]. Modules with guidance on the concept of method validation and how to bring it to the practice in NAA have been added to the IAEA e-leaning course on NAA.

6.5 SELF-RELIANCE OF PARTICIPATION IN INTERLABORATORY COMPARISONS

It is not uncommon that a fee is raised by providers of interlaboratory comparisons, which may be up to a few hundred euros per round. Indications can be found via the European Proficiency Testing Information System [40]. Several NAA laboratories confirmed during the Vienna workshop in 2013 that such fees might be an impediment in participation, and therefore welcomed IAEA's support for interlaboratory comparison.

The NAA laboratories in Asia and the Pacific have also the opportunity of participating in interlaboratory comparisons organized by project groups under the Forum for Nuclear Cooperation in Asia [41, 42].

Overall, any NAA laboratory may consider a bi- or multilateral exchange of samples with other NAA laboratories, e.g., in their region, as an easy and relatively inexpensive alternative. This could be done with any type of material suitable to NAA and with sufficient degree of homogeneity.

7. OUTCOME AND IMPACT

This section discusses the outcome and impact of the proficiency testing exercises organized, including comparisons of performance within each region, and comparison of performance between NAA and other techniques.

7.1 GENERAL

Many NAA laboratories have expanded their knowledge of the metrology of their technique, and the importance of implementing QA/QC procedures to increase the probability that valid results with a known degree of trueness are reported. Several laboratories increased and demonstrated their technical competence, which contributes to their reputation and credibility as a trustworthy partner for element measurements in view of scientific research and third party service efforts. It is an indispensable asset in eventual application for accreditation of implemented (quality) management systems e.g. for conformity with the ISO/IEC 17025 [1].

Some NAA laboratories still face analytical and organizational sources of error that hinder them in the consistent generation of valid results. The problems needs to be effectively resolved before using the technique for acquiring data for scientific studies or embarking on third party service activities.

Technical competence and valid results are also important to the IAEA as stakeholder of the NAA laboratories, especially if the laboratories apply for further IAEA support such as technical cooperation projects or involvement in coordinated research projects.

Newcomer NAA laboratories have shown how the IAEA support resulted in achieving satisfactory to excellent results within a short time after starting up. This is an important outcome in view of the large number of countries embarking on their first research reactor projects, where NAA is expected to be one of the main applications for scientific research and for services of socioeconomic relevance.

7.2 PERFORMANCE OF NAA LABORATORIES COMPARED TO OTHER NUCLEAR ANALYTICAL TECHNIQUES

Reviews of the characteristics of analytical methods such as number of elements measurable, degree of accuracy and (often) limit of detection have been made for many years but most of them are semi-quantitative (e.g. see Refs [43–46]). Moreover, they are often biased by selected potential applications or by focusing on only a few characteristics such as limit of detection or integral duration of an analysis. Direct comparison of the performance of analytical methods in terms of degree of accuracy (trueness and precision and uncertainty of measurement) can be derived from the reports of laboratory intercomparison exercises. The annual reports of WEPAL provide an overview of the aggregated results, in terms of z-scores and performance of each participant, and information is also available on the technique applied.

However, one major difference in analytical techniques is the ability of analysing the test item either ‘as received’, i.e., without further modifying the physicochemical integrity, or ‘as modified for the practice’, which often implies digestion and dissolution of solid materials and may include dilution and/or chemical modification of solutions. A direct comparison of all analytical characteristics of techniques suitable for analysing test items ‘as received’ is rarely done.

The IAEA interlaboratory comparisons for proficiency testing allows for such comparison of the degree of trueness obtained by laboratories using NAA and laboratories using methods based on XRF which are both able to analyse the test item 'as received'. Various approaches based on XRF have been used by the laboratories submitting results for the IAEA interlaboratory comparisons such as based on ED-XRF, milli-XRF, Total Reflection XRF, micro-XRF and PIXE (See Table 1 in Ref. [30]). In order to keep the overview organized, the laboratories using XRF and PIXE have been considered as one category for the comparison. The range of the $|z,z'|$ values for the results by laboratories using NAA, XRF and a few using other techniques in the PTNATIAEA20 round for the soil type and biological type material are shown in Figs 43–52.

It can be derived from Figs 43–52 that a few laboratories using XRF based techniques reported results with a validity equivalent to those reported by the majority of the NAA laboratories, especially for the soil type material. The performance by most XRF laboratories for the biological type material is worse than by the NAA laboratories which can be most likely attributed to the low level of trace elements in this material.

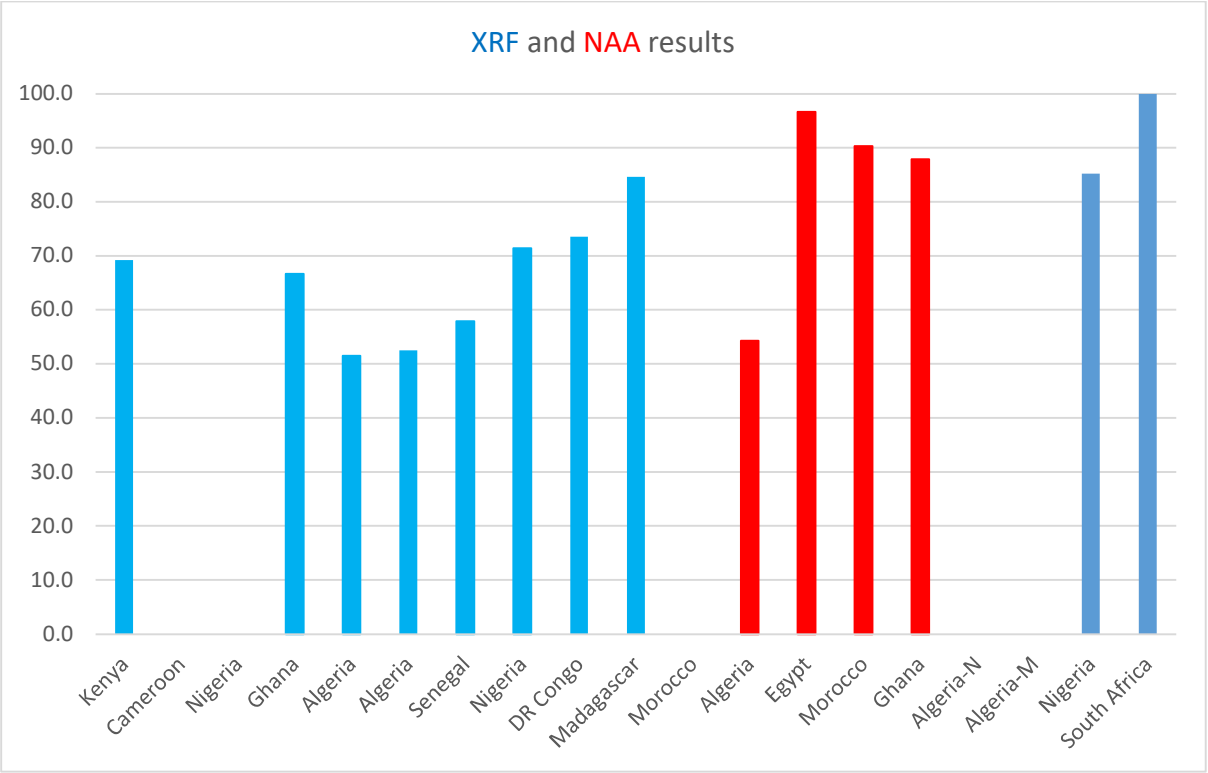


FIG. 43. Comparison of percentage performance indicators for laboratories from Africa using NAA and, XRF-based techniques for the analysis results of soil type material in PTNATIAEA20 interlaboratory comparison.

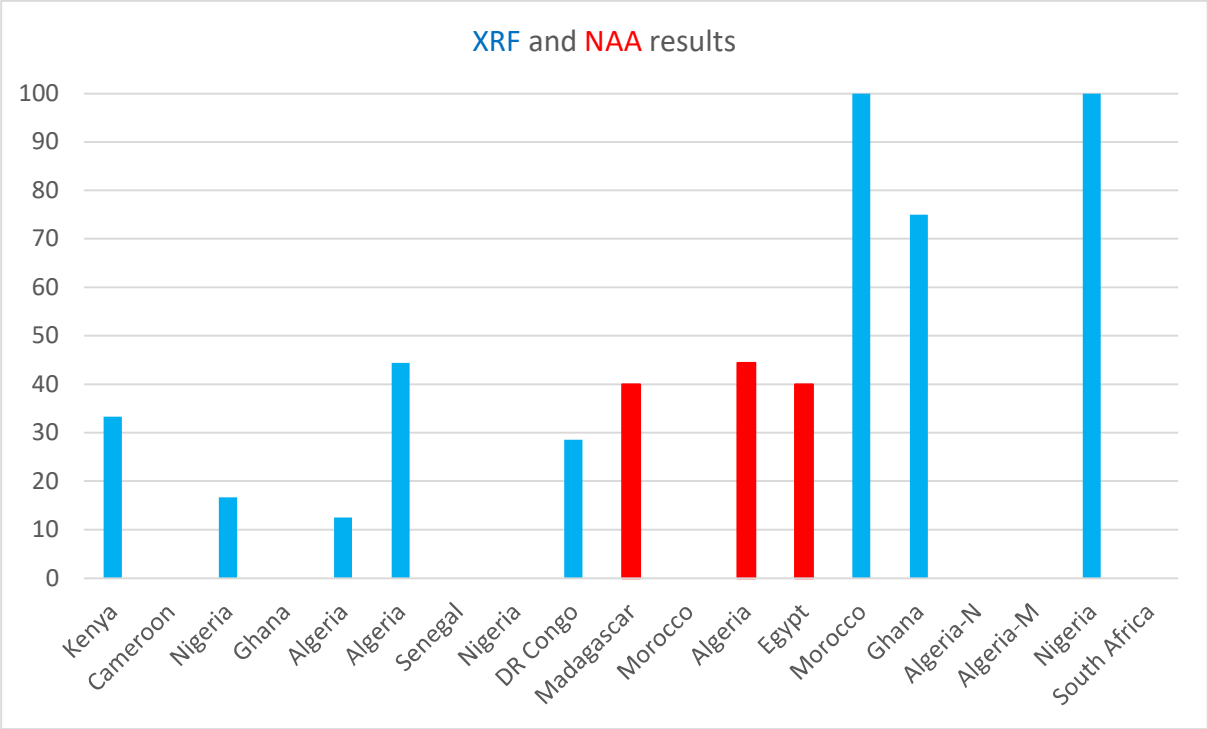


FIG. 44. Comparison of percentage performance indicators for laboratories from Africa using AA, XRF-based techniques for the analysis results of biological type material in PTNATIAEA20 interlaboratory comparison.

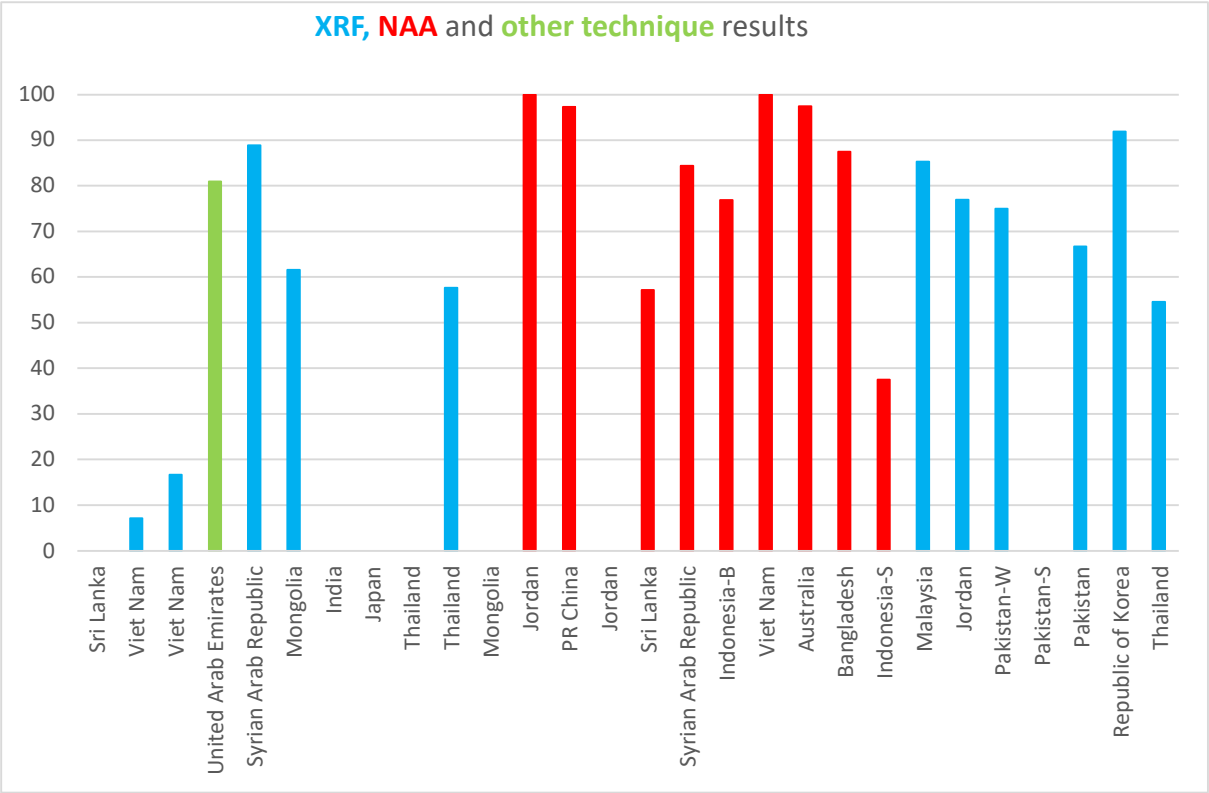


FIG. 45. Comparison of percentage performance indicators for laboratories from Asia and the Pacific using NAA, XRF-based and other techniques for the analysis results of soil type in PTNATIAEA20 interlaboratory comparison.

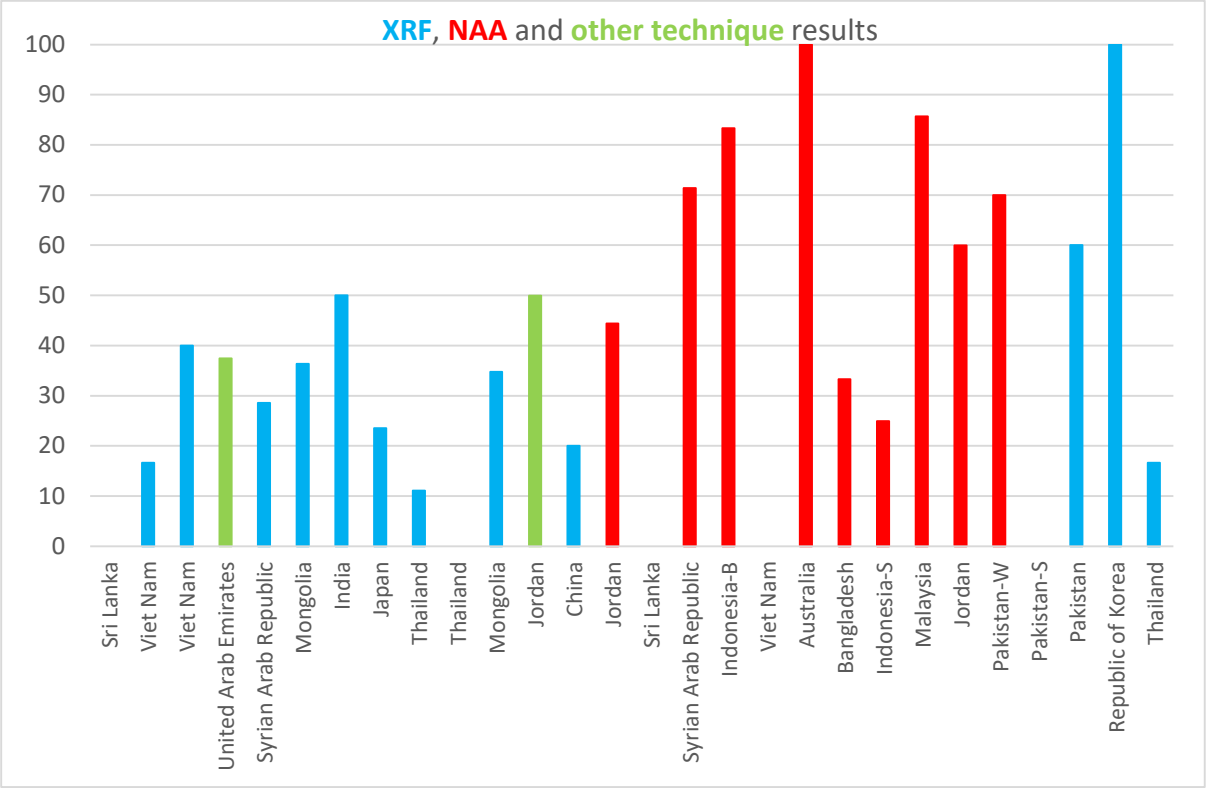


FIG. 46. Comparison of percentage performance indicators for laboratories from Asia and the Pacific using NAA, XRF-based and other techniques for the analysis results of biological type material in PTNATIAEA20 interlaboratory comparison.

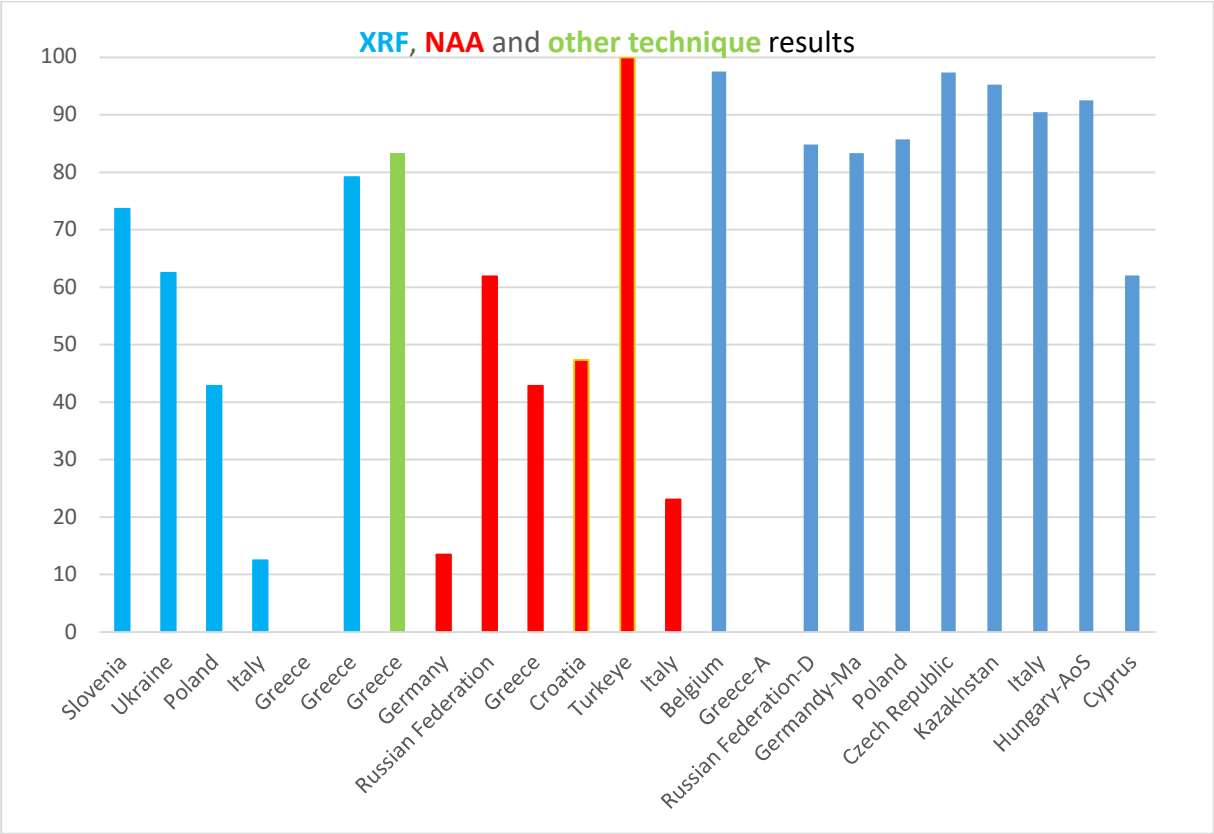


FIG. 47. Comparison of percentage performance indicators for laboratories from Europe using NAA, XRF-based and other techniques for the analysis results of soil type material in PTNATIAEA20 interlaboratory comparison.

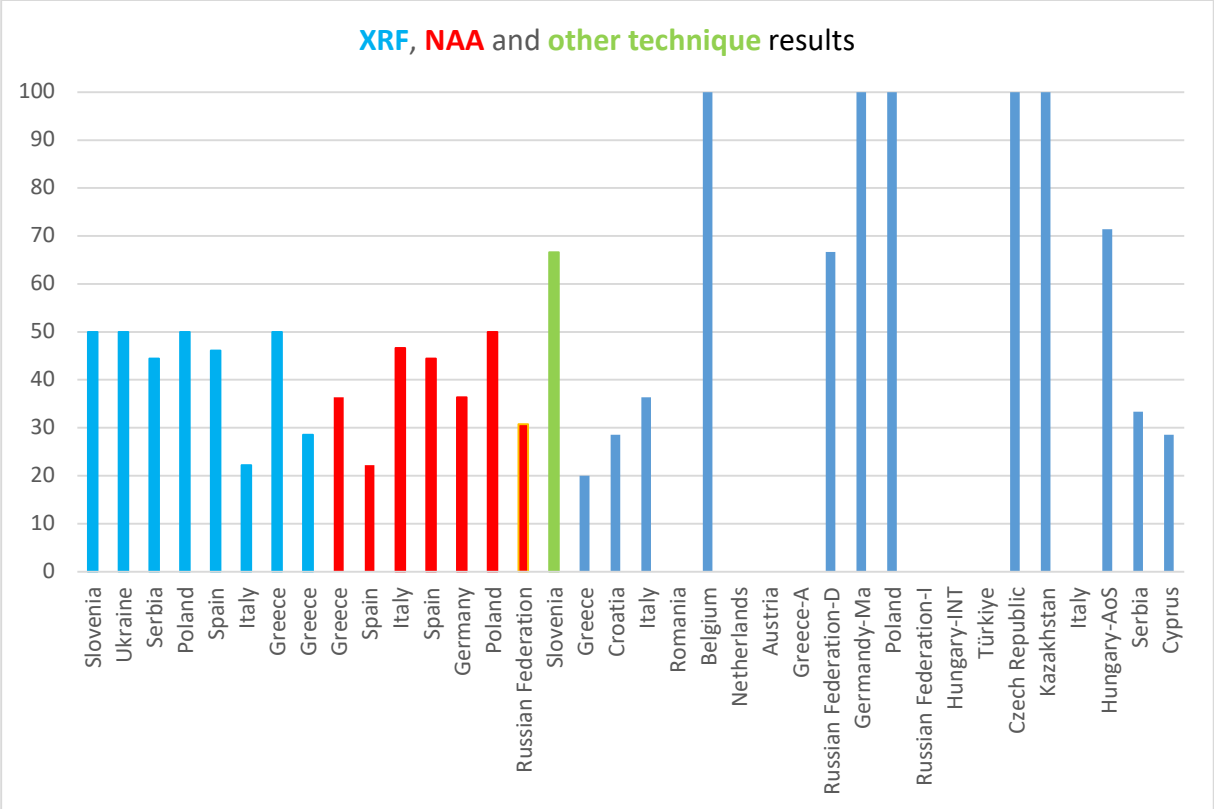


FIG. 48. Comparison of percentage performance indicators for laboratories from Europe using NAA, XRF-based and other techniques for the analysis results of biological type material in PTNATIAEA20 interlaboratory comparison.

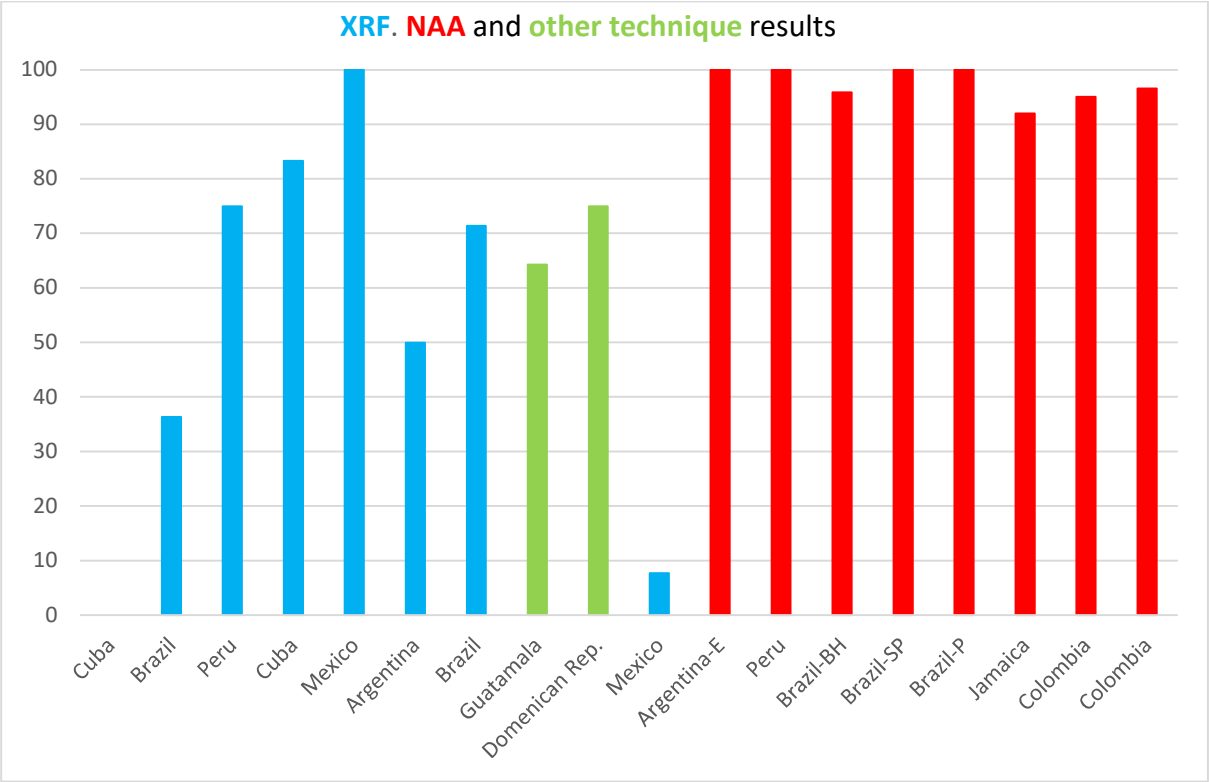


FIG. 49. Comparison of percentage performance indicators for laboratories from Latin America and the Caribbean using NAA, XRF-based and other techniques for the analysis results of soil type material in PTNATIAEA20 interlaboratory comparison.

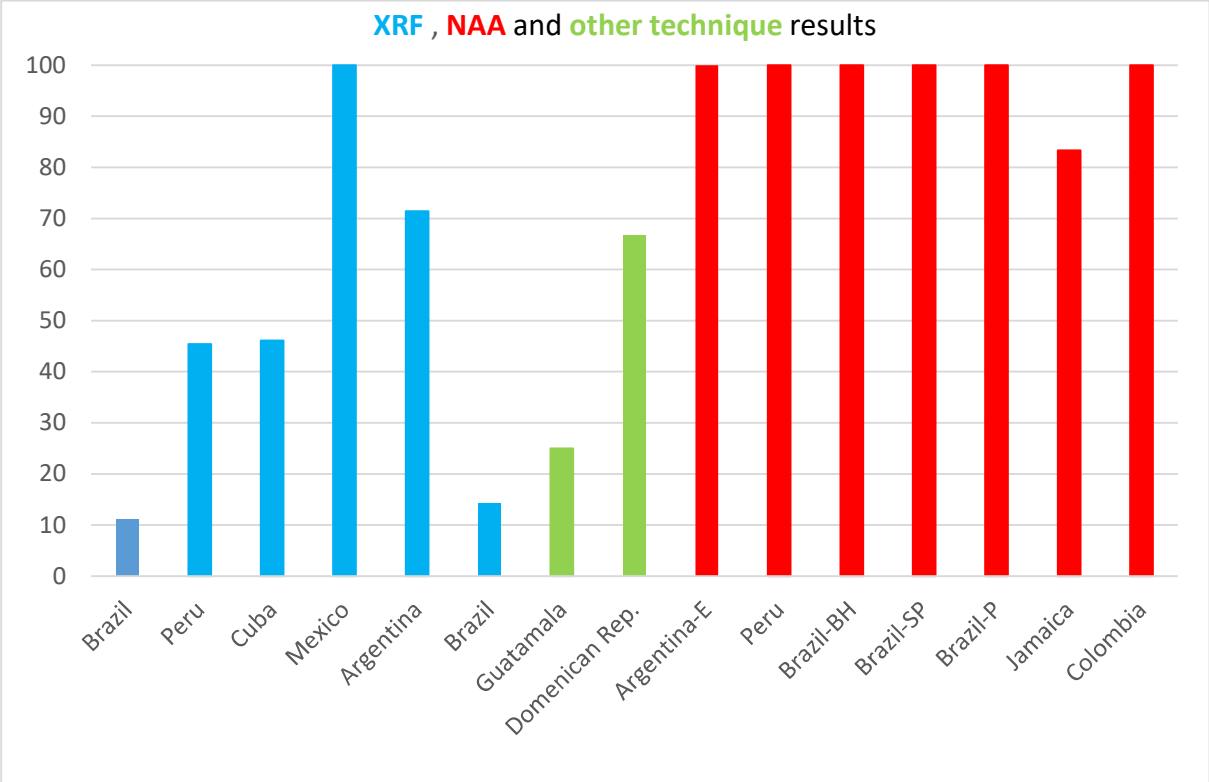


FIG. 50. Comparison of percentage the performance indicators for laboratories from Latin America and the Caribbean using NAA, XRF-based and other techniques for the analysis results of biological type material in PTNATIAEA20 interlaboratory comparison.

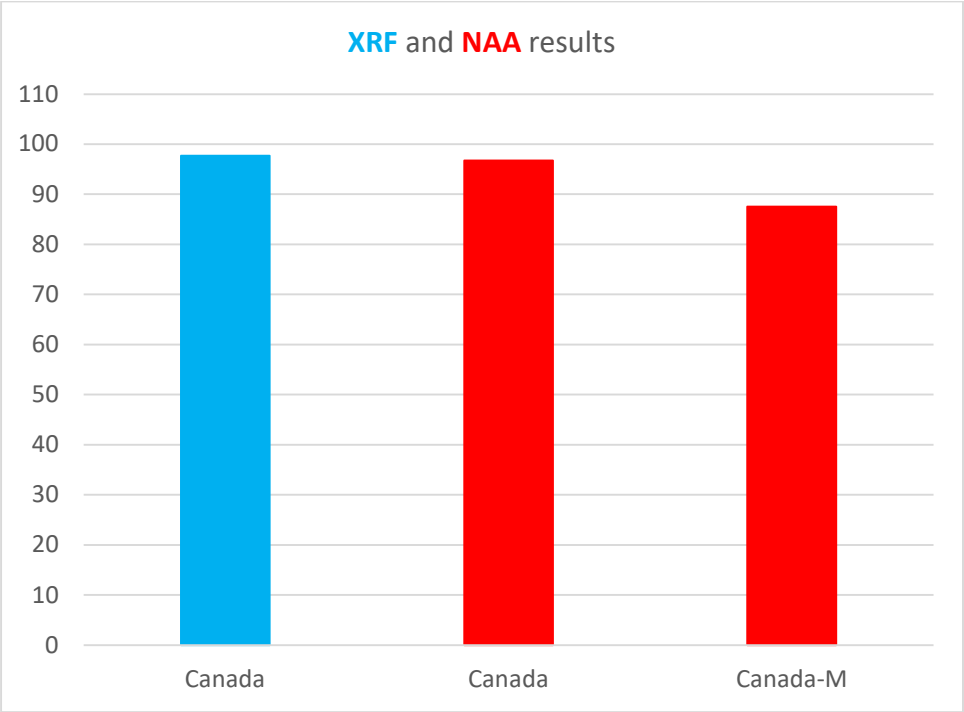


FIG. 51. Comparison of percentage the performance indicators for laboratories from North America using NAA and XRF-based techniques for the analysis results of soil type material in PTNATIAEA20 interlaboratory comparison.

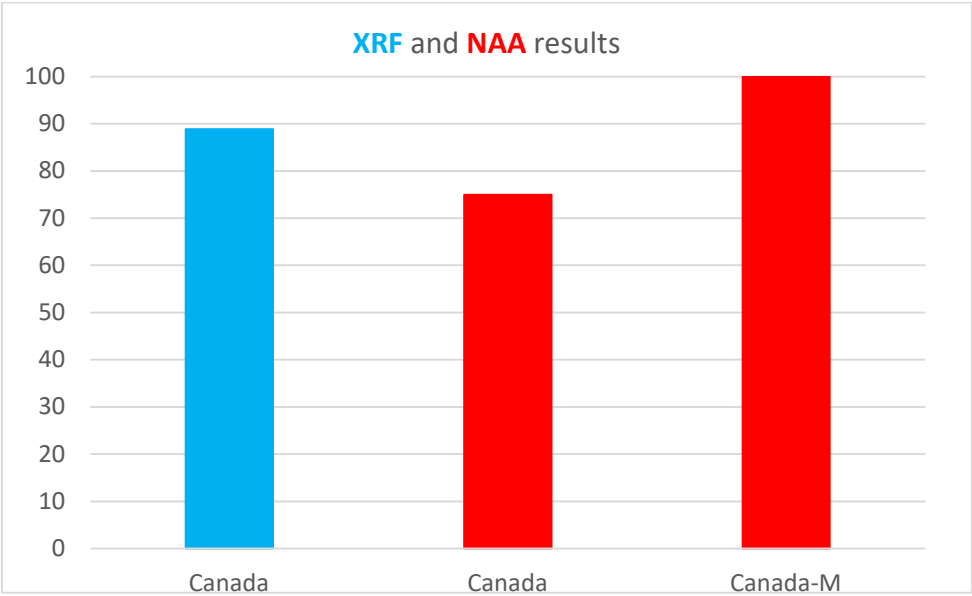


FIG. 52. Comparison of percentage performance indicators for laboratories from North America using NAA and XRF-based techniques for the analysis results of biological type material in PTNATIAEA20 interlaboratory comparison.

However, the results of many other XRF laboratories deviate from the assigned values with many $|z,z'|$ scores > 3, sometimes even with deviations larger than 10 for both types of material as indicated by the box-whisker plots of the range of the (z,z') scores in Figs 53 and 54. The laboratories are ordered by region in these plots. Please note that some data might not been shown since they lie out of the range of the scale of the graphs. The whiskers, i.e. the thin vertical lines associated with the boxes, cover all data points that are considered valid. The data points beyond (lower or higher) the whiskers are the data points considered to be outliers. The

horizontal green dotted lines in Figs 53 and 54 mark the range of $z,z'| < 3$ that forms the basis of the performance indicator.

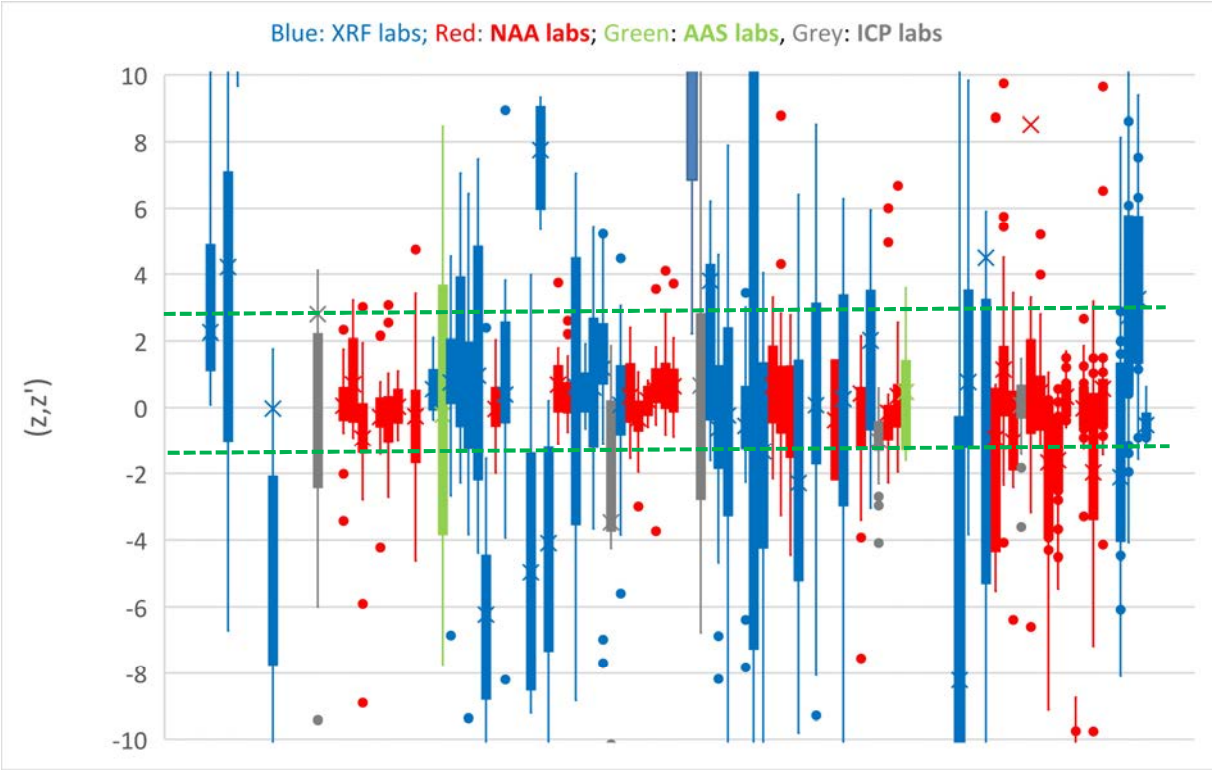


FIG.53. Box-whisker plot representing the ranges of the (z,z') scores of results of analysis of the soil type material submitted in PTNATIAEA20 by laboratories using different analytical techniques.

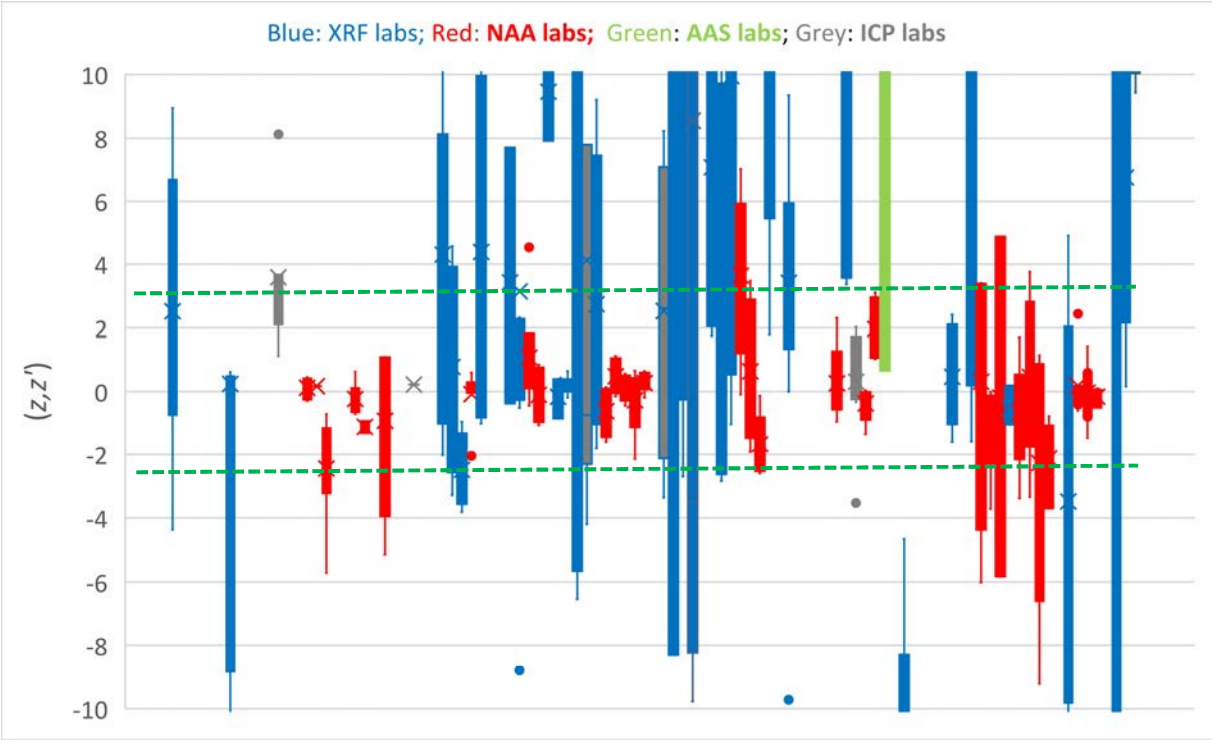


FIG. 54. Box-whisker plot representing the ranges of the (z,z') scores of results of analysis of the biological type material submitted in PTNATIAEA20 by laboratories using different analytical techniques.

Any comparison needs to be balanced against the laboratory's 'customer requirements' and the (*z,z'*) scores need also to be interpreted in terms of R values, the ratio between measured and

assigned value. It was shown in Fig. 1 that even a |z,z'| score of 5 may still imply a difference of up to a factor of 2 or less from mass fractions up to 0.1 $mg \cdot kg^{-1}$. This might be still acceptable if the analyses are done for screening purposes. Nonetheless, several XRF laboratories demonstrated in this interlaboratory comparison that they are able to achieve results equivalent to the best performing NAA laboratories, which indicates that limitations in reaching accurate results do not seem to be technique specific.

8. CONCLUSIONS

The IAEA initiative between 2010 and 2022 to facilitate the participation of NAA laboratories in interlaboratory comparisons for proficiency testing together with feedback meetings resulted in an increase in the analytical and organizational performance of most of the participating laboratories. Several NAA laboratories demonstrated the consolidation of their excellent performance in this period.

Improved performance was achieved by an increase in awareness on potential sources of error, technical and/or organizational, and by implementation of related procedures for QA and QC.

NAA laboratories base their results on calibration against a reference material, the 'relative' or 'comparator' method, or on basis of empirical established physical parameters, the 'k_0 method'. Most laboratories participating in the interlaboratory comparisons are using the 'relative' method in spite of ample promotion of the k_0 method as superior by various experts and the availability of specific IAEA software for this method. Moreover, the results of the laboratory intercomparisons show that many laboratories operating the relative method perform equally well as laboratories operating the k_0 method, and that less good performance occurs in both categories too.

The expected outcome of continued participation in interlaboratory comparisons is further improvement of the analytical performance. Several NAA laboratories mentioned they did not have sufficient own funding and other resources and that they are dependent on the IAEA for this.

It has been observed that retirement and/or leave of experienced staff often results in gaps in the metrological principles of the techniques and the appropriate practical conduct of NAA and, consequently, insufficient ability in identifying of sources of error and designing effective approaches for preventing recurrence. This might be one of the reasons that some NAA laboratories did not achieve a significant improvement of their performance.

The implemented IAEA programme for participation in interlaboratory comparisons for proficiency testing has several important advantages:

— The speed of feedback on laboratory performance through data processing and report preparation within one month of the deadline for submitting results means that any corrective actions that may be necessary can be implemented quickly.
— The compliance of the IAEA's statistical evaluation with ISO 13528 [3] and implemented criteria for identification of outliers and blunders provides an objective and impartial evaluation.
— The feedback and QA/QC workshops provide a unique platform for detailed evaluation of (the origin of) the unsatisfactory results and further discussion on (monitoring and identification of) potential sources of error, and for approaches to effectively minimize their recurrence. It also presents an opportunity for further improvement of the IAEA training material on the metrology and practice of NAA.

The IAEA's interpretation of the performance of NAA laboratories on the fraction of submitted data with $|z|<3$, and subsequent categorization is not based on any international accepted convention. Changing the criterion from $|z|<3$ to $|z, z'| \leq 2$ would bring this performance indicator in better agreement with international accepted interpretations as described in ISO/IEC 17043 [2] and ISO 13528 [3]. It will hardly result in major changes in the

categorization of laboratories under the benchmarks > 90% for ‘excellent’ classification and > 70% for ‘satisfactory”. How the performance indicator could be made independent of the number of elements for which results are submitted also needs to be evaluated, as this would correct the mutual comparison of performances of the laboratories and trends therein.

Analyses of samples from interlaboratory comparison may often be carried out, intentionally or unintentionally, with more care than for routine analyses, e.g., by the use of replicates. In principle, this may even affect the degree of equivalence in the performance of the laboratories. Different laboratories may have different reasons for participating, for example:

— To assess performance under best working conditions, and compare it with other laboratories that also use NAA or that use a different technique; or
— To assess results obtained under routine conditions, to identify unknown sources of error and to optimize the quality of routine work.

This means that the results of the proficiency testing exercises can indicate ‘good’ or ‘bad’ performance but cannot by themselves lead to such categorization. The laboratory itself has to draw conclusions on its performance in view of its mission and its customers’ requirements.

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ANNEX. DETAILS OF PARTICIPANTS

This annex contains the details of participants in the IAEA facilitated interlaboratory comparisons for NAA proficiency testing 2017–2022.

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1029	MASHA	205	Centre de recherche nucléaire de Draria (CRND)	Algeria-M	soil type	X	X		X	X	
					biological	X	X				
1110	NOUSSE	217	Centre de recherche nucléaire de Birine (CRNB); Commissariat à l'énergie atomique (COMENA)	Algeria-N	soil type				X	X	
					biological					X	
1031	ETRR	178	Egypt Second Research Reactor ETRR-2; Atomic Energy Authority (AEA)	Egypt	soil type	X	X	X	X		X
					biological	X	X	X			X
1032	GAEC	182	National Nuclear Research Institute; Ghana Atomic Energy Commission (GAEC)	Ghana	soil type			X	X	X	X
					biological					X	X
1033	CNES	194	Centre national de l'énergie, des sciences et des techniques nucléaires (CNESTEN)	Morocco	soil type		X	X	X	X	X
					biological		X	X		X	X

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1034	CERT	245	Centre for Energy Research and Training (CERT); Ahmadu Bello University (ABU)	Nigeria	soil type	X				X	X
					biological	X				X	X
1035	NECSA	218	South African Nuclear Energy Corp. of South Africa (NECSA)	South Africa	soil type						X
					biological						X
		166	TU Wien, Center for Labelling and Isotope Production, TRIGA Center Atominstitut	Austria	soil type		X			X	
					biological		X			X	
		237	SCK-CEN, NAA lab	Belgium	soil type					X	X
					biological					X	X
1089	NPIAS	176	Nuclear Physics Institute (NPI); Academy of Sciences of the Czech Republic (ASCR)	Czech Republic	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
		224	Nuclear Physics Institute (NPI); Academy of Sciences of the Czech Republic (ASCR)	Czech Republic-2	soil type			X			
					biological			X			
		223	Nuclear Physics Institute (NPI); Academy of Sciences of the Czech Republic (ASCR)	Czech Republic -3	soil type			X			
					biological						

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
			Nuclear Physics Institute (NPI); Academy of Sciences of the Czech Republic (ASCR)	Czech Republic-P	soil type		X				
					biological		X				
		84	National Technical University of Athens, Nuclear Engineering Department - School of Mechanical Engineering	Greece-A	soil type			X		X	
					biological		X			X	
1090	ARIST	94	Aristotle University of Thessaloniki	Greece-T	soil type						
					biological						
		181	Institut für Kernchemie, Johannes Gutenberg-Universität Mainz	Germany-Ma	soil type		X			X	X
					biological		X			X	X
			Radiochemie München, TU München	Germany-Mu	soil type		X				
					biological		X				
1091	KFKI	183	NAA laboratory,Nuclear Analysis and Radiography Department, Hungarian Academy of Sciences	Hungary-AoS	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
1092	REAK	184	Institute of Nuclear Techniques, Technical University Budapest	Hungary-INT	soil type		X	X	X		
					biological		X	X			

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1111	LNIP	191	Laboratory of Applied Nuclear Energy (LENA), University of Pavia	Italy	soil type	X	X		X		
					biological	X	X				
1094	DENNAA	192	Institute of Nuclear Physics; National Nuclear Center of the Republic of Kazakhstan (NNC)	Kazakhstan	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
		248	Delft University of Technology, Reactor Institute Delft	Netherlands	soil type					X	
					biological					X	
		196	Food and Environmental Laboratory, Institute of Nuclear Chemistry and Technology	Poland	soil type		X		X		X
					biological		X				X
1095	SACAV		Instituto Superior Técnico, Instituto Tecnológico e Nuclear; Ministry of Science, Technology and Higher Education (MCTES)	Portugal	soil type						
					biological						
1096	CAMPU	197	Institute for Nuclear Research - Pitesti; Romanian Authority for Nuclear Activities (RAAN)	Romania	soil type		X		X	X	
					biological					X	

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1139	NURES	203	IREN research facility, Joint Institute for Nuclear Research, Frank Laboratory of Neutron Physics - complementary methods	Russian Federation-D	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
		221	Joint Institute for Nuclear Research, Facility REGATA at the IBR-2 reactor	Russian Federation-Z	soil type				X	X	
					biological			X		X	
		234	IREN research facility, Joint Institute for Nuclear Research, Frank Laboratory of Neutron Physics - complementary methods	Russian Federation-P	soil type				X		
					biological			X			
1099	TEFA	198	Jozef Stefan Institute, Department of Environmental Sciences	Slovenia	soil type	X	X	X			
					biological	X	X	X			
1109	YAZA	228	Energy Institute Ayazaga Campus; Istanbul Technical University	Türkiye	soil type				X		
					biological						
1208	SAREZ		Institute of Nuclear Physics AS RUZ	Uzbekistan	soil type						
					biological						

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1102	ACTIVA	165	NAA laboratory at RA6 reactor at Bariloche, Comisión Nacional de Energía Atómica	Argentina-B	soil type	X	X				
					biological	X	X				
1103	TECNUC	61	Técnicas Analíticas Nucleares, Comisión Nacional de Energía Atómica (CNEA), Centro Atómico Ezeiza	Argentina-E	soil type	X	X	X		X	X
					biological	X	X	X		X	X
1136	VOLVI	169	Centro de Desenvolvimento da Tecnologia Nuclear, Comissão Nacional de Energia Nuclear (CDTN)	Brazil-BH	soil type	X	X	X		X	X
					biological	X	X	X		X	X
		171	Laboratório de Radioisótopos, Centro de Energia Nuclear na Agricultura, Universidade de São Paulo	Brazil-P	soil type		X		X	X	X
					biological		X			X	X
1104	IPCN	170	Instituto de Pesquisas Energeticas e Nucleares (IPEN); Comissão Nacional de Energia Nuclear (CNEN)	Brazil-SP	soil type	X	X	X		X	X
					biological		X	X		X	X
1138	CASIL	139	Comisión Chilena de Energía Nuclear (CCHEN)	Chile	soil type	X	X	X			X
					biological	X	X	X			X

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1105	CANCRA	175 or 245	Servicio Geológico Colombiano	Colombia	soil type		X	X	X	X	X
					biological		X	X		X	X
1106	INDIES	55	Centre of Nuclear Sciences; University of the West Indies (ICENS)	Jamaica	soil type	X			X	X	
					biological	X		X		X	
1107	CN5238M	193	Instituto Nacional de Investigaciones Nucleares (ININ)	Mexico	soil type		X	X			
					biological		X	X			
1108	DESAR	40 or 232	Instituto Peruano de Energia Nuclear, (IPEN)	Peru	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
1202	NUCLT	167	Institute of Nuclear Science & Technology	Bangladesh	soil type	X	X			X	X
					biological	X	X		X		X
			Neutron Activation Analysis Laboratory, China Institute of Atomic Energy	China	soil type		X				
					biological		X				
1204	SARIN	187	National Nuclear Energy Agency (BATAN), Yogyakarta	Indonesia-Y	soil type	X	X				
					biological	X	X				
1205	SCIENA	149	National Nuclear Energy Agency (BATAN), Bandung	Indonesia-B	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1209	SELAT	186 or 240	Center for Science and Technology of Advanced, Serpong	Indonesia-S	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
1335	SRDEP	188	NAA group, MNSR Dep., Reactor School	Islamic Republic of Iran-E	soil type	X	X				X
					biological	X	X				X
	Fathivand		Neutron Physics lab, Nuclear Science and Technology Research Institute (NSTRI), Atomic Energy Organization of Islamic Republic of Iran (AEOI)	Islamic Republic of Iran-T	soil type	X	X				
					biological	X	X				
			INAA Lab, Radiation Applications Research School, Nuclear Science and Technology Research Institute	Islamic Republic of Iran-P	soil type		X				
					biological						
	JRTR	219	Jordan Atomic Energy Commission (JAEC), Jordan research and training reactor (JRTR)	Jordan	soil type	X				X	X
					biological	X				X	X
		220	Korean Atomic Energy Research Institute	Republic of Korea							X
											X

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
1206	AGBAN	152	Malaysian Nuclear Agency	Malaysia	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
1336	ACGDIV	195	Environmental Chemistry Group (ECG), Pakistan Institute of Nuclear Science and Technology (PINSTECH)	Pakistan-S	soil type	X	X	X		X	X
					biological	X	X	X		X	X
	Wasim	99	Pakistan Institute of Nuclear Science and Technology (PINSTECH), MNSR Neutron Activation Analysis Lab	Pakistan-W	soil type	X				X	
					biological	X				X	
1100	SYRAT	199	Department of Physics; Atomic Energy Commission of Syria (AECS)	Syrian Arab Republic	soil type	X	X	X	X	X	X
					biological	X	X	X		X	X
1194	PTNAA1	76	Thailand Institute of Nuclear Technology (TINT)	Thailand	soil type	X		X			
					biological	X		X			
1207	LUCST	202	Dalat Nuclear Research Institute, Center for Analytical Techniques	Viet Nam	soil type	X	X		X	X	X
					biological	X	X			X	
		215	Australian National Science and Technology Organisation (ANSTO)	Australia	soil type			X	X	X	X
					biological			X		X	X

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
		172	SLOWPOKE NAA Laboratory, Polytechnique Montreal	Canada-M	soil type		X	X	X	X	X
					biological		X	X		X	X
		222	SLOWPOKE NAA Laboratory, Polytechnique Montreal	Canada M-2	soil type			X			
					biological			X			
			SLOWPOKE-2 Facility, Royal Military College of Canada	Canada-RMC	soil type		X				
					biological		X				
		257	Bureau Veritas Laboratory Mississauga, Environmental Laboratories	Canada-O	Soil type						X
					Biological						X
	UoL		Department of Environmental, Earth & Atmospheric Sciences, University of Massachusetts Lowell	United States of America-L	soil type		X				
					biological		X				
	UoT		University of Texas in Austin, Nuclear Engineering Teaching Lab	United States of America-T	soil type	X	X				
					biological	X	X				
		211	University of Utah, Reactor Laboratory	United States of America-U	soil type			X			
					biological			X			

TABLE A-1. PARTICIPANTS, WEPAL LABORATORY NUMBER AND CODES, IAEA CODES AND FREQUENCY OF PARTICIPATION FROM 2017–2022 FOR SOIL TYPE AND BIOLOGICAL TYPE MATERIALS (Cont.)

WEPAL Nr.	WEPAL Labcode	IAEA code	Laboratory	Member State identifier in this report	Material type analysed	2017-3	2018	2019	2020	2021	2022
		213	Penn State University, Penn State Department of Nuclear Engineering	United States of America-P	soil type			X			
					biological			X			
		214	Chemical Sciences Division, NIST, Chemical Process and Nuclear Measurements Group	United States of America-N	soil type			X			
					biological			X			

ABBREVIATIONS

CRM	Certified reference materials
IPE	International Plant-analytical Exchange
ISE	International Soil-analytical Exchange
ISO	International Organization for Standardization
k_0-NAA	Neutron activation analysis with the k_0 method
NAA	Neutron activation analysis
NDA	Normal distribution approximation
PGAA	Prompt gamma activation analysis
QA	Quality assurance
QA/QC	Quality assurance and quality control
QC	Quality control
XRF	X ray fluorescence

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Training Workshop on Intercomparison Feedback of Neutron Activation Analysis Proficiency Tests Performed in 2017, Ljubljana, Slovenia, 6–10 November 2017

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Training Workshop on Optimization of Performance and Processes in Neutron Activation Analysis, Vienna, Austria, 30 November – 4 December 2020

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