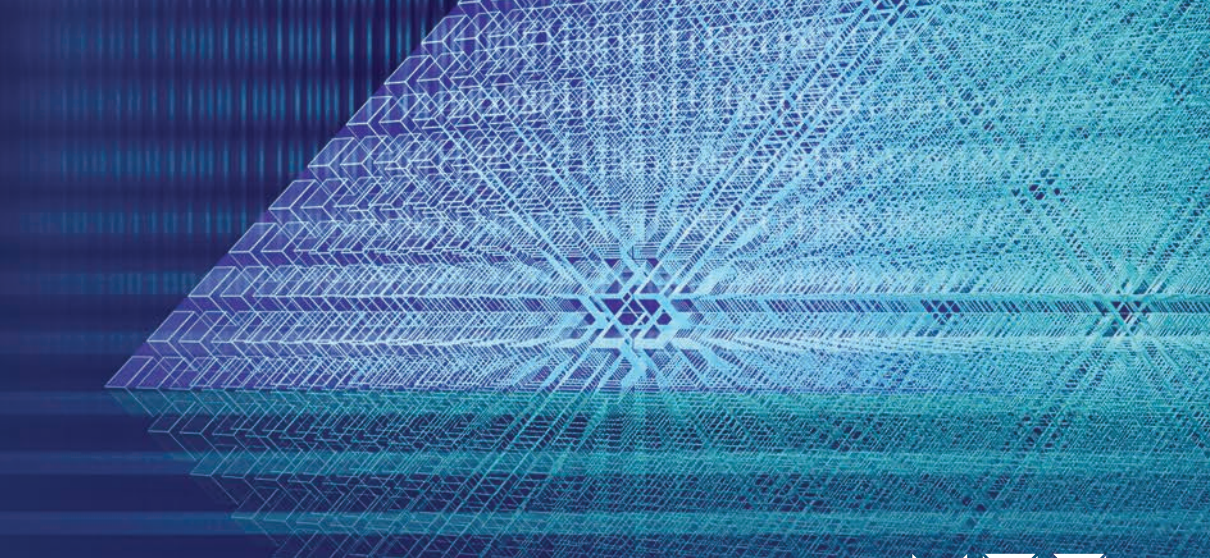

TECHNICAL REPORTS SERIES NO. 487

Quality Assurance and Quality Control in Neutron Activation Analysis: A Guide to Practical Approaches

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QUALITY ASSURANCE AND QUALITY CONTROL IN NEUTRON ACTIVATION ANALYSIS: A GUIDE TO PRACTICAL APPROACHES

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TECHNICAL REPORTS SERIES No. 487

QUALITY ASSURANCE AND QUALITY CONTROL IN NEUTRON ACTIVATION ANALYSIS: A GUIDE TO PRACTICAL APPROACHES

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FOREWORD

Neutron activation analysis (NAA) is the most common technique practised in research reactors. According to the IAEA's Research Reactor Database, NAA is practised in 50 countries, in approximately half of all operating research reactors. The IAEA has a long history of supporting NAA groups in its Member States. By sustaining and expanding the utility of research reactors, benefits to society can be maximized.

In the past decade, the IAEA has coordinated research on the analysis of large intact samples, an area that significantly expands the scope of application of NAA. It has also coordinated the development of an integrated approach to routine automation of NAA, leading to measurable gains in the capacity of laboratories.

For many years, the IAEA has supported the enhancement of the quality of NAA analytical services by providing training in quality assurance and quality control (QA/QC). This has included developing and delivering reference materials and organizing and facilitating participation in interlaboratory comparisons. Since 2010, the IAEA has organized feedback workshops following interlaboratory comparisons, during which participants analyse and identify sources of error, both technical and managerial, and present methodologies for their elimination through improved QA/QC approaches. This strategy has led to rapid and sustained improvement in the analytical performance of many NAA laboratories. In several cases, the lack of improvement in laboratory performance levels — or the inability to sustain improvements — has been attributed to the rapid turnover of personnel and the consequent loss of knowledge and expertise.

To address the issue of knowledge preservation, the IAEA has developed a comprehensive modular e-learning course on NAA. The course covers basic and advanced issues and is therefore appropriate for both newcomers to the technique and experienced practitioners. The course, on-line since 2017, has participants from nearly all countries in which NAA is practised. Several modules address specific sources of error, focusing on how these errors are detected, how the probability of their occurrence is minimized and, if they do occur, how their impact is mitigated.

This publication complements the IAEA e-learning course on NAA by providing guidance on QA/QC approaches in the NAA laboratory. The information provided is intended to be used in the day to day practice of NAA at research reactors. Potential sources of error and associated QA/QC actions are listed, including for the handling and preparation of samples and test portions, calibration, irradiation, decay, measurement, feasibility of analysis and selection of the analytical protocol, and laboratory management. A description of the theoretical background of the sources of error is complemented with references

to the dedicated modules in the IAEA e-learning course. The supplementary files, available on-line, provide additional information and practical QA/QC tools for use in the laboratory.

The IAEA wishes to acknowledge the assistance provided by the contributors and reviewers listed at the end of this publication, especially P. Bode (Netherlands) and P. Vermaercke (Belgium). The IAEA officer responsible for this publication was N. Pessoa Barradas of the Division of Physical and Chemical Sciences.

EDITORIAL NOTE

CONTENTS

1. INTRODUCTION

1.1. BACKGROUND

Since the early stages of the development of neutron activation analysis (NAA), practitioners have sought to explain the reasons for unsatisfactory or inaccurate measurement results. This effort has to some extent been aided by the fact that NAA quantification is based on physical principles, which facilitates an understanding of the causes of inaccuracy. Several sources of error[1] could be identified and quantified during the pioneering years of the 1950–1970s [1–6]. The first key publications on activation analysis [7, 8] addressed major sources of error, such as differences in counting geometry, neutron flux variations and the effects of high counting rates. NAA has been a valuable technique in the characterization of candidate reference materials. Interlaboratory comparisons, also combined with other methods, have further contributed to knowledge on sources of error and to approaches for prevention and correction [9, 10]. Reports on the cause of inaccurate results, and solutions to identify the corrections, continue to be published [11–14]. However, rather than identifying significant new sources of error, in many cases these reports discuss causes that had been identified during the pioneering years.

Since 2010, the IAEA has supported a new approach to interlaboratory comparisons for NAA proficiency testing by organizing feedback workshops at which laboratory representatives discuss measurement results [15]. Unsatisfactory results have often been attributed to both technical and/or managerial errors. For example, it has been noted that the preservation of knowledge and expertise in NAA is not always optimal owing to the usually short overlap period between the retirement of senior staff and the recruitment of new staff members from a younger generation. Moreover, many laboratories do not have the resources for unlimited access to leading NAA journals. As a result, newcomers to NAA do not always have the opportunity to study earlier papers that describe sources of error and explain how they can be detected and what can be done to either prevent them or at least mitigate their impact.

One of the lessons learned from the interlaboratory comparisons facilitated by the IAEA [15] is that errors occur just as frequently during the management of the NAA process as they do during the actual performance of the technique.

[1] ‘Error’ and ‘uncertainty’ refer to different concepts. Errors include mistakes, blunders and also systematic and random variations, all resulting in a systematic or random bias (i.e. a difference between the measurement results and a reference value). Uncertainty of measurement describes the dispersion of measurement results if repeated.

Errors noted during the management of the NAA process occurred at different steps and include the following:

— Incorrect decision on the feasibility of an analysis by NAA for a given sample type and elements to be measured in view of the resources available (i.e. human, equipment, time);
— Selection of an inappropriate analytical protocol (choice of sample mass, irradiation–decay–measurement time, sample–detector distance during measurement);
— Linkage discrepancies (e.g. between samples and their results, samples and their test portions, samples and the customer codes);
— Errors during transcription and reporting;
— Analysis by insufficiently qualified or insufficiently trained persons.

Errors during the performance of NAA were noted at each step, and include the following:

— Insufficient or no homogenization;
— Lack of knowledge or disregard for the dry mass correction;
— Geometrical differences between sample and calibrator during irradiation and/or measurement;
— Failure to consider nuclear interference, spectral interference and coincidence summing effects;
— Contamination and element losses during sample preparation and during irradiation;
— Failure to consider self-attenuation effects during irradiation and measurement.

Monitoring for the possible occurrence of errors, minimizing their probability and mitigating their impact are accomplished through quality control (QC) and quality assurance (QA), respectively. Quality assurance is process oriented and focuses on defect or risk prevention, while QC is oriented towards the product or analytical result and focuses on the identification of defects. The international standard ISO 9000:2015[2] defines QC as "part of *quality management* focused on fulfilling *quality requirements*" and QA as "part of *quality management* focused on providing confidence that *quality requirements* will be fulfilled" [16]. However, these definitions do not easily transfer to the

[2] ISO 9000:2015 [16] describes the fundamental concepts, principles and associated vocabulary of quality management systems.

application of QA/QC in practice. The International Union of Pure and Applied Chemistry (IUPAC) Gold Book [17] provides the following definitions:

Quality control:

"The maintenance and statement of the quality of a product (data set, etc.) specifically that it meets or exceeds some minimum standard based on known, testable criteria."

Quality assurance:

"The guarantee that the quality of a product (analytical data set, etc.) is actually what is claimed on the basis of the quality control applied in creating that product. Quality assurance is not synonymous with quality control. Quality assurance is meant to protect against failures of quality control."

An interpretation of the definition of QC is that it is an 'end of the pipeline' activity, performed at any point at which it has to be decided if a result (e.g. degree of trueness, uncertainty of measurement, limit of detection) is in agreement with predefined specifications [18]. Specifications may be set by a laboratory or by an end user, or may also be of a legal nature. Quality control is therefore a binary process: the result of an assessment can only be 'acceptable' or 'not acceptable'.

An interpretation of the definition of QA is that it represents all actions implemented to minimize the probability of rejection in QC. It is therefore a proactive process and comprises the following:

— Identification of potential sources of error at any step of the analytical procedure;
— Implementation of methodologies and techniques to minimize the probability of occurrence of these errors;
— Implementation of methods and techniques to monitor their occurrence;
— Definition of acceptance and rejection criteria;
— Implementation of non-conformity management with root cause analysis.

The two concepts of QC and QA cannot be separated. The implementation of a QC mechanism to verify whether or not a result meets specifications can also be considered to be a QA step, as it minimizes the probability that unacceptable results will be used or reported.

1.2. OBJECTIVE

Despite the significant body of work available, discussing the considerations mentioned above on the recurrence of errors in NAA is the primary objective of this publication. Errors may occur during each step of NAA, from the review of the request for analysis up to the reporting of the measurement results. Therefore, to minimize the probability that incorrect results are reported, QA/QC needs to be considered for each of these steps. Examples of such errors and associated practical QA/QC approaches are provided in this publication. It will also be shown that errors may be difficult to detect and that in some cases QC is (almost) impossible. Finally, this publication aims to serve as a guide to QA/QC in NAA by addressing the questions: 'What can go wrong in NAA?', 'What can be done to prevent mistakes?', and 'How do we know if things went right or wrong?'.

Guidance provided here, describing good practices, represents expert opinion but does not constitute recommendations made on the basis of a consensus of Member States.

1.3. SCOPE

This publication is intended for practitioners of NAA and aims to serve as a reference guide during daily activities. It is therefore concise and does not attempt to cover the various aspects of NAA that are needed for an understanding of related issues. More detailed information is provided in the IAEA e-learning course on NAA [19]. The course covers both basic and more advanced issues that are appropriate for newcomers to the technique as well as for experienced practitioners. It is structured according to thematic areas, including calibration, instrumentation, quality and NAA practice, with each area containing modules on a range of subjects. Extensive reference is made in this publication to various modules of the e-learning course. A guide can be found in the Appendix, and further technical and scientific information can be obtained from relevant literature (e.g. Ref. [20]). Annexes I and II are dedicated to basic principles of control charts and to examples demonstrating the inherent QC of NAA.

Quality assurance procedures (minimizing the probability of an error) and QC procedures (observing an error) can contribute to preventing the eventual reporting of incorrect results, but not all types of error are covered (e.g. although processed simultaneously, element losses due to volatilization may occur in real samples but not in control samples such as reference materials). Both QA and QC are based on answers to certain questions, such as the following:

— Are we sure about this?
— Can something go wrong?
— How do we minimize the probability that it will go wrong?
— How do we detect if something went wrong?
— When is the shortcoming unacceptable?
— How do we manage the things that went wrong?

This publication addresses these questions and, in general, how QA/QC can be applied to the various steps of NAA.

1.4. STRUCTURE

Following this introduction, eight sections address the most common types of error in the planning, performance, organization and management of NAA, with examples given of QA/QC approaches to minimize the probability and mitigate the impact of these errors. Each section includes a table summarizing the types of error that may occur and the opportunities for implementing QA/QC. These tables are not intended to be complete: NAA practitioners should use their technical competence and experience to identify additional potential errors, and their creativity to find approaches to deal with them in their own laboratory. Note that most errors can be attributed to human failure.

The tables demonstrate that QA and QC are integrated areas. For example, a QA action may consist of the implementation of a specific QC procedure; the performance of that procedure is, in fact, a QC action.

These sections are followed by conclusions and an appendix on the IAEA's e-learning course on NAA. Annexes I and II focus on the basic principles of control charts and on examples demonstrating the inherent QC of NAA. Annex III presents the tables in a user-friendly format for daily use in laboratories. All three annexes are available on-line only as supplementary files.[3]

2. SAMPLE HANDLING AND PREPARATION OF TEST PORTIONS

The size of the laboratory sample (i.e. the material provided by the customer) is typically much larger than that needed for the test portion (i.e. the amount to be

[3] Available on the publication's individual web page at www.iaea.org/publications

irradiated). Subsampling is needed to obtain a test portion that is representative of the laboratory sample (i.e. ensuring that the variance of an intralaboratory comparison, or intermediate precision, of the property value of interest in such test portions is less than a predefined value). Quality assurance procedures are needed to ensure the following:

— Unambiguous identification of the measurand [21] and appropriate documentation, such as labelling, to ensure that all objects and files have unique name tags.
— Verification of repeatability[4] against a predefined acceptance value in order to confirm the degree of homogenization (if applicable) [22, 23]. As an example, a repeatability standard deviation of less than 2% could be set.[5]
— No interchange of samples and test portions during the procedure.
— Anticipation of potential contamination (e.g. by iron, chromium, manganese, cobalt from stainless steel) during sample size reduction from milling and crushing machines and other tools; of element losses due to volatilization, wall adsorption or heating; and of other factors (NAA e-learning module P10 [19]).
— Measurement of the moisture content in the samples. Mass fractions are usually expressed on a dry mass basis. The moisture content may vary from a few per cent for soil samples to 10% or more for plant materials, human and animal tissue and other organic matter. Documentation for certified reference materials (CRMs) may contain information on moisture measurement procedures (NAA e-learning modules C6 and P10 [19]).
— Anticipation of possible sample mass changes by moisture uptake during the weighing of extremely hygroscopic materials. The finer the particles, the more hygroscopic are the materials. Rice, hair and nails all serve as examples of such (very) hygroscopic materials. Filter papers (e.g. for liquid test portions) are also hygroscopic.
— Anticipation of weighing problems caused by static electricity. This may lead to difficulties in the weighing of very small amounts of material in

[4] Repeatability is the closeness of the results obtained with the same sample (or subsamples of the same sample) using the same measurement procedure, same operators, same measuring system, same operating conditions and same location over a short period of time [21].

[5] The larger the number of replicates, the smaller the standard error of the mean and the smaller the contribution of sampling uncertainty to the overall measurement uncertainty. If homogenization is not possible, a first indication of the contribution of the sampling uncertainty, at least for elements in food and feedstuffs, may be seen using the equation of the Horwitz uncertainty or similar approaches [22, 23]. Statistical evaluation of the results of five to ten replicates may be needed.

plastic capsules, and/or a distribution of such amounts throughout the capsule. The geometry is then not well defined.

— Recording of the filling height of the vials, thickness of the pellet, material composition and apparent density. This information will be needed for geometry correction, gamma ray self-attenuation effects and neutron self-shielding during calculation (NAA e-learning modules B8 and C5 [19]).
— Anticipation of potential problems during irradiation, such as losses owing to volatilization and migration of volatile elements (e.g. bromine, mercury) through the walls of polyethylene containers. This results in both loss of mass with the test portion and contamination of the container's environment and possibly other containers (including the blank). In addition, impurities in the encapsulation material (e.g. chromium in polyethylene) may result in contamination of the test portion with ^{51}Cr during irradiation owing to recoil from the polyethylene. Such problems may be resolved by irradiation in sealed, ultrapure quartz ampoules.

Quality control opportunities exist for monitoring the adequacy of several, but not all, QA measures implemented to minimize the occurrence of errors. Errors that may typically occur in the preparation of sample test portions, and suggestions for related QA actions, are given in Table 1. More information on sample preparation can be found in Ref. [24] and in the NAA e-learning modules P2 and P10 [19]; concepts dealing with control charts are described in Annex I and NAA e-learning module Q4 [19].

TABLE 1. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST PORTION PREPARATION AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Interchange of samples	— Use an unambiguous coding system	— Perform a visual check — In case of doubt of sample interchange, check the initial weighted mass of the samples
Insufficient homogenization	— Expand the work instruction for homogenization to include experimental verification through the analysis of replicates — Ask customer for details of sample homogeneity	— Perform a statistical evaluation of the results of five to ten replicates
Moisture content ignored	— Use common sense — Create and use a checklist for review of request — Measure moisture fraction — Check samples for constant weight during drying process	— If drying and moisture correction is applied routinely, check the registration form. Otherwise, none
Element loss during drying	— Ensure appropriate drying temperature for volatile elements — Use common sense — Use freeze drier or desiccant drying technique	None
Hygroscopic behaviour ignored	— Use common sense — Measure moisture fraction	None

TABLE 1. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST PORTION PREPARATION AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Contamination during production of test portion	— Follow appropriate practices in the laboratory — List sources of contamination and associated elements — Use blanks — Use common sense — Use laminar flow hoods	— Analyse procedural blanks
Weighing; balance malfunctioning (instability, invalid calibration, non-linearity, etc.)	— Perform balance calibration — Conduct regular intermediate performance tests	— Create and use control charts of performance tests and of the periodic (yearly) calibration
Static electricity during weighing	— Use anti-static electricity devices, such as alpha radiation emitting sources	— Perform a visual check
Small sample masses (e.g. a few milligrams) are distributed through the capsule, affecting irradiation and counting geometry	— Use dedicated encapsulation — In some cases glue a few grains to the capsule	— Perform a visual check
Wrong encapsulation material	— Review irradiation requests for elements that may cause problems	None
Sampling handling/test portion preparation	— Perform internal QC: process a portion of a (certified) reference material and a blank capsule simultaneously with the real samples	— Analyse results of control materials and compare with reference values

3. CALIBRATION

3.1. ENERGY, EFFICIENCY AND ELEMENT CALIBRATION

Calibration[6] is required to ensure accuracy in the weighing of test portions; the temperature of ovens used for drying; the volumes of glassware and pipettes used for making in-house solutions of working calibrators; and detector photopeak efficiency.

Calibrations form an essential part of any QA programme, as their absence may have an immediate negative impact on the trueness of measurements taken with instruments. Although weighing or moisture content are not, in most cases, large contributors to the overall uncertainty of NAA, metrological traceability of the related instruments is nevertheless recommended. Metrological traceability of parameters, such as sample weight and temperature, will need to be demonstrated. This can be achieved by using accredited calibration laboratories. A less expensive but more labour intensive alternative is that the laboratory performs these calibrations internally using calibrated reference weights (of the same range as the samples or standards) or calibrated reference thermometers for oven and refrigerator verification. In this case, the laboratory needs to keep records of these calibrations and calculate the associated calibration measurement uncertainty.

However, the most important calibration is the photopeak efficiency. The full energy response of the semiconductor detector (photopeak efficiency) is obtained by measuring the response of the detector to the emission of a metrologically traceable gamma ray source (NAA e-learning module I4 [19]). This may be a single or mixed radionuclide source, and it may be a 'point' source or a source of extended geometry, reflecting the geometry of frequently measured sample test portions.

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[6] Ref. [21] defines calibration as an "operation that, under specified conditions, in a first step, establishes a relation between the **quantity values**, with **measurement uncertainties** provided by **measurement standards** and corresponding **indications** with associated **measurement uncertainties** and, in a second step, uses this information to establish a relation for obtaining a **measurement result** from an indication.

NOTE 1 A calibration may be expressed by a statement, calibration function, **calibration diagram**, **calibration curve**, or calibration table. In some cases, it may consist of an additive or multiplicative **correction** of the indication with associated measurement uncertainty.

NOTE 2 Calibration should not be confused with **adjustment of a measuring system**, often mistakenly called 'self-calibration', nor with **verification** of calibration.

NOTE 3 Often, the first step alone in the above definition is perceived as being calibration."

There may be situations in which it is almost impossible to determine the efficiency curve experimentally (e.g. when very large, complex or irregular shaped sample types have to be analysed). In such cases, an estimate of the photopeak efficiency may be calculated (e.g. using Monte Carlo based calculations). Some detector manufacturers provide software to perform such calculations when the detector is procured.

The relationship between the multichannel analyser channel and the gamma ray energy, together with the determination of the peak shape parameters (NAA e-learning module I3 [19]), is often denoted as 'detector calibration'. However, it is primarily an energy calibration and a verification of the detector's performance through peak shape parameters. Most gamma ray energy measurements of the radiation emitted by the sources used are not derived from calibration against a metrological standard but rather from empirical assessments. Moreover, in most cases, the uncertainties in the gamma ray energy measurements are not given.

Element calibration is the term often used for determining the proportionality factors between the net peak area in the gamma ray spectrum and the mass of an element. These factors are experimentally measured in NAA. In the relative method, the calibration is done using test portions from single element standards or CRMs[7]. In the k_0-NAA method, tabulated factors are mainly used. More information can be found in NAA e-learning modules C1, C2 and C6 [19].

Typical errors that may occur during calibration procedures, as well as suggestions for related QA/QC actions, are summarized in Table 2 [25–27].

3.2. STANDARD (CALIBRATOR) PREPARATION

In addition to the measures described above for managing errors during sample handling, test portion preparation and calibration, further QA/QC measures are needed that address the use of standards (calibrators). Such measures depend on the type of calibration.

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[7] Whereas a reference material (RM) is defined as a material that is sufficiently homogeneous and stable with regard to specified properties and that has been established to be fit for its intended use in measurement [17], a CRM adds the dimension of documentation (a certificate) issued by an authoritative body and provides one or more specified property values with associated uncertainties and traceability using valid procedures. In this publication, the term 'reference material' is used in addition to the term 'certified reference material' to emphasize that there are many cases in which the analysis of an RM is sufficient, and the analysis of a CRM unnecessary.

TABLE 2. POTENTIAL ERRORS DURING CALIBRATION PROCEDURES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Calibration not acceptable (e.g. to conformity assessment bodies)	— Use, preferentially, CRMs produced in accordance with the requirements of ISO 17034 providing metrological traceability to SI, or otherwise RMs with metrological traceability to an appropriate reference [25, 26][a] — Arrange for calibrations to be performed by conformity assessment bodies	— Check the results of CRM analysis
Invalid calibration status	— Conduct regular performance tests after and between calibrations — Check calibration status on instruments — Add date for recalibration in on-line calendar	— Conduct internal audits — Create and use control charts
Coincidence effects not accounted for in the photopeak efficiency calibration	— Determine the total efficiency curve, enabling coincidence correction calculations — Use validated software [26][b]	— Check the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II) — Compare the ratio of coincidence-free radionuclides (e.g. Cr-51, Zn-65) with non-coincidence-free radionuclides (e.g. Co-60, Se-75) at different positions from the detector

TABLE 2. POTENTIAL ERRORS DURING CALIBRATION PROCEDURES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Voluminous efficiency not accounted for in the photopeak efficiency calibration	— Use validated software or appropriate Monte Carlo software packages	— Check the results of CRM analysis and proficiency testing
Inadequate fit of the photopeak efficiency	— Use validated software	— Verify the chi-square of the residuals of the fit — For k_0-NAA: analyse appropriate (synthetic) multi-element materials [27]
Unacceptable certificates of calibration sources	— Describe criteria for acceptable certificates — Conduct incoming check of all calibration certificates	— Conduct internal audits

[a] ISO 17034 [25] specifies general requirements for the competence and consistent operation of RM producers. It covers the production of all RMs, including CRMs, and aims at confirming or recognizing the competence of RM producers. According to ISO/IEC 17025 [26], in order to ensure that measurement results are traceable to the International System of Units (SI), values of CRMs provided by a competent producer can be used. RM producers fulfilling the requirements of ISO 17034 are considered to be competent.

[b] ISO/IEC 17025, paragraph 7.11.2 [26], adds in Note 2 that "Commercial off-the-shelf software in general use within its designed application range can be considered to be sufficiently validated." However, it is also recommended in paragraph 7.11.2 that "Whenever there are any changes, including laboratory software configuration or modifications to commercial off-the-shelf software, they shall be authorized, documented and validated before implementation." The latter may also be derived from its fitness for purpose demonstrated in scientific publications.

3.2.1. Comparator or relative NAA

For comparator or relative NAA (see NAA e-learning module C1 [19]):

(a) An equal isotopic abundance has to be present in the calibrator test portion and in the sample test portion of the element to be measured. Variations in isotopic abundance are known to exist in elements such as sulphur, calcium and uranium (and also lithium and boron, both of less importance to NAA). Many uranium chemical compounds can be depleted (or enriched) in ^{235}U isotopic content, sometimes as low as 0.4% instead of approximately 0.7%. The use of such compounds for estimating the quantity of fission products produced would yield values lower than achieved when irradiating materials containing uranium in natural isotopic abundance. Use of uranium compounds with certified isotopic abundance is therefore the only option for measuring the quantities of fission products and obtaining valid correction factors for the related lanthanide isotopes.
(b) Appropriate certificates of the metrological qualities of the calibrator material need to be available if metrological traceability of property values to the International System of Units (SI) is necessary. Calibrators will therefore preferably be CRMs of pure elements with certificates containing information on the specified property value, its associated uncertainty and metrological traceability to SI. CRMs of a composed character (i.e. that imitate the type of material under study) are also useful, although the uncertainty of the property value is higher than when using pure element standards for calibration.
(c) Attention needs to be given to the approximate degree of equivalence in average atomic number of samples and calibrators (relevant in view of gamma ray self-attenuation) and to the presence of large quantities of neutron and/or gamma ray absorbers.

It is also possible to prepare a calibrator based on a combination of aliquots of pure element calibrators, although it has been outlined that a minor impurity in one calibrator may be a significant interfering element for another calibrator [28].

Calibrators without certified property values are not to be used in NAA because the stoichiometry can be ambiguous. This applies, in particular, to cases such as off the shelf chemicals containing metal compounds, in which the metal content has to be derived from the compound's chemical formula [28].

Ideally, the physical dimensions of the calibrator portions are equivalent to those of the sample test portion to minimize errors during the measurement of the induced radioactivity.

3.2.2. k_0-NAA

Assuming that the k_0-NAA method has been implemented and validated (see NAA e-learning module C2 [19]), the routinely used calibrator in this technique is the monitor for the thermal and epithermal neutron flux.

Typical errors that may occur in the preparation of calibrator test portions, as well as suggestions for related QA/QC actions, are summarized in Table 3. Further information on standards, calibrators and reference materials in NAA can be found in NAA e-learning module C6 [19].

TABLE 3. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST PORTION PREPARATION FOR CALIBRATION PURPOSES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL
(in addition to those in Table 1)

Source of error	Action	
	Quality assurance	Quality control
Differences in the isotopic abundance of some elements in real samples and some elements in standards	— Refer to checklist upon receipt of samples that contain elements that may have isotopic abundance different from the standards — Use RMs from producers conforming to ISO 17034	— None, except for U-235 through measuring the ratio of fission product activity and Np-239 activity — Use inherent QC of NAA (see Annex II)
Stoichiometry of pure element standards differs from expectation	— Use CRMs of single element standards	None
Mass of the (certified) RM used is too small	— Use minimum mass as prescribed in the RM's certificate	— Conduct independent verification of masses used

TABLE 3. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST PORTION PREPARATION FOR CALIBRATION PURPOSES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL
(in addition to those in Table 1) (cont.)

Source of error	Action	
	Quality assurance	Quality control
Differences in filling the height of material in capsules	— Measure filling heights of samples and standards — Pelletize samples and calibrators	— Verify filling or pellet height — Density (for solid materials): verify with the material composition and published data on density
Certificates of calibrators do not meet metrological requirements	— Define criteria in the work instruction — Verify the certificates of the calibrators (e.g. on the web sites of producers)	— Inspect existing certificates
Interference by impurities in self-made calibrators	— Verify the purity of the substance before use	— Check analysis results

3.3. NEUTRON FLUX GRADIENT MONITORS

Neutron flux gradient monitors for thermal, epithermal and, sometimes, fast neutron fluxes are needed in both the relative NAA method and the k_0-NAA method. Flux monitors allow for radial or axial fluence rate gradient corrections, if needed, as well as for specific neutron self-shielding effects, if relevant. Abnormal values for the comparator factor or neutron fluence rate for some flux monitors compared with other flux monitors might indicate the presence in the sample of materials containing large quantities of neutron self-shielded elements such as cadmium or gadolinium.

Quality assurance procedures consist of sandwiching the test portions between portions of the neutron flux monitor to account for gradients in the irradiation position (see NAA e-learning module B8 [19]). Various neutron flux

monitors have been described in the literature but it is beyond the scope of this publication to review them in detail here.

As these neutron flux monitors have to be prepared in the NAA laboratory, errors similar to those made for test portion and calibrator preparation may occur. The associated QA/QC actions apply in a similar fashion.

A selection of errors and proposals for QA/QC actions are given in Table 4.

TABLE 4. POTENTIAL ERRORS IN NEUTRON FLUX MONITOR PREPARATION AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL
(in addition to those in Tables 1 and 2)

Source of error	Action	
	Quality assurance	Quality control
Weighing of flux monitors	— Perform balance calibration — Conduct regular intermediate performance tests	— Create and use control charts of performance tests
Mass (fraction) of comparator/monitor	— Buy from RM producers conforming to ISO 17034	— Create and use control chart of flux at fixed positions and thermal–epithermal ratio
Neutron flux gradient in irradiation container underestimated	— Sandwich sample test portions between neutron fluence rate/calibrator test portions	— Check analysis results of CRMs — Check for abnormal results in the flux results (see Section 6.2)
Neutron spectrum gradients in irradiation container underestimated	— Use monitors sensitive to detecting differences between epithermal and fast neutron fluence rates	— Check analysis results of CRMs for elements with a high resonance integral
Contribution of fast neutrons underestimated	— Select and add an appropriate flux monitor for fast neutrons	— Check analysis results of CRMs for elements with activation products through fast neutron reactions

TABLE 4. POTENTIAL ERRORS IN NEUTRON FLUX MONITOR PREPARATION AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL
(in addition to those in Tables 1 and 2) (cont.)

Source of error	Action	
	Quality assurance	Quality control
Non-repeatable or non-reproducible results for thermal–epithermal flux ratio and alpha parameter	— Select and use an appropriate flux monitor depending on the degree of neutron thermalization in the facility	— Check analysis results of CRMs for elements with a high resonance integral — Create and use control chart for thermal–epithermal flux ratio and alpha parameter — Analyse appropriate (synthetic) multielement materials [27]

4. IRRADIATION PROCEDURE

Differences in neutron exposure between sample and calibrator/flux monitor are the main sources of errors during neutron irradiation. Sandwiching samples between flux monitors provides acceptable QA to compensate for the differences.

The timing of the duration of irradiation may be a critical factor when very short half-life radionuclides are measured, such as ^{20}F (half-life approximately 11 s) or ^{77m}Se (half-life approximately 17.5 s). In contrast to other types of NAA, in such analyses the sample is seldom irradiated with its equivalent element calibrator, since the activity of the calibrator will have decreased by the time it can be measured (i.e. following measurement of the activity of the sample). In such cases, samples and calibrators are irradiated consecutively.

The moment of arrival of the irradiation container near the reactor core, and the moment of its removal, have to be determined with a precision of typically 0.1 s or better. A systematic error owing to imprecise timing may be indicated using a set of RM analyses at increasing irradiation times (e.g. at 0.5, 1 and 2 times the half-life of the radionuclide of interest).

Elements like bromine, arsenic, selenium and mercury are known to volatilize during irradiation. The degree depends on their chemical speciation

and is to some extent unpredictable. In addition, once volatilized, these elements may migrate through the walls of polyethylene containers and be lost from the sample while the activation products contaminate other containers. Encapsulation in sealed quartz ampoules is often the only possible solution. The addition of an ionic liquid, such as thiocyanate on a filter paper within the same encapsulation, has shown to be successful for retaining volatilized mercury [29].

Errors resulting from other effects, such as burnup of the target nuclei, neutron flux depression due to strong absorbers inside the sample resulting in a lower neutron fluence rate outside the sample (at the position of the monitor), and neutron self-shielding can only be addressed if information on the probable contents of the sample to be analysed is available. An IAEA publication [30] provides guidelines for QA in the areas of reactor operations and facilities, preparation for irradiation, irradiation process and performance of analyses.

Errors that may typically occur during irradiation and suggestions for related QA/QC actions are summarized in Table 5.

TABLE 5. POTENTIAL ERRORS DURING THE IRRADIATION PROCEDURE AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Thermal/epithermal/fast neutron flux gradients	— Sandwich samples and flux monitors	— Check results of flux monitors to observe flux gradients
Strong neutron absorbers	— Anticipate during review of request for analysis	— Observe possible neutron flux depression and lower flux in the flux monitors surrounding the sample — Observe possible deformation or melting of plastic capsules due to heating that may occur in the presence of exceptionally high amounts of boron

TABLE 5. POTENTIAL ERRORS DURING THE IRRADIATION PROCEDURE AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Imprecise timing of duration of irradiation	— Mostly for NAA with short half-life radionuclides: Verify imprecision experimentally — Other types of NAA: Use internal QC samples and calibrator/ fluence rate monitors	— Short half-life NAA: Observe systematic differences in the trueness of the results obtained for short half-life radionuclides — Long half-life NAA: Compare results of different radionuclides from the same element
Element losses due to volatilization and migration through plastics (e.g. mercury, bromine)	— Use common sense — Encapsulate in quartz — Include an ionic liquid such as thiocyanate on a filter paper within the same encapsulation	— Use blanks
Neutron self-attenuation (thermal, epithermal), neutron flux depression	— Use common sense — Request review checklist — Use validated software such as MATSSF [31]	— Sandwich samples between flux monitors to provide information if the unperturbed flux gradients are known
Recoil contamination of samples	— Perform advance analysis of encapsulation material	None
Burnup of target isotope	— Use common sense — Set maximum irradiation time	— Analyse results of internal QC samples

5. DECAY

The incorrect synchronization of time measuring instruments is probably the only error that can occur during the decay period (i.e. from the end of irradiation to the start of measurement). This error is most probable for procedures in which radionuclides with very short half-lives are measured. Shifts to and from daylight saving time (or summer time to winter time) may lead to a one hour error in the decay correction if the irradiation and counting occur in different periods. However, this error mostly affects the measurement of radionuclides with half-lives of less than one or two days. Typical errors that may occur during the decay period and suggestions for related QA/QC actions are summarized in Table 6.

TABLE 6. POTENTIAL ERRORS DURING THE DECAY PERIOD AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Incorrect synchronization of clocks at the reactor and in the counting room	— Use radiofrequency controlled clocks — Verify synchronization regularly	— Use analysis results of RMs and correlation of deviations with half-lives of the radionuclides measured
Daylight saving time	— Irradiate and count in the same period — Use common sense	— Use analysis results of RMs and correlation of deviations with half-lives of the radionuclides measured
Wrong correction of decay before and during measurements	— Use validated software	— Use inherent QC of NAA (see Annex II)

6. MEASUREMENT

6.1. GAMMA RAY SPECTRUM MEASUREMENT

Gamma ray spectrometers need to be inspected regularly to ensure adequate measurement performance. Possible fluctuations in electronic noise, changes in gain and direct current level, sample–detector geometry, and detector background are all factors that may affect the shape of measurement peaks. Quality assurance is based on the measurement of the gamma ray spectrum of one or more sources with known emission rates and gamma ray energies distributed regularly over the energy range of interest. The radionuclide ^{152}Eu is often used for this purpose. Peak positions, energy resolution and source activity are ideally recorded in control charts to verify agreement with expected values and to detect trends indicating instability or error [32].

Peak shape analysis is essential for quick identification of the sources of malfunction, and a significant quantity of information can be derived from the presence of a pulser peak in the spectrum [33, 34]. The width and shape of this pulser peak is only affected by electronic noise and not by processes in the detector crystal. Visual inspection of the low and high tails of the peaks is an important QA step as it leads to quicker identification of the problems than, for example, just inspecting the ratio of full width at half maximum/full width at one-tenth maximum (FWHM/FWTM).

The gamma ray spectrometer performance test can be extended by measuring the detector background to determine if the detector or the test portion holder are contaminated and if other sources stored inside the counting room are causing interference.

Regular cleaning of the interior of the high voltage power supply helps to minimize high frequency interference caused by dust accumulation and resulting in peak broadening in the spectrum.

Errors also occur if the counting conditions for sample and calibrator differ in: (i) dead time and pile-up; (ii) physical dimension and effective distance to the detector; and (iii) gamma ray self-attenuation.

Count rate induced effects such as dead time and pile-up can be accommodated through instruments, using either the pulser method [35] if the dead time does not significantly change during counting, or electronic modules such as a dead time stabilizer [36] or a loss free counting unit [37, 38] (see also NAA e-learning module I5 [19]).

Quality assurance procedures include registering the distance between the detector and an unambiguous point (e.g. the bottom of the vial) on the sample test

portion and on the calibrator test portion, respectively, as well as registering the filling heights of both vials.

The composition of the calibrator test portion is known. The gamma ray self-attenuation in the calibrator test portion can therefore be estimated by integrating the attenuation over the source's dimensions. The gamma ray self-attenuation in the sample test portion can be estimated from existing information on its composition and tabulated attenuation coefficients for simulated 'real' materials [39] (see also NAA e-learning module C5 [19]).

Coincidence effects are similar for the sample test portion and the calibrator test portion in the relative method. In the k_0-NAA approach, corrections for coincidence summing effects are needed for both the sample and the calibrator (like the flux monitor), which may be cumbersome and require additional calibration and access to specific software. The problems may be largely circumvented by measurements at distances from the detector at which corrections become insignificant.

Typical errors that may occur during the measurement of the gamma ray spectrum and suggestions for related QA/QC actions are summarized in Tables 7 and 8 (see also NAA e-learning module I6 [19]).

Detailed information on problems with gamma ray spectrometers and their potential causes can also be found in, for example, Ref. [39] and in section I, Instrumentation, of the NAA e-learning course [19]. Examples of actions to avoid difficulties with gamma ray spectrometers include the following:

— Bundle cables, such as preamplifier power, preamplifier output, and high voltage power supply, to minimize radiofrequency pick-ups. Even lead shields (especially if supported by iron) may act as antennas.
— Guide liquid nitrogen exhaust far away from the preamplifier.
— Ensure adequate ventilation through the nuclear instrumentation module crate.
— Do not rely only on liquid nitrogen monitors.
— Do not misinterpret claims that the dewars have a three week holding time; they are likely to be empty after three weeks.

TABLE 7. POTENTIAL ERRORS IN THE MEASUREMENT OF THE GAMMA RAY SPECTRUM AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL: SPECTRUM

Source of error	Action	
	Quality assurance	Quality control
Peak shifts and doublets in the spectrum owing to gain instability	— Use spectrometer performance control and control charts for peak position at low, medium and high energy — Ensure proper management of spectrometers[a]	— Check for the existence of peaks in the spectrum that do not match energy calibration
Increased spectral interferences owing to poor detector resolution	— Use spectrometer performance control and control charts for FWHM at low, medium and high energy. If needed, also for FWTM — Ensure proper management of spectrometers[a]	— Perform a visual check of the results of spectrum fitting
Non-Gaussian shaped peaks	— Use spectrometer performance control and control charts — Ensure proper management of spectrometers[a]	— Perform a visual check of the results of spectrum fitting (peak tailings may result in poor fits or satellite peaks) — Look at residuals after peak fitting
Gamma ray self-attenuation	— Use common sense — Make first order estimates on basis of estimate of major element composition	— Check that the results of RM analysis and inspection if results measured on low energy gamma rays (e.g. <200 keV) concur with results measured for the same radionuclide at higher energy gamma rays — Prepare and use appropriate (synthetic) multielement materials [27]

[a] See also Ref. [39] and section I, Instrumentation, of the NAA e-learning course [19].

TABLE 8. POTENTIAL ERRORS IN THE MEASUREMENT OF THE GAMMA RAY SPECTRUM AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL: DETECTION

Source of error	Action	
	Quality assurance	Quality control
Wrong source–detector distance	— Minimize the possibility of measuring at different distances (e.g. by using fixed measurement positions)	— Check dead time (too high if counting too close) — Check whether there are peak shifts in the measured spectrum or whether peaks are too small owing to high count rates
Wrong efficiency curve selected	— Use automatic read-out of the measuring position	— Use results of RM analysis — Use appropriate (synthetic) multielement materials [27]
Geometry (size) differences between calibration, sample and flux monitor	— Fill height registration and correction — Immobilize test portions inside their encapsulation — Use validated software	— Perform a visual check
Dead time variations during counting	— Zero dead time system/ loss free counting — Set a maximum dead time based on a double source validation technique	— Perform a visual check during measurement
Coincidence summing	— Calculations, measurement at larger source–detector distance — Measure sample and calibrator at same distances and count rates — Use validated software	— Use results of RM analysis and inspection for consistent results on all gamma rays emitted by the same radionuclides, especially for radionuclides decaying by gamma rays in cascade (e.g. Co-60, Se-75)

TABLE 8. POTENTIAL ERRORS IN THE MEASUREMENT OF THE GAMMA RAY SPECTRUM AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL: DETECTION (cont.)

Source of error	Action	
	Quality assurance	Quality control
Contamination of detector end cap	— Cover detector end cap with household wrapping foil — Always keep spacer between source and end cap	— Measure detector background with no sources present — Create and use control chart of background for well chosen regions of interest

6.2. RESULT VALIDATION

One of the advantages of NAA is its self-validating character owing to the use of several dimensions such as intensity and energy and a third dimension that most analytical techniques cannot use, which is time, whereby decay is used as another possibility of inherent QC. Many measurement problems are revealed if multiple measurements are taken at different decay intervals, at different channels, at different counting positions, etc. Further possibilities of inherent QC include using other dimensions such as different radionuclides from the same element or adding redundancies such as different measurement positions, different irradiation channels, different sample geometries, or using a wisely chosen combination of all these dimensions.

Calculated masses (or mass fractions) from each gamma ray peak emitted by radionuclides from the same element will ideally be the same for every measurement. Minor but interference free peaks can therefore be used to an advantage if major peaks overlap with interfering peaks. This approach is based on the assumption that the photopeak efficiency curve is correct, and that gamma ray self-attenuation, coincidence summing corrections, threshold corrections and fission corrections are insignificant or adequately applied.

Annex II presents several examples of the inherent QC of NAA.

6.2.1. Using the dimension of energy

Observation of the different gamma lines of the same radionuclide can be considered as repeat measurements of that radionuclide. Coherence between the results from the different gamma lines confirms the following:

— Coincidence corrections were applied adequately (coincidence corrections are different for each gamma ray);
— Interference corrections were applied adequately;
— The efficiency calibration of the germanium detector was selected and calculated correctly;
— Corrections due to gamma self-absorption at low energies were applied;
— Only correct efficiency curves were selected.

6.2.2. Using the dimension of time (decay)

Different measurements at different decay times can be considered as repeat measurements. Often these measurements are performed after a short decay, after an intermediate period of time, and after a long decay. They are often measured at different positions. Coherence between all these measurements confirms the following:

— Corrections for count rate effects, such as dead time and pulse pile-up, were applied adequately (often the first measurement has a much higher dead time than the last measurement).
— Coincidence corrections were applied adequately (often the first measurement is made at a high position with low coincidence effects, while the last measurement is made at close to end cap positions).
— Spectral interference corrections were applied adequately as most of the radionuclides likely to be interfering have different half-lives.
— Decay corrections were applied adequately.
— Only correct source–detector distances (and associated efficiency curves) were selected.

6.2.3. Using different radionuclides produced from the same element

Upon activation, more than one radionuclide is often produced from one element [40, 41]. Examples of this are in the activation scheme of zinc, selenium, copper, bromine, germanium, rubidium, strontium, zirconium and antimony (for zinc: $^{64}Zn(n,\gamma)^{65}Zn$ and $^{68}Zn(n,\gamma)^{69m}Zn$; for copper: $^{63}Cu(n,\gamma)^{64}Cu$ and $^{65}Cu(n,\gamma)^{66}Cu$; for selenium: $^{74}Se(n,\gamma)^{75}Se$ and $^{76}Se(n,\gamma)^{77m}Se$, etc.). Coherence

of results obtained from different radionuclides of the same element assures the NAA operator that no calculation errors have been made and that all corrections were applied adequately.

6.2.4. Using fission interference corrections

When uranium is present in the sample, several elements that need to be determined (such as lanthanum, molybdenum and zirconium) will suffer from fission interference. As such, NAA operators have defined several fission interference corrections. Once the fission interference correction has been validated, it can also be used for result validation.

6.2.5. Using different irradiation channels and threshold interference corrections

If the irradiation channel has a strong, fast neutron gradient, threshold interference corrections need to be applied. Pre-validation of these corrections is necessary, usually by using specific pure standards. Once the threshold interference corrections have been validated, they can also be used for result validation.

6.3. VALIDITY CHECK AND REPORTING

The analysis results of the CRM(s) and blank capsules need to be compared with reference values such as certified property values or median values of repeat measurements by the laboratory. The laboratory has to decide which statistical approaches, if any, are needed to assess objectively whether the results are within preset specifications. Such statistical approaches include, for example, z-score, zeta (ζ) score, E_n-score, percentage difference or analysis of variance. However, these assessments only indicate the occurrence or absence of potential systematic errors; random errors may still have affected the results of either the control samples or the real samples. Common sense is needed to identify random (or even systematic) deviations in the real sample results that are not reflected by deviations in the control sample results. A practitioner of NAA will always identify mistakes, such as transposing errors or even interchange of samples, if an RM is involved (as the values are known in advance), but this identification is less probable with real samples.

The results and all data subject to transposing errors need to be checked independently, and not just by the person who performed the analysis. This double checking needs also to cover the customer's requirements with respect to

the format of reporting, which may differ from the laboratory's normal routine. As an example, customers may request that mass fractions are reported in $g \cdot kg^{-1}$, whereas a laboratory might routinely report in $mg \cdot kg^{-1}$.

Errors that may occur during gamma ray spectrum interpretation, validity checking and reporting, and suggestions for related QA/QC actions, are summarized in Table 9. NAA e-learning modules in section Q, Quality, provide detailed information on validity checks and reporting [19].

TABLE 9. POTENTIAL ERRORS IN GAMMA RAY SPECTRUM INTERPRETATION, VALIDITY CHECKING AND REPORTING AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Calculation errors	— Use validated calculation sheets or commercial software codes (validated in international literature)	— Use the results of CRM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Peaks not assigned to radionuclides	— Use spectrometer performance control to avoid drifts — Verify decay time independently	— Use the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Intensity ratios between peaks do not match theoretical values	— Prevent counting close to end cap (and coincidence effects) — Include verification of efficiency curve in method validation	— Use the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Interfering nuclear reactions	— Use common sense — Calibrate	— Use the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)

TABLE 9. POTENTIAL ERRORS IN GAMMA RAY SPECTRUM INTERPRETATION, VALIDITY CHECKING AND REPORTING AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Typos and transposing errors; interchange of samples and customer codes	— Verify all manually entered data independently	— Laboratory management performs final verification and authorization — Use inherent QC of NAA (see Annex II)
Contamination	— Use a blank	— Create a control chart of the blank measurements
Neutron flux gradient correction	— Prescribe sandwiching of samples and flux monitors	None
Trueness unclear	— Use CRM with each batch, even if the batch consists of one sample	— Use the results of CRM analysis and proficiency testing
Unsatisfactory results	— Perform non-conformance reporting and root cause analysis	— Participate in proficiency testing
Reporting format differs from customer request	— Ensure effective internal communication	— Evaluate customer satisfaction
Reporting beyond deadline	— Incorporate a safety margin in planning	— Conduct internal audits — Establish a key performance indicator on reporting time
Reporting results even though results of control materials are not satisfactory	— Laboratory management performs final verification and authorization	— Conduct internal audits

7. REVIEW OF REQUESTS FOR ANALYSIS

7.1. FEASIBILITY OF ANALYSIS

The International Standard ISO/IEC 17025 [26][8] requires in Clause 7.1, Review of Requests, Tenders and Contracts, that customer requirements need to be defined, documented and understood; that the laboratory has the capability and resources to meet those requirements; and that the appropriate methods or procedures are selected and are also capable of meeting those requirements.

An NAA laboratory needs to be familiar with the customer's requirements for the measurement of the elements, the reporting deadline, and the many compositional aspects of the sample to be analysed. This knowledge minimizes the risk that the laboratory will face analytical difficulties, report wrong results, or even fail entirely in meeting the customer's requirements. It is therefore embedded in the QA practices of the laboratory.

An example of a checklist of issues that can be used to assess the feasibility of analysis is provided in Table 10. It should be noted that not all questions are always answered by the customer. In addition, other material characteristics and management issues are evaluated in the feasibility assessment; these are also listed in Table 10. Such a checklist may also be used, and even extended, to determine participation in proficiency testing exercises.

TABLE 10. EXAMPLE OF A CHECKLIST FOR THE ASSESSMENT OF THE FEASIBILITY OF AN ANALYSIS REQUEST FOR NEUTRON ACTIVATION ANALYSIS

Questions to customer	Issues to consider
Specific elements or panoramic analysis	
Indication of the expected mass fractions	
Any indication of interfering elements (which interfering elements varies on a case-by-case basis)	

[8] ISO/IEC 17025:2017 specifies the general requirements for the competence, impartiality and consistent operation of laboratories. It is the basic standard with which NAA laboratories need to comply if the laboratory wants to confirm or recognize, through peers or accreditation bodies, its technical and organizational competence.

TABLE 10. EXAMPLE OF A CHECKLIST FOR THE ASSESSMENT OF THE FEASIBILITY OF AN ANALYSIS REQUEST FOR NEUTRON ACTIVATION ANALYSIS (cont.)

Questions to customer	Issues to consider
Requested degree of trueness and uncertainty Requested limit of detection	— Performance indicators for the methods used at the laboratory (e.g. specificity and selectivity, trueness, precision, robustness, uncertainty, traceability) — Contamination risks during sample preparation and irradiation
Date of reporting	— Availability of reactor — Availability of human resources for all parts of the procedure — Availability of equipment within the time window needed for performing the analysis
Available amount of sample	— Preferably at least twice the amount the laboratory normally uses for analysis
Any indication of the degree of homogeneity	
Possible moisture fraction	
Presence of components (e.g. proteins) that may decompose during irradiation, causing pressure buildup Presence of significant amounts of neutron absorbing elements (e.g. boron, lithium, samarium, gadolinium, iridium) Presence of significant amounts of gamma ray absorbing elements (lead, thallium, bismuth) Presence of uranium (fission)	— If the customer is not aware of such components and/or elements, and the presence of significant amounts cannot be excluded, the laboratory may have to perform a series of tests (such as gamma ray transmission), or test irradiations with increasing amounts of material; to consider analysis by an alternative method (such as X ray fluorescence analysis); or to consider rejecting the request for analysis
Aspects of safety in material handling (e.g. carcinogenic, contagious), safeguards	
Special reporting format requirements	
Accept price and delivery conditions (if any)	

7.2. SELECTION OF AN ANALYTICAL PROTOCOL

An analytical protocol is a mix of sample mass, irradiation, decay and counting conditions. Optimization in NAA is the selection of an analytical protocol that provides the highest signal to background (or to spectral interference) ratio within the constraints of customer requests, including total analysis time and costs. Many adjustable variables can be used: irradiation, decay and counting time; detector type (e.g. low energy detector, well type detector, regular coaxial detector); source–detector distance; and different gamma ray lines from the same radionuclide, or from the different radionuclides produced from the same element, if applicable.

The feasibility assessment forms the basis for the selection of the analytical protocol. Consulting scientific literature for examples of analytical protocols is one option. Another option is using the experience gained by the laboratory from previous analyses and the relevant documentation. Approaches using advanced computer prediction programs have been published, by which the shape of the gamma ray spectrum is simulated for a given protocol [42–44]. This allows the laboratory to assess the possibility of detecting a peak from the radionuclide of interest despite the presence of peaks and background radiation from other radionuclides.

A typical error that may occur in the selection of the analytical protocol and suggestions for related QA/QC actions is provided in Table 11.

TABLE 11. POTENTIAL ERRORS IN THE ANALYTICAL PROTOCOL AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Wrong combination of sample size, irradiation, decay, measurement times and counting geometry	— Maintain a database of past measurements — Study literature — Test irradiations — Use advanced computer prediction programs	None

8. LABORATORY MANAGEMENT

8.1. MANAGEMENT ISSUES

Documentation and records of experimental parameters and observations are a cross-cutting aspect of management in an NAA laboratory. The records need to contain all applicable information that will facilitate, where possible, the identification of factors affecting uncertainty, and enable the test or calibration to be repeated under conditions as similar as possible to the original [26]. Records can be kept as logbooks, forms, checklists and computer files, as well as audio, photographic and video formats. Good record-keeping, with a view to repeating a test under conditions as similar as possible to the original, is key for a technique like NAA in which all test portions remain intact (non-destructive analysis), allowing experiments to be repeated using the same test material.

Many QA procedures include go/no-go decisions when parameters are compared with predefined, unambiguous specifications (criteria). If the specification is not met, the laboratory needs to investigate the root cause of the non-conformity and its possible effect on other parts of the NAA procedure. Once the results of the investigation are known, remedial action needs to be taken to correct the non-conformity and, if a recurrence is likely, corrective action is needed in the form of a change to the procedure or the management system. Management of non-conformities results in a better understanding of the technique and of the possible sources of error.

Associated QC on the effectiveness of management issues is typically achieved through random or systematic inspections, which may be organized as internal audits. Typical major shortcomings that may occur, and suggestions for related QA/QC actions, are summarized in Table 12.

8.2. TECHNICAL COMPETENCE

Successful QA in NAA requires educated practitioners trained in both common chemical analysis, such as sample size reduction, (freeze) drying, moisture fraction measurement, weighting, dilution, pipetting and blank control, and the relevant parts of the theory and practice of NAA, such as radiation detection and gamma ray spectrometry. Technical competence also includes the ability to monitor and identify deviations from normal experimental conditions and in measurement results, and to contribute to the evaluation of the root cause of such problems.

TABLE 12. POTENTIAL ERRORS IN THE MANAGEMENT OF THE NEUTRON ACTIVATION ANALYSIS LABORATORY AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Details of how past measurements were performed are not known	— Register and record all conditions/parameters	— Conduct internal audits
Confusion on how work has to be done and what has to be registered	— Refer to flow charts, forms and training material	— Conduct internal audits
Recurring errors and mistakes	— Register and manage non-conformity — Maintain and check an experience database	— Conduct internal audits
Random decisions at go/no-go points	— Define objective assessment criteria	— Conduct internal audits

The IAEA e-learning course on NAA can contribute to the continuous education of NAA practitioners [19, 45]. Practical skills may be further developed by hands-on training. The common goal is for NAA practitioners to acquire adequate competence for the assigned tasks.

9. PROFICIENCY TESTING

Quality assurance for participation in interlaboratory comparisons for proficiency testing starts with a systematic evaluation of the information provided by the proficiency testing provider, similar to the feasibility assessment for NAA that is described in Section 7.1. In addition to all relevant information on material, measurands, reporting deadline, etc., the laboratory may also wish to know the expected number of participants and types of techniques employed, the costs of participation and an indication of the turnaround time for reporting by the proficiency testing provider. Although ISO/IEC 17025 does not formally oblige the use of accredited proficiency testing providers, it does state that proficiency

testing providers meeting the requirements of ISO/IEC 17043 can be considered to be competent [46].[9]

Eventually, the laboratory has to evaluate the provider's report from the exercise and verify that the objectives for participation have been fulfilled. To this end, the laboratory may consider conducting a separate evaluation against its own specifications for the acceptance of results (see also NAA e-learning module Q5 [19]). Errors that may occur during participation in proficiency testing exercises (not in the results thereof), and related QA/QC actions, are summarized in Table 13.

TABLE 13. POTENTIAL ERRORS DURING PARTICIPATION IN PROFICIENCY TESTING EXERCISES IN THE NEUTRON ACTIVATION ANALYSIS LABORATORY AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Non-conformity owing to wrong report format	— Conduct independent verification before submitting results	— Laboratory management performs final verification
Results not accepted because report received after deadline	— Incorporate a safety margin into planning	— Conduct internal audits
No conclusions possible after receipt of report from the proficiency testing provider because of too few results and/or absence of information on techniques used	— Prepare work instructions for selection of proficiency testing schemes	None
Acceptance of provider's report plus follow-up on non-conformity without any self-assessment by the laboratory	— Prepare work instruction for self-assessment (e.g. based on ζ score instead of z-score)	— Conduct internal audits

[9] ISO/IEC 17043:2010 specifies general requirements for the competence of providers of proficiency testing schemes and for the development and operation of such schemes. ISO/IEC 17025:2017 requires suitable proficiency testing services.

TABLE 13. POTENTIAL ERRORS DURING PARTICIPATION IN PROFICIENCY TESTING EXERCISES IN THE NEUTRON ACTIVATION ANALYSIS LABORATORY AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Only NAA laboratory; all others use 'destructive' techniques	— Prepare work instructions for selection of proficiency testing schemes	None

10. CONCLUSIONS

It needs to be emphasized that QA/QC is only effective if the approach chosen by the laboratory reflects actual needs. Determining what is needed may be based on an evaluation of the risk of a potential error and the balancing of this against the efforts and costs of QA/QC. Various approaches for risk assessment and evaluation are described in ISO 31000 [47].

All QA/QC actions involve the management of the NAA laboratory. In the past, this concept was referred to as 'quality system', but this term has been replaced by 'management system'. Guidance on how to implement such a system can be found in Ref. [48]. Although the underlying international standard (ISO/IEC 17025) has been revised [26], the concepts underpinning the requirements and the approaches to fulfil them remain largely unchanged.

Other nuclear analytical techniques, such as particle induced X ray emission, and analytical techniques based on mass spectrometry, such as inductively coupled plasma mass spectrometry, are comparable to NAA in terms of sensitivity and ability to measure a wide range of elements. However, the techniques are not the same as the elements measured and the sensitivity for each one is different. Mass spectrometry is destructive, requiring dissolution of the portion of the sample that is analysed. Nevertheless, to ensure the long term sustainability of NAA as a competitive chemical analytical technique, its technical development needs to be accompanied by the enhancement of the quality of NAA analytical services provided by laboratories.

The guidance provided in this publication can be used by NAA laboratories at research reactors, as well as accelerator based and isotopic neutron sources to establish or reinforce their QA/QC practices. It is left to the discretion of the laboratories to effectively apply such practices in their day to day work and to demonstrate a sustained improvement in their analytical performance.

Appendix

IAEA E-LEARNING COURSE ON NEUTRON ACTIVATION ANALYSIS

The IAEA e-learning course on NAA [19, 45] has been available on-line on the IAEA's Cyber Learning Platform for Network Education and Training (CLP4NET)[10] since October 2017. The first major review was completed in May 2019 and a second major review was completed in August 2021. The course is a tool for human capacity building in NAA, as well as a method to contribute to the overall sustainability of the technique. It covers all aspects of NAA and is directed at young specialists or beginners who lack experience in conducting NAA independently. Experienced practitioners who wish to refresh or develop their knowledge in specific areas can also benefit from it. Of particular relevance to this publication is the thematic area on quality. Modules in other thematic areas, such as calibration, instrumentation and NAA practice, are also relevant to QA/QC and are cited in this publication.

The IAEA's NAA e-learning web site [19] includes an 'NAA Community' page intended to serve as a forum for the global NAA community for discussion and the sharing of experience. The syllabus of the course is presented in Fig. 1. A quick reference guide to the modules most relevant to this publication is provided in Table 14.

[10] The IAEA's Cyber Learning Platform for Network Education and Training (CLP4NET) can be found at https://www.iaea.org/resources/databases/cyber-learning-platform-for-network-education-and-training-clp4net

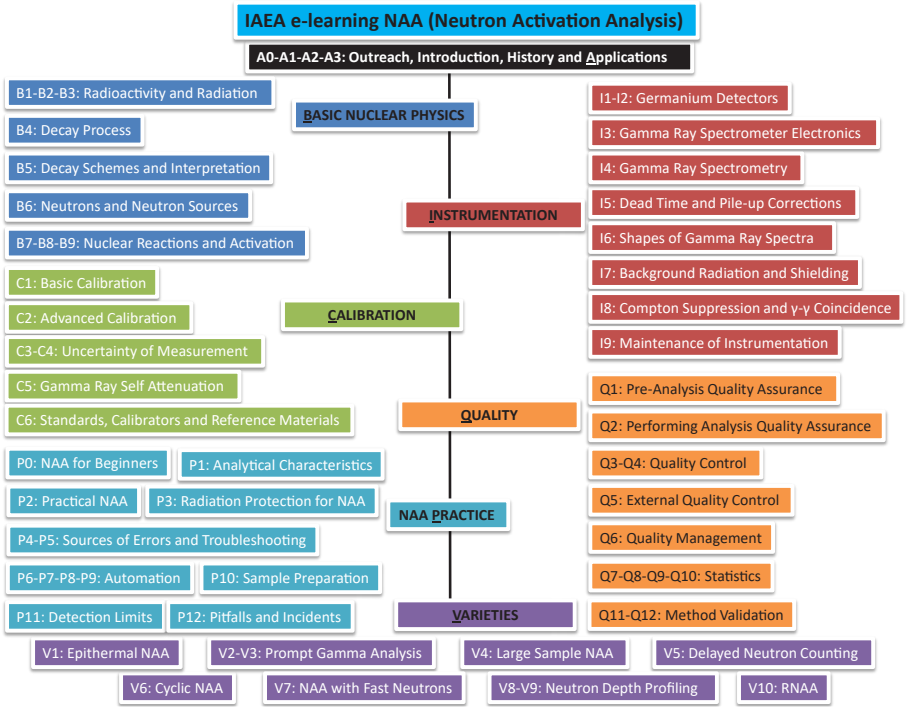

FIG. 1. Syllabus of the IAEA e-learning course on NAA.

TABLE 14. QUICK GUIDE TO THE MODULES OF THE IAEA E-LEARNING COURSE ON NEUTRON ACTIVATION ANALYSIS MOST RELEVANT TO QUALITY ASSURANCE AND QUALITY CONTROL

Module	Issues covered
B8: Nuclear Reactions and Activation	Neutron flux gradients, self-shielding
I1–I2: Germanium Detectors	Selection of the appropriate germanium detector for a given application
I3: Gamma Ray Spectrometer Electronics	Preventive maintenance of electronics
I4: Gamma Ray Spectrometry	Efficiency, coincidence effects
I5: Dead Time and Pile-up Corrections	Hardware and software based corrections
I6: Shapes of Gamma Ray Spectra	Differentiation between photo peaks and other peaks in the gamma ray spectrum
C1: Basic Calibration	Absolute method and comparator method, CRMs
C2: Advanced Calibration	Single comparator method, k_0-NAA, modelling and internal mono-standard method
C5: Gamma Ray Self-Attenuation	Basic principles and experimental determination
C6: Standards, Calibrators and Reference Materials	Use of standards and reference materials for calibration, method validation and quality control
Q1: Pre-Analysis Quality Assurance	Basic concepts of QA/QC, sample preparation
Q2: Performing Analysis Quality Assurance	Implementation of QA procedures for irradiation and measurement
Q3–Q4: Quality Control	Instrument performance tests, handling non-conformity, control charts, internal QC, report checking
Q5: External Quality Control	Proficiency testing and laboratory intercomparison

TABLE 14. QUICK GUIDE TO THE MODULES OF THE IAEA E-LEARNING COURSE ON NEUTRON ACTIVATION ANALYSIS MOST RELEVANT TO QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Module	Issues covered
Q6: Quality Management	ISO/IEC 17025:2017
Q11–Q12: Method Validation	Basic approach, method validation in practice
P2: Practical NAA	Sample receipt and documentation, sample and standards preparation, irradiation, unloading and counting procedures
P4–P5: Sources of Errors and Troubleshooting	Sources of problems and examples of solutions, guidance for troubleshooting
P10: Sample Preparation	Contamination and element losses, preparation of test portions, representativeness and homogenization
P12: Pitfalls and Incidents	Pitfalls in sample preparation, irradiation, counting and spectrum analysis, and others

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ANNEXES: SUPPLEMENTARY FILES

Annexes I–III are available as on-line supplementary files and can be found on the individual web page of this publication at www.iaea.org/publications.

Annex I

BASIC PRINCIPLES OF CONTROL CHARTS

Control charts are one of the key tools in QC and are to be routinely used in the NAA laboratory. This annex introduces the basic concepts for the use of control charts in QC.

Annex II

INHERENT QUALITY CONTROL OF NEUTRON ACTIVATION ANALYSIS — EXAMPLES

One of the advantages of NAA is its self-validating character owing to the use of several dimensions such as intensity and energy and a third dimension that most analytical techniques cannot use, which is time, whereby decay is used as another possibility of inherent QC. Section 6.2 has further information on this subject. This annex presents examples of the inherent QC of NAA, illustrating the concepts in Section 6.2.

Annex III

SUMMARY OF POTENTIAL MAJOR ERRORS

This annex contains duplicates of the tables included in this publication, but in a user-friendly format. They are intended to be used in the day to day practice of NAA at research reactors.

ABBREVIATIONS

CLP4NET	Cyber Learning Platform for Network Education and Training
CRM	certified reference material
FWHM	full width at half maximum
FWTM	full width at one-tenth maximum
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
k_0-NAA	neutron activation analysis with the k_0 method
NAA	neutron activation analysis
QA	quality assurance
QC	quality control
RM	reference material
SI	International System of Units

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Neutron activation analysis (NAA) is the most common technique practised in research reactors worldwide. To enhance the quality of NAA services in the laboratory, the IAEA promotes and conducts annual interlaboratory comparisons for proficiency testing. It also develops specialized publications and capacity building instruments such as the IAEA e-learning course on NAA.

Referring to the most up to date international standards and practices, this publication provides guidance on QA/QC approaches to be used in the day to day practice of NAA at research reactors. Potential sources of error and associated QA/QC actions are provided for all main areas of NAA practice, including as easy to use tables intended for direct reference in the laboratory.

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