

# IAEA TECDOC SERIES

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## **Development of an Integrated Approach to Routine Automation of Neutron Activation Analysis**

*Results of a Coordinated Research Project*



**IAEA**

International Atomic Energy Agency

DEVELOPMENT OF AN INTEGRATED  
APPROACH TO ROUTINE AUTOMATION  
OF NEUTRON ACTIVATION ANALYSIS

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# DEVELOPMENT OF AN INTEGRATED APPROACH TO ROUTINE AUTOMATION OF NEUTRON ACTIVATION ANALYSIS

RESULTS OF A COORDINATED RESEARCH PROJECT

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For further information on this publication, please contact:

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

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## FOREWORD

Despite a decrease in the number of research reactors around the world, many existing facilities are not used to their full potential. There is a need to develop strategies for using such facilities more effectively on a national, regional and international basis. This is particularly important in the case of small and medium sized research reactors, which can be used for educational and training activities but present few opportunities for neutron beam research or radioisotope production. One option could be increasing the number of neutron activation analysis (NAA) activities undertaken.

Through its technical cooperation programme and coordinated research projects (CRPs), the IAEA has encouraged NAA groups throughout the world to focus on fields of application where there are large numbers of samples for analysis. In this respect, the lack of automation limits the analytical capacity of many NAA laboratories, which cannot analyse the required number of samples within the required turnaround time. There is considerable scope for automation throughout the NAA process — from receiving the samples to issuing the analysis report — in areas such as sample changers, irradiation facilities, data management, data analysis, and quality assurance and quality control.

Automation of NAA is challenging and requires combinations of components that improve not only the efficiency of analysis but also, by reducing human intervention, its reliability. Although a few laboratories have successfully implemented highly automated systems, many still do not have sufficient in-house experience to know the best practices to follow.

In response to requests from the NAA community, in 2012 the IAEA implemented a CRP on the development of an integrated approach to routine automation of NAA. Eighteen Member States participated in the project, which ran from 2012 to 2015 and involved both laboratories with proven facilities as well as newcomers. Participants built automated facilities in research reactors for irradiation as well as gamma ray spectrometry, and developed approaches for automating and integrating data streams in NAA processes, significantly increasing capacity and minimizing human error.

This publication presents the results of the CRP concerning automation of NAA and will serve as a reference for practitioners, experts and research reactor personnel in the field. It will also be of interest to various stakeholders and users interested in the basic research and services that can be provided with NAA. The international network established during the CRP will contribute to the automation of NAA not only in the CRP partner laboratories but also in other laboratories around the world. The individual country reports submitted by the Member States are available on the CD-ROM accompanying this publication.

The IAEA expresses its appreciation to all the participants in the CRP for their efforts leading to this publication, and to the international experts who contributed to its drafting and review. The IAEA officers responsible for this publication were D. Ridikas and N. Pessoa Barradas of the Division of Physical and Chemical Sciences.

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# 1. INTRODUCTION

## 1.1. BACKGROUND

Neutron Activation Analysis (NAA) has been confirmed to be one of the most suited opportunities for the commercialization of research reactor (RR) services [1, 2]. Whereas the markets for NAA laboratories may have been identified, and quality may have been established, an underestimated problem remains the absence of automation, which limits tremendously the analytical capacity. Most NAA laboratories have only one or two detectors, and commercial sample changers are considered too expensive. The capacity is also limited by the time-consuming data handling due to the lack of associated automation. Analysts often have to transfer the output of the analyser — a list of gamma-ray energies and peak areas, sometimes topped with element assignment — through various file transformation programs: firstly to the spectrum interpretation software for qualitative analysis, next to a program for quantitative mass fraction calculations, and finally to the reporting software. Lack of automation limits the ability of NAA laboratories to service the needs of specific fields, such as archaeology and epidemiology where analysis is required of a large number of samples. Whereas NAA is the often preferred technique for such studies, there have been cases in which requests had to be rejected because of limited capacity in automation and data processing.

NAA is a multi-element technique, in which often two to three measurements are done and all the intermediate results have to be considered for the final output. Most of the commercially available software has been made for gamma-ray spectrometry and not for NAA. In this linear chain of processes, the administration of samples and customers is often a missing link, and it may be present only in a spreadsheet. This is illustrated in Figure 1, showing the various sources of data to be combined in an NAA procedure in comparison to those that can be covered by commercially available software. The information from the other sources has to be either entered/combined by hand, or using self-made programs.

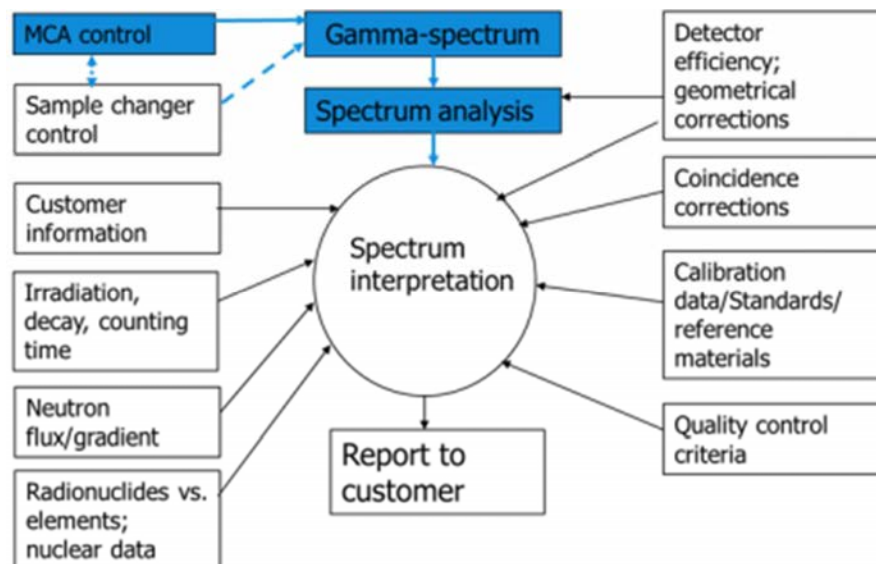


FIG. 1. Types of data that have to be combined in an NAA procedure (MCA= Multichannel pulse-height analyser). Commercially available packages are available for the data handling in the coloured boxes; dotted lines indicate potential for communication (courtesy of Mr. P.Bode).

Automation in NAA requires replacement of the majority time-consuming human manipulations in the analytical procedure by machines and software. Examples are balances that can be read-out remotely to automated encapsulation tools, automated systems for successive or cyclic irradiation of samples and, of course, sample changers at gamma-ray spectrometers. Software is also needed for managing and bridging all relevant data flows as shown in Figure 1. Such an automation of NAA introduces irradiations and measurements around the clock, i.e. also outside office hours, thus increasing the analytical capacity.

Only a few sample changers are commercially available. Not to mention their high costs, implementation thereof is difficult or impossible due to differences in vial sizes used in irradiation and required by the sample changer. In addition, there is no common interface that allows them to be integrated with local systems available in specific labs. Many laboratories suffer from a lack of in-house technical and engineering support for constructing their own sample changer and realizing integrated data management.

Automation of NAA therefore requires a significant effort for the development and implementation of software, hardware and data management. This often represents an insurmountable barrier for individual NAA laboratories that are typically staffed by only a few people, hindering the efficient use of the facility.

There is an additional aspect of automation in NAA that is often overlooked. Once the increased experimental capacity has led to an increase in requests for samples to be analysed, the number of gamma-ray spectra also increases; and with it the potential for conflicting issues in the interpretation of the results. More quality control materials will have to be analysed and equally so a larger number of unacceptable deviations from the expected results may occur. Automation demands that gamma-ray spectrometers work reliably, and there will be stress in the facility if performance tests indicate problems. This stress is related to the turnaround time of the analysis — the time between the acceptance of the request and reporting — which should be kept the same even after the throughput increases significantly. Consequently, the NAA facility may face a situation in which the number of spectra to be analysed and interpreted within the same timespan may double or even triple; and with it, the spare time for resolving problems may reduce substantially. An effective and efficient troubleshooting mechanism is therefore also essential for automated NAA facilities, and should be based on a thorough understanding of the physical and analytical concepts of NAA. That is to say, quality assurance and quality control (QA/QC) must be implemented taking into account the demands of an automated system.

## 1.2. OBJECTIVE

A Consultants' meeting (December 2009) on "Preparation of Guidelines on Implementation of Routine Automation in Advanced Neutron Activation Analysis Laboratories" recommended that a Co-ordinated Research Project (CRP) could be developed to address this pressing need for automation within the NAA community. As a result, the IAEA implemented the CRP 1888 / F1.20.25 on "Development of an Integrated Approach to Routine Automation of Neutron Activation Analysis" (2012–2015).

The overall objective of this CRP was to contribute to enhanced and sustainable RR utilization by increasing NAA capacity through automation, resulting in more opportunities to engage in scientific research and commercial services. The specific objectives of the CRP were:

- To identify centres of expertise that can be called upon by laboratories wishing to improve automation within their laboratories;
- To provide detailed examples of designs and components suitable for the automation of hardware;
- To develop standardized communication protocols (software) and make them available as open source codes;
- To develop detailed guidelines for the integration of hardware, software, data management and related Quality Assurance and Quality Control (QA/QC) in automated NAA facilities;
- To increase the level of automation in participating laboratories, according to the guidelines produced in the CRP;
- To facilitate the establishment of long-term cooperation (network) between participating laboratories on automation in NAA.

### 1.3. SCOPE

#### 1.3.1. General

The participants in the CRP focused on three specific areas allowing to increase the analytical capacity of NAA laboratories, namely development and implementation of (a) automation of irradiation facilities, (b) sample changers with gamma-ray spectrometers and (c) integration of sample changers and gamma-ray spectrometers; and on implementing the hitherto missing tools for automated data processing and analysis reporting. All these activities included QA/QC procedures as a cross-cutting component. By bringing together a range of laboratories with different skills, experience and needs, the CRP was successful in enabling an effective network to perform the significant amount of research and development required.

Automation of irradiation facilities is especially relevant if short half-life radionuclides are of interest in NAA. Automation typically consists of combining a capsule loader to a pneumatic transfer system and a receiving end of this system near a radiation detector to measure the activity in the capsules induced by irradiation. Several participants were successful in realizing such automation. It should be noted that not all existing facilities can be modified towards an automated one (mainly due to specific facility design constraints).

Automated irradiation facilities are a common approach in delayed neutron counting [3, 4], which is in fact a form of neutron activation analysis on basis of short half-life radionuclides (fission products of  $^{235}\text{U}$  such as  $^{87,88,89,90}\text{Br}$  ( $t_{1/2} = 55.7 \text{ s}, 16.3 \text{ s}, 4.4 \text{ s}, 1.9 \text{ s}$ ; or  $^{137,138,139}\text{I}$  ( $t_{1/2} = 24 \text{ s}, 6 \text{ s}, 2.3 \text{ s}$ ) emitting neutrons rather than gamma-rays.

Existing or new facilities can be equipped with diverters that allow sending the activated capsule either directly to an appropriate radiation detector (this could be a gamma-ray spectrometer as well as a delayed neutron counter), to a decay position or a waste storage. One of the difficulties is that the irradiation capsule is also activated and, in addition, may contain radioactive contamination on its outside. This is of no relevance for delayed neutron counting, but it interferes with the measurement of the activity of the test portion inside this capsule. Converting an existing system for delayed neutron counting into a dual facility for NAA on basis of short half-life gamma-ray emitters may therefore have limitations making it impractical for the desired type of NAA as the sample vial may have to be removed manually from the irradiation capsule.

No systems have been reported in this CRP for automatic separation of irradiation capsule and sample vial after irradiation. Activity of the capsule by contamination inside metal (aluminium) transfer tubes can be prevented by use of, for instance, carbon-composite tube material instead. Other important aspects of automated irradiation systems are the timing of the arrival of the irradiation capsule in the irradiation position, and safety aspects to facilitate the unattended operation of the system.

Two basic approaches have been used in the design of sample changers (sample transport to the detector): (i) the use of robotic arms, or (ii) a pneumatic system. Samples may be made available in a tray or in a magazine stack. In some cases the tray may move in the X-Y plane to reduce the degrees of freedom needed in the robotic arm, in others the tray is fixed and the arm has three degrees of freedom. Where robotic arms are used, the transport system may be based on a linear stepping motor, where pulses are counted, or an optical sensor (proportional encoder). There are also multiple methods of positioning the sample in front of the high purity germanium detector, including placing it on a plate, holding it in the robotic arm or delivering it to a holder which can be independently and automatically positioned. The latter two methods allow for the position to be optimized on the basis of detector dead-time. In one case, a sample changer can deliver to two detectors.

With respect to the control system, participants have considered three components: the part that determines the work programme (which samples, to be measured when and for how long, etc.); the part that controls the spectrometer; and the part that controls the sample changer. It was concluded at the start of the CRP that it is not feasible to design a single software package for NAA that will address all users' needs. It was also recognized that most existing software packages for data acquisition and/or analysis contain modules that would be useful to many users. Such modules from various origins should then be able to communicate through well-defined file formats that are available to all via open source software. Users with home-made sample changers or multi-channel analysers will thus be able to use existing modules where possible, and create their own ones where necessary. Exchange of modules within the NAA community is expected, greatly facilitating automation of existing as well as new laboratories for NAA.

A certain fraction of the results will be outside the specifications set by the laboratory itself and/or its customer. Part thereof can be corrected for by remediation actions; in addition, corrective actions may be needed in the analytical procedure and/or management of the facilities. In all cases, the laboratory will have to assess the root cause of the problem. The effectiveness thereof depends strongly on the metrological expertise of the staff involved. It has been observed already that there are gaps in this expertise, especially in facilities where their most experienced staff have retired, and in facilities in which there is insufficient or no back-up by nuclear physicists.

NAA has a long history spanning over 80 years, well documented on its metrology and with ample documented examples in scientific literature of potential sources of errors. However, the relevant information is scattered over many papers published over several decades, and it is difficult to retrieve. Moreover, most such publications have not been made for educational purposes. As such, the participants agreed to compile the metrology, the theoretical and practical aspects of NAA — including sources of error and how to anticipate on them — into an e-learning tool to support the trouble-shooting in NAA by providing guidance to the fundamentals aspects of NAA. This e-learning tool is now on-line available on the IAEA Cyber Learning Platform for Network Education and Training (CLP4NET) [5]. It addresses most analytical process steps in NAA by a modular design using PowerPoint presentations,

supporting video clips, suggestions for self-practising, assignments for self-testing the knowledge acquired and links to relevant literature for in-depth study. The e-learning tool is expected to become a 'living document', to be continuously updated and expanded. More details are given in the Appendix.

### **1.3.2. Outcome of the CRP**

Increase of the analytical capacity such as the number of samples to be irradiated per unit of time, or number of samples to be counted per unit of time, was the prime objective of this CRP. Upon completion of the CRP, participants reflected on the impact of their accomplishments to their own laboratories by comparing the relevant status before the CRP started and once it was completed. The survey included resulted in the following feedback:

- Laboratories that worked on automated irradiation facilities reported increases of their irradiation capacity ranging from 1.5 to 4 times their capacity before the CRP;
- Laboratories that worked on automatic sample changers reported increases of their sample counting capacity ranging from 1.5 to 3 times their capacity before the CRP;
- Laboratories reported increased number of samples analysed through automation is data management and quality assurance ranging from 1.6 to 3 times to their capacity before the CRP;
- Laboratories indicate that the shared information on automation in NAA, as well as the networking during this CRP allows them to continue further in automation after completion of the CRP;
- The networking during the CRP provided the laboratories more insight in software development resulting in time savings varying from 1 day/week to 2–4 months/year, depending of course on the existing local availability of data management software;
- The newly developed E-learning modules will contribute to the continuous capacity building in the NAA laboratories;
- Overall, the relevance of the CRP and the information exchanged was considered most valuable for (in order of significance) the development of sample changers, e-learning, NAA practice (including QA) and integrated data processing, management, and for automation of irradiation facilities.

Participants also provided feedback on how the information and material, developed and disseminated during the project, are sufficient for a specific step in automation to be made without hesitation already, or if that step needs some or substantially more work to be done. From this feedback it was concluded that:

- The information on irradiation facilities, sample changers and QA in NAA will enable the laboratories to realize automation thereof (if relevant) and implementation without much additional effort;
- Implementation of E-learning can be brought to practice without hesitation in most laboratories, whereas some hesitation is noticed in others, mostly because of language proficiency issues (the E-learning tool is in the English language);
- More work will be needed on development and implementation of the software (modules) for automation and integrated data management in NAA.

Finally, participants indicated that they will continue their efforts on automation in all aspects of NAA facilities and analytical procedures, including the use of the new IAEA E-learning tool [5].

#### 1.4. STRUCTURE

The present report consists of this introduction, two technical sections describing first the concepts and then the benefits of automation in NAA, an appendix on the E-learning tool for Neutron Activation Analysis, list of references, and list of individual paper contributors together with their affiliations and individual paper titles.

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

- Annex I: List of participants in the CRP;
- Annex II: Individual paper contributors.

The individual reports are available on the CD-ROM attached to this publication.

## **2. BENEFITS OF AUTOMATION**

NAA, prompt gamma analysis and charged particle or photon induced activation analysis are multi-elemental nuclear analytical techniques that are unique in that there are specific parameters that can be altered to significantly enhance detection limits and reduce uncertainty. While tedious sample digestion is not needed, there are many steps required in NAA to achieve reliable results in a timely fashion. Automation is any process that reduces the need for operator intervention.

An ultimate concept would be to remove the need for any human intervention, with *machine* talking to *machine* controlled by software. As many independent modules should be created as possible, to give the analyst the greatest flexibility in its use in a given situation. Automation in NAA can enhance the quality of results by reducing human error, increase the revenue-generating capabilities of a laboratory, raise the socio-economic benefits and increase reactor utilization. All of these areas can intensify the visibility and impact of the facility. New markets may be opened by increasing capacity or particular limiting steps may be overcome.

Automation is of most benefit where large numbers of similar samples need to be analysed or may also be advantageous where there is a high demand to run small batches of samples. For example, throughput is significantly increased if jobs can be run around the clock, rather than being limited to running during working hours.

### **2.1. APPLICATIONS OF AUTOMATION IN NAA**

#### **2.1.1. Sample preparation**

Perhaps the first place where automation can take place is in the preparation of samples, which always requires weighing of samples, standard reference materials and calibration standards in the milligram to gram range. Automated balances can now automatically transfer the weight to a spreadsheet, which minimizes human error in transmitting visual results of weight to a spreadsheet or paper copy.

#### **2.1.2. Irradiation Facilities**

One critical step in the automation of NAA is the precise accounting of irradiation times for short-lived isotope production. This is particularly true for durations in the order of seconds. For instance, a one second uncertainty in a 10-second irradiation leads to an automatic 10% error in the final result. While this error decreases with longer irradiation times, often this is not possible due to the activity of the returning sample. An automated electronic system can accurately determine the irradiation time within 0.1 seconds. The top module displays the irradiation time while the bottom modules display the decay times. In such a system the worker can irradiate up to two samples, with one sample being irradiated while the other one is being counted. This type of procedure can greatly increase the number of samples processed in one day.

#### **2.1.3. Irradiation Automation for Prompt Gamma Analysis**

An automated sampler changer can also be incorporated in a prompt gamma facility. The key benefits include:



- Reduction of the time required to change samples from several minutes to fewer than 10 sec;
- Creation of a system that would be fully automated requiring no user interaction, i.e. a system that can communicate with the existing detector software;
- Creation of a system that would have a capacity of up to eight samples as shown in Figure 2.

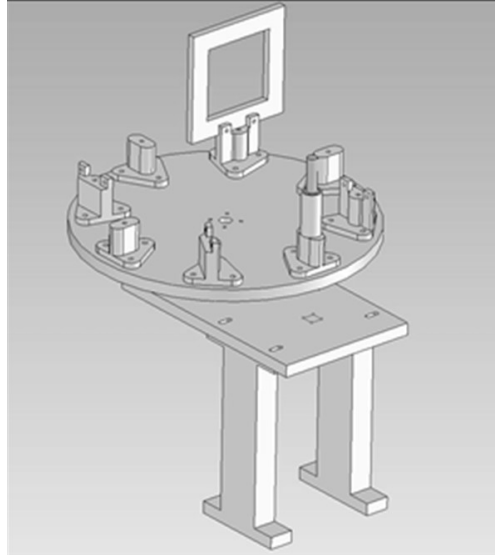


FIG. 2. Automated irradiation facility for prompt gamma analysis (courtesy of University of Texas, USA).

#### 2.1.4. Cyclic Activation Analysis

The employment of cyclic activation analysis [6] is rather limited in the number of elements that can be determined as compared to routine NAA. However, cyclic NAA has proven to be useful in several analytical situations particularly for fluorine, oxygen, selenium, silver using both thermal and epithermal neutrons. A comprehensive irradiation facility that is capable of handling up to thirty samples and the computer controlled commands are depicted in Figure 3. The cyclic system can also be used in “single mode”, when only one irradiation is needed for routine NAA. A tremendous benefit is that the operator/worker does not need to handle the sample, thus greatly reducing the dose exposure to the hands and body.

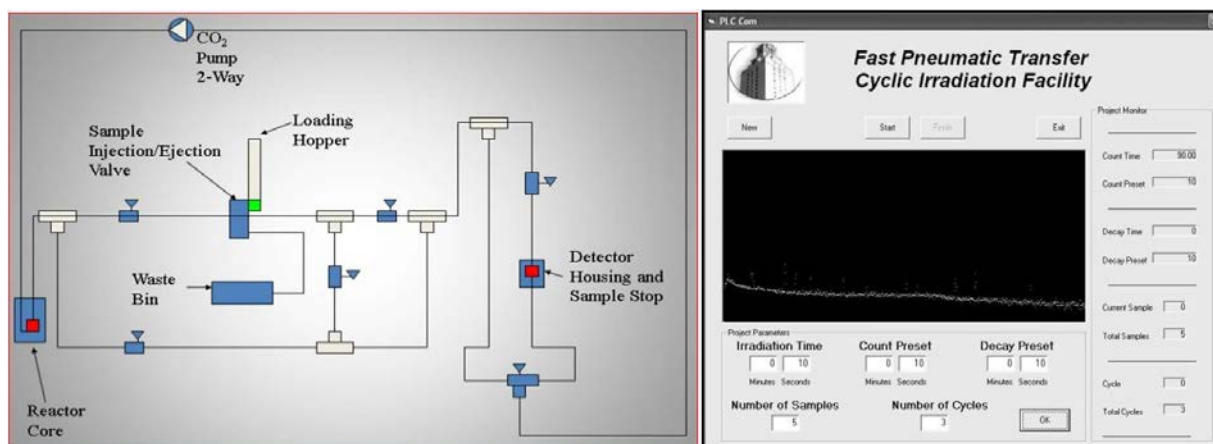


FIG. 3. Overview of cyclic activation system (left) and irradiation, decay and counting control system (courtesy of University of Texas, USA).

### 2.1.5 Sample Changers

While sample changers have a long history of use in NAA not all the laboratories have functional systems. Currently a few companies such as CANBERRA [7], ORTEC [8] and Changer Labs [9] manufacture and sell sample changers for use with gamma-ray spectrometry and NAA, respectively. All other sample changers are developed and built “in house” requiring the needs of electronic and mechanical technicians. However, once a sample changer is commissioned it can work around the clock not requiring a worker to put on and take off a sample from the detector manually. This greatly reduces human error since the sample changer interacts with the multichannel system output to automatically store the spectrum with a specific identification, remove the sample from the detector and process the counting of the next one. Sample changers have the benefit relieving staff employees from tedious manual sample changing as they can position the sample very reproducibly, and allowing them to perform other duties.

Sampler changers now also exist for complex structures such as Compton suppression systems, as shown in Figure 4.



*FIG. 4. Compton suppressor gamma system with automatic sample changer (courtesy of Comisión Chilena de Energía Nuclear, Chile).*

### 2.1.6. Data Analysis, Quality Control and Quality Assurance

Automation of data analysis can take on various forms besides the classic peak fitting routines. Storage of all sample weights, irradiation, delay and counting parameters and geometry can be automatically logged into spreadsheets that can be accessed within a laboratory or remotely. Algorithms can be set up to monitor results for standard reference materials thus alerting the worker that a problem may exist in some aspect of the data analysis or signalling that a sample which may have an unusually high dead time thus compromising the final result. Quality assurance can also be embedded in algorithms that may include counting a sample more than once (such as for long-lived NAA). Automation can also include report writing with final concentrations and uncertainties.

## 2.2. SUMMARY

Automation, in all its hardware and software forms, remains the key ingredient to safeguard that NAA remains competitive to produce high quality results in a time manner. There is no single uniform way to implement all aspects of automation in the individual worldwide NAA laboratories. However, a culture to advance the efficiency of NAA through improvements in automation is needed to ensure NAA continues its importance in the analytical field.

### 3. CONCEPTS IN AUTOMATION

The concepts in automation of NAA are further described in this Chapter together with some examples of the CRP participants' accomplishments in the various categories, automation in irradiation, in gamma-ray spectrometry, automation in auxiliary laboratory instruments, integration of data management and automation of data processing, with quality assurance/quality control as cross-cutting concepts. The individual contributions in Annex II and the country reports on the CD-ROM contain more detail on these subjects.

#### 3.1. AUTOMATIC IRRADIATION FACILITIES

There is no generally applicable set of detailed instructions on how to design an automatic irradiation facility. However, the particular reactor design, local safety regulations and the NAA laboratory requirements are the key factors in outlining the main features of the system. Modification of existing facilities is often difficult, and/or not allowed or will not result into the desired specifications, for instance due to long transfer time, contamination of the irradiation container, inability to integrate with the gamma-ray spectrometer. Stemming from those constraints and limiting factors, the intended purpose of the facility should delineate the final details in designing a custom-built facility.

An automated irradiation system allows for performing (successive) irradiations without human intervention. The automation consists of (at least) an automatic loading device for the irradiation capsules, a controller for the valves that provide the pressurized gas resulting in the propulsion of the irradiation capsule, monitors to detect the arrival of the capsule in the reactor and in the return position, timers and a storage system of the irradiated capsules. Pneumatic transfer systems are relatively easy to automate for this purpose. They are valuable if:

- The irradiation capsule of the pneumatic transfer system can hold only a few sample containers; automation thus eases the handling of large batches;
- Irradiations have to be done outside office hours and support from reactor operators is not possible;
- Short half-life radionuclides (half lives in the order of less than one minute) have to be measured by cyclic procedures; this requires the arrival of the irradiated capsule at a counting position near a radiation detector such as a gamma-ray spectrometer or delayed neutron counter;
- Short half-life radionuclides (half lives up to several minutes) have to be measured in procedures with overlap of measurement time and irradiation time of successive samples;
- Large batches of samples have to be manually processed after irradiation, and reduction of the radiation exposure of the workers is desired.

Pneumatic transfer systems are commonly 'designed-in' with the research reactor and, with exception for the most recently built reactors, not automated upon commission of the reactor. Sometimes the unattended irradiation of capsules is not allowed for safety considerations; this depends on the local safety rules, design and outcome of a risk assessment. Other pneumatic transfer systems are difficult to modify towards automation, for instance, because the capsule loader cannot easily be integrated, or because it is not possible to connect the transfer system to the desired counting position.

A tailored new automatic transfer system may be realized in some reactors; both as pool-type or inside a (radial, tangential) beam tube.

### 3.1.1. The tubing system

Pneumatic transfer systems at research reactors are commonly made with aluminium tubing, for radiation resistance, ease of construction, economics and decommissioning considerations. Corrosion of the tube's inner surface results in the scraping off of dust during the transfer of the irradiation capsule and contamination thereof, mostly by  $^{28}\text{Al}$ ,  $^{65}\text{Zn}$ ,  $^{60}\text{Co}$  and other activation products from impurities in the aluminium alloy used. The induced activity from this contamination may be so high that the facility is not suitable for direct connection with the gamma-ray spectrometer; capsules have to be opened immediately after irradiation, thus reducing the attractiveness of the automate system unless it is also achieved by automation. In addition, corrosion products may also originate from the piping connecting the gas supply and valves to the transfer system.

It is difficult or practically impossible to improve existing pneumatic transfers for such contamination problems. As such, existing systems may only be suitable for full automation for delayed neutron counting but not for gamma-ray spectrometry immediately following the irradiation (and thus not for cyclic NAA). Eventually, automatic separation of sample container and irradiation capsule could be considered [10] but such systems do not allow for cyclic activation (see paragraph 2.1.4).

A new pneumatic transfer system may be a better alternative; one could select materials for the tubing other than aluminium, such as plastic (polyethylene, polypropylene, polyvinyl chloride, etc.), for the part not exposed to neutrons and reactor gamma-rays, and an inert material such as quartz or carbon-carbon composite (inserted inside an Al-tube as the composite is not water-tight) for the irradiation end of the system [11–13]. Such a system can be realized as a pool-side facility or built inside a reactor beam-tube.

### 3.1.2. Irradiation capsules

The irradiation capsules may form another issue with existing facilities. Typically, these are made of polyethylene which may contain impurities such as Al, Cr, Mn that all result in induced activity, a potential interference to the measurement of gamma-ray emission by the activated sample. In addition,  $^{41}\text{Ar}$  is produced in the air inside the capsule, resulting in an increase of the background during counting. Of course, both interferences are not relevant if delayed neutron counting is done.

Polyimide and carbon are alternatives for polyethylene, both with at least a similar radiation resistance. However, in both cases these types of capsules may not be commercially available at the desired sizes; used thereof thus depend on the availability of technical support exists to manufacture them.

Automatic separation of irradiation capsule and sample container upon arrival near the counting position have been reported in literature [10] but, except for the complexity but as mentioned in the above, such systems do not allow for cyclic activation. In addition, some technology is needed to remove both the irradiation capsule and the sample container from the system once the radiation measurement has been completed.

### 3.1.3. Propulsion

Propulsion of the capsules can be done by (1) overpressure or by (2) underpressure (vacuum); again, one will encounter a given system with existing facilities. However, it should not be too

complicate to modify the propulsion art and to change from underpressure towards overpressure. For measurement of very short half-life radionuclides (less than one minute) overpressure propulsion systems are preferred, as the pressure differences up to 10 bars result in a very high transfer speed of the capsules, reaching easily less than one second over distances up to 25 m.

Compressed air is used in some facilities but this is highly undesirable for systems that are integrated with a gamma-ray spectrometry system due to the  $^{41}\text{Ar}$  production. More favourable gases are  $\text{N}_2$ ,  $\text{CO}_2$  [14] or even He [15]; all of those using gas cylinders. A combination of vacuum and  $\text{CO}_2$  overpressure can also be applied for propelling capsules. In general,  $\text{CO}_2$  is often less preferred because of the production of  $^{14}\text{C}$ ; He is an expensive propellant.

#### **3.1.4. Timing**

The shorter the half-life of the radionuclide of interest, the more important is the precise timing of the arrival of the irradiation capsule in the irradiation position. For instance, a one second uncertainty in a 10-second irradiation leads to an automatic 10% error in the final result. Most pneumatic transfer systems are equipped with sensors as close as possible to the active zone to ‘observe’ the passing of the capsule in the transfer tube shortly before it ends in the active zone.

However, for practical reasons, these sensors are sometimes positioned on top of the reactor pool, i.e. approximately 6–8 m from the irradiation end. As these sensors also cause a start of the irradiation timer, their imprecision may cause an error of several hundreds of milliseconds in the real exposure to neutrons. Such errors become relevant for NAA on basis of radionuclides with half-lives of several seconds; for NAA using radionuclides with longer half-lives, the error in the irradiation may be not significant. Still, it is important to have good insight in the error cause by the too early start of the irradiation time, triggered by the sensor observing the irradiation capsule passing. For achieving reliable timing information, optical or acoustic sensors may be preferred to relying on repeatability of actuators.

#### **3.1.5. Sample loader**

There are different designs of capsule loaders for pneumatic systems. Examples are

- The stack loader capsules are loaded in a stack one above the other and subsequently fed into an irradiation system by means of gravity. The most common design is a simple vertical stack, but for space limitations, sometimes Z-shaped stack is applied. Such a loader may have a capacity up to 100 capsules [16]; the actual number depends of course on the application;
- The revolver drum or rotating wheel design where, by rotating the device, the capsules are positioned in front of the transfer tube. As the drum or wheel has fixed positions, the device may be combined with a system to read-out the position in the wheel from which the capsule was fired. This also contributes to the quality assurance.

Eventually (see the examples in paragraph 2.1.3), the same drum/wheel may be used for receiving the activated capsules, either for intermediate decay or for final storage upon completion of the analysis.

At large, it is highly recommended to have also a system that stores the activated capsules in the same order as they have been irradiated. This can be accomplished by either a Z-type storage stack, or using a similar (or the same) drum or wheel as used for loading the capsules.

### 3.1.6. Integration with radiation detector

Automated pneumatic transfer systems must have a receiving end near the radiation detector, either the gamma-ray spectrometer (especially if cyclic activation analysis is pursued) or the delayed neutron counter. Depending on the type of detector, the last part of the (existing) transfer tube may have to be modified in its orientation, towards a vertical positioning for delayed neutron counting and horizontal dipstick Ge detectors, and towards a horizontal positioning in vertical dipstick Ge detectors are used. The distance between receiving end and Ge detector may have to be adjustable depending on the activity to be measured. As such, it may be considered to have the last part of the transfer tube made of flexible hose.

Several difficulties may be encountered if an automated pneumatic transfer system is used for NAA using gamma-ray spectrometry. The problem of the interfering activity from impurities in the irradiation capsule and contamination on its outside were already mentioned. In addition, the air in the transfer tube will most likely contain  $^{41}\text{Ar}$ . In order to reduce its contribution to the radiation background, a short flushing with clean air or nitrogen gas of the tube part near the detector could be considered.

### 3.1.7. Diverters

Diverters are used to route samples between the irradiation facility and the various counting stations in the system. e.g. from the reactor to either the gamma-ray detector, or the delayed neutron counter, or the intermediate storage for decay and a successive measurement; and for sending the capsule, upon completion of the measurement, either to the final storage or to an intermediate storage pending a second measurement [17,18].

It can be considered, in some cases, that the diverters provide the essential link between automated irradiation facilities and automated counting devices. The diverter has a special importance in pneumatic cyclic neutron activation analysis (CNAA) for measurement of very short lived radionuclides for accurate detection of the irradiation, decay and counting times.

Such diverters are typically equipped with of pneumatic or electrical device positioning part of the transfer tube to a further connecting tube towards the desired destination (see Figure 5).

### 3.1.8. Controller

The controller is the heart of an automatic irradiation facility. It is used for setting and controlling the transfer, duration of the irradiation, duration of intermediate storage (if any) and duration of the measurement. It communicates with the capsule loading device and the radiation detection systems, and interacts with the overall data management in NAA [7].

There are several approaches in use, based on: (1) personal computers with additional input-output boards, (2) programmable logic controllers (PLC) or (3) programmable automation controllers (PAC). These are more powerful and flexible to communicate with other systems. Each approach has its strengths and weaknesses; some are more robust, the others are easier to program, another category might be easier to expand and so on.

Once the hardware components have been selected and the system is constructed, the software part will determine how well and how easily the facility will operate. It is important that the designer decided about the adjustable and fixed parameters before the software development phase; and the manual operation is always possible, e.g. in case of sample jamming or other



*FIG. 5. Example of a diverter in the fast pneumatic system at Reactor Institute Delft. The diverter is pneumatically operated. Optical sensors can be seen on the left and right of the diverter (courtesy of TU Delft, Netherlands).*

system failure. It is also advantageous to provide possibility for further future expansion of the system either by some additional sensors or other sub-systems. In general, the controller also measures time, as it sends orders to actuators and receives signals from the sensors.

Ideally, the controller should have a means to automatically recognize the code of the capsules. However, this is difficult to accomplish; bar-code labels, as in use in sample changers, cannot easily be applied as they may be not resistant against the reactor radiation and/or may contain unwanted impurities. The CRP participant from China reported the use of a radio frequency identification tag which for identifying and/or tracing the sample route or location.

The controller should have a slave module in the reactor control room so that the operator can provide clearance on the use of the system, can always interact and, if needed, can intervene with the operation of the system.

### **3.1.9. Safety related aspects**

Automated irradiation systems, especially those operating without direct attendance, must be equipped with a device that returns a capsule from the irradiation position immediately in case of a power failure in the controller, or in case of loss of propellant or, in the worst case, both. This may be accomplished by having a buffer tank in the system for the propellant that is not used during normal operations, but can be applied, for instance, using both valves that are closed with power applied and open without power; as well as with manually operated valves as back-up.

### **3.1.10. Examples of automatic irradiation facilities**

Examples of automated irradiation facilities existing at, and/or developed by the participants in the CRP are shortly described in this paragraph. More details can be found in the references and the individual country reports on the CD-ROM belonging to this document.



### 3.1.10.1. Beam-tube automatic pneumatic irradiation system Reactor Institute, Delft University of Technology, Netherlands

Two fast pneumatic transfer systems (PTS) are operational using N<sub>2</sub> overpressure as means of capsule transport. The oldest (pool-side) automated system is a straightforward blow-in/blow out system (transfer time 1 s) that is automated for cyclic activation, but suffers from contamination of the transfer capsules by the abrasion inside the aluminium transfer tube. The sample containers have to be removed from the transfer capsule before counting; shortest decay time is therefore ca. 15 s. Another system is fully automated (Table 1) and equipped with a sample changer. This system is located in a radial beam tube, has plastic tubing outside the irradiation zone and a carbon-carbon composite irradiation end. Contamination of the transfer capsule is practically absent [11, 12]. The sample can rotate around its vertical axis during counting. Figure 6 shows the scheme of the facility.

### 3.1.10.2. Automated existing pneumatic irradiation transfer system at Jožef Stefan Institute, Slovenia

The TRIGA Mark II facility comprises one fast overpressure PTS and one standard underpressure PTS; both of them are automated (See Tables 2 and 3). The Standard PTS at JSI TRIGA Mark II reactor is presented in Figure 7.

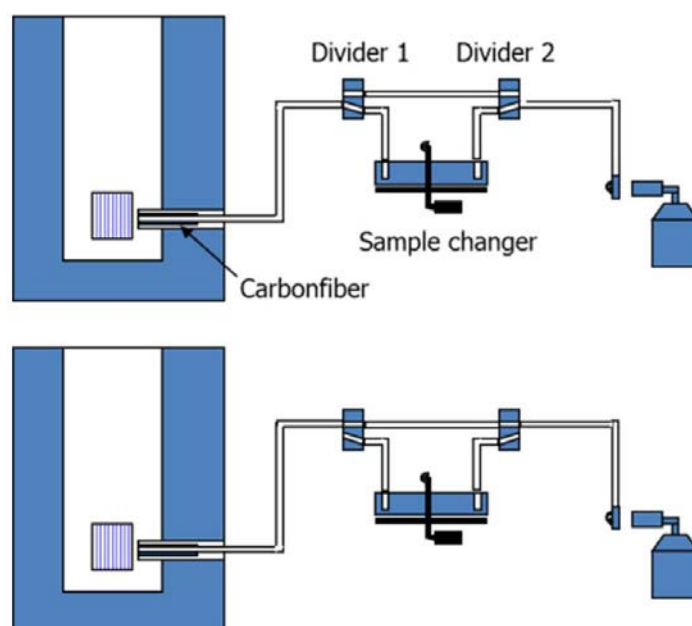


FIG. 6. Delft Carbon fiber autonomous facility for irradiation and analysis (CAFIA) facility with sample changer (courtesy of Delft University of Technology, Netherlands).

TABLE 1. AUTOMATED PTS AT THE DELFT CAFIA FACILITY

Neutron flux (total)	$4 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
Tubing material	Polyethylene / carbon-carbon composite
Propellant gas	N <sub>2</sub> , 3 bar overpressure
Transfer time	2 s
Loader capacity	64
Controller	PLC + PC

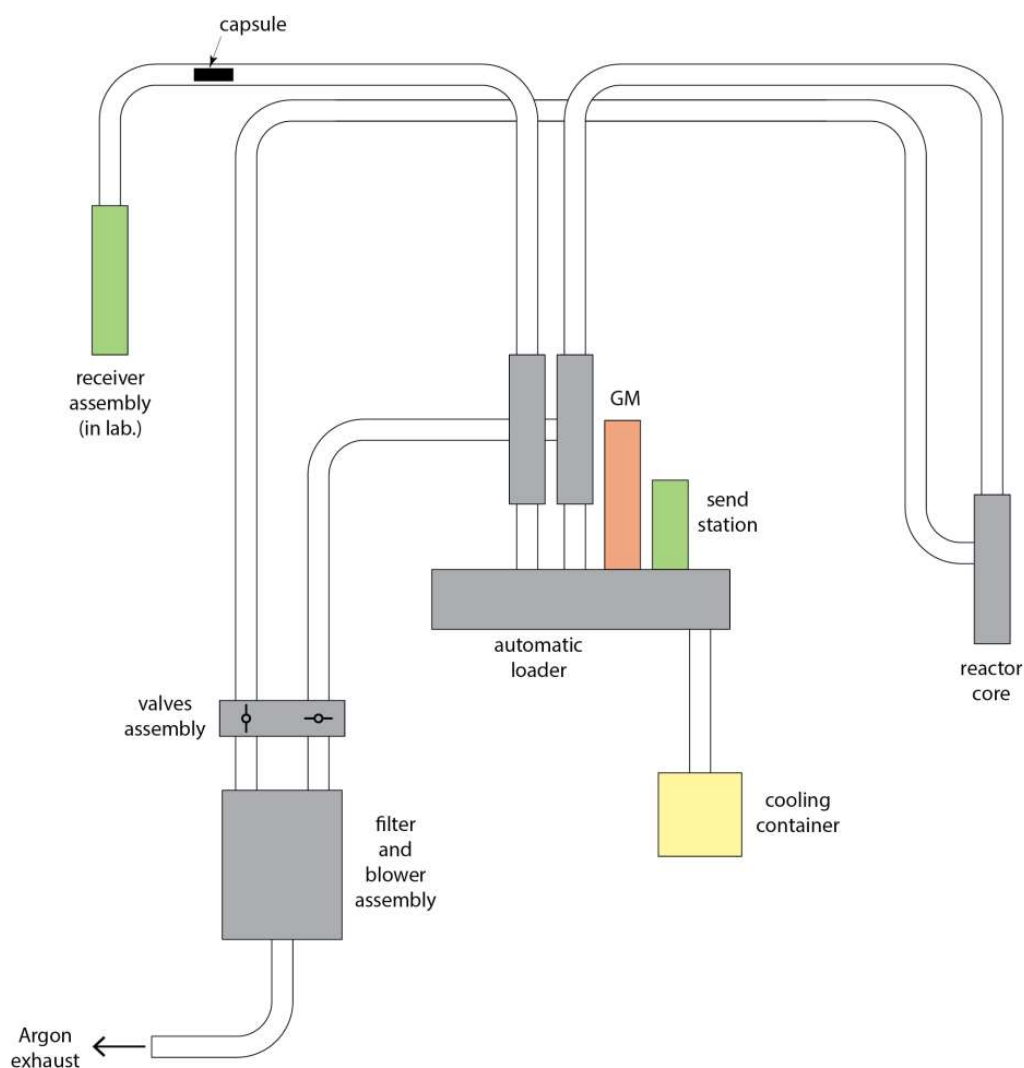


FIG. 7. Standard PTS at JSI TRIGA Mark II reactor (courtesy of Jožef Stefan Institute, Slovenia).

The system was upgraded to increase the level of automation in the laboratory. The new control system records the required and actual irradiation times as well as all actions, including the reactor power during irradiation and will, at a later stage, transfer data to a central database. The loading station comprises an automatic capsule loader with capacity of 20 capsules, with the possibility of further extension. The system allows for changing the settings and changing the irradiation order of the capsules waiting for irradiation, according to the user authorization. The PT system has sensors for monitoring correct transport of capsules from the reactor building to the laboratory. The whole process is controlled by a Programmable Automatic Controller (PAC).

TABLE 2. FAST PTS at the JSI TRIGA Mark II reactor

Neutron flux (thermal)	$3.3 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
Tubing material	Polyethylene / aluminium
Propellant gas	Air, 5 bar overpressure
Transfer time	<1 s
Loader capacity	100, stack
Controller	PAC + PC

TABLE 3. STANDARD PTS AT THE JSI TRIGA MARK II REACTOR

Neutron flux (thermal)	$3.3 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
Tubing material	Aluminium
Propellant gas	Air, underpressure
Transfer time	3 s
Loader capacity	20, revolver drum
Controller	PAC + PC

*3.1.10.3. Automated existing pneumatic irradiation transfer system at Instituto Peruano de Energía Nuclear, Peru.*

Another sample changer with a capacity of 20 capsules was integrated with the pneumatic transfer system (see Figure 8). The system has two receiving ends which allows irradiation and temporary shielded storage of samples on weekends when the reactor operates, meaning that the process of analysis can continue even in the absence of the NAA staff.

*3.1.10.4. Newly designed automatic pneumatic irradiation system with multiple receiving ends, China Institute of Atomic Energy (CIAE), P.R. of China*

A new instrumental neutron activation analysis automatic measurement system has been established at the China Advanced Research Reactor (CARR) consisting of full integration of the pneumatic transfer system, a delayed neutron counter and several gamma-ray spectrometers. The system even consists of two transfer systems, one larger diameter system for long irradiations of multiple samples and one for short irradiations, one sample at-a-time; both systems are built into one reactor irradiation end.

The irradiation and counting capsule contains an RFID (radio frequency identification) tag which is used for identifying and/or tracing the sample route or location. The pneumatic transfer system is operated with filtered fresh air with a maximum pressure of 0.4 MPa. The system contains a sample holder block with a capacity of 20 counting capsules in each of ten sets, and a sample changer, which was designed with manual and automatic sample loading inlets and four sample transfer outlets.

Three detectors can be run simultaneously and continuously for 24 hours; sample-detector distances can be automatically adjusted. The RFID tag enables real-time tracking of the samples and ensures high consistency between a spectrum and the counted sample.

### 3.2. SAMPLE CHANGERS FOR GAMMA-RAY SPECTROMETRY

While sample changers have a long history of use in NAA, not all the laboratories have yet functional systems. A few sample changers that can serve gamma-ray spectrometers are commercially available; one is designed for moving the types of samples typically encountered in NAA specifically [3], whereas others are commonly used for moving relatively large samples such as Marinelli beakers [2]. Given the relatively high price of commercial sample changers (which is in the order of the price of a large volume Ge-detector), difficulties to handle the types of sample containers used in the NAA laboratory as well as difficulties in communication with other software in use, most NAA laboratories develop and realize their own system.

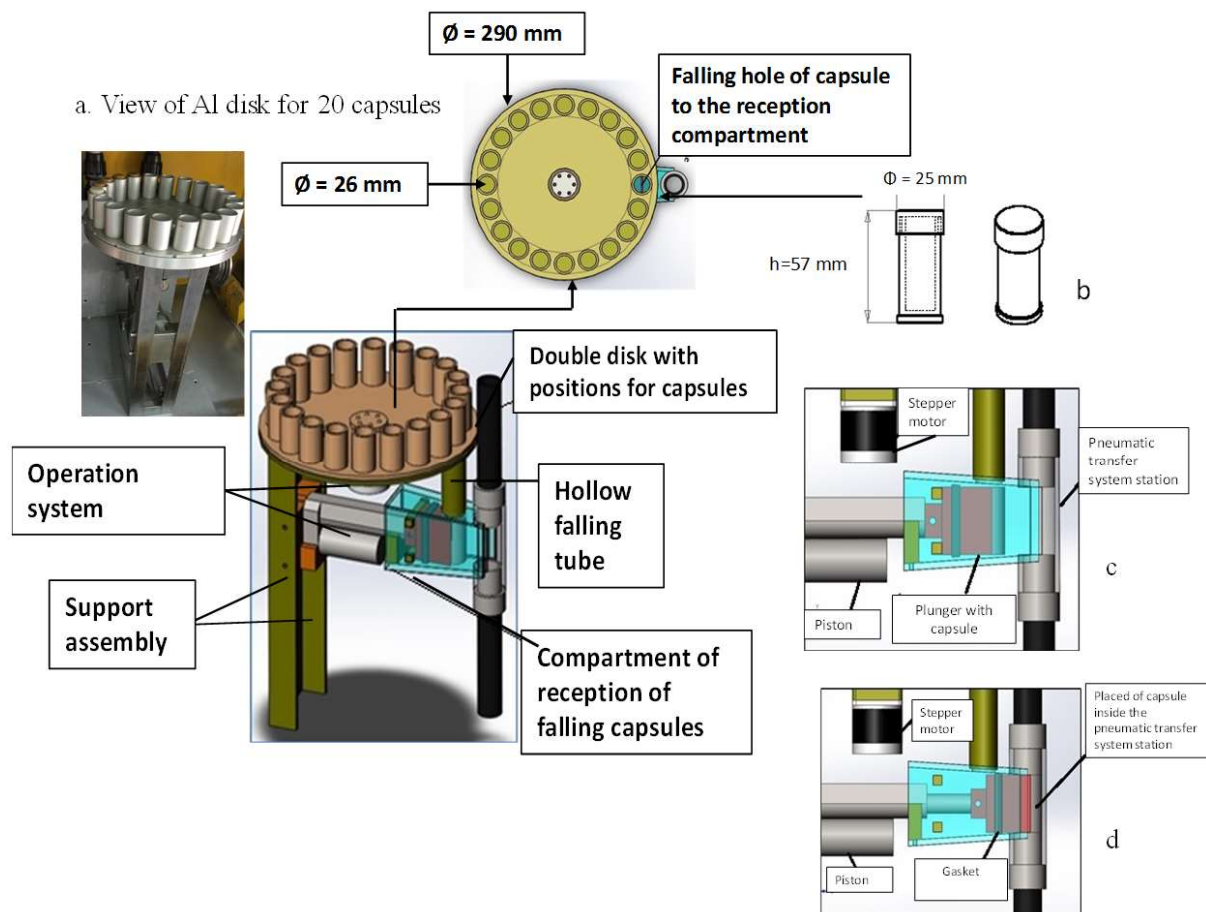


FIG. 8. Details of the sample changer in the existing pneumatic irradiation transfer system at IPEN, Peru (courtesy of Instituto Peruano de Energía Nuclear, Peru).

Sample changers developed and built “in house” require support by electronic and mechanical technicians. However, once a sample changer is commissioned, it can work around the clock not requiring a worker to put on and take off a sample manually. This greatly reduces human error since the sample changer interacts with the multichannel system output to automatically store the spectrum with a specific identification, remove the sample from the detector and process the counting of the next one. Sample changers have the benefit relieving staff employees from tedious manual sample changing and allowing them to perform other duties.

### 3.2.1. Sample Changer Design

#### 3.2.1.1. General design aspects

The design of a sample changer is set by a few constraints:

- The type of detector to be serve: horizontal or vertical dipstick Ge detector, well-type detector or Ge detector with Compton suppression shield;
- The distance between sample container and detector end-cap: adjustable or fixed;
- The distance between the sample storage and the detector, and estimate of shielding required;
- The maximum number of samples to be stored in the changer;
- The type and size of the sample container;
- The communication with the gamma-ray spectrometer.

The transfer of sample containers between a storage position (such as a stack loader or a storage tray) can be done using a pneumatic transfer system, equipped with a robotic system in which the necessary movement is simplified by a device that can grab a container and moves it via X, Y and Z directions from a tray to a detector and back, or otherwise.

### 3.2.1.2. *Pneumatic sample changers*

Sample changers for gamma-ray spectrometry can be based on the identical concept as a pneumatic transfer system used for irradiations. The advantage of such sample changers is that the storage rack can be positioned at a large distance from, or even in a different room than the detector, which partly reduces the need for shielding between storage and detector. Another advantage is that the system allows for positioning the counting end of the transfer tube at different distances from the detector, irrespective if this is a vertical or horizontal dipstick detector. Such a system is operational with the CRP participant at CCHEN in Chile (see Figure 9). Compact sample changers with pneumatic transfer system have been developed at BAEC in Bangladesh (see Figure 10) and at the Malaysia Nuclear Agency (see Figure 11).

A disadvantage of pneumatic sample changers could be that the sample containers may have to be re-packed (for safety considerations) in transfer capsules; and that this type is difficult to realize for well-type detectors.



FIG. 9. Counting room at CCHEN, Chile, showing (left) shielded Ge detector and part of the tubing of the pneumatic transfer system of the sample changer and (right) stack loader of the sample changer (courtesy of Comisión Chilena de Energía Nuclear, Chile).

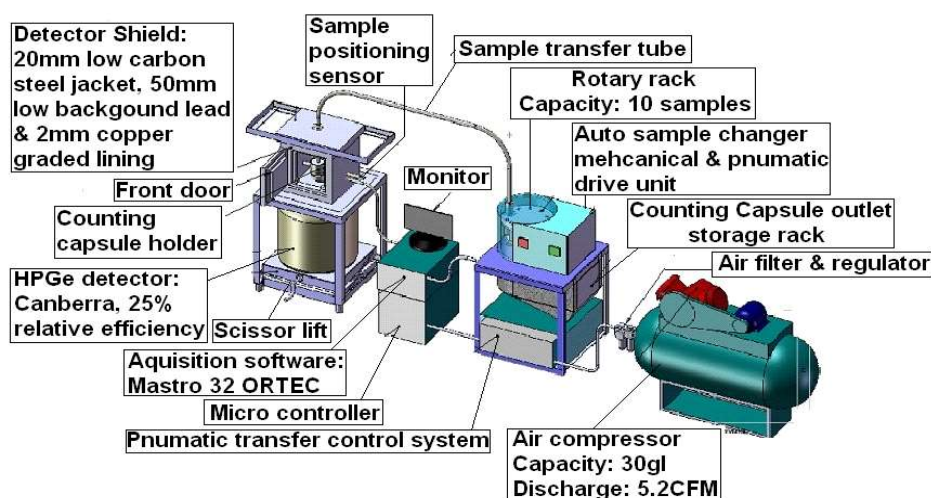


FIG. 10. Pneumatic sample changer, developed at Bangladesh Atomic Energy Commission in Bangladesh (courtesy of Institute of Nuclear Science & Technology, Bangladesh)

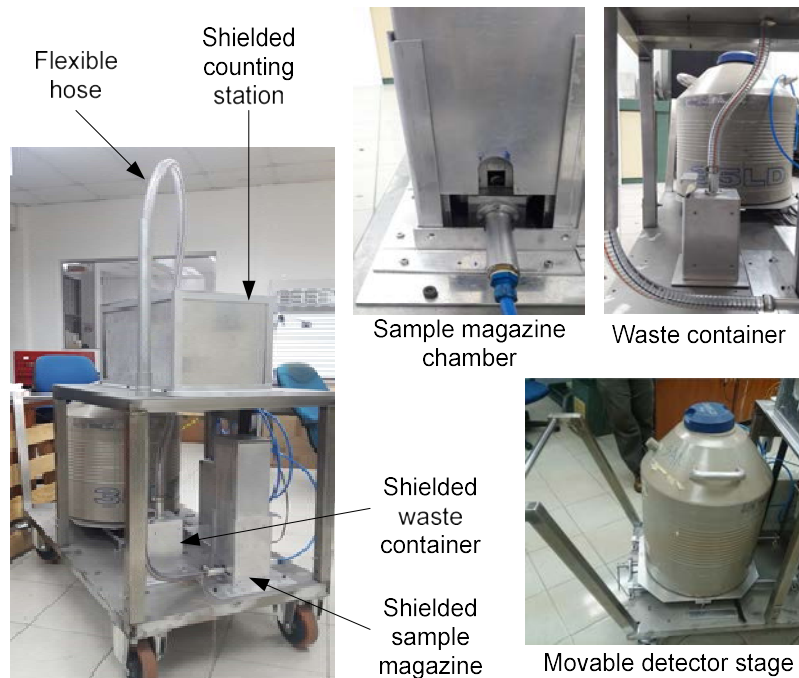


FIG.11. Pneumatic sample changer, developed at Malaysia Nuclear Energy in Malaysia (courtesy of Malaysian Nuclear Agency, Malaysia).

### 3.2.1.3. Robotic sample changers

A sample changer should meet several design criteria, such as

- A sufficient capacity (number of samples) to operate unattended during the required period (for instance overnight or even during a full weekend);
- Operate with most of the gamma ray detector types (i.e. horizontal or vertical looking detectors);
- Communicate with the multi-channel analyse for unattended acquisition, data storage and on-line analysis of the gamma-spectra;
- Have adequate shielding to accommodate a variation in activity of the samples;
- Provide ease of operation;
- Have an outlook for ease of maintenance and robustness in view of long-term reliability;
- Have an affordable price.

‘Industrial’ robotic arms are commercially available and have been used to serve gamma-ray spectrometers. Such robotic arms are compact, flexible in operations but relatively expensive whereas programming may have to be done by specialists.

### 3.2.1.4. Self-designed systems with linear actuators

Several sample changers developed during the CRP were based on a design from the Reactor Institute Delft (see Figure 12). The basic starting point in the Delft design was to use mainly commercially available components so as to limit the need for workshop engineering. The samples are positioned on an X-Y table, vertical lifted and moved along an axis towards the detector location where the vertical actuator lowers the sample.

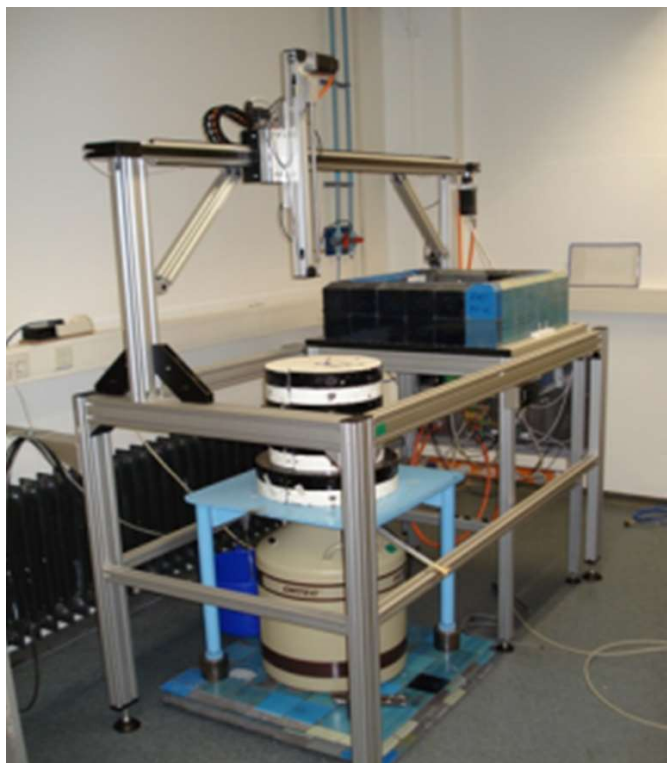


This type of sample changer has been developed by several CRP participants. It basically consists of a bilateral axis (two-axis linear positioning X and Y-axis) such as in the design of JINR (Russian federation) (Figure 13a), but others operate with movement along three axes (X, Y and Z- axis) such as at AECS (Syria) (Figure 13b). The participant from COMENA/CBRN, Algeria developed such a sample changer serving two detectors (Figure 13c). The axes are made from an aluminium square profile, a construction material commonly in use with optical benches. Some sample changers use a stepping motor to move their components whereas others operate with and some of them use the compressed pressured air. The two methods enable to move and position samples between the various defined positions with a high degree of accuracy.

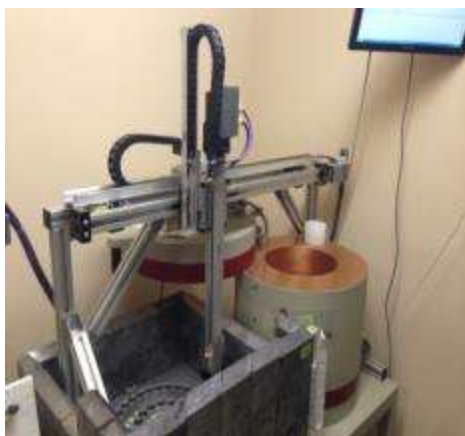
The axes are equipped with sensors for precise positioning of all actuators, both near the sample storage and near the detector. Movement between tray (samples slots) and gamma ray detectors relies on sensors placed on the axes. Other sensors may be used for allow controlling the diminution, to prevent any damage to the equipment during the movement of the axes and to control the movement speed during the transfer of the samples. More details on these types of samples changers are found in the country reports on the CD-ROM.

#### *3.2.1.5. Gripper*

An important part of this type of sample changer is the device replacing the human hand during the transport of the samples: the gripper. The design of the gripper depends on the type of sample containers in use. Various designs have been demonstrated, such as the use of a rubber seal and under-pressure which ‘sucks’ the container to the seal (Figure 14); mechanical fingers or clams (Figure 15) to hold either the sample container or a special container in which the sample container is positioned.



*FIG. 12. Sample changer, designed and operational (since 2006) at Reactor Institute Delft, Netherlands (courtesy of TU Delft, Netherlands).*



*a) JINR (Russian federation) design.*



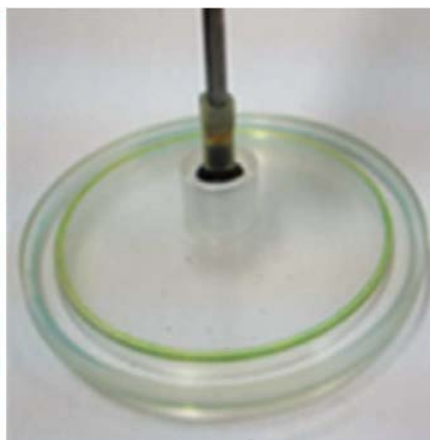
*b) SAE (Syria) design.*



*c) NRC Birin (Algeria) design*

*FIG. 13. Different types of laboratory developed robotic sample changes with linear actuators (courtesy of JINR, Russian federation, Atomic Energy Commission of Syria and Nuclear Research Centre of Birine, Algeria).*

There may be a need for adjustable measurement geometry, in particular the distance from the sample to end cup of gamma detector, in some of the sample changer designs. This can be accomplished by having the vertical actuator to stop at the required height but without releasing the sample.



*FIG. 14. Left: Rubber seal at the end of a tube connected to an underpressure system; capsule is sucked to this rubber (TU Delft, Netherlands); Right: similar system, positioned on top of a sample vial (courtesy of Centro de Desenvolvimento da Tecnologia Nuclear, Brazil).*





FIG. 15. Mechanical fingers (left: courtesy of COMENA/CNRB, Algeria; right: courtesy of BATAN, Indonesia).

#### 3.2.1.6. Sample tray

Samples can be stored on squared/rectangular trays (see Figure 16a) or in discs (see Figure 16b). Either the robotic arm moves by the X-Y-Z controller to the desired position above the tray or disc and lowers its gripper, or the tray/disc is moved by its own controller (such as an X-Y actuator under a tray or a stepping motor driving the disc, or by pneumatic means) under the gripper. In both cases, precise programming of the positions of the tray is important. The spacing between samples on sample tray depends on gripper size.

The number of samples in the storage depends on the physical size of the sample containers, the time the system should run unattended and the number of samples to be counted in that period. As an example, if a system has to be loaded only once per day for samples have to be counted for 1 h each, the capacity chosen would be in the order of 24; if the system has to run at full capacity during a weekend, from Friday afternoon to Monday morning (ca. 72 hours), its capacity should be obviously larger.

Please note that the maximum capacity also depends on the total allowed activity to be stored, the quality of the shielding as well as the dose rate to the user when loading all samples.



FIG. 16. a) Left: Rectangular sample storage on an X-Y positioning table (courtesy of TU Delft, Netherlands); b) Right: Rotating disc containing samples (courtesy of JINR, Russian federation).

### *3.2.1.7. Sample changer container*

Sometimes irradiated sample vials are re-packed into sample changer containers prior to measurement, either because of local radiological safety regulations (minimizing risk of accidental opening of the vial and dispersion/contamination by the content); and/or to minimize the risk of contamination of the storage tray/disc and/or the detector; and/or to ease the gripping. Such an additional sample changer container inside the sample changer can be made from any suitable (and/or disposable) material. Another advantage is that these containers allow for adding a code (such as a bar code label) that can be read-out. The sample container can be made of polyethylene (vial) or aluminium. Spacing between the samples in sample tray depends on gripper size.

Samples can be loaded into their sample carriers manually. At large, it is recommended having a routine procedure in which a not-irradiated vial is measured in between batches, to inspect for possible contamination of the sample changer's gripper.

### *3.2.1.8. Compressed air requirement*

A properly prepared compressed air facility helps to prevent faults in pneumatic components and to prevent any malfunctions or damage to systems. Use of clean compressed air increases the service life of the components and reduces machine failures and downtime, thereby increasing the process reliability. The compressed air should be unlubricated and filtered, and the air pressure should not exceed 6 bars.

### *3.2.1.9. Shielding*

The sample storage has to be shielded, obviously. Detectors may have a cylindrical or a rectangular lead shield. Commercially available cylindrical ones are usually equipped with either a door on the side or a door on the top of the shield. Such doors can also be opened and closed using (pneumatic, hydraulic or electrical) actuators, but these have to be designed together with the sample changer.

### *3.2.1.10. Other types of sample changers*

Another type of sample changer is operational in Peru. The device consists of a disc containing a small number of samples which rotates over a collimator in the lead shield of the detector (see Figure 17). Possibly this is one of the simplest designs requiring only a minimum on investment and assistance by workshops.

For shielding considerations, such a design can hold only a limited number of samples although this may be ample to measure overnight continuously.

A sample changer serving a Compton suppression spectrometer is also operational at CCHEN in Chile. The scintillation plug detector is combined with a sample holder and lifted from its position for (un)loading the samples. The samples are loaded from a stack loader (see Figure 18).



FIG. 17. Lead shield with sample disc on top (courtesy of Instituto Peruano de Energía Nuclear, Peru).

### 3.2.2. Sample changer software

A self-designed sample changer needs software to operate and control its actuators so that the sample can be moved; and it needs software to communicate with the gamma-ray spectrometer. Several approaches have been reported during this CRP for the control of the actuators of the sample changers (such as the gripper, sample tray/disc mover, X-Y-Z mover, etc.). Programmable Logic Controllers (PLCs) have been used; others combined this with software such as Sample Changer Control Software GUI, SYR-Sample-Changer.exe, Visual Basic or LABVIEW.

As an example, the participant from Indonesia reported that BATAN Data Acquisition was written in Visual Basic and used for setting the parameters of measurement (ID sample, number of samples, measuring time, batch file name, etc.), setting and control of the measurement and evaluation of the samples. This software uses the PLC control and manages the entire acquisition process. Commands can be sent through USB connections to an external microcontroller to change the samples, to the gamma-ray spectrometer to start the data acquisition and to monitor whether the measurement has been completed.

The gamma-ray spectrometer (and the multi-channel analyser) itself can be programmed to save the measured spectra, using the tags on e.g. sample code entered as described in the above. At large, the control software depends on the type of electronics used as well as the available human capacity and expertise in programming and controlling such devices.

### 3.2.3. Summary

With support by the IAEA through this CRP, all participating laboratories now have insight in the design principles of automatic sample changers for gamma-ray spectrometry. Most laboratories successfully developed and installed on their own their first sample changer during the CRP.

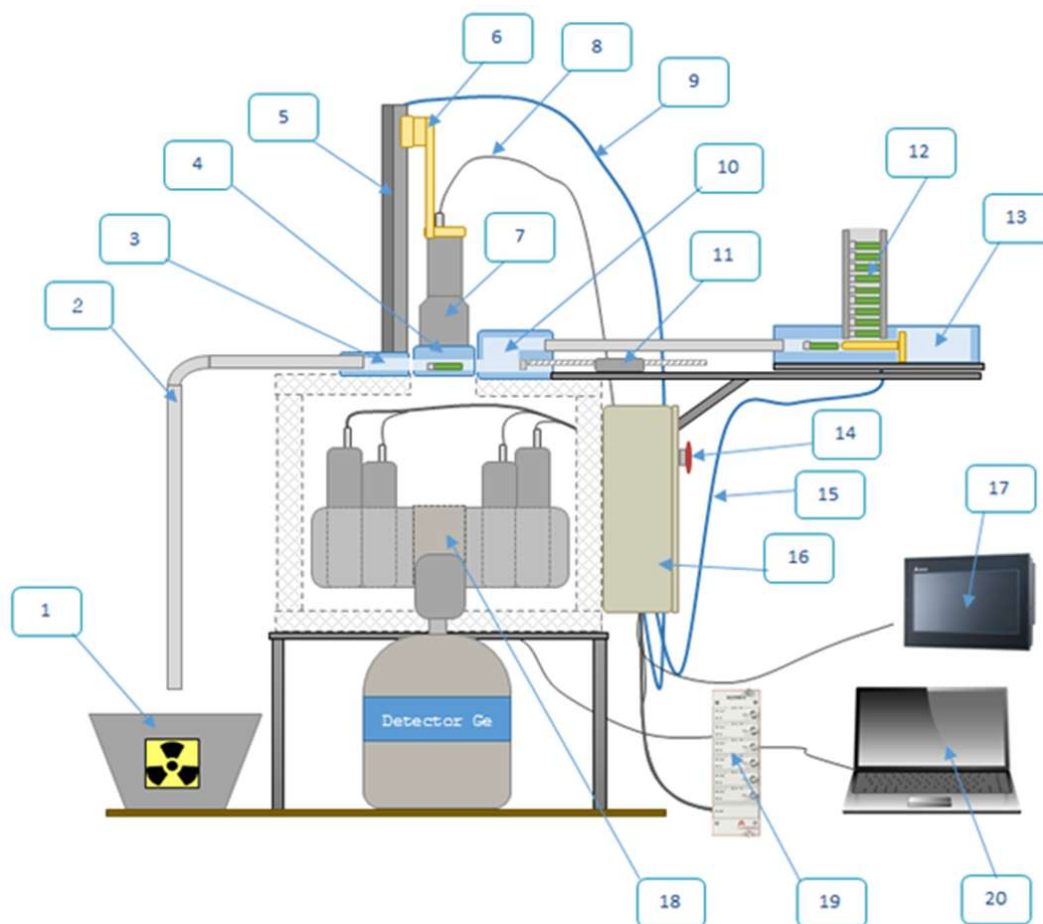


FIG. 18. Sample changer for Compton suppression spectrometer. 1.Waste container; 2. Pneumatic tube discharge; 3. Pneumatic tube connection; 4. Sample holder; 5. Piston rod less linear guide; 6. Upper support detector; 7. Sodium Iodide upper detector; 8. Signal cable detectors; 9. Compressed air lines; 10. Capsule receiver; 11. Linear guide; 12. Capsule loader; 13. Sendstation; 14. Emergency stop; 15. Compressed air line; 16. Control panel; 17. HMI Screen; 18. Countingsamples; 19. Multiport II; 20. Computer (courtesy of Comisión Chilena de Energía Nuclear, Chile).

### 3.3. OTHER DEVICES FOR AUTOMATION

Besides automatic irradiation faculties and sample changers, automation in other equipment involved in NAA facilitates analyses of large number of samples with various weights and matrices. This equipment includes, for example, the sample registration system, micro-balance, micro-pipette and diverters.

#### 3.3.1. Sample identification systems

The first stage in the neutron activation analysis method after receipt of the samples is the sample preparation. The automation in sample preparation enhances laboratory productivity and accuracy. Data management during the sample preparation can be controlled using a PC-based data entry software. This method would replace redundant manual data entries that need to be completed by laboratory personnel. The software can automatically generate a sample code for each sample, create printable registration forms for administration purposes and store selected parameters that will be passed on to the sample analysis program [14].

### 3.3.2. Balance

Samples are weighted and recorded, given ID and packed in vials for the irradiation. The weighing stage involves weighing the samples, standards, certified reference materials (CRM) and blanks. The accuracy of the weight of the samples is usually one tenth to one hundredth of a milligram. Most NAA laboratories carry out these procedures manually, which is time consuming since all data regarding the sample preparation has to be registered manually and then entered in a personal computer for the concentration determinations of elements.

Automatic recording of the balance reading eliminates the error associated with manual data entries and therefore enhances the accuracy. In this case, the micro-balance is connected to a personal computer for automating the reading of the balance and for saving weighing records. Examples of commercial software for automatic recording of a micro-balance are given in Refs. [19–21]. It can be accomplished with the use of an interface card and some self-made software, as has been demonstrated by the CRP participant at IPEN, Peru; see Figure 19. The card communicates with in-house data management software.

However, all NAA laboratories often have their own requirements and features for data management. Such cases need either modification in the commercial software, which is often not possible, leaving as viable alternative the creation of compatible software.



FIG. 19. Interface card and its connection to the balance at IPEN, Peru (courtesy of Instituto Peruano de Energía Nuclear, Peru).

### 3.2.3. Pipette

The accuracy of the preparation of the standard solution is critically affected by the measured volume. Manual pipettes are not efficient for preparation of a large numbers of solutions. Use of automated electronic micro-pipette enables maximum liquid handling performance with high levels of accuracy and precision. In addition, it avoids human errors and increases the throughput of the laboratory.



### 3.3. INTEGRATED DATA MANAGEMENT

#### 3.3.1. Data management concepts

##### 3.3.1.1. *Importance of data management*

The previous sections have all demonstrated ways in which the various hardware components of an NAA facility can be automated. The ability to process a higher number of samples in a shorter time frame and/or with less human intervention will ultimately lead to a greater generation of intermediate data, often requiring manipulation in order to produce the final desired output.

Many of the competing technologies in the field of elemental analysis are often "complete packages" supplied and supported by a single vendor. However, no such "complete NAA" systems currently exist, where a single vendor supplies a complete integrated system from the irradiation by the neutron source through to the data analysis and report preparation. In reality, NAA facilities are essentially modular systems built from components which rarely communicate directly with each other.

In order to maximise the investment in hardware automation, the data produced by each component should ideally be brought together during the calculation stage of analysis, in the most efficient way possible, to convert raw data into meaningful results. Without an effective way to handle at the analysis stage the larger amounts of data produced by automated components, any advantages may be effectively undone if data is poorly handled, both from an efficiency and possibly from a quality point of view as well. Even laboratories not employing devices capable of high sample throughput could benefit from robust integrated data management.

##### 3.3.1.2. *Data types in the NAA process*

Data produced during typical NAA processes include client/sample information, sample weights, irradiation facility and timing information, raw gamma spectra and deconvoluted peak table files. Software packages are then used to process all of the relevant data, cross-referencing nuclear data libraries, into final analysis results in the form of masses or mass fractions (see Figure 1).

Each component typically produces data "in isolation" in terms of awareness of the overall calculation process and traditionally it has been up to the analyst to "match" the various sources of data and present it to the different software packages. For example, client samples may be logged into a laboratory register, sample masses might then be hand-written into a separate laboratory note-book. Irradiation times may be recorded (possibly also by hand) in another location while gamma spectra would be saved on a computer. The analyst must then retrieve all of this data from the various locations in order to produce the desired output. This is illustrated by the approach in use with the CRP participant from ANSTO, Australia and shown in Figure 20.

Such a process may be adequate for smaller-scale operations but would not be suitable for a laboratory aspiring to increase its throughput. Not only would such a method be slow, inefficient and poorly matched to hardware capable of producing relatively large amounts of data, but is susceptible to mishaps such as transcription errors and data loss, particularly in the case of hand-recorded data. An integrated data system should therefore be considered as

important to achieving higher throughput in NAA as automatic irradiation facilities and sample changers.

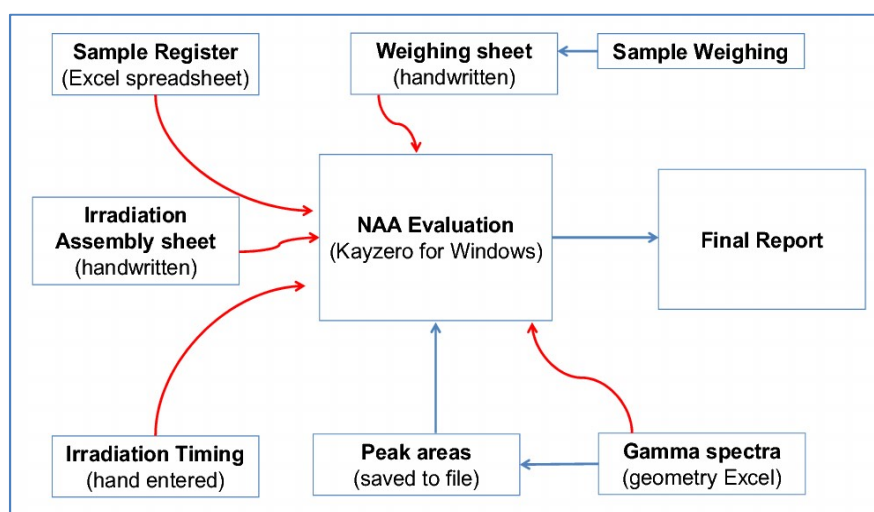


FIG. 20. Workflow at the NAA laboratory of ANSTO, Australia, before implementing automation (courtesy of Australian Nuclear Science & Technology Organisation, Australia).

### 3.3.1.3. Laboratory Information Management Systems

Many of the concepts of integrated data management are not unique to NAA and are common to all laboratories providing analysis services of any kind, namely the registry and tracking of samples. Many facilities employ commercial Laboratory Information Management Systems (LIMS) to fulfil this requirement.

Commercially available LIMS are highly configurable and can incorporate features essential to handling large numbers of samples including label printers, barcode readers and PC-connected analytical balances to directly transfer sample masses into the system. Such commercial systems may prove too costly to implement and maintain for NAA laboratories (ongoing maintenance costs are usually associated with such systems).

However, with careful planning, a very effective LIMS can be assembled using software included in commonly used office suites, some of which are free. Key to effectively managing data associated with NAA is to cross-link all of the relevant particulars, such as weighing data to irradiation data to measurement data. This facilitates ease of retrieval of the information required to perform the analysis, whether it's to be done semi-interactively by an analyst or even fully automatically by software. For example, upon selecting a particular job or batch of samples, a well-integrated system should automatically retrieve the associated sample masses, irradiation start and end times, irradiation channel data (if required, for instance where  $k_0$  or absolute standardization is being used) and measurement data (start, measurement and dead times, detector number and measurement position), plus the corresponding data for the associated standards.

As has been demonstrated by several of the participants of this CRP, using a relational database is a logical way of implementing this. Indeed, commercial LIMS are built upon on such databases. Well-constructed databases have many advantages over using purely file-based record keeping including better efficiencies (such as elimination of data duplication) and robust architectures maximising data integrity and security.

### 3.3.2. Specific considerations for data management in NAA

#### 3.3.2.1. *Data protocols*

Prior to an NAA laboratory developing or implementing a LIMS (or some mechanism for centralising all of its data), protocols should be agreed upon within the facility to ensure that all information is recorded in a consistent manner by all involved workers. For example, the way in which samples are numbered and labelled or the way in which spectrum files are saved should be standardized within a laboratory and not depend on the individual performing the task. This is particularly important for facilities which have a relatively high turnover of workers, such as in universities, or where multiple staff are present and have well-defined, individual roles that don't necessarily cross-over frequently.

Data should ideally be stored in an agreed, central location that is frequently backed up using best IT security practices. Another important feature is the ability for multiple users to interact with the LIMS simultaneously. Systems built on the common database architectures generally have this capability, while purely file-based systems such as a spreadsheet have at best limited multi-user capabilities.

#### 3.3.2.2. *Data retrieval and manipulation*

Similarly, consideration should be given to how the centralized data will be extracted and utilized in the final NAA calculations. Few complete NAA software solutions exist and many laboratories use either their own codes or even spreadsheets. The latter is in some ways the easiest to mesh with a LIMS, where an appropriate database query can produce the required input in exactly the form needed which could then be "cut and pasted" into the calculation spreadsheet. This approach still involves a reasonable amount of data manipulation which may prove to be a hindrance above a certain level of throughput.

Laboratories using "in-house" NAA software may need to update their programs in order to efficiently take input from the LIMS. Software that uses text files for input can, as with the previous example, create a query that would produce the input files exactly as needed. Software that relies on direct user input only should be updated to use file-based inputs, or better yet, to retrieve the required data from the LIMS directly.

At time of writing this report, current versions of commonly available NAA software packages as used by multiple laboratories do not integrate optimally with LIMS. The commercial package Kayzero for Windows [22] takes most of its input by direct user interaction and relies on peak table files to be placed in a certain user-defined directory. The ability to input data from text files is however being considered. The  $k_0$ -IAEA package [23] is in some ways more conducive to working with a LIMS as it already uses a database architecture to store data, although not in the form of a centralized system. Both systems ultimately store all the information necessary for the analysis, but their workflows are best when all the data is already available (including at least sample weights), rather than being built-up step by step (e.g. register samples in database, weigh samples, irradiate samples, etc.). For this reason, an independent LIMS is still recommended when using such software packages.

#### 3.3.2.3. *Analytical balances*

Many hardware devices typically in use by NAA laboratories can be interfaced into LIMS with varying degrees of communication. The higher the level of integration desired, the more effort



is needed on the part of the laboratory. Most of the major analytical balance suppliers offer the ability to interface their devices with computers. The interface software provided by the vendor sometimes only offers rudimentary connectivity, such as copying the balance readout to the computer's clipboard. Even this level of connectivity has benefits in terms of reducing transcription errors and potentially providing better data security. Higher levels of integration generally require custom programming. Fortunately, most balance manufacturers provide good documentation detailing the serial commands transmitted and received by their equipment, allowing interactivity with such devices to be programmed directly by the end users provided they have such expertise.

#### *3.3.2.4. Irradiation systems and data isolation*

Interfacing automatic irradiation systems with LIMS is also possible. Such systems are commonly based on well-supported scientific or industrial control systems, which often have the ability to interface directly with the common database architectures. Challenges may arise when such irradiation systems are integrated with reactor control systems which require data isolation from internet connected networks for security reasons. Unidirectional network (so-called "data diodes"), which allow data to travel only in one direction, are required in these cases if irradiation information is to be fed directly into a LIMS while preventing information to be fed to the control system of the reactor.

#### *3.3.2.5. Gamma spectrometry data*

The gamma spectrometry component of NAA produces the most complex form of data which in turn can be the most difficult to integrate into a LIMS. Gamma spectrometers record their data as proprietary files, both as raw spectra and as peak tables.

There are several approaches to incorporating this type of data into a LIMS. Spectrum and peak table files can be saved into some databases, although this may result in unnecessary bloating of the database, potentially slowing it down. As many NAA software packages use these files directly, storing these files separately alongside the LIMS may be preferable.

A hybrid approach may be considered, where only the key metadata associated with gamma spectra such as start, end and dead times may be recorded. The commercial gamma analysis package Hyperlabs [24], on the other hand, uses a database at its back-end to store all information read and derived from gamma spectra, including the raw gamma spectra, peak table data and virtually all types of associated metadata. This approach is arguably the most powerful, as the resulting database is accessible by outside software.

### **3.3.3. NAA and beyond**

#### *3.3.3.1. Multi-capability facilities*

Many NAA laboratories do not just do NAA alone but often provide additional services such as natural radioactivity counting, neutron irradiation services (including geochronology and other purposes) or provide other forms of elemental analysis. In these circumstances a well-designed LIMS should take this into account and be applicable to all services offered; certain stages of analysis such as sample registry and weighing are common to all of these services.

Retaining data in a centralized, cross-linked relational database has additional benefits beyond supporting automation and higher sample throughput. Depending on the detail of the information stored in the database, powerful internal reporting capabilities can be implemented

to provide statistical feedback to the laboratory. This can be useful for not only straightforward statistics such as how many samples of a certain type were analysed, but the automation of QA and QC checks and procedures can be automated.

### 3.3.3.2. *Hardware and software integration*

As with hardware automation, various degrees of software automation can be achieved depending on the requirements of the laboratory and the means available to implement them. Even with limited hardware automation available, implementing a simple LIMS using tools already available on most computers can produce efficiency gains and potential improvements in quality. A higher level of software integration can be attained by exploiting tools provided by hardware vendors such as analytical balance or gamma spectrometry manufacturers to achieve a more "hands-off" workflow, yet still requiring human intervention at key points.

More ambitious facilities could invest more heavily in both software and hardware automation to potentially achieve complete automation from irradiation to report generation, at least for simpler analysis scenarios such as measuring single elements where interferences are not anticipated. Higher levels of automation such as this would certainly require more sophisticated analysis software, potentially needing to communicate directly with hardware components such as spectrometers, as opposed to calling upon the vendor-supplied software to perform certain tasks using scripting languages.

For instance, a high level of integration was achieved at the reactor IBR-2 of the Frank Laboratory of Neutron Physics, JINR, Russian Federation. A software package for automation of neutron activation analysis was developed, including the following programs [25, 26] (see Figure 21):

- (a) Database of information about all steps of analysis — *NAA database*.
- (b) A set of service programs to automate and facilitate the completion of the database:
  - (i) Environment of NAA;
  - (ii) Information about clients;
  - (iii) Information about samples;
  - (iv) Weight of each sample;
  - (v) Journal of measurements;
  - (vi) Search for most appropriate CRM.
- (c) A program for automation of measurement of the induced activity using the program for spectra analysis Genie-2000 and Batch Support Tools S561 Genie-2000 [27].
- (d) A program for calculation of element concentrations based on analysis of gamma spectra with the program Genie-2000.

At the NAA Laboratory of ANSTO, Australia also use was made of a multi-dimensional database. This has led to a different integration of the NAA procedures (see Figure 22) which can be compared to the approach before automation as shown in Figure 20. It resulted in time-savings of between 10% and 30% in performing various tasks.

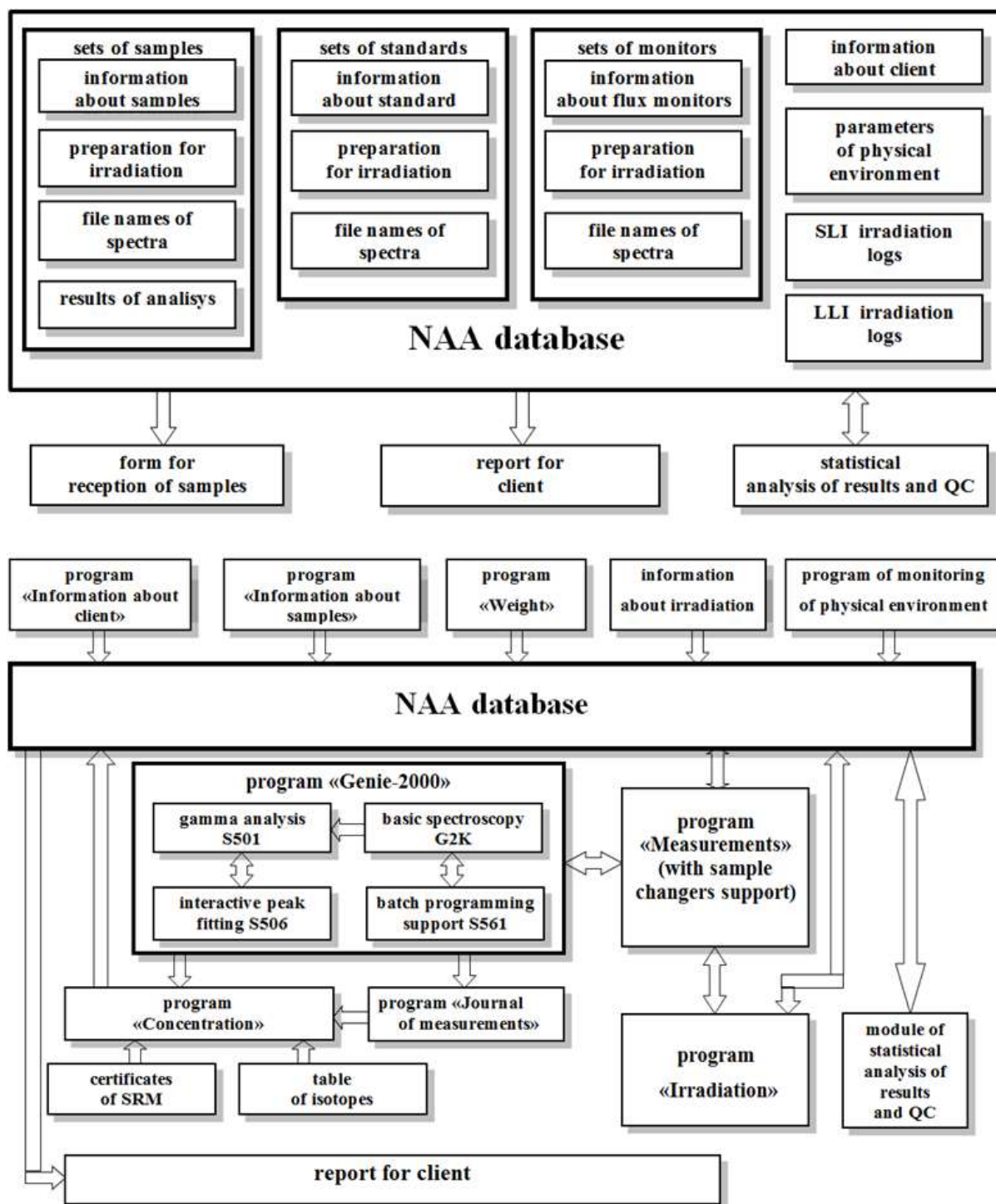


FIG. 21. Structure of the NAA database and NAA software package, as developed at JINR, Russian Federation (courtesy of Joint Institute for Nuclear Research, Dubna, Russian Federation).

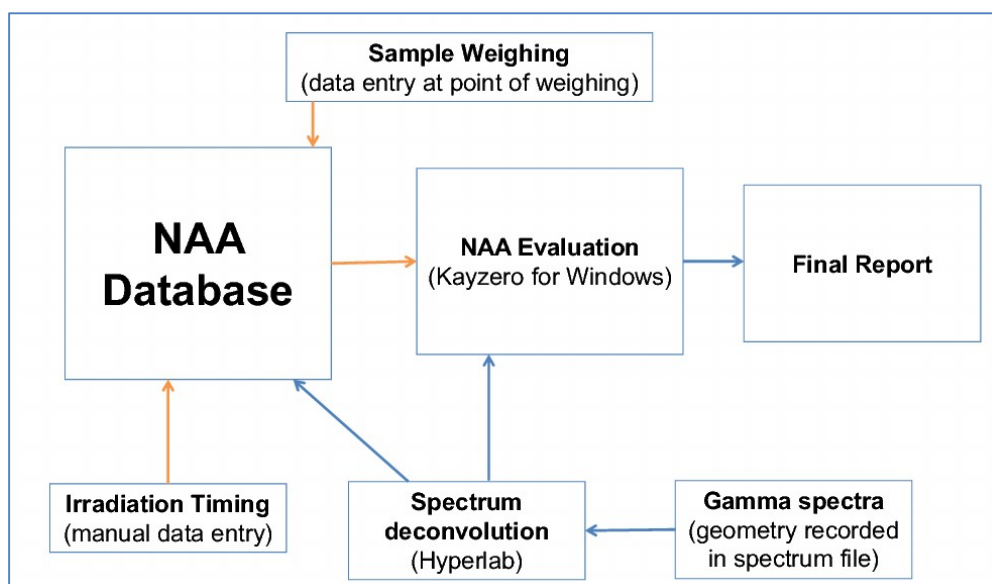


FIG. 22. Workflow at the NAA laboratory of ANSTO, Australia, after reorganization and automation using a multi-dimensional database (courtesy of Australian Nuclear Science & Technology Organisation, Australia).

### 3.3.3.3. Open standards

Today no readily available NAA software can achieve the full automation as described above. To be able to directly interact with the digital spectrometers as supplied by the major vendors of today requires the purchase of additional software toolkits at not insignificant expense. Each supplier uses proprietary methods, specific to their brand only. Jamaica's OpenNAA concept [28] developed within this CRP aims to alleviate this problem by having created a set of open standards.

The OpenNAA framework specification will define a common programming interface between an application and various classes of operations in the NAA process such as: system management; data acquisition and control; data input / output; spectrum processing; nuclide library management; reference library management; and analysis data management. Figures 23 and 24 present some of the features of OpenNAA.

If adopted by hardware vendors, would enable programmers to directly address the common functions of compatible hardware using these common protocols.

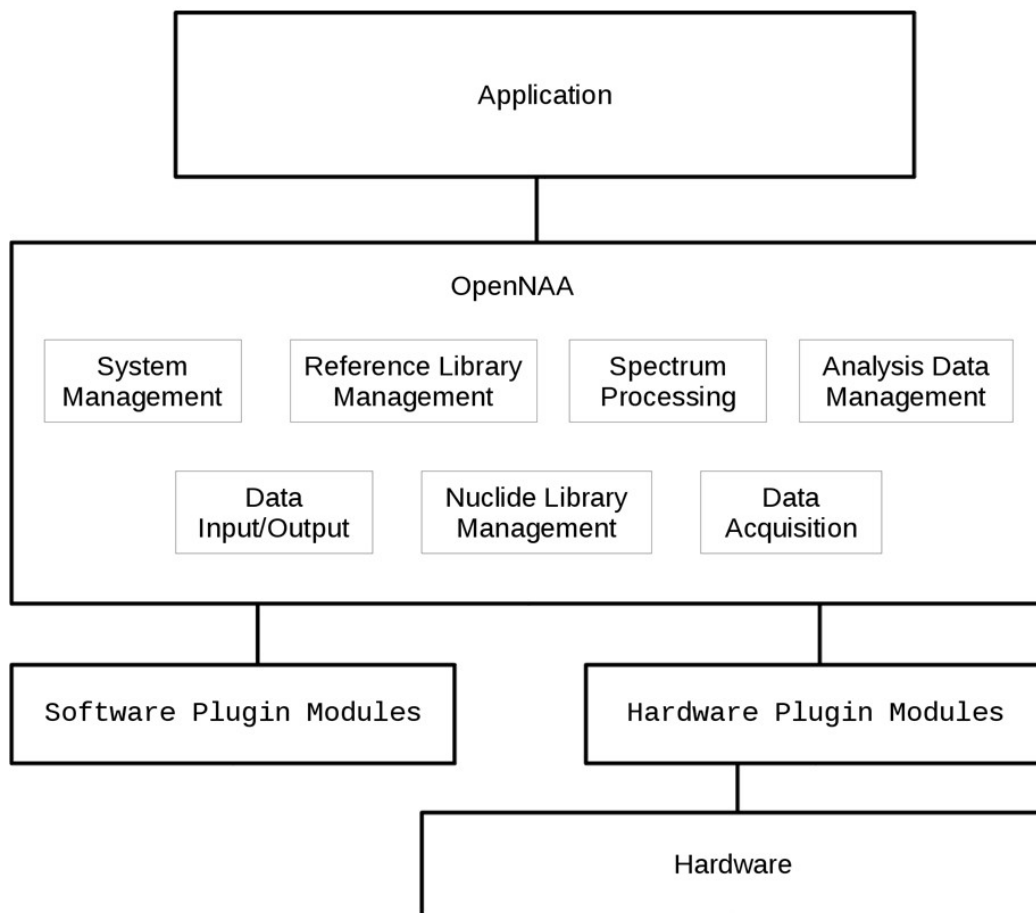


FIG. 23. OpenNAA architecture (courtesy of International Centre for Environmental and Nuclear Sciences, Kingston, Jamaica).

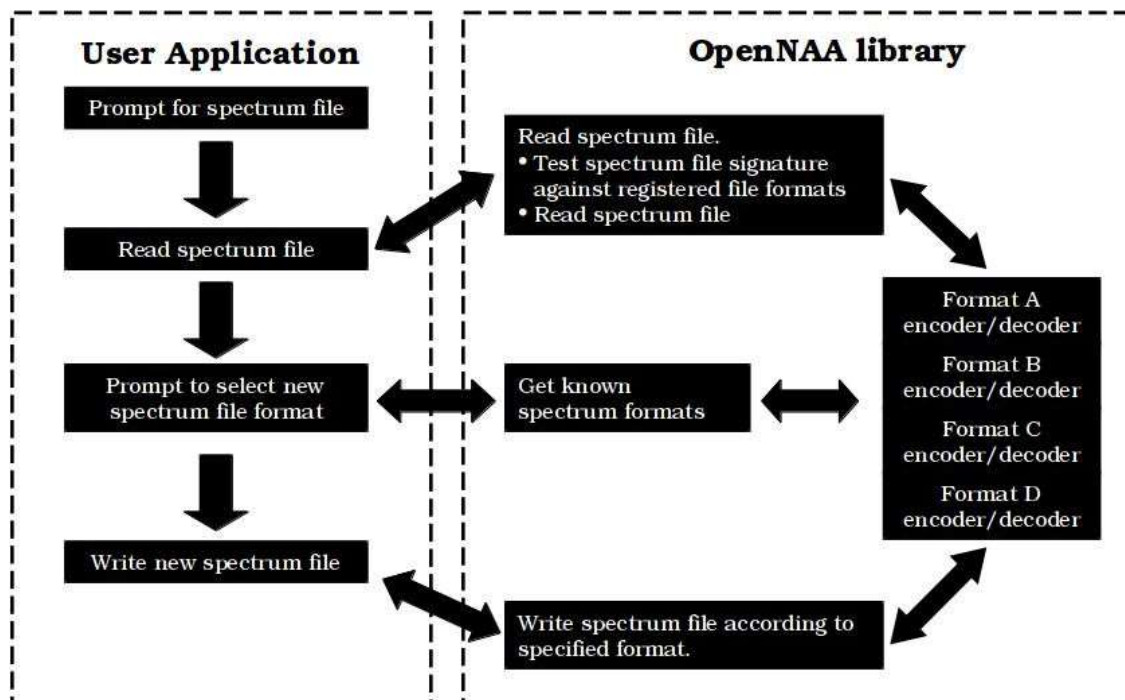


FIG. 24. Using OpenNAA to implement a spectrum analysis program (courtesy of International Centre for Environmental and Nuclear Sciences, Kingston, Jamaica).

### 3.4. QUALITY ASSURANCE AND QUALITY CONTROL

In the usual definitions, quality assurance encompasses all those planned and systematic actions undertaken by the organization necessary to provide adequate confidence that a product or service will satisfy given requirements for quality, while quality control refers to the operational techniques and activities that are used to fulfil requirements for quality [29].

These definitions are not very transparent; the concepts, however, are simpler. Quality assurance consists of procedures that contribute to minimize the occurrence of defects — mistakes and errors — during all the steps in the analysis procedure. Quality control consists of practical procedures that enable the analyst to identify such defects when they occur. Quality control is a binary process: the inspected component either passes or fails specified compliance criteria.

An effective and efficient quality assurance program is imperative once automation leads to an increase in samples to be analysed and spectra to be processed and interpreted. Any mistake or error detected by the quality control will result in non-quality costs and affect the facility's operations by

- Time needed for remedial or corrective actions;
- Repetition of work;
- Exceeding deadlines;
- Disturbed planning of other work;
- Stress, and a potential for more errors;
- Unhappy customers;
- Bad reputation.

#### 3.4.1. Quality Assurance

A successful quality assurance programme requires an open mind towards the unavoidable fact that nonconformities (mistakes, errors or any nonfulfillment of a requirement) will occur, irrespective of the experience of the analyst and of the simplicity or complexity of the operation. Measures have to be implemented that help to minimize the occurrence of such nonconformities, such as:

- Trained, skilled and qualified staff. Staff should understand the physico-chemical principles of the various steps in the process and the operational principles of the instruments used, and demonstrate that they can perform within certain boundary conditions such as analytical quality, time and even cleanliness. In addition, staff should be knowledgeable on the potential errors and mistakes, methods to monitor them and how to respond to the occurrence of nonconformities;
- Registration of conditions under which the experiment is conducted, such as instrument settings, environmental conditions, sample-specific treatments etc.;
- Calibration of instruments used;
- Performance checks of instruments used. Performance checks are needed to verify the validity of the calibration status and to ensure that an instrument functions as required;
- Preventive maintenance of instruments such as mechanical sample changers and/or automated irradiation systems, HV supply units;
- Temperature stabilization ('control') in balance weighing rooms and counting rooms;

- Anticipation on technical errors including but not limited to moisture content correction, size and geometry differences between samples and calibrators, neutron flux gradients, neutron spectrum shape variations (essential for  $k_0$ -NAA), counting geometry differences especially when counting close to the detector end-cap and spectrum interpretation errors;
- Anticipation on human errors, such as transcription errors and exchange of samples, together with risk assessment of their occurrence;
- Independent checks of manually entered data;
- Nonconformity management. This contributes to the reduction or re-occurrence of the same mistakes and errors. Nonconformity management is built on registration of defects, assessment of the root-cause, risk assessment of its impact and re-occurrence and implementation of remedial (i.e., ‘repairing’ the problem, for instance by re-adjusting the pole-zero cancellation when peak tailing has been observed) and/or corrective actions (i.e., changing the procedure because of intrinsic problems);
- Developing a database of NAA measurement results and experimental conditions to be used as a reference on the evaluation of analysis requests;
- Implementing a planning system covering human resources, reactor schedule, measurement time and sample changer availability.

One should note that in principle there is no absolute need<sup>1</sup> to describe each quality assurance step in extensive detail in a documented ‘standard operating procedure’ (SOP). A laboratory should decide on basis of a documented risk assessment if and how such steps should be documented. This can be made on basis of a ‘classic’ SOP or on basis of flowcharts and forms, to name but a few options. A facility may decide to make such SOPs for training purposes if a regular turnover of personnel is expected, but qualified staff will usually not consult them in daily practice. Maintenance of SOPs requires a significant amount of effort, which may be conflicting to the time needed for managing the increased sample throughput. The laboratory will already benefit much from smart documentation of observations and experimental conditions, even without a formalized SOP.

### 3.4.2. Quality Control

As outlined in the above, quality assurance is needed to minimize the occurrence of mistakes and errors; it is not a warranty that they will not occur. No matter how thoroughly implemented and how well all staff complies to quality assurance procedures, mistakes will still be made, and some measurement data will be out of specifications.

Various defects may and will occur during an analysis procedure, the most common being:

- Transcription errors of manually registered information, such as customer codes, lab sample codes, masses, irradiation time and duration, decay time (counting time is usually stored by the spectrum acquisition software), selected counting geometry code;
- Instruments working outside specifications;
- Incomplete or no registration of experimental conditions;
- Contamination of the test portion during handling prior to the irradiation; in some cases also during the irradiation;
- Wrong or absence of moisture correction;
- Irradiation geometry errors (neutron flux gradients);

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<sup>1</sup> Even though it is not explicitly required by the International Standard ISO/IEC17025, some national accreditation bodies demand that all aspects in the work-process are described in SOPs if a facility intends to be accredited for compliance of its management system to this standard.

- Exchange of samples before the irradiation or after the irradiation and prior to counting;
- Counting geometry differences between sample and calibrator;
- Spectrum analysis and interpretation errors; for instance, due use of wrong energy calibration curve, wrong photopeak efficiency curve;
- Coincidence summing effects, gamma-ray self-attenuation effects;
- Unpredictable human errors.

The occurrence of such defects cannot always be identified at the end of the analysis procedure. Several of them may be systematic, applying to all samples. They can be observed by processing samples with known property values (element mass fractions). Certified reference materials suit this purpose. ‘Blanks’ — i.e., encapsulations similar as used for the samples but without any material in it — are essential to detect contaminations. It may be obvious that such quality control samples should be handled simultaneously with the real samples through all steps in the analytical procedure.

Replicate testing is another opportunity, provided there is ample confirmation that the test portions are homogeneous. Replicate testing should be done at least in triplicate; duplicate sample analysis is a waste of time since it is not possible to draw any conclusion from the results when two measurements significantly differ from each other considering their respective uncertainties. One, or both, could be wrong, or the real experimental uncertainty could be higher than the estimated uncertainty.

Quality control should be implemented throughout the entire analysis procedure, not just once the analysis report is available. Quality control is needed in each step at which the analyst has to decide whether the output of this step is good or wrong. As an example, performance checks of instruments (balances, pipettes, gamma-ray spectrometers) result in a series of measurement results; the comparison thereof with the specifications is a form of quality control as it allows for the identification of defects in such steps.

Once the number of samples analysed increases due to automation — and with it the number of spectra to be analysed — the quality control should, where possible, also be automated. As an example, the evaluation of the analysis results of the quality control samples (reference material and blank) can be automated in the software. The measured mass fractions can be compared to the certified values and performance scores can be quickly and automatically calculated to assess whether the values are within specifications.

Results from performance checks (such as the ‘energy calibration’ or energy resolution data) can be entered manually into software (for instance, Excel sheets or control charts) for immediate — and objective — evaluation once specifications have been pre-set. Great care is required for using control chart software or charts that are automatically generated by gamma-ray spectrum analysis software, as in some cases the warning and action limits vary with the date entered — which is conceptually wrong.

An overview of potential mistakes, quality control and quality assurance options for integration in automated NAA is given in Table 4.



TABLE 4. EXAMPLE OF MISTAKES TO BE DETECTED BY QUALITY CONTROL, ASSOCIATED QUALITY ASSURANCE AND OPPORTUNITIES FOR INTEGRATION IN AUTOMATION IN NAA

Mistake	Quality Control	Option for automated control?	Minimized risk by Quality assurance?
Transcription errors of manually registered information, such as customer codes, lab sample codes, masses, irradiation time and duration, decay time, selected counting geometry code.	Double checking of all data by person, different from the one who entered the data.	No	Yes: Routine procedure of double checking
Contamination of the test portion during handling prior to the irradiation; in some cases also during the irradiation.	Use of blank (empty encapsulation) and specifications based on well characterized blank	Yes, by automated calculation of zeta scores using blank reference data	Yes: if routine use of blanks in each procedure is implemented
Wrong or absence of moisture correction.	Only in analysis of CRM	No	Yes: if routine procedure is implemented
Irradiation geometry errors (neutron flux gradients)	Only if multiple flux monitors are used	Yes, if multiple flux monitors are used and criteria exist for maximum variation along flux monitors	Yes: sandwich samples between flux monitors
Exchange of samples before the irradiation or after the irradiation prior to counting.	Only if bar code labels are used on vials, but wrong labelling cannot be detected, except with CRM is exchanged with another sample	Only if bar code labels are used on vials	Yes, by double checking when loading and unloading sample changer; by using stack receiver in automated irradiation systems
Counting geometry differences between sample and calibrator.	Visual inspection	No	Yes, e.g. by pelletizing; by calculus; by counting on large distances
Spectrum analysis and interpretation errors; e.g. due to use of wrong energy calibration curve, wrong photopeak efficiency curve.	From analysis of CRM	Yes if geometry indicator is automatically added to spectrum file or spectrum filename	Yes, e.g. by automated read-out of geometry indicator, to be linked to relevant efficiency curve.
Coincidence summing effects, gamma-ray self-attenuation effects.	Problematic mainly in $k_0$ NAA. Check multiple peaks from the same radionuclide; possible via analysis of CRM	No	Yes, by calibration or measurement on large distances only

## 4. CONCLUSIONS

Participants in the IAEA CRP 1888 / F1.20.25 “Development of Integrated Approach to Routine Automation of Neutron Activation Analysis” (2012–2015) developed and shared their approaches towards automation of irradiation facilities, sample changers for gamma-ray spectrometry, automation of auxiliary instruments and integration of data stream and automation of data processing. The final individual country reports, available on the CD-ROM of this publication, thus provide a wealth of information on how automation in NAA has been accomplished with ample detailed descriptions. The various examples may be inspiring to others since there is not one uniform way to implement all aspects of automation in the individual NAA laboratories worldwide.

Automation of existing irradiation facilities is not always technically possible or even allowed due to specific design features related constraints. Sometimes the design and implementation of a new, dedicated automated irradiation facility could be a preferred approach. An automated irradiation facility with fast transfer of the capsules is a prerequisite for applying cyclic activation analysis to enhance the sensitivity for some elements. Nonetheless, some laboratories reported an increase in their irradiation capacity up to a factor 4 by automation of their irradiation facility.

The CRP participants presented different creative designs of sample changers for gamma-ray spectrometry and were able to construct and test these systems. It allows them now to count samples ‘around the clock’. This has resulted in an increase in their measurement capacity (number of samples analysed per unit of time) up to a factor of 4, thus matching the increase in irradiation capacity.

However, such a higher analytical capacity also brings a higher workload on the processing of the gamma-ray spectra. Individual NAA-specific laboratory information management systems have been developed and implemented and the benefits of data integration and automation are estimated as 10–30 % time savings. However, these approaches were found to be difficult to implement at other facilities. Hence there is great interest in a modular approach based on open source software as tested and demonstrated during this CRP. This would eventually result in sharing and exchange of controllers, processors and other software to communicate locally developed hardware. Still, whereas it has been shown that automation and integration of hardware could be realized within the three year timeframe of the CRP, automation of data processing requires more time.

Automation, in all its hardware and software forms may contribute significantly to keep NAA competitive in serving end-users. Automation provides opportunities for a larger analytical capacity and a shorter overall turnaround time if large series of samples have to be analysed. The degree of accuracy of the results will increase too because of the reduced human intervention during the analytical process and, consequently, the reduced probability of errors.

Participants have implemented, expanded and improved quality control and quality assurance as cross-cutting concepts of their (automated) NAA procedures. As non-conformities like data outside specifications can never be fully avoided, effective trouble shooting is required once the number of samples measured and analysed per unit of time increase. The modular e-learning tool initiated during this CRP stems from the participants’ needs for a practical and pragmatic modern approach to life-long learning on the principles and details of NAA, and on the associated sources of error in the practice and methodologies to overcome them. The course was well received by all participants, and is now available on the IAEA Cyber Learning

Platform for Network Education and Training (CLP4NET). More details are given in the Appendix.

It can be concluded from the above and from the contents of this document that the first five objectives (see for details in section 1.2) have been met at the completion of the CRP. Only the future can tell if the last objective, a long-term cooperation network between participating laboratories has become stronger and will continue among the NAA practitioners.

## APPENDIX: E-LEARNING IN NEUTRON ACTIVATION ANALYSIS

Since the middle 1990's electronic technology (e-learning) has slowly infiltrated and now is part of education at all levels. E-learning encompasses a wide array of deliveries from simple introductions of animations in a teaching class to Massive Open Online Courses (MOOCs) and Modular Object-Oriented Dynamic Learning Environment MOODLEs. Even the definition of e-learning can be elusive [30] while an overview of all the definitions of e-learning is given by Wikipedia [31]. E-learning may include some or all of the following aspects: internet availability, supervised exams, a tutor's support and advice to the students by electronic means such as e-mail, telephone, some (proprietary or open source, commercial or free) software systems, and multimedia designs.

E-learning has transformed education from the Traditional Classroom Course to a) Synchronous Distributed Course in which web-based technologies are used to extend classroom lectures and discussions to students at remote sites in real time; b) Web-Enhanced Course in which online course activity complements class sessions without reducing the number of required class meetings; c) "Emporium" Course where this model is designed for on-campus use, eliminates all class meetings and replaces them with a learning resource centre featuring online materials and on-demand personalized assistance; d) Hybrid Course where online activity is mixed with classroom meetings, replacing at least 20 percent, but not all required face-to-face meetings; and e) Online Course where all course activity is done online and there are no required face-to-face sessions within the course and no requirements for on-campus activity.

E-learning takes an extra challenge where the subject matter is highly scientific and engineering oriented encompassing multiple complex mathematical notations and concepts. A straightforward presentation of equations in PowerPoint is a recipe for an unsuccessful delivery of lectures. The challenge exists of how to deliver these concepts without the use of a traditional blackboard.

There are currently 216 operational research reactors in the world and another 22 in temporary shutdown (status: September 2017) with the largest portion of usage being in NAA: 114 reactors in over 50 Member States [32]. With a large fraction of neutron activation analysis scientists and engineers retiring, it behoves the national and international communities to pass on the information in a non-traditional format, namely e-learning. There has been no comprehensive book written about NAA since the early 1990's (Susan Parry in 1991 [33] with a second edition in 2003 [34], and Activation Analysis Volumes 1 and 2, Alfassi, 1990 [35]), and the most comprehensive one written by Soete, Gijbels and Hoste in 1972 [36] which is still considered the foremost reference work on NAA. It is well understood that with the advancement of e-learning, another book would be less useful using current technologies.

The e-learning tool for NAA has been developed by the IAEA for the Member States, to pass on the basics and more advanced forms of the technology in a manner such that both novices starting their careers in this area and higher level staff members and academics can utilize these modules to refresh themselves or to teach others. The overall philosophy included a team approach with multiple discussions, draft documents, vivid presentations, detailed animations, videos and randomized questions which included multiple choice, and true and false categories. The next phase included outside experts to assess final draft with a follow up meeting at the IAEA to finalize a workshop that was held in October 2016. The modules were written by two NAA experts, Mr. Peter Bode (Netherlands) and Mr. Sheldon Landsberger (United States of

America), with two other experts invited to prepare the modules on prompt gamma analysis and neutron depth profiling.

The IAEA e-learning tool will not have teachers or points of contact with learners, as these will come from over fifty Member States with different skillsets, and will be going online to follow the course. Thus the success of such an endeavour relies on the individual's self-motivation to study effectively and to use additional information on the internet or library to assess published articles and review papers. The overview of the e-learning scheme is described in Figure 25.

These modules in the seven categories, which include Introduction, Basic Nuclear Physics, Instrumentation, Calibration, Quality, NAA Practice and Varieties of NAA, have been designed to be self-contained. In such a manner an individual may choose to take one, some or all of the categories depending on the duties of the work or research. For instance, a staff member or researcher whose work mainly involves instrumentation may choose only this section to study in great detail, while only cursorily studying the other sections. On the other hand a person who is only involved in sample preparation or data output may do likewise by only studying the Quality category.

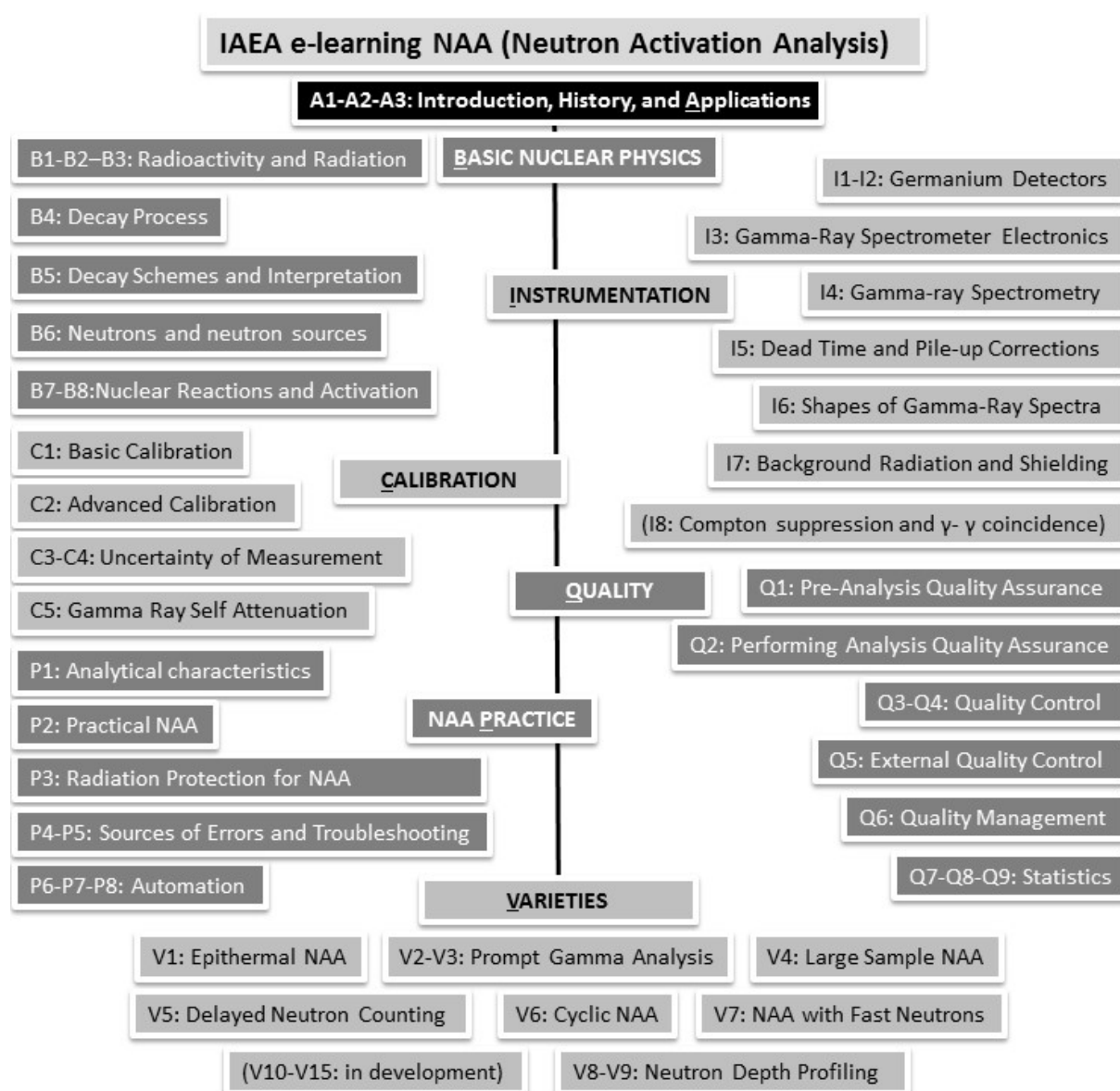


FIG. 25. The overview of the E-learning NAA modules.

One should note that the NAA e-learning tool is intended to be a living course. Continuous improvement will be done based on feedback forms included in the tool, and biannual Training Workshops are planned, where feedback from NAA practitioners is expected to lead to useful suggestions for improvement. In fact, expansion is already foreseen, with several modules already planned: in the Instrumentation category, a module on Compton suppression and  $\gamma$ - $\gamma$  coincidence will be added. Several Varieties of NAA are planned to be also included: Radiochemical NAA, In vivo NAA, Derivate NAA, Industrial NAA, Photon activation analysis and Charged particle activation analysis.

The e-learning tool was made available on the IAEA Cyber Learning Platform for Network Education and Training (CLP4NET) in October 2017 [5].



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## ANNEX I: LIST OF CRP PARTICIPANTS

M. TOUIZA	Commissariat à l'énergie atomique (COMENA); Centre de Recherche Nucléaire de Birine (CRNB) B.P. 180 17230 Birine, Djelfa Algeria Email: crnb@wissal.dz
J. W. BENNETT	Australian Nuclear Science and Technology Organisation (ANSTO) New Illawarra Road Lucas Heights, NSW 2234 Australia Email: John.BENNETT@ansto.gov.au
S. M. HOSSAIN	Bangladesh Atomic Energy Commission (BAEC); Atomic Energy Research Establishment (AERE); Institute of Nuclear Science and Technology (INST) Ganakbari, Savar P.O. Box 3787 Dhaka 1000 Bangladesh Email: syed9495@yahoo.com
A. DUTRA NETO	Centro de Desenvolvimento da Tecnologia Nuclear / Comissão Nacional de Energia Nuclear (CDTN/CNEN) Avenida Presidente Antônio Carlos, no 6627, CEP 31270-901 Caixa Postal 941, CEP 30161-970 Belo Horizonte, Minas Gerais Brazil Email: dutraa@cdtn.br
C. CHILIAN	Ecole Polytechnique de Montreal 2900, boul. Édouard-Montpetit Université de Montréal Campus 2500, Chemin de Polytechnique Montreal, Québec H3T1J4 Canada Email: cornelia.chilian@polymtl.ca
L. MUÑOZ ANRIQUE	Comisión Chilena de Energía Nuclear (CCHEN) Amunátegui 95 Casilla 188-D Santiago Chile Email: lmunoz@cchen.cl
Bangfa NI	China National Nuclear Corp. (CNNC); China Institute of Atomic Energy (CIAE) Xinzhen, Fangshan P.O. Box 275-58 Beijing 102413 China Email: bfni@ciae.ac.cn

N. MOHAMED	Atomic Energy Authority (AEA); Egypt Second Research Reactor ETRR-2 13759 Abo Zabal, Kalubya Egypt Email: mnader73@yahoo.com
S. SUTISNA	National Nuclear Energy Agency (BATAN); R & D Centre for Materials Science & Technology P3IB Kawasan Puspiptek Serpong, Tangerang 15314 Indonesia Email: sutisna@batan.go.id
J. A. PRESTON	University of the West Indies; International Center for Environmental and Nuclear Sciences Mona Campus, Mona Road P.O. Box 104 Kingston 7 Jamaica Email: icens@uwimona.edu.jm
N. YUSSUP	Malaysian Nuclear Agency (Nuclear Malaysia) Bangi Komplek Selangor Darul Ehsan 43000 Kahang, Selangor Malaysia Email: nolida@nuclearmalaysia.gov.my
M BOUNAKHLA	Centre national de l'énergie, des sciences et des techniques nucléaires (CNESTEN) B.P. 1382 10001 Rabat, Agdal Morocco Email: moussabounakhla@yahoo.fr
P. BODE	Delft University of Technology; Faculty of Applied Sciences; Department Radiation Science & Technology Mekelweg 15 2629 JB Delft  Currently: NUQAM consultancy, 3284LK Zuid-Beijerland, The Netherlands Email: peter.bode@ymail.com
P. BEDREGAL	Instituto Peruano de Energia Nuclear (IPEN) Av. Canada N° 1470 – Lima 41 Peru Email: pbedregal@ipen.gob.pe
M. FRONTASYEVA	Joint Institute for Nuclear Research, JINR Joliot-Curie ulitsa 6 141980 Dubna, Moskovskaya Oblast Russian Federation Email: marina@nf.jinr.ru

B. SMODIŠ

Jožef Stefan Institute  
Jamova cesta 39  
1000 LJUBLJANA  
SLOVENIA  
Email: borut.smodis@ijs.si

A. SARHEEL

Atomic Energy Commission of Syria (AECS), Department of Physics  
Kafar Sousah, 17 Nissan Street  
Damascus  
Syrian Arab Republic  
Email: atomic@aec.org.sy

S. LANDSBERGER

University of Texas at Austin; Nuclear Engineering Laboratory  
PRC 159, 10100 Burnet Road  
Austin, TX 78712  
United States of America  
Email: s.landsberger@mail.utexas.edu



## ANNEX II: CONTENTS OF INDIVIDUAL PAPER CONTRIBUTIONS

All contributions are available on the attached CD-ROM.

<b>TOPIC 1: AUTOMATION IN IRRADIATION</b>		
Main author	Affiliation	Title of the paper
P. Bedregal	Instituto Peruano de Energía Nuclear. Subdirección de Investigación Científica, Lima, Perú	Design and manufacture of a sample changer made in-house for irradiation automation in neutron activation analysis
S. Landsberger	University of Texas at Austin, Nuclear Engineering Teaching Lab, Austin, Texas, USA	Maximizing utilization of neutrons at a research reactor by employing automation of irradiation and counting procedures for thermal and epithermal NAA, cyclic NAA and Compton suppression
B. Smodiš	Jožef Stefan Institute, Ljubljana, Slovenia	Automation of a pneumatic transport system for neutron activation analysis
<b>TOPIC 2: AUTOMATION AT SAMPLE CHANGER FOR GAMMA SPECTROSCOPY</b>		
Main author	Affiliation	Title of the paper
S.M. Hossain	Institute of Nuclear Science & Technology, Atomic Energy Research Establishment (AERE), Dhaka, Bangladesh	Development of an auto sample exchanger for gamma counting to enhance NAA capacity of BAEC
Bangfa Ni	Department of Nuclear Physics, China Institute of Atomic Energy, Beijing, China	Design of INAA automation at CARR
Sutisna	Center for Science and Technology of Advanced Material, Indonesia	Improvement of neutron activation analysis laboratory by applying the automation system
A. Sarheel	Atomic Energy Commission of Syria, Nuclear Engineering Department, Damascus, Syria	Automated sample changer system connected with gamma spectroscopy system
<b>TOPIC 3: AUTOMATION AT DATA HANDLING AND ANALYSIS</b>		
Main author	Affiliation	Title of the paper
J. Preston	International Centre for Environmental and Nuclear Sciences, University of the West Indies Mona Campus, Kingston, Jamaica	Development of standardized protocols for communication between various components of the NAA process
N. Yussup	Technical Support Division, Malaysian Nuclear Agency, Selangor, Malaysia	Development of process automation in the neutron activation analysis facility in Malaysian Nuclear Agency
M. Bounakhla	National Center of Nuclear Energy, Sciences and Techniques, Morocco	Development of a Modular Laboratory Information Management System (LIMS) for NAA laboratories using open-source developing tools

M. Frontasyeva	Joint Institute for Nuclear Research, Dubna, Russian Federation	Automation system for neutron activation analysis at the reactor IBR-2, Dubna, Russia
<b>TOPIC 4: OVERALL AUTOMATION</b>		
<b>Main author</b>	<b>Affiliation</b>	<b>Title of the paper</b>
M. Touiza	Nuclear Research Centre of Birine, Algeria	Achievement of routine automation OF NAA systems around Algeria research reactors
A. Stopic, J. W Bennett	Australian Nuclear Science & Technology Organisation, Lucas Heights, Australia	Final report of activities by ANSTO relating to the development of an integrated approach to routine automation of neutron activation analysis
M. Nader	Atomic Energy Authority, ETRR-2, 13759 Abuzabal, Egypt	Development of an integrated approach to routine automation of neutron activation analysis at ETRR-2
P. Bode	Delft University of Technology, Reactor Institute Delft Mekelweg 15, 2629JB Delft, Netherlands	Input for E-learning for laymen's use of automated neutron activation analysis
A. Dutra Neto1	CNEN-Comissão Nacional de Energia Nuclear, Brazil	Sample changer: for better neutron activation analysis efficiency at CDTN/CNEN, Brazil
L. Muñoz	Comisión Chilena de Energía Nuclear, Santiago, Chile	Updates and improvements in the automation of the neutron activation analysis laboratory in Chile

## ABBREVIATIONS

AAS	Atomic absorption spectroscopy
CLP4NET	IAEA cyber learning platform for network education and training
CNAA	Cyclic neutron activation analysis
CRM	Certified reference material
GUI	Graphical user interface
ICP	Inductively coupled plasma spectrometry
IT	Information technology
$k_0$ -NAA	Nuclear activation analysis with the $k_0$ method
LIMS	Laboratory information management system
MCA	Multi-channel pulse-height analyser
MOOC	Massive open online course
MOODLE	Modular object-oriented dynamic learning environment
NAA	Neutron activation analysis
PAC	Programmable automation controllers
PC	Personal computer
PLC	Programmable logic controllers
PTS	Pneumatic transfer system
QA	Quality assurance
QC	Quality control
RFID	Radio frequency identification
RR	Research reactor
SOP	Standard operating procedure
XRF	X ray fluorescence





## CONTRIBUTORS TO DRAFTING AND REVIEW

Barradas, N.P.	International Atomic Energy Agency
Bennet, J.	Australian Nuclear Science and Technology Organisation (ANSTO), Australia
Bode, P.	Delft University of Technology, Netherlands, currently NUQAM Consultancy, Netherlands
Landsberger, S.	University of Texas at Austin, United States of America
Nader, M.	Atomic Energy Authority, Egypt
Preston, J.	University of the West Indies, Jamaica
Ridikas, D.	International Atomic Energy Agency
Sharheel, A.	Atomic Energy Commission of Syria (AECS), Syrian Arab Republic
Smodiš, B.	Jožef Stefan Institute, Slovenia
Stopic, A.	Australian Nuclear Science and Technology Organisation (ANSTO), Australia
Yunikova, A.	ROSATOM CICE&T, Obninsk, Russian Federation

### **Research Coordination Meetings**

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## **RUSSIAN FEDERATION**

### ***Scientific and Engineering Centre for Nuclear and Radiation Safety***

107140, Moscow, Malaya Krasnoselskaya st. 2/8, bld. 5, RUSSIAN FEDERATION

Telephone: +7 499 264 00 03 • Fax: +7 499 264 28 59

Email: [secnrs@secnrs.ru](mailto:secnrs@secnrs.ru) • Web site: [www.secnrs.ru](http://www.secnrs.ru)

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