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Quality aspects of research reactor operations for instrumental neutron activation analysis

Report of an Advisory Group meeting held in Accra, Ghana, 18–22 October 1999

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QUALITY ASPECTS OF RESEARCH REACTOR OPERATIONS
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FOREWORD

This publication is intended to provide guidance on quality aspects of research reactor utilization with emphasis on neutron activation analysis (NAA). It is written to provide users with the practical information required to improve their work and to help reactor staff understand the quality assurance requirements that users need from their facilities.

The need for such a publication was identified during a co-ordination meeting of an IAEA African Regional Project on Research Reactor Operation, Safety and Utilization held in Algiers, Algeria, 12–14 April 1999. Subsequently, an Advisory Group meeting was held in Accra, Ghana, in October 1999 to draft this report. Group members comprised experts from Algeria, Egypt, Ghana and South Africa working under the guidance of P. Bode of Delft University of Technology, Netherlands. While the report is intended to take into account the situation of research reactors in Africa, it should also be of value to other facilities.

This publication is applicable to the establishment and implementation of quality aspects at various stages of the utilization of research reactors with emphasis on NAA. It is not intended to be complete and it should be considered as a stepping stone for improvement. It includes references to other documentation that is readily available. This TECDOC provides guidelines for practical quality assurance in the areas of reactor operations and facilities, preparation for irradiations, the irradiation process and conduct of analyses. It also covers areas of general consideration in quality management and includes recommendations for monitoring, registration, correction and prevention for the effective implementation of the programme. It is expected that the guidelines in this report will also be useful in establishing effective co-operation between reactor operators and experimenters for improved and more reliable utilization.

The IAEA officer responsible for this publication was B. Dodd of the Division of Physical and Chemical Sciences.

EDITORIAL NOTE

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CONTENTS

1. THE INTERFACE BETWEEN REACTOR OPERATION AND REACTOR UTILIZATION

Research reactors are the main suppliers of the neutrons needed for neutron activation analysis (NAA). In many institutes, especially those with low power reactors (thermal power <250 kW), NAA may be the only application for which the reactor is being used. It is often generally stated that NAA is a 'mature' method, well understood and well documented via numerous publications in refereed scientific journals, textbooks as well as publications issued by the IAEA. Many of these documents deal with the identification of sources of error and provide practical solutions to prevent them and/or to apply proper corrections.

Many NAA groups are requested to identify beneficiaries (customers) for their technique and are encouraged to start 'commercial' activities. The principal reasons are budget cuts and/or the fact that (industrial) customers are considered important for public justification of the existence and continuation of the reactor. Several laboratories have already identified a problem of acquiring credibility with intended beneficiaries because of the lack of objective evidence on the quality and reliability of their operations. The IAEA, through its Analytical Quality Control Services (AQCS), provides NAA laboratories with certified reference materials for quality control purposes. However, intercomparison rounds still demonstrate poor performance of some NAA laboratories. This may indicate that there is still insufficient awareness and/or control of potential sources of error that may affect the accuracy of the results.

There may be several reasons why mistakes are still being made during the analysis process. These could include the need to determine very low element levels and the implicit problems of contamination, blank and/or element loss, insufficient insight into the correct operation of equipment and principles of the software in use, as well as the wrong interpretation of quality control and quality assurance concepts. In addition, NAA requires a co-ordination of the activities of the experimentalist and the provider of the neutrons, viz. the reactor operations group. Sometimes the latter group is insufficiently aware and/or not well informed of the requirements of the NAA group with respect to the conditions under which irradiations have to be carried out. This in turn may ultimately lead to wrong results.

Quality practices and quality assurance systems are increasingly implemented throughout the world in nuclear research facilities, both in reactor operations and management as well as in analytical laboratories. Often, these developments follow separate paths, partly because the basis of the quality systems is different (viz. ISO-9000 series of standards for reactor operations and ISO/IEC Guide-25 for analytical laboratories). As a result, there may be a mismatch at the interface of these communities; a crucial interface where the supplier and user of the neutrons meet.

This publication aims to serve as a tool for both the supplier of neutrons, the reactor operations group, and for one of the users of neutrons, the NAA group. Its purpose is to enable them to understand each other's requirements regarding quality assurance for their individual activities. This in turn will identify where and how these requirements interact and how they have to be tuned to one another.

This publication is not intended to provide a complete listing of all steps and potential sources of error for which quality assurance has to be introduced to reduce the probability of mistakes. However, it may be consulted if quality systems have to be developed both in reactor

operations and in utilization of the reactor, or for verification and fine-tuning of this interface between existing quality systems. To this end, typical sources of problems and mistakes have been identified for the following:

— Reactor operations and instrumental NAA (INAA) facilities;
— Preparation for irradiation;
— Irradiation process;
— Conduct of analyses.

Recommendations are given for the implementation of quality assurance in order to reduce the probability of occurrence of problems or their negative effects on the final quality of the analysis. Where relevant, references are given to further useful material on a particular topic.

2. BASIC PRINCIPLES AND ACCURACY OF INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS

2.1. Overview of neutron activation analysis

Neutron activation analysis allows for the qualitative and quantitative determination of elements. The method is based upon the conversion of stable atomic nuclei into radioactive nuclei by irradiation with neutrons and the subsequent measurement of the radiation released by these radioactive nuclei. Amongst the several types of radiation that can be emitted, gamma radiation offers the best characteristics for the selective and simultaneous determination of elements.

By neutron activation, radionuclides may be produced from all elements present in the sample, albeit at sometimes strongly different production rates. This mixture of radioactivity can be analyzed in two broad ways:

— The resulting radioactive sample is chemically dissolved, and by chemical separations the total number of radionuclides is split-up into many fractions each having a few radionuclides. This is called destructive or radiochemical neutron activation analysis (RNAA) and will not be discussed further in this publication.
— The resulting radioactive sample is kept intact, and the radionuclides present are determined by taking advantage of differences in the decay rates and measuring the samples at different decay intervals, utilizing equipment with a high energy resolution for gamma radiation. This is called non-destructive or instrumental neutron activation analysis (INAA).

An INAA procedure is characterized by (i) activation via irradiation with reactor neutrons, (ii) measurement of the gamma radiation after one or more decay intervals, and (iii) interpretation of the resulting gamma ray spectra in terms of the elements present and their concentrations. Many textbooks can be consulted on the underlying principles and analytical characteristics of INAA [1–3].

INAA has found its usage in many fields of science. Particular advantage is taken of the fact that the samples do not have to undergo any chemical treatment, neither prior, nor after the activation. It is in this sense that INAA is 'non-destructive'. In addition, light elements such as H, C, N, O, Si which in many materials belong to the major matrix components, do not

produce radioactive products upon neutron activation. This means that they cannot interfere with the determination of the other activities. This often enables the observation of trace elements at detection limits in the $mg.kg^{-1}$ to $\mu g.kg^{-1}$ level in matrices composed of these light elements. In addition, the high selectivity of gamma ray spectrometry allows for simultaneous evaluation of many radionuclides.

The non-destructive character of INAA makes the technique attractive for application in geochemistry and related sciences. Because of the limited sample handling operations, there is also a lower risk of contamination compared to element analysis methods in which the sample has to be dissolved. This advantage has been exploited in many biological applications of INAA and for the analysis of minute quantities of material such as atmospheric and cosmic dust.

Since the signals in INAA are related to the properties of the atomic nucleus, the results in INAA are not affected by the chemical and physical state of the elements. The method is well described by physical laws and selectivity is unambiguous for all elements. The reason is that the combination of the nuclear properties such as the decay constant (often converted to half-life) and the energies and intensities of the gamma radiation is uniquely characteristic for each radionuclide. This all contributes to a high degree of accuracy that makes INAA well acknowledged for analyses related to such areas as the certification of reference materials.

2.2. Accuracy of INAA

The factors affecting the accuracy in INAA are briefly highlighted in the following paragraphs. Accuracy is defined as the closeness of the agreement between the result of a measurement and a true value of a measurand [4].

2.2.1. Accuracy of qualitative analysis

The accuracy of the QUALITATIVE analysis, viz. the identification of the target elements is governed by the quality of the catalogues of radionuclide-related gamma ray energies and intensity ratios, and the knowledge of the nuclear transformations involved in the production routes. The qualitative accuracy may be affected such factors as: peaks being assigned to erroneous radionuclides due to instrumental drift of the spectrometer from the calibration conditions; gamma ray spectrum interferences (including narrowly spaced peaks, sum-peaks and self-absorption phenomena); and under-estimations of interfering nuclear reactions.

Corrections can be made for many of these effects, including the instrumental drift problem. Multiple peaks instead of single peaks can be used for nuclide identification. Multiple radionuclides can be considered for element identification and escape energies and coincidence sum-peaks can be employed and compared to the gamma ray catalogues.

In principle errors may be made by assigning a single gamma ray peak to one or more, wrong nuclides or wrong elements. However, the number of nuclides for which such a situation may occur is very small and is reduced to zero when also differences in half-life are taken into consideration. Additionally, errors in element assignment may occur due to interfering production reactions.

The assignment of gamma ray lines to radionuclides can be considered to be possible with a very high degree of accuracy. The accuracy of the final identification of the target elements

depends on the knowledge of the production mechanisms, the interfering reactions, and the ability to discriminate between other potential target elements.

2.2.2. Accuracy of quantitative INAA

The degree of accuracy in the QUANTITATIVE analysis has to be assessed by how closely the concentrations obtained by analysis approximate the true concentration values. Since the analytical results are in fact estimates of the true values, the results have to be accompanied by a statement of uncertainty. The sources of error contributing to this uncertainty can be rather completely assessed in INAA since many aspects of the procedure (activation, decay, measurement) are described by well defined physical laws. Traditionally, three types of errors can still be distinguished as affecting the final concentrations:

(1) *Random errors.* These vary in sign and magnitude and are unpredictable. Since they have a statistical character, they affect the precision of the analysis. Examples are differences in neutron flux between sample and standard caused by badly defined positions during irradiation, sample inhomogeneities and counting statistics.
(2) *Systematic errors.* Systematic errors are always of the same sign and magnitude, and produce bias between the obtained result and the true value. Systematic errors thus affect the accuracy of the analysis. Examples of sources of systematic error are contamination, moisture content, erroneous standardization, dead-time and pile-up losses, differences in geometry between sample and standard, errors in the photopeak efficiency of the detector, blank and natural background corrections, neutron self-shielding, neutron self-moderation and gamma ray self-absorption, as well as errors in half-lives.
(3) *Additional errors.* These may include the choice of the wrong method for drying with consequent loss by volatilization, erroneously entered sample weights, contamination, or errors made during spectrum analysis. These errors may either have a random or systematic character.

The type of errors related to sample preparation (contamination, element loss due to volatilization, incomplete drying) are not unique for INAA but are applicable to any method of chemical analysis. In this respect INAA has the additional advantage that the samples do not require any pre-treatment such as dissolution or mixing with an inert material for pelletizing, thus largely eliminating errors due to the blank, contamination and losses. Errors due to neutron- and gamma ray self-absorption as well as neutron self-thermalization can largely be allowed for and can eve be neglected when processing small samples. Since the principles of INAA are based on effects taking place in the atomic nucleus, the results are not affected by the chemical or physical state of the elements. Errors made in standardization have often the largest effect on the accuracy of INAA. This source of error includes errors due to unknown purity and stoichiometry of chemical compounds, standard preparation errors (e.g. inaccuracy in pipetting and similar errors as mentioned with sample preparation).

An estimate of the accuracy of INAA can be obtained by comparison of the results of an analysis of certified reference materials with the data in the certificates. The accuracy is expressed as a standardized difference *'z'* thereby taking into account the uncertainties of the obtained result and the uncertainty in the certified value:

$$z_i = \frac{C_i - C_{ref,i}}{\sqrt{\sigma_i^2 + \sigma^2_{ref,i}}}$$

in which:

C_i , σ_i = the observed concentration and its uncertainty respectively
$C_{ref,i}$, $\sigma_{ref,i}$ = the 'actual' concentration and its uncertainty in the reference material.

However, for quite a few elements there are no reference materials available with certified concentrations. If this is the case for an element under consideration, an indication of the degree of accuracy may be obtained by participation in laboratory intercomparison rounds or in proficiency testing.

3. BASIC PRINCIPLES OF QUALITY MANAGEMENT, QUALITY ASSURANCE AND QUALITY CONTROL

3.1. Quality management

Management policies, strategies and responsibilities should be clearly defined for development, implementation and monitoring of a quality assurance programme. The quality assurance programme should provide an interdisciplinary approach involving all of the organizational components.

Senior management should ensure that the organizational structure, functional responsibility levels of authority and interfaces (locally and internationally) for those managing the various levels of work are adequate. Effective measures should also be implemented to verify effectiveness and continuous improvement of the quality management programme as well as integration with all nuclear related systems such as environmental management, radiological protection, safety and security.

3.2. Quality assurance

Quality assurance is all those planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality [4]. For the analytical laboratory, quality assurance describes the overall measures that a laboratory uses to ensure the quality of its operations. For example, typical items involved with quality assurance are: quality control, appropriate equipment, trained and skilled staff, documented and validated procedures, requirements for calibration, standards and reference materials, traceability, proficiency testing, non-conformance management, internal audits, and statistical analysis.

Commitment towards the quality assurance system should be visible through all levels of staff, since they need to 'buy into' the system. Strategic planning is necessary to define direction and an organization's performance as well as the work performance and effectiveness of management also need to be subject to the system. Quality systems should be audited at regular intervals to determine the level of maintainability, effectiveness and implementation. The quality assurance systems should provide the individuals performing their work with the necessary tools, support and encouragement to perform their allocated responsibilities properly.

3.3. Quality control

Quality control is the operational techniques and activities that are used to fulfil the requirements of quality [4]. The basic quality control principle is to ensure that the processes or measurement methods implemented in day-to-day work activities is sufficient to assure conformance to specified requirements. These would include such things as the customer needs, nuclear safety and regulatory requirements. Personnel should be trained in the work procedures, statistical control processes and facilities in order to control work activities effectively. Documented evidence should be kept for all activities such as reactor operations, statistical analysis, safety analysis, design and changes in design, and quality of irradiated material to clearly indicate the quality of the product and work performed. For the analytical laboratory, quality control implies analysis of reference materials, blind samples, blanks, spiked samples, duplicates and other control samples.

4. GUIDELINES FOR PRACTICAL QUALITY ASSURANCE

4.1. Reactor operations and INAA facilities

4.1.1. General requirements

A good collaboration between the reactor operator and the experimentalist is important for the safe operation of facilities. Start-up of the reactor or the experimental devices, removal of radioactive equipment from the reactor and other operations are apt to cause difficulties if the experimentalist and operators do not keep each other informed.

As at all levels in quality management, responsibilities should be well defined. The reactor management is, depending on the organizational structure, responsible for:

— safe operation of the reactor and its facilities;
— scheduling and control of reactor utilization;
— all safety aspects of the preparation and implementation of a modification or experiment.

For every experiment, including irradiations for INAA, the reactor operations group and the experimentalists must be aware and have available information necessary for the safe operation of the experiment. This includes information which may be needed in the event of a safety problem or operating difficulties with the experiment or with the reactor. This requires preparation of a safety evaluation/report that should be reviewed by the radiation protection officer and approved by the reactor safety committee, as applicable. The same officials also have to approve modifications or changes in the status of the experiments or irradiations.

Reactor operators should have a list of personnel trained and qualified for the various tasks associate with sample irradiations such as packaging of samples for irradiation and loading/unloading of the irradiation facilities (e.g. rabbit systems). Moreover, there should be a list available of persons to contact if difficulties arise.

An operating schedule needs to be issued on a regular basis to indicate the operating cycles of the reactor and its associated flux and/or power levels. Since INAA activities may require timely planning of irradiations, a schedule covering several months is highly preferable.

Again, good communication is needed to harmonize the schedules of the reactor, the experimentalist and the detectors.

It is important to announce through a public address system what the status of the reactor is, e.g. “starting-up”, “at a power level of”, “shut down”. The use of warning lights or other visible signs in experimental areas are desirable to indicate that the reactor is operating.

4.1.2. Characterization of irradiation positions

An evaluation is required for the thermal neutron flux in the irradiation positions but also for the epithermal and fast neutron fluxes. This neutron spectrum characterization can be performed via multi-foil irradiation such as with Au + Zr, Ge, Fe (Mn) foils [5–15]. Characterizations should be made for each core configuration as well as for various power levels and as a function of operating time. Axial and radial gradients should be determined. An indication of the gamma ray heating can also be useful to the user. Characterizations should be repeated as soon as possible after each change of the reactor-core configuration.

Recommendations for good QA:

— Stock control of flux monitors and characterization thereof (coding, masses, etc.).
— Standard operating procedure (SOP) for neutron flux monitor irradiations and measurements. Here, the INAA group may be involved as well.
— SOP for calculating the flux and spectrum characteristics and for the use of unfolding codes.
— Suitability of flux monitors for the purpose of measurement.
— Planning of irradiation and measurements after core changes.
— Record keeping of critical variables and experimental conditions.
— Calibration status of control rods.
— Adjustment of water level and water temperature.
— Communication with users.

4.1.3. Pneumatic transfer systems

A pneumatic transfer system is used for relatively short irradiations (up to several hours) and if short transfer times are needed. The systems vary with different reactor types. Irradiation positions may be located in the reactor’s reflector (water, beryllium or graphite), inside a beam port or in between the fuel elements (in-core position). The system should be operated, characterized, and maintained in accordance with appropriate procedures and checklists (see Annex II). It is important to anticipate problems such as a stuck capsule or melting and rupture of a sample encapsulation.

Recommendations:

— Regular inspection and maintenance should be performed.
— Correct reactor operating parameters should be adjusted according to established values for in-core positions.
— Facility operators should be qualified and trained to operate the pneumatic transfer system according to a standard operating procedure.
— Reactor and facility parameters (i.e. pressure, speed of capsule) should be recorded at regular interval in the logbook and compared with previous results.

— Radiological protection personnel should evaluate and establish the necessary radiation levels and advise according to the established radiobiological protection programme.

4.1.4. Reactivity effects of in-core experiments

The safety evaluation of an in-core experiment should include an evaluation of the reactivity worth of the experiment. This evaluation needs to also account for any changes in reactivity worth by accident, failure or neutronic changes during the period that the experiment remains in the core. The main safety consideration is that the induced reactivity worth should not alter the overall excess reactivity of the core greater than the shutdown margin or lead to an impermissible reduction of reactor power.

Recommendations:

— Samples should not contain any absorbers such as cadmium, or boron in quantities that can alter the overall reactivity of the reactor. This must be controlled by the reactor safety committee.
— Information on material containing absorbers must be forwarded to the reactor operation personnel prior to irradiation so that they may take the relevant corrective/preventive action.
— The reactor shutdown margin must be taken into consideration for abnormal changes.

4.1.5. Pressure build-up due to radiolysis

Radiolysis is the effect in which H_2O decomposes into H_2, O_2 and H_2O_2 under the influence of gamma radiation. Radiolysis may also lead to decomposition of proteins into gaseous compounds. The gases produced by radiolysis cause pressure build-up in the irradiation container. Because of the explosion hazard, samples are preferably dried prior to irradiation. However, it is still important to have an indication of the maximum pressure that can be expected when irradiating unknown products that may be expected to undergo radiolytic decomposition. Such an indication can be obtained by irradiating in quartz ampoules, for increasing irradiation times and measuring the pressure in the ampoule after the irradiation by breaking the ampoule. An alternative is to perform test irradiations with de-ionized water as the sample in order to demonstrate the effectiveness of a particular encapsulation. Typically, this is done for a greater integrated flux than that for which it will eventually be used. Leaving a significant volume inside the encapsulation for expansion also helps reduce the pressure.

Recommendations:

— SOP prescribing under which conditions pressure build-up measurements should be done.
— A facility available for the measurement of pressure build-up due to radiolysis.
— Development of a reference document with a history of all irradiated substances for which pressure build-up measurements have been carried out.
— A document detailing the tested and approved encapsulation methodologies and their constraints.

4.2. Preparation for irradiation

4.2.1. Impurities in irradiation containers or sample encapsulation

The presence of impurities in irradiation containers contributes to the problems associated with blanks and thus will affect the accuracy of the analysis. Therefore impurities should be evaluated by irradiating and analyzing several of the capsules.

Recommendations:

— Perform a test analysis before procurement and/or receipt of a new production batch.
— Develop a control chart.
— If possible keep containers thoroughly washed with appropriate reagents e.g. for polyethylene use dilute HNO_3.
— Make an inventory list of the various batches and implement a procedure for stock control.

4.2.2. Estimation of induced radioactivity and radiation dose rate

For radiation protection purposes it is important to estimate the induced radioactivity and associated dose rate after irradiation. This necessitates having a fair knowledge of the composition of the sample. It is also important to have an estimate of the maximum amount of radioactivity that may be induced in case a container gets stuck in the irradiation position.

Recommendations:

— Get the best composition information available from customer or have the customer/experimenter perform the calculations.
— Estimate induced radioactivity utilizing appropriate tabulations [7], [16].
— If is information not available, carry out test irradiations at a lower neutron fluence.
— Develop an in-house database with information on observed induced radioactivity in typical samples to facilitate future predictions.

4.2.3. Encapsulation of liquids

There is the need to develop a procedure for leakage testing of containers used for liquid samples. This test may also be of use if powdered material is being irradiated, since it could easily result in dust-like contamination if the capsule leaked. As discussed earlier, longer irradiations with de-ionized water as the liquid provides a safe testing methodology.

Recommendations:

— Submerge the capsule in hot water to test for leakage.
— If the container contains acetous or alkaline liquids, a wipe test with pH paper may also indicate leakage.
— Test capsule sealing methodology with aqueous irradiations.
— Improve the heat-sealing procedure if leakage is found.

4.2.4. Contamination of capsules

Heat sealing of plastic containers is necessary to prevent accidental opening during irradiation and handling as well as for use in pneumatic facilities. This is important not only for radiation protection purposes, but also to ensure that there is no loss of sample mass. This sealing is often done using conventional soldering tools, but this may introduce impurities in the containers.

Recommendations:

— Heat seal polyethylene capsules with quartz tools. Clean the capsule after sealing.
— If possible, transfer the sample after irradiation into a clean capsule and re-weigh the sample remainder.

4.2.5. Moisture content

The moisture content of a sample needs to be determined because samples usually have to be dried prior to analysis. Moreover, control samples such as certified reference materials also typically have to be dried in accordance with the prescribed procedures in the certificate.

Recommendations:

— Determine the moisture content of the reference material by weighing before and after drying in accordance with the procedure in the certificate. Note that the moisture determination always has to be done on a sample that is different from the one to be analyzed. Note also that the certificate typically also includes the correct use of the reference material, and sometimes the drying procedure prior to analysis may be different.
— Determine the moisture content of the samples in the same way, and treat them, if applicable, in the same way as the control material.

4.2.6. Moisture uptake during weighing

Some samples are hygroscopic and are likely to take up moisture during weighing. This can be monitored by the change in mass during weighing

Recommendation:

— To correct for this change in mass there is a need to record the weighing time for each sample and extrapolate them to the same point in time for all samples.

4.2.7. Evaporation losses during drying

Certain elements that are volatile may be lost during drying (As, Se, Halogens, Sb, Hg).

Recommendations:

— Inspect the recommendations in the certificates for use and drying of reference materials of similar matrix composition.
— Inspect scientific literature [17].
— Use common sense.

4.2.8. Quality of homogeneity

Often the amount of material received from the customer is much larger than the analytical portion to be used. This implies the need for sub-sampling and often necessitates sample-size reduction. To ensure the quality of homogeneity of samples homogeneity tests should be carried out.

Recommendations:

— About 6–10 replicate samples should be analysed.
— Statistical test should be applied to the analyzed data.
— If necessary, apply a different homogenization procedure.
— Propagate the variance in the replicates towards the uncertainty of the entire analysis.

4.2.9. Quality of standards

In order to verify the purity and the stoichiometry of the standards used it is necessary to obtain the requested information from the supplier. Purity as given in certificates is often based on the total level of measured impurities and often the detection limits for the unmeasured elements are not included in this. If impurities are specified with an uncertainty, it can be confusing as to what this uncertainty really means.

For neutron activation analysis it is important to realize that in some cases the isotopic abundance of elements may be different from the values, specified in references such as the Chart of Nuclides. For example this is true of Li, B, S, Ca and U. It is well known that almost all commercially available U-compounds are depleted in ^{235}U. Fission product corrections on basis of a standard derived from such a compound may thus lead to incorrect results.

Recommendations:

— There should be a careful selection of chemicals (see Ref. [17]).
— Acquire an indication of the quality stated on the purity certificate before procurement.
— Be alert for differences in isotopic composition.
— If purity certificates can be made available, obtain information on the meaning of the specified purity and/or uncertainty. Do not forget to ask for detection limits in the analysis method applied.
— Propagate the uncertainty in the purity towards the uncertainty of the entire analysis.

4.2.10. Quality of the balance used

There are many ways to distort the proper functioning of a balance, which rapidly may lead to wrong results because of non-linearity or time-dependent changes. This is in particular true if samples and standards are not weighted within a short time interval, or if the samples and the standards are weighed in containers with significantly different masses.

Recommendation:

— The balance should be routinely inspected and calibrated to obtain an accurate indication of the weighed masses.
— A functional check should be made before work is commenced.

— Use control masses for inspection and for development of control charts to inspect for drifting with time.
— Eventually, have the balance calibrated by a body, officially certified to calibrate balances. Some manufacturers have such a certification (ISO-9000 series).

4.2.11. Pipette calibration

Pipettes are often used as a gravimetric tool rather than as a volumetric instrument. However, in the latter case, the reliability of the readings from the pipettes depends on the accuracy of its calibration. This can be verified by analyzing internal quality control samples.

Recommendations:

— Take 6–10 readings with the pipette of the same volume of an internal quality control sample and apply statistical analysis to obtain a control chart.

4.2.12. Effect of differences between samples and standards

In almost every irradiation facility there are axial and radial neutron flux gradients. In addition, the neutron spectrum (ratios of the fluence rates of thermal neutrons over epithermal and fast neutrons) may also vary with time and space. These changes can result in differences in the activation rate between a sample and a standard for which corrections need to be made. Similarly, if samples and standards differ greatly in physical geometry errors may be introduced by assuming they are the same. Finally, differences in activation rate may result from variations in the concentrations of strongly neutron absorbing components such as B, Cd and Gd.

Recommendations:

— Make an experimental verification of the neutron flux gradients and correct for the experimental results.
— Make an experimental verification of the neutron spectrum using triple monitors [5]. Correct where applicable (review the contribution of the epithermal and fast neutron activation using tabulations, such as those in Ref. [18].
— If applicable, make a series of measurements with samples and/or standards of different physical size.

4.3. The irradiation process

4.3.1. Irradiation requests

The user should complete an irradiation request containing the relevant information needed for the facility used, sample characteristics, container/capsule contents and obtain the necessary approval of the applicable control personnel (see Annex II) The reactor safety committee may review the request.

Recommendations:

— The measured activation based on initial dose rate must be compared with the required activity.

— A significant deviation should be regarded as a non-conformance report (see Annex II).
— Confirmation on neutron flux levels and the duration of the irradiation should be recorded.
— A visual inspection of the holder/sample should be performed (i.e. inspect for things such as physical damage, leaks, or cracking) before commencement of the irradiation or loading the sample/holder.
— Coincidence between the reactor operation schedule and the irradiation programme.
— Any abnormal situation during the operation should be reported as a nonconformance report.

4.3.2. Integrity and identification of sample holders

Each sample holder should be uniquely and clearly identified. Samples should be handled carefully in order to prevent damage and/or deformation, which could lead to a sample getting stuck. Also, the material of the holder must not contain or be contaminated with foreign material or impurities, which could affect the reactivity of the reactor during operation.

Recommendations:

— Instructions should clearly prescribe the orientation and handling conditions of samples.
— If the irradiated sample could release airborne contamination a handling process to prevent this release should be developed (e.g. by keeping material in a leak-tight container or by providing a system with negative pressure or with filters).
— A careful inventory system should be kept of the material samples, equipment and the devices put into the reactor. These will need to be retrieved and accounted for at the end of an irradiation.

4.3.3. Routine and non-routine samples

A list of routine samples should be prepared in advance, which needs to contain information about the physical properties in material. Samples contained in this list could be evaluated and receive generic approval by the reactor safety committee.

For non-routine samples, a request would need to be submitted to the reactor safety committee for approval or necessary modification in accordance with the irradiation request form. In this manner, non-routine samples are required to be subjected to a full safety evaluation, which addresses all the salient safety considerations such as cooling, post-irradiation handling and test irradiations.

Recommendations:

— A list of approved samples should be distributed to the various laboratories as well as reactor operational staff.
— The list should be regularly updated for completeness.
— In the event of any problems or difficulty experienced, a non-conformance report should be issued.

4.4. Conduct of analysis

4.4.1. Synchronization of clocks

To accurately correct for radioactive decay it is important to know the synchronization of the clocks used for registration of the start of the irradiation time and its duration as well as the start of the counting. Differences in timing manifest themselves by erroneous results since the decay correction is not accurate. This especially applies to short half-live radionuclides.

Recommendations:

— Verify the synchronization and if necessary, apply corrections. Document the correction factor in the relevant SOP and, if applicable, introduce it into the quantitative analysis process (e.g. analytical spreadsheet).
— Develop SOPs for regular verification of the synchronization (e.g. once per month or if needed upon every irradiation) and keep a record in the log books.
— Document in an SOP what times should be recorded during the complete experiment cycle and how precise they need to be. Agree if summer-time or winter-time is recorded.
— If power failures cannot be excluded, avoid clocks that run on AC power. Try to use clocks running on batteries but then develop a SOP for preventive, timely, replacement of the batteries.
— Consider using clocks that are adjusted via radiofrequency to a transmitter, based on and/or traceable to an atomic clock.

4.4.2. Sample and standard physical dimensions

In comparative INAA, often elemental standards are made by pipetting small quantities onto filter paper. Thus the physical geometry of the standard does not necessarily match the geometry of the real sample and errors will be introduced if counting close to the detector's end cap. Such effects will also manifest themselves in the results of internal control samples.

Recommendations:

— Develop an SOP for the preparation of the sample and standard in the same geometry, for instance by pelletizing.
— Document in the SOP what should be recorded and where it should be recorded. This should include such factors as the counting geometry and the filling heights of sample and standard.
— Develop an SOP that prescribes under which conditions counting should be done at larger source to detector distances
— Document in the SOP that the standard should be flipped half way through the counting if the counting is being done at short sample-detector distances and if the sample and standard do not have matching geometries.
— Make a filling height correction curve and apply the corrections in the quantitative analysis process.

4.4.3. Ill-defined geometry of sample and/or standard

Sometimes the sample material may be distributed randomly throughout a (plastic) encapsulation due to electrostatic effects or because only very little material is available (e.g.

mineral fragments). Similarly, a filter paper may be not well fixed in a capsule. This all results in errors if counting close to the end-cap.

Such a situation can be inspected visually and manifests itself, in the case of problems with the standard, in wrong results in the control sample.

Recommendations:

— Use control charts to inspect the results of the control sample. Apply the Westgard rules for objective decisions [19].
— Document in an SOP which observations on the sample should be recorded and where. These can include problems with well defined distribution in the capsule and electrostatic effects during sample preparation and counting.
— Develop a technique, and document it in an SOP for fixing filter papers within capsules, e.g. by using inert filling material.
— Develop a technique, and document it in an SOP for fixing small pieces of material in a well defined position in a capsule (before irradiation), e.g. by using glue.
— Develop an SOP that prescribes when (e.g. in case of electrostatic behavior) and how capsules should be flipped 180^{o} half-way through the count if using vertical dipstick style detectors.
— Develop an SOP that links the size of the capsule to the amount of material to be analyzed, and if necessary, use smaller capsules.

4.4.4. Dead-time effects

Sometimes the live-time correction circuit in multi-channel pulse height analyzers (MCA's) does not work properly in combination with pile-up correction circuits or if using a pulse-generator. This effect is more dominant at higher dead-times/count-rates. It will manifest itself by wrong results for all elements in the control sample.

Recommendations:

— Develop an SOP for inspection of the proper operation of the dead-time correction circuit using the multiple sources method. Use, if available, ^{57}Co and ^{60}Co at a fixed position to the detector and vary the total dead time using an additional ^{137}Cs source. Plot the various peak areas in a control chart.
— Develop an SOP that prescribes under which conditions, in case of unknown samples, test analyses should be done to inspect for the amount of induced radioactivity and count rate to be expected. Use the results to decide on the analysis protocol. Options are irradiation of smaller portions than usual or for shorter irradiation periods. Another option is to irradiate at a lower reactor power level.
— Include as corrective action in the relevant SOP that, if the sample is much more radioactive than anticipated, the sample should be measured at larger source/detector distances (lower dead-time) than originally planned.

4.4.5. Variable count rate during counting

If short half-life radionuclides are used for INAA, the total activity may decay significantly during the measurement. The live-time correction of the MCA will then not be correct. The problem is that this effect may be different for the sample, the standard and the control

sample. It may manifest itself in erroneous results for the control sample, but it may not necessarily be obvious. Therefore, for short half-life radionuclide INAA, the user has to be alert for such effects.

Recommendations:

— Include in the relevant SOP that the dead-time should be recorded at the beginning and at the end of the counting, and/or the deadtime at the beginning of the count compared with the deadtime printed by the MCA.
— Use common sense in the planning of the experiment. Estimate which radionuclides will have the main contribution to the total radioactivity, check their half lives and compare these with the planned measurement time. If the half live of the dominant radionuclide(s) is shorter or comparable to the duration of the measurement, problems may be anticipated.
— Develop an SOP for application of mathematical routines to correct for these effects [20].
— Consider the procurement of a loss-free counting module (dead-time stabilizer).

4.4.6. Pile-up corrections

Pile-up at high count rates is the consequence of the spectrometer's electronics being unable to distinguish successive pulses anymore. This is evidenced by elevation of the underground in the gamma ray spectrum at high gamma ray energies.

Recommendations:

— Include in the relevant SOP that such observations would be recorded on a non-conformance form.
— Check the operation of the pile-up correction circuit in the amplifier (see the manual of the amplifier).
— Consider the use of a pulse-generator to correct for this effect [21].
— If necessary and if possible, consider using a radionuclide at a fixed position to the detector for pile-up evaluation. This radionuclide should have a long half life (e.g. ^{137}Cs), with a non-interfering gamma ray line and with not too much effect on the Compton background of the spectrum. The peak area of this gamma ray line can then be used as a time-base for the measurements.
— Consider measuring the sample at lower count rates or at larger source-detector distances than originally planning. Such an action should be documented in the relevant SOP as a corrective or even preventive action.

4.4.7. Co-incidence or cascade summing effects

An activated sample will usually contain various radionuclides, typically more than will be produced by irradiating the standard. As a result, the gamma ray spectrum of the sample will be more complex. If counting in a geometry close to the end cap, coincidence summing effects of certain radionuclides may result in a sum peak interfering with the gamma ray line of interest in the spectrum of the sample, but not in the spectrum of the standard. This may manifest itself in the spectrum analysis (poor fit results, doublets), but not necessarily in the spectrum of the control sample.

Recommendations:

— It should be noted that the absolute efficiency of the detector is the determining factor, and not the distance of the sample to the detector. In other words, effects may be observed for example if counting at 3 cm in front of a 10% Ge detector and not at 10 cm distance, but they may be observed again if counting at 10 cm distance from say a 50% Ge detector.
— Use tabulations of gamma ray lines with sum-peaks [22], or check decay schemes to inspect for potential interferences.
— Prescribe in the SOPs which radionuclides should be considered for measurement at larger sample-to-detector distances (e.g. ^{82}Br interference on the ^{60}Co line at 1173 keV)
— Prescribe, via documentation in the SOP, which other interference-free gamma ray lines should be used for quantification.
— Develop an SOP for application of a mathematical coincidence correction [23].

4.4.8. Gamma ray self-absorption effects

Gamma ray self-absorption occurs in all samples, but becomes more dominant in materials with high average atomic numbers (such as minerals or mineral separates) and materials with a relatively high density. The effect is usually mainly of importance in the energy region below 200 keV. Self-absorption effects are manifested by a change in the intensity ratio between the low energy and high energy peaks of a given radionuclides.

Recommendations:

— Develop an SOP for generating a control chart of intensity ratios of non-perturbed peak areas of radionuclides with gamma ray energies in the region below 150 keV and above 500 keV.
— Check the observed intensity ratios with these control charts and apply Westgard rules [19].
— Include in the SOP dealing with the analysis methodology a request that as much information as possible should be obtained from the customer on the expected composition of the sample before planning the analysis. Then use common sense, for example deciding to use smaller sample masses.
— Develop an SOP for the experimental determination of the effective mass attenuation coefficients and apply corrections. This may be done by irradiating the suspected material, and measuring it with and without an inactive portion (of known thickness) of the same material between the radioactive material and the detector. The resulting mass attenuation coefficients can be used to calculate the self-absorption correction factors (see Ref. [24]).

4.4.9. Background, shielding and contamination problems

If low level measurements have to be carried out, peaks occurring in the background spectrum may have a significant effect on the detection limits. The background is affected by the occurrence of sources in the counting room, reactor operation or other radiological activities in the surrounding areas and by natural radioactivity such as radon emanation from concrete. These effects may vary with time, which may show up in residuals if a ‘standard background’ spectrum is subtracted from the measured spectrum.

Sometimes the samples have to be counted on top of the detector's end cap. This increases the risk of contamination of the detector, which may be seen as abnormal blank results.

Recommendations:

— Develop an SOP, prescribing the generation of control charts for the most dominant peaks in the background spectrum and routinely inspect for trends using the Westgard rules.
— Develop an SOP for 'good housekeeping' in order to limit the number of radioactive sources in the counting room.
— Apply a disposable plastic cover on the detector (e.g. wrapping in household foil or clingfilm).
— Flush the inner part of the lead shield around the detector with N_2 from the vent of the dewar flask.
— Fill the empty space inside the lead shield with inert material of low atomic number (e.g. polyfoam as used in packaging or sealed bags filled with inert N_2).
— If relevant, include in the SOP for carrying out measurements that the background should be measured between every batch of samples processed.

4.4.10. Exchange of samples

One of the most feared mistakes in any analytical laboratory is the inadvertent exchange of samples, i.e. reversing unintentionally and without knowing the actual order of the samples from the recorded order. It is a typical human error that is difficult to anticipate. Sometimes it is the result of too high a workload or lack of concentration while performing a task that has become routine. It may become obvious if duplicates are being analyzed, but only if they are placed randomly in the sequence, or via the analysis of the control sample, or perhaps via customer complaints.

Recommendations:

— Develop an SOP requiring that the capsule or sample codes be verified before and after counting, as well as being recorded and signed by the analyst.

4.4.11. Nuclear reaction interferences and gamma ray spectrum interferences

Actual samples may contain elements from which radionuclides are being produced that are similar to the radionuclide coming from the element of interest. Perhaps an unusual fast neutron reaction is involved. Such nuclear reaction interferences may manifest themselves in the control samples by erroneous results; however they may be difficult to find in real samples if only one element is determined at a time.

Recommendations:

— Include in the SOP dealing with the analytical request from the customer that as much information as possible should be obtained in advance regarding the expected elemental composition of the sample.
— Inspect tabulations for interfering reactions [7].

— Develop an SOP for the experimental determination of correction factors for these contributions by analyzing standards of the interfering elements. Include in this SOP, if relevant, that these correction factors should be redetermined after core changes.
— If possible, determine whether irradiation with epithermal neutrons might give better results [25].

4.4.12. Differences in irradiation geometry between samples and standards

In nearly all irradiation facilities axial and/or radial flux gradients occur. Sometimes the neutron spectrum may also vary over small distances [26], resulting in a different ratio of the epithermal and fast neutron flux to the thermal neutron flux. Differences in the thermal neutron flux between the sample and standard may be seen by a high variability in the control charts of control samples.

Recommendations:

— Develop an SOP for the experimental determination of the neutron flux gradient in the irradiation position. To this end, use material for which the mass has been determined with high precision (weighed rather than pipetted).
— Establish if neutron spectrum variations are relevant for the application. Inspect the relation between neutron absorption cross section with the neutron energy of the element of choice (check the resonance integral).
— Develop an SOP, if necessary, for the experimental verification of the variation of the neutron spectrum in the irradiation position using triple monitors (e.g. $^{95,97}Zr + {}^{198}Au$, [5]).
— Include in the SOP for packaging the sample for irradiation that the samples should be sandwiched between standards or flux monitors. Include in the SOP for calculation of the result that the results of these sandwiching standards should be interpolated towards the sample.

4.4.13. Differences in neutron absorption between samples and standards

Standards and control samples usually have a reasonably well-known elemental composition and it is highly unlikely that they will contain significant levels of elements with strongly neutron absorbing properties such as B, Cd, In, Gd, Eu, Dy, Au and Ir. However, the experimental samples themselves may indeed contain these elements, resulting in stronger neutron self-absorption than in the standard or in the control sample. Such strong self-absorption may not always manifest itself in a flux depression in the surrounding standards.

Recommendations:

— Include in the SOP dealing with the analytical request that it should be verified with the customer, before starting the analysis, whether or not the material contains significant levels of elements such as B, Cd, In, Gd, Eu, Dy, Au and Ir.
— Develop an SOP for test-analysis of small portions, or analysis of a series of samples with varying mass in case such knowledge is not available or if the presence of such elements cannot be excluded on the basis of common sense.
— Include in the SOP dealing with the quality control inspection of the samples that the specific activity of the surrounding standards should be compared with the mean value in the respective control chart. Apply the Westgard rules.

4.4.14. Malfunctioning gamma ray spectrometers

Even though it is assumed that the gamma ray spectrometer has been verified to be 'fit for the purpose', it is possible that instabilities may occur during the measurement, ultimately resulting in wrong results. Typical examples of problems are: count rate, temperature- or humidity-dependent gain jumps and shifts, DC shifts, excessive tailing due to high count rates and inappropriate baseline restoration or power (mains) instability. Such effects may be monitored from the shape of the gamma ray spectrum and the shape (symmetry) of the most prominent peaks.

Recommendations:

— Develop an SOP for the inspection of the symmetry of the most prominent and most likely interference-free peak in the gamma ray spectrum and record the number of channels above background on both sides from the centroid of the peak.
— Develop an SOP for a visual inspection looking for the existence of 'shadow' peaks, i.e. peaks in the gamma ray spectrum resulting from gain or DC jumps.

5. GENERAL CONSIDERATIONS IN QUALITY MANAGEMENT APPLIED TO REACTOR OPERATIONS AND INAA FACILITIES

5.1. Documentation generation and control

Traceability of activities by documentation is a key component in quality assurance and quality management. While there may be regulatory requirements for full documentation it is also good practice, since it also contributes to the identification of sources of error, and thus to continuous improvement. Traceability of operations requires documented attention to the management aspects as well as the technical aspects of a quality assurance system.

Recommendations:

— *Coding system.* A unique sample and file coding system must be used. This should enable unambiguous identification, quick location of samples, identification of standards, control samples and blanks. The identification of spectra needs to be related to the different measurements of the same sample such as those made after 1 week and 3 weeks decay time for example.
— *Analysis reports.* Reporting should be clear and unambiguous and should include such data as: the uncertainty of results, significant digits, experimental conditions (irradiation, decay and counting) as well as customer and laboratory codes. It should be made clear what is the meaning of the number behind the +/- sign. It should be made clear what the meaning is of the number behind the < sign. It must be noted that if quality assurance activities are to comply with ISO requirements (ISO-International Standards 17025 [27]) the associated requirements on the content of the analysis reports must be met. These are considerably more elaborate.
— *Approval of final results.* There should be acceptance criteria for the approval of results. One option is to compare the results of control samples with expected values (e.g., derived from control charts) and to calculate the modified 'z-score' ('u-score'; see Section 2). This can be done for the blank as well to decide if there are any contaminants.

— *Transposition errors.* There should be an independent, verifiable check that all input parameters were properly transposed into the computer files. These parameters include the sample/standard code, sample/standard mass, filling heights, irradiation start-time and duration and start-time of counting and duration.
— *Hand calculations.* There should be an independent and verifiable check on hand calculations in which the raw input data cannot be visually verified (e.g. if using pocket calculators).
— *External documentation.* Records of the irradiation schemes, core configurations and the like should be retained.

The documents subject to control are any written or pictorial information prescribing, defining or specifying activities, requirements, forms used for observations, availability of drawings and schemes of facilities (all 'as built') or procedures for certain jobs or activities ("standard operating procedures"). There are numerous examples and suggestions for the paragraphs to be included in standard operating procedures. They might typically include: title, purpose, explanation/background information, responsibilities, references to other documents, safety precautions, starting requirements, operations, non-conformance and permitted deviations, registrations, archiving, contribution to uncertainty budget, schemes and drawings, page number and total number of pages, code, date of verification, date of issue, names of initiator, verifier and authorizer (see also Annex II).

Recommendations:

— During the generation of procedures, the document should be regularly reviewed for adequacy, completeness and corrections by a competent person other than the author. Jargon should be avoided. If possible (i.e. if national language allows), try to differentiate between "shall" and "should".
— The documents describing the sequence of operation and the instructions for operating the equipment should be known to the personnel and available throughout the time of handling, dismantling, post-irradiation examination and storage of irradiated elements until final disposal.
— The responsibility for the generation of documents in the field of reactor utilization is the reactor manager, head of the experiment and the safety committee.

5.2. Acceptance criteria

Quantifiable criteria are necessary to make unambiguous decisions at critical points in the various procedures. These include criteria for acceptance of equipment, criteria for accepting the quality of the analysis results and so on. Acceptance criteria may depend to a certain extent on customer requirements in terms of accuracy and precision, state of the art of facilities and equipment, operability and economics.

Recommendations:

— Acceptance criteria may be derived in a variety of ways, including for example, quality control charts (Westgard rules), comparison with previously obtained results (to be defined and documented in advance, e.g. difference <10%), using z-scores or u-scores, via statistical analysis (t-test, F-test, χ^2-test, ANOVA [28], [29]) or from the specifications of the manufacturer or vendor.

— Criteria also depend on the contribution of the related step in the analytical process to the uncertainty budget of the analysis.

5.3. Non-conformance management

Non-conformances are deviations from normal conditions, both in conduct of work and in observations. These may be errors detected in data, calculations, reasoning, assumptions, programming and measurement; differences between anticipated results, actual results and from similar tests; and failure or incident during testing. Procedures are needed for corrective action on non-conforming items, as well as preventive action for the recurrence of non-conformance.

Recommendations:

— It should be clear in the SOPs whether or not it is allowed to deviate from a procedure, and under what conditions.
— All personnel should report on detecting non-conformances and perform investigations to establish the corrective and preventive actions required (see Annex II). Unique record numbers should be allocated during this process.
— Specify in SOPs what to do in case of non-conformance, viz. either: record and continue; record, take corrective action and continue, or: record and stop.
— The relevant manager/responsible employee should co-ordinate and approve an investigation, propose corrective and preventive actions.
— Preventive and corrective actions need to be followed up and closed out in order to ensure that the non-conformance treatments are effective.
— A register should be kept to control the above activities and non-conformances are best reviewed at regular intervals to determine the effectiveness of procedures and to determine any trends or problem areas.
— Report mechanisms for non-conformances need to be effective and must be communicated to all parties involved (i.e., laboratory assistant, operating personnel, maintenance).

5.4. Training and qualifications

Under all circumstances it must be possible to demonstrate that the personnel involved is technical competent to perform the activities.

Recommendations:

— The personnel performing handling, dismantling, post irradiation examination and storage of experimental devices need to be given the necessary training in all aspects of these operations including, where necessary, exercises with mock-ups, before the work with irradiated objects is undertaken.
— A method for determining the effectiveness of training should be in place.
— A reactor training committee should be established to oversee the effective training and certification of reactor operators and users of the facilities. A method for determining the effectiveness of training should be in place.
— The personnel who work in the experimental laboratories must be educationally qualified and have general training in radiological practices and emergency plans.
— Specific training should be provided for the experimenters which includes:

(i) Operating procedures for the experiments such as sample preparation, sample packaging, use of reference materials, estimation of induced radioactivity, checking hand calculations, completion of irradiation forms, receiving samples after irradiation, unpacking of samples and opening of irradiation containers;
(ii) Use of laboratory equipment including pipettes, balances, ovens, freeze driers, sample size reduction machines, gamma ray spectrometers, sample changers and software;
(iii) Radiological rules and instructions associated with the performance of the experiments in the facility.

— Consider also a systematic programme for training and qualification in the conduct of analysis. The programme for such an internal training should be documented in a standard operating procedure. This procedure should also include the assignment and identification of instructors. Internal training is facilitated by documented procedures. On-the-job training and simulator exercises are needed for the personnel performing the preparation and operation of the experimental facility.
— All documents describing the sequence of operations and the instructions for operating the equipment in different laboratories should be known to the experimenters and be available throughout the time of the handling, dismantling, post-irradiation examination and storage of irradiated elements until final disposal.

5.5. Design changes

5.5.1. Reactor safety committee

The reactor safety committee (RSC) should oversee the safe operation of the reactor and its associated facilities and be responsible for:

— Evaluation of new experiments, experimental facilities (INAA) and any modifications to them.
— A formal approval mechanism such as issuance of a RSC approval certificate (see Annex II) before a facility or experiment may be commenced.
— Deciding whether additional information is required in order to grant an approval. Such additional information will usually comprise a safety and or risk analysis report that prescribes the safety measures for protecting equipment and personnel.
— Ensuring that all approved experiments comply with operational constraints and acceptance criteria. Approval may be withdrawn if the terms of the approval are not adhered to for any reason.
— Investigatation of relevant non-conformance.

5.5.2. General design changes

Design activities are applicable to physical facility changes and may include new designs as well as modification of previous designs. Included in design activities is the preparation, checking and approval of the changes. Design and development of facilities are made in response to customer needs, safety considerations and equipment considerations. The assignment of design activities needs to be made to competent persons. These activities may include checking, comments, inputs to design as well as independent verification of the design.

Recommendations:

— Any changes, modifications or additions to the INAA facilities (including procurement of any new facilities or part thereof) which may have an impact on the nuclear safety or operation of the facility should be assessed and approved by the reactor safety committee.
— The person responsible for the design activity needs to co-ordinate the interfaces necessary for the activities to function effectively.
— Drawings generated as a result of changes in existing facilities or of new facilities being procured should be controlled in accordance with the specific procedural requirements to maintain the build and/or revised status of the facility.

5.5.3. Control of design changes to INAA facilities

A risk analysis is required for changes that may have an impact on nuclear safety and it is the responsibility of the reactor safety committee to obtain approval from the regulatory body or authority as applicable.

Recommendations:

— A facility change proposal should be prepared and approved by the reactor safety committee. Such a facility change proposal should set out or reference the following as applicable:

  (i) Identification of the facility to be changed.
  (ii) Details of the problem or deficiency that needs to be addressed and/or the improvement required.
  (iii) Details of the proposed change, including any proposed new equipment or materials.
  (iv) Details of any design and/or calculations that have already been carried out or that are required.
  (v) A statement of the possible impact on nuclear, radiological, criticality and other safety issues as well as a statement of possible environmental impact.
  (vi) Design changes or modifications that could affect the characteristics of the irradiation position. Examples of such modifications are proposals to:

    — Change the core configuration,
    — Change the pneumatic tube position in the reactor core,
    — Refuel, or shuffle elements in all or part of the core,
    — Changes during conversion from high enrichment fuel to low enrichment fuel,
    — Changes in horizontal tubes,
    — Introduction of a highly absorbing material in an irradiation position close to INAA positions. In such a case, characterization of the irradiation position should be performed to determine if it has a significant impact on the end user.

5.6. Equipment control and maintenance

5.6.1. Checklists for pneumatic transfer systems

In order to ensure safe operation of equipment used for the operation of rabbit systems, periodic inspection of the system and components are necessary.

Recommendations:

— Inspections must be carried out regularly (e.g. weekly) using a checklist for the rabbit system and the pneumatic control loop (see Annex II).
— Periodic maintenance must be performed for the various components of the rabbit system to ensure availability of the system (see Annex II).

5.6.2. INAA equipment

Equipment for INAA should be fit for the purpose. The laboratory itself decides on the acceptance criteria (see Section 5) of the equipment which depends on, amongst things, the state of the practice, the environmental conditions and mission of the laboratory.

Recommendations with regard to sample preparation:

— *Balances.* If possible, use a dedicated balance table, carry out regular adjustments and have the balance calibrated regularly. An alternative is to use a set of calibrated masses. For day-to-day checks, a set of home-made masses can be used (e.g. made from brass). The results of tests and checks should be documented in control charts. It helps to inspect for trends or shifts.
— *Pipettes.* It is recommended to use them as a gravimetric tool rather than as a volumetric tool. Calibrate them regularly and calibrate using the solvent of interest.
— *Ovens and furnaces.* Check the quality of the built-in thermometer, since reference materials will need to be dried at a specified temperature. Apply correction curves if necessary.
— *Dessicators:* Always have fresh silica gel in stock (in the oven) and replace it regularly, even if the material still colors blue.

Recommendations with regard to gamma ray spectrometers:

— Check the high voltage (HV) supply performance in case of power (mains) failures. Turn the HV to zero if liquid nitrogen supply is not reliable and if detector is not being used.
— Apply the common checks for functionality of the equipment, such as inspection of the full-width half-maximum (FWHM), FW(0.1)M, and symmetry (left and right tailing) both in the low energy and high-energy region of the gamma ray spectrum [30].
— Apply the common check on linearity of the channel-number-gamma ray energy relationship. Stabilize the temperature before use. Record environmental conditions during these measurements.
— Convert all observations into control charts and inspect for trends.
— Use an uninterruptible power supply (UPS) to ensure data protection in case of power (mains) failure.
— Inspect gamma ray software performance using the IAEA-test spectra [31].

6. SAFETY AND WASTE MANAGEMENT

6.1. In-core facilities

The safety analysis of an in-core experiment needs to include an evaluation of the cooling capability for the particular arrangement (i.e. geometry, heat generation). Experiments in which the target material is fissile or fissionable should receive special attention with regard to cooling. This is because heat is produced within the sample, over and above the heat deposited there by the neutron and gamma absorption. Care is needed to confirm that the integrity of the sample container or cladding will not be lost under any operational or accident condition, especially with loss of flow, and consequently release fission products into the reactor or pool coolant systems. Similarly, attention is needed for experiments in which the sample material may decompose due to radiolysis (see para. 4.1.5).

6.2. INAA operations

It is assumed that the irradiation process and the conduct of INAA are carried out with common radiological safety measures. Still, there are several implications for these activities that requires special attention and/or safety measures as discussed earlier.

Recommendations:

— Use a fumehood and enclosure for opening sample containers to avoid contamination and to prevent inhalation of radioactive gases.
— Monitor and record radiation dose rates and radioactive contamination levels of the laboratory. Be alert for samples with high levels of halogens and Hg. These elements tend, upon irradiation, to migrate through polyethylene containers and thus, in principle, may result in radioactive contamination.
— Estimate induced radioactivity before irradiation. An independent check on calculations, recording and archiving data as well as useing feedback from radiation dose rate monitoring helps to provide better predictions.
— Estimate ^{41}Ar production, release and dose effects during opening of irradiation capsules.
— Be aware of the possible presence and approximate quantity of elements with high neutron absorption cross sections such as B, Cd, In, Eu, Dy, Au and Ir. Also maintain alertness for the presence of, and approximate quantity of fissionable elements such as U and Th. Awareness of components that may decompose by radiolysis, such as moisture and proteins will help prevent difficulties. Specifically ask for such information on the irradiation request form.
— Assure facilities are available for intermediate storage and traceability of irradiation capsules.
— Include in SOPs a safety paragraph on liquid N_2 filling of dewer flasks and brief people adequately.

6.3. Waste management

Because of the costs of radioactive waste disposal, it is important to estimate the final radioactive waste product characteristics. In addition, it may be fruitful to estimate the maximum induced radioactivity both in the irradiation container (including samples) as well as in the construction materials of the irradiation facility.

Recommendations:

— Estimate the remaining radioactivity in the samples after a given decay time, on the basis of measured radiation levels and results of the INAA performed.
— Keep a record of stored radioactive samples, their location, identification and date of final disposal. Storage locations should be as far away as possible from the counting room and occupied areas.
— Decide on the minimum storage time (e.g. 3 months after reporting the analytical results) before final disposal of radioactive samples.
— Agree in advance with customer what to do with the remaining original material. Decide if it should be returned, disposed of (who will pay for it), and how long to keep it before disposal.
— Estimate the maximum induced radioactivity in the irradiation facility (i.e., typically the part directly facing the reactor-core) on basis of the known composition of the construction materials. Include this information in the decommissioning plan file for the reactor.

There may be regulatory requirements to the safe handling of waste and eventually it may be required to comply with ISO-14000 regarding the quality system for waste management. This would, however, imply also that an ISO-9000 compliance is required for the quality system that describes the management of the organization and the associated facilities. More information on these issues can be found e.g. on the ISO Website [32].

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ANNEX I
DEFINITIONS AND ABBREVIATIONS

The definitions presented in this report are intended principally for use in this publication and do not necessarily conform to definitions adopted elsewhere for other use.

Acceptable limits

Limits acceptable to the regulatory body.

Corrective action

Any action such as a continuous operation, reject, network of repair to ensure that normal condition is restored and that the final product meets the specified requirements.

Disposal

The emplacement of waste in a repository, or a given location, without the intention of retrieval.

Experiment (or experimental equipment)

Equipment installed in or around the reactor to utilize its neutron flux and/or ionizing radiation for research, isotope production or any other purposes.

Experimental facility

Permanent equipment such as beam ports, including extension tubes with shield, in-core irradiation apparatus, pneumatic tubes and in-pool irradiation facilities.

Maintenance

The activity of keeping equipment in good operating condition, including both preventive and corrective (or repair) aspects.

Modification

Any change in, or an addition to, the existing experimental facility, with potential safety implications.

Neutron activation analysis (NAA)

A method for the determination of elements and their concentrations based on the conversion, upon irradiation with neutrons, of stable isotopes into radioactive isotopes and subsequent measurement of the induced radioactivity.

Non-conformance

A non-conformance is a non-compliance of a product or service with respect to specified requirements.

Non-routine experiment

An experiment that has not been carried out before or for which there is no valid approval document issued by the Reactor Safety Committee.

Normal operation

Operation of a research reactor and associated experimental devices within approved operational limits and conditions, including start-up, power operation, shutdown.

Preventive action

Action required to eliminate or to reduce the recurrence of a non-conformance, incident or occurrence.

Quality assurance

All planned and systematic actions necessary to provide adequate confidence that an item or service will satisfy given requirements for quality.

Rabbit system

A device installed in and around the reactor to enable samples to be rapidly inserted and removed from an irradiation position.

Reactor manager

The member of the operating organization who has the responsibility and authority for directing the operation of the research facility.

Reactor operator

The licensed person who operates the reactor and performs other reactor facility operations.

Reactor Safety Committee

An oversight committee usually consisting of responsible persons from the reactor utilization, safety, health physics, radiation protection, maintenance, quality assurance and isotope production groups. The committee will usually be involved with safety-related items, and approval of procedures, documentation or operating procedures.

Research reactor

A nuclear reactor which includes experimental facilities used mainly for the generation and utilization of neutron flux and ionizing radiation for research, isotope production and other purposes.

Routine experiment

An approved experiment that has been carried out before and which has had a safety analysis evaluation performed prior to its approval.

Standard operating procedures (SOPs)

A term used for an approved document that describes procedures, work-instructions or operating instructions.

Traceability

The property of a system which enables the ready retrieval of the different elements of a record to allow unambiguous correlation with an uniquely identified sample.

Utilization (or reactor utilization)

The use of the reactor or of experiments or experimental devices during operation of the reactor.

Waste management

All activities, administrative and operational, that are involved in the handling, treatment, conditioning, transportation, storage and disposal of radioactive waste.

ANNEX II

EXAMPLES OF WORKSHEETS

II.1. REQUEST FORM FOR NEUTRON IRRADIATION

Lab	NAA	Rabbit	Other
End User			

Sample No.			
Sample Label			
Routine		Non Routine	

Information about Irradiation Facility

Source of Irradiation	Reactor		Power(MW)	Date of start-up	Time of start-up	Time of shutdown
	External Source		Flux(n/cm^{2}s)			

Information about Sample

Material Type	gas		Dimensions (cm)		Weight (g)		Homogeneity		Volume (cm^3)		
	solid										
	liquid										
Neutron (barn) absorption		Severity	Toxic		Required Activity (μCi)		Duration of irradiation	Hours			
			Explosive					Min.			
			Others					Sec.			
Place of irradiation	Rabbit System	A		Date of irradiation	Measured Activity (μCi)		Flux (n/cm^{2}s)	Dose Rate (mSv/h)	transportation		
		B									
	Vertical channel	Wet			Error (%)				Unshielded		
		Dry									
	Beam channel No.			Control of waste disposal	H.Ph				shielded		
					other						

Information about the Container / Capsule/Rabbit system

	Material	Impurity	Dimensions	Absorption cross section (barn)	Availability
Container					
Capsule					
Rabbit					

Personnel Control

	Name	Signature
Prepared by :		
Reactor Manager		
Safety Officer		
Lab. Physicist		
Rad. Protection officer		

II.2. NEUTRON IRRADIATION REQUEST FORM

LONG IRRADIATIONS

THIS SECTION TO BE COMPLETED BY THE REQUESTER	REQ. INFO	Request No. Date Requested by: Affiliation:	
	MATERIAL DESCRIPTION	Name of Material: _____ Chemical Form: _____ No. of Samples per Rabbit:_____ Physical Form: _____ Sample total (grams) in Rabbit:_____ Other info: BE SURE TO COMPLETE IRRADIATION TRACKING LOG Be sure to fill out calendar schedule book.	
	IRRADIATION FACILITIES AND TIME REQUESTED	Facilities Requested (circle one) Rabbit System A Rabbit System B Power level requested: _____ Duration of Irradiation: Long: with an irradiation time of :_____ day(s). NOTES:	
	ISOTOPE INFO	Expected Radioisotope (list) and Amount (μCi or mCi or kBq) Sample: _____ Container: _____ Impurities: _____ Flux (n/cm^{2}-s used to calculate activities): _____	
	RABBIT DISPATCH	Health Physics: _____ Date: _____ Reactivity Effect: Negligible Otherwise: _____ Reactor Manager: _____ Date: _____	
	RABBIT STORAGE	Dose Rates (mSv/hr): _____ Unshielded: _____ Shielded _____ Date: _____ Time: _____ Storage Location: _____ HP measurements taken by: _____	
	RELEASE TO LABORATORY	Dose Rates (mSv/hr) _____ Beta _____ Gamma _____ Beta + Gamma @ distance of : _____	Destination : _____ Log Book Notation: [Y/N]:____S.No.:____ Date :_____ Time: _____ HP Signature: _____

II.3. NON-CONFORMANCE REPORT

	NON-CONFORMANCE REPORT	Page 1 of 1
	REPORT No. NCR..... /	DEPARTMENT:
1. ALL PERSON	DESCRIPTION OF NONCONFORMANCE / INCIDENT:	
	ITEM/FIELD/LOT AFFECTED: QUANTITY: PREPARED BY DATE: SUPERVISOR: DATE:	
2. ALL	INVESTIGATION : (Reports and other documents to be added)	
	PERFORMED BY: POSITION: DATE:	
3. RELEVA DEPT	RECOMMENDED CORRECTIVE / PREVENTIVE ACTION: PREVENTIVE: CORRECTIVE:	
	RECOMMENDED BY: POSITION DATE:	
QA PERSONNEL	FOLLOW UP CORRECTIVE / PREVENTIVE ACTIONS.	
	CORRECTIVE ACTION IMPLEMENTED: (REMARKS) FOLLOWED-UP BY: DATE	
	PREVENTIVE ACTION IMPLEMENTED: FOLLOWED-UP BY: DATE	

II.4. SAMPLE REGISTRATION FORM, PART I

FORM NO. .....

CUSTOMER… .....
Contactperson .....
tel.....
fax.....
address.....
.....
.....
.....

Quotation no.
Invoice no.....
Price agreed.....
Date receipt of samples.....
Date analysis ready.....
Final date for mailing report.....
Mailing by
o fax o diskette

SAMPLES
Received by :
1 .....
2 .....
date :.....
total number of samples.....
total number of sub-series.....

description .....
type : o geological o biological o plastic
o .....
coding by customers : o yeso no

samples already weighted ? o yes o no
weighing form enclosed ? o yes o no
complete? o yes o no

special information on
sample handling and preparation o yes o no

remainders return to customer ? o yes o no
units o mg/kg o % o else.....
suggestions for storage.....
.....

Delivery OK : o yes o no intials.....

SAMPLE TYPE ANALYSED BEFORE ?
o yes
o no : have analysis protocol authorized
ELEMENTS TO BE DETERMINED
o all
o shorts
o medieum
o longs
o some, viz.....
.....

EXPECTED CONCENTRATIONS (IF KNOWN) :
.....
.....

SUGGESTIONS FOR REF.MAT/CONTROL SAMPLE
.....
DETECTION LIMITS.....
.....
.....

Comments for inclusion on 1st page of final report

II.4. SAMPLE REGISTRATION FORM, PART II

CONDUCT OF ANALYSIS

Name analist .....-
.....

Code samples :.....

Irradiation contracted-out ?
o yes, register no. .....
Analysis contracted-out ?
o ja, register no. .....

ANALYSIS PROTOCOL
o routine o new, authroisation by head of laboratory).....
Planned conditions (first estimates) :
Irradiation plan no..... d.d. .....
Shorts : t_i .....s t_w .....s t_c .....s geom. .....
Mediums: t_i.....h t_{w1} .....d t_{c1} .....h
detector : o Ge-1 o Ge-2 o Ge-3 geom.....
longs : t_{w2} .....d t_{c2} .....h
detector : Ge-o 1 o Ge-2 o Ge-3 geom.....

SAMPLE PREPARATION
o drying, oven .....h op .....°C o freeze-drier
o milling o crushing o ball-mill o LN_2
o different ....-
.....
date of sample preparation : .....
REFERENCE/CONTROL SAMPLE : .....

SAMPLE STORAGE :
First storage .....
Radioactive storage .....
Remainders .....
Final storage .....
Date of waste disposal *.....
Date or return remainders.....
* fill in date after reporting results

NON-COMPLIANCE (non-compliance form no....)
.....
.....
.....
.....
.....
.....
.....
.....

FILL IN THIS PART ONLY IF REPORT CAN NOT BE RELEASED :

Date of contact with customer : .....
Agreement with customer.....
.....
.....
.....
.....
.....

nitials: .....

QUALITY CONTROL :

- report complete : o yes o no
- ref./control sample OK : o yes o no
- blank OK : o yes o no
- fluxmonitors OK : o yes o no
- irradiation date and time checked with hard-copy information ? :
o yes o no
- weights in report checked with weighing-form information ? : o yes o no

Qualitu control OK ? : o yes o no

date : ..... initials .....

If Quality control not OK, bring report to the Quality Manager for fuurther action and file a non-compliance form

Quality Control OK after control by quality manager and corrective actions
o yes o no
date :..... initials .....
initials QA manager.....
If not OK, then contact customer

II.5. REACTOR SAFETY COMMITTEE APPROVAL

Doc. N^{o}	

Approval is hereby granted for the following irradiation/activity
Subject to the following conditions
Reactor Manager: Date:

II.6. MAINTENANCE DOCUMENTATION
PNEUMATIC LOOP-RABBIT SYSTEMS A&B

Date	System	Unit	Fault	Action	Operation/ Remarks

II.7. WEEKLY CHECK LIST
RABBIT SYSTEMS AND PNEUMATIC CONTROL LOOP

DATE:.....

System	Unit	**Operating Condition**		**Description of Fault**
Rabbit system type A	Power on indicator light			
	Large capsule line: 1. Sample - In 2. Sample-Out			
	Small capsule line: 1. Sample-In 2. Sample-Out			
	Water knocking gas strainer			
	Pressure regulating valve			
Rabbit system type B	Power-On indicator light			
	Divider 1: positioning ejecting			
	Divider 2: positioning ejecting			
	Divider 3: positioning ejecting			
	Combiner: positioning ejecting			
	Transformer: positioning ejecting			
	Filter-Pressure Reducer			
	Oil Sprayer			
	Air Compressor:			
	1. Level of lubrication oil 2. Pressure gauge 3. Triagle belts 4. Filter 5. Discharge valve			
	Glove Box: stripping resetting sample-in sample-out			

CHECKED BY: .....
QA:.....

II.8. ROUTINE MAINTENANCE SCHEDULE

DATE:.....

System	Unit	**Operating Condition**		**Description of Fault**
Rabbit system type A	Power on indicator light			
	Large capsule line: 1. Sample - In 2. Sample-Out			
	Small capsule line: 1. Sample-In 2. Sample-Out			
	Water knocking gas strainer			
	Pressure regulating valve			
Rabbit system type B	Power-On indicator light			
	Divider 1: positioning ejecting			
	Divider 2: positioning ejecting			
	Divider 3: positioning ejecting			
	Combiner: positioning ejecting			
	Transformer: positioning ejecting			
	Filter-Pressure Reducer			
	Oil Sprayer			
	Air Compressor:			
	1. Level of lubrication oil 2. Pressure gauge 3. Triagle belts 4. Filter 5. Discharge valve			
	Glove Box: stripping resetting sample-in sample-out			

CHECKED BY: .....
QA:.....

ANNEX III
EXAMPLE OF A STANDARD OPERATING PROCEDURE

In this annex the term standard operating procedure (SOP) is used as a descriptive quality document covering both '*procedures*' and '*instructions*'. A *procedure* describes how a component of the work process has been organized, and which arrangements have been agreed upon in this process and between employees involved. An *instruction* comprises prescriptive details for the conduct of work, or for a small unit operation, or for the operation of equipment or facilities.

A quality document should have a unique identification and version number. In addition the page number and total number of pages should be given. It is often preferred to specify the persons who drafted, verified and approved it, and the date of approval. Sometimes the cover page also includes a distribution list. Colored lay-outs are sometimes used to distinguish originals from photocopies.

Here, some suggestions for the contents of an SOP are given.

1. Objective

2. Scope

3. References

— Equipment manuals etc.
— Internal documents (e.g. reactor operating schedule, loading scheme of facilities, neutron spectrum characteristics)
— Relevant paragraph in normative standards (such as ISO 9000 or ISO-17025)
— Relevant other quality documents (e.g. instructions for packing of samples and irradiation containers, filling-in irradiation forms, maintenance of the irradiation facility etc.)

4. Requirements

— Requirements as to qualifications of personnel
— Requirements as to e.g. quality of irradiation containers (material characteristics, incl.impurities)

5. Responsibilities

— List of contact and responsible persons
— Availability of personnel outside office hours

6. Safety

— A reference to the relevant Safety Rules (correct packaging, estimate of induced radioactivity and dose rates, test-irradiations, pressure build-up tests etc.)
— Control of radioactive samples and waste disposal.

Made by : E.X. Perimentator
Verified by V.E.Rificator
Approved by : B.Oss
Date : November 15, 1999

Distribution :
- all reactor operators, date : November 16, 1999
- health physics group, date : November 16, 1999
- qualified employees of NAA laboratory, date : November 16, 1999

7. Operation

A step-by step description (in logical order) of the operations. Indicate — as far as language allows — which steps SHALL be followed. Be careful with too precise prescriptions (e.g. too many significant "digits or specifications of materials); if relevant, refer to use terms as "…or equivalent…", and specify which items need to be registered to ensure trackability of events. If relevant, refer for certain actions to other instructions.

Preparation

— Planning the irradiation: assuring availability of facility, reactor operators, reactor power, neutron spectrum characterization (if relevant), fulfilling all paperwork requirements, final check of correct packing or irradiation capsule and container, final check of estimated induced radioactivity
— Obtaining permission for irradiations

Preparation of the system for irradiation

— Enable the control system
— Assurance of synchronization of irradiation timer with other times related to the conduct of analysis.
— Assurance of convention with respect to daylight saving time + registration thereof
— Setting of the irradiation timer

Irradiation

— Set the system for automatic processing of rabbit
— Run the system automatically with the rabbit
— Register start of irradiation, date, operator, reactor power, irradiation facility and/or position
— Register end of irradiation, date, operator
— Register (if possible) dose rate after irradiation
— Disable the control system

Made by : E.X. Perimentator
Verified by V.E.Rificator
Approved by : B.Oss
Date : November 15, 1999

Distribution :
- all reactor operators, date : November 16, 1999
- health physics group, date : November 16, 1999
- qualified employees of NAA laboratory, date : November 16, 1999

8. Problems

— Handling of system errors during the automatic cycle
— Verification of the experimental data
— Handling of facility defects and problems
— A list of persons to contact outside office hours

9. Deviations and corrective action

— An indication under which conditions deviations from the normal procedure are allowed
— An indication which persons have the authority to decide so and/or approve this

10. Registration and Administration

— A list of important observations that should be registered in all cases
— An indication where the registers are kept

Made by : E.X. Perimentator
Verified by V.E.Rificator
Approved by : B.Oss
Date : November 15, 1999

Distribution :
- all reactor operators, date : November 16, 1999
- health physics group, date : November 16, 1999
- qualified employees of NAA laboratory, date : November 16, 1999

ANNEX IV
EXAMPLES OF A PRE-IRRADIATION CALCULATION RESULT

Irradiation plan no. 99-01
Date : November 15, 1999
Time : 15.22
Made by : E.X. Perimentator

Irradiation facility : pneumatic tube 1
Thermal flux $4.2 . 10^{16}$ $m^{-2}s^{-1}$
Epithermal flux1. $1.0 . 10^{15}$ $m^{-2}s^{-1}$
Fast flux $3.7 . 10^{15}$ $m^{-2}s^{-1}$

Irradiation time : 4 h Cooling time: 0 h resp. 3 h
Material type : coal Total mass : 250 mg
****
Expected composition in ppm or % (major activable elements) :
Al: 0.85% Fe: 0.76% Mg: 400 ppm K: 750 ppm Na: 500 ppm
Mn: 10 ppm Ca: 0.20 % Co: 2 ppm
****
Total induced radioactivity:
After 0 h cooling: 50 MBq
After 3 h cooling: 2 MBq
Total radiation dose due to gamma radiation, at 30 cm :
After 0 h cooling: $1.3 . 10^{2}$ μSv/h
After 3 h cooling: 7.5 μSv/h
****

Radioisotopes produced, after 0 h cooling:

Element	Nuclide	Half-life	Activity (Bq)	Gamma dose rate (μSv/h)
Al	^{28}Al	2.23 m	$4.8 . 10^{7}$	$1.2 . 10^{2}$
	^{27}Mg	9.45 m	$7.1 . 10^{4}$	
	^{24}Na	15 h	$2.2 . 10^{3}$	
Fe	^{59}Fe	45 d	$8.2 . 10^{2}$	
	^{55}Fe	2.7 y	$1.3 . 10^{3}$	
	^{56}Mn	2.6 h	$4.9 . 10^{3}$	
	^{54}Mn	313 d	$1.3. 10^{1}$	
Mg	^{27}Mg	9.45 m	$4.2 . 10^{4}$	
	^{25}Na	60 s	$5.1. 10^{1}$	
	^{24}Na	15 h	$1.8. 10^{2}$	
K	^{42}K	12.4 h	$2.4 . 10^{5}$	
Na	^{20}F	20 s	$9.6 . 10^{2}$	
	^{24}Na	15 h	$1.3 . 10^{6}$	7.1
	^{23}Ne	37.6 s	$1.9. 10^{3}$	
Mn	^{56}Mn	2.6 h	$1.24 . 10^{6}$	

Element	Nuclide	Half-life	Activity (Bq)	Gamma dose rate (μSv/h)
Ca	^{37}Ar	35 d	$3.8.\ 10^{2}$	
	^{45}Ca	163 d	$4.9\ .\ 10^{2}$	
	^{49}Ca	8.7 m	$6.9.\ 10^{4}$	
	^{47}Ca	4.5 d	$1.9\ .\ 10^{1}$	
Co	^{60m}Co	10.5 m	$5.1\ .\ 10^{5}$	
	^{60}Co	5.3 y	$5.8\ .\ 10^{1}$	

Radioisotopes produced, after 3 h cooling:

Element	Nuclide	Half-life	Activity (Bq)	Gamma dose rate (μSv/h)
Al	^{28}Al	2.23 m		
	^{27}Mg	9.45 m		
	^{24}Na	15 h	$1.9\ .\ 10^{3}$	
Fe	^{59}Fe	45 d	$8.2\ .\ 10^{2}$	
	^{55}Fe	2.7 y	$1.3\ .\ 10^{3}$	
	^{56}Mn	2.6 h	$2.2\ .\ 10^{3}$	
	^{54}Mn	313 d	$1.3.10^{1}$	
Mg	^{27}Mg	9.45 m		
	^{25}Na	60 s		
	^{24}Na	15 h	$1.5.\ 10^{2}$	
K	^{42}K	12.4 h	$2.1.\ 10^{5}$	
Na	^{20}F	20 s		
	^{24}Na	15 h	$1.1\ .\ 10^{6}$	6.2
	^{23}Ne	37.6 s		
Mn	^{56}Mn	2.6 h	$5.5\ .\ 10^{5}$	1.4
Ca	^{37}Ar	35 d	$3.8.\ 10^{2}$	
	^{45}Ca	163 d	$4.9\ .\ 10^{2}$	
	^{49}Ca	8.7 m		
	^{47}Ca	4.5 d	$1.9\ .\ 10^{1}$	
Co	^{60m}Co	10.5 m	3.6	
	^{60}Co	5.3 y	$5.8\ .\ 10^{1}$	

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