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Quality System Implementation for Nuclear Analytical Techniques

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FOREWORD

In recent years, the International Atomic Energy Agency has helped many institutions in developing Member States to set up nuclear analytical laboratories through assistance via technical co-operation and co-ordinated research projects, expert services, and fellowship awards. Some of these laboratories have now matured to approach close to self-sustainability by providing service analysis for customers in many fields, including geological prospecting, environmental contamination survey or biomedical investigations. Particularly in the fields related to international trade, harmonization of analytical results is required to assure mutual recognition and prevent large financial losses through erroneous results. International bodies, such as the Association of Analytical Communities (AOAC International), EURACHEM, International Union of Pure and Applied Chemistry (IUPAC), European Union (EU) and International Organization for Standardization (ISO) have made considerable efforts to establish guidelines for a general quality system to be applied in analytical laboratories to trace and document the results in such a way that compatibility between laboratories can be obtained. One of the latest documents describing the requirements on how to achieve quality assurance/quality control (QA/QC) in analytical laboratories is the ISO/IEC/EN 17025, giving a comprehensive guideline for the implementation of quality system.

There is, however, still the need for practical assistance in explaining the ISO/IEC/EN 17025 procedures and recommendations for the individual nuclear techniques on how to start and proceed with the establishment of an efficient quality system. A guidebook to support beginners and users of the ISO/IEC/EN 17025 for application in nuclear analytical laboratories is presented in this publication. This training material aims to facilitate the implementation of internationally accepted quality principles and to promote attempts by Member States' laboratories to obtain accreditation for nuclear analytical techniques.

The methodology provided is appropriate for:

(a) Analysis of radionuclides as in alpha, beta, and gamma spectrometry for environmental and human-made radioactivity investigations;
(b) Analysis of trace, minor and major elements using nuclear and related analytical techniques such as neutron activation analysis, X ray fluorescence, PIXE, etc.

This training guidebook can be used by staff of analytical laboratories as a starting kit to better understand the quality assurance and quality control principles as prescribed in the ISO 17025 standard. It follows a logical order related to practical laboratory work rather than the formal clauses as given by the standard. It can be used as a stand alone textbook. However, in some cases, cross-reference is given to the ISO 17025 clauses hence it is recommended to consult the ISO standard for exact wording of specific requirements.

Through this material some misunderstandings concerning the complexity of complete quality system implementation and formalistic approach of the ISO standard might be reduced. Also, it is hoped that more and more analysts can be convinced of the benefits of working in accordance with internationally accepted quality standards.

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

EDITORIAL NOTE

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1. INTRODUCTION

Quality assurance in the context of analytical research, notably nuclear analytical work, seems redundant as everybody involved performs work with utmost care and implements quality control measures providing confidence on correctness of analysis results. From time to time, however, doubt arises when comparing results with other laboratories. “Why results are biased?” “What happens to Cr whereas Hg is correct in a particular matrix?” “Obtained results agree well with the certified values the test sample gives difference by factors?” In such cases the analyst has to struggle hard to keep confidence in his own work and trust in his analytical technique. Additionally such cases imply re-analysis, consume time and labour cost; precious samples might be lost and, if analysis was performed for a customer, can seriously damage the reputation of a laboratory.

In order to minimize such incidents quality control and quality assurance concepts have been developed to assist the laboratory personnel to achieve a higher degree of transparency of procedures, minimize potential sources of error, standardize the handling of samples, instruments and data, and in the end, decrease the rate of non-conformance results.

Quality of analytical data is not only expressed by the closeness of result to a fictive “true value” but also in a realistic estimate of the uncertainty of the results and a comprehensive documentation on how the results was obtained. It is still not common knowledge that any analytical result is associated with a specific uncertainty due to the matrix heterogeneity, the method performance fluctuation, the uncertainty of values assigned to the standards, and so on. The combined uncertainty of these factors can be estimated by repetitive analysis of independent aliquots and has to be shown together with the result.

The sample preparation, analytical procedures and other factors, which might have an influence on the result, should be carefully documented to allow re-evaluation of results if doubts about their reliability come up. Particularly for sensitive materials this has been recognized as early as the 1950s in the nuclear industry, and concepts have been developed to assure tractability of materials, traceability and, of course, reliability of results. Many different branches of industry have adopted, refined and further developed these concepts because it has been realized that a formal quality control and quality assurance system helps to achieve a stable level of high quality output. It was only in the late 80s that similar strategies became attractive to research laboratories and, in particular, EURACHEM and ISO started to set up guidelines and norms for the implementation of these measures (e.g. the ISO 9000 series).

Finally the idea of a formal accreditation of laboratories following these guidelines was developed and now it is widely accepted that — through the advent of international trade and exchange — an internationally agreed procedure for accreditation of analytical laboratories through national accreditation bodies is a proper means to enhance acceptance and legal recognition of analytical results worldwide. The economic dimension of this important development should not be underestimated.

As a matter of fact, the increased quality awareness has still not found wide reflectance in university curricula and technical training courses. But as with any change in general paradigms — from focus on increased sensitivity to focus on increased quality — analytical chemistry is facing a generation gap. It seems to be only a matter of time till the principle of QA/QC in routine service and research analytical laboratories will be commonly accepted and

the benefits of its implementation will be deemed superior to the efforts needed for its completion.

A common attitude to carry on the QA/QC in a systematic way needs to be developed and supported by all personnel involved, from the laboratory technician, the laboratory head, to the lab management and the director of the organization

1.1. QA/QC is a continuous process, not a goal to be achieved once and forever.

No doubt, increased efficiency and effectiveness, a lower rate of false results, higher grade of transparency of procedures will definitely foster the confidence and reliability, enhance the productivity and improve the reputation of a laboratory towards its clients. The credibility of a laboratory, which should be one of the major concerns of any laboratory management, is increasingly dependent on the documented evidence of QA/QC implementation according to the international standards. In accordance with the national and international demands appropriate QA/QC implementation is indispensable for the survival of an analytical laboratory in a long run. Laboratory managers have to cope with the requirements of setting up a complete quality system in a particular area of an analytical laboratory.

The costs of repetition of measurements are enormous. Direct tangible costs are the consumption of quality control samples and/or reference materials, reactor irradiation costs, vials, standards, etc. Labor cost for repletion work is the heaviest burden in this respect and increased effectiveness by having a concise quality system in place will pay off the initial costs for its setting up within a relatively short period.

A laboratory's continuity with respect to expertise, know-how and standard should be made independent of its personnel development as far as possible. The older generation working in nuclear chemistry laboratories is about to retire in many places and they should document how they were doing their work. A great help to achieve this independence is provided by a comprehensive record of written procedures, detailed as necessary to provide newcomers with sufficient information on all aspects, such as handling of samples, sample preparation, sample treatment, digestion and dilution, analysis and reporting of results. Particularly at university laboratories frequent changes in staff are hampering continuity in research activities. In these cases written procedures for e.g. method validation, homogeneity studies, development of new research materials, or new sample preparation techniques would greatly help to support metrology in chemical measurements and foster the scientific approach in training of new staff members. Nuclear analytical laboratories are more sensitive in this respect since anything related to the word 'nuclear' deals with the focus of the entire world.

Taking account human resource development, upgrading of laboratories, improving in methods and techniques is evident that the QA/QC is a continuous process, not a goal to be achieved once for ever.

1.2. Driving forces top-down

Laboratories, particularly those involved in analysis of goods for import/export or of forensic materials, might be fully aware about the advantages of having an official certificate about proper performance in compliance with international standards, being regularly audited

and re-evaluated. Such an accreditation helps to persuade customers and to credit analytical methods. Also for obtaining particular licensing for special analytical tasks (e.g. precious materials, sensitive materials, or acting as a reference laboratory) a formal accreditation will help and should be strived for by thorough implementation of a stringent quality management system.

Related to the laboratory's policy, a QA/QC system can be tailored to closely meet the specific needs. Such a system, if properly presented to the stack holders or sponsors of a laboratory can be decisive for the perspective of analytical laboratories, in particular for the competition of nuclear analytical methods.

Driving forces from management's point of view for the implementation of a quality system are mostly related to a profit oriented approach. If a simple calculation of the financial loss due to mistakes in laboratory performance is carried out for a certain period of time it becomes obvious how much of the resources can be spent to avoid these blunders. The most straightforward approach in this respect will be the initiation of a complete QA/QC system covering all relevant aspects of the laboratory's performance. According to existing experience this investment will pay off within a few months of its completion.

If a laboratory is more research oriented the scientific reputation and, related to this, the expected funding of new research projects is necessary. Nowadays not only major scientific journals but also large granting societies are aware about the importance of quality control and documented evidence of quality assurance in analytical research. It is becoming more and more difficult to compete on the scientific market place if QA/QC principles are disregarded. In such a case the support of metrology in analytical chemistry should be considered and it should not be overlooked since there is scientific challenge in this subject as well: The International Journal of Accreditation and Quality Assurance (Springer publishers) is particularly dedicated to publish contributions from the area and a wealth of information on QA/QC can be found in articles of this journal.

The image of a laboratory is not only governed by the personality of its leaders but to a great extent depends on the continuous high quality of its output. To enhance this superiority it is required to formalize to a certain extent procedures, and create a general awareness of the criteria necessary for high quality products. Identification of weak points in an action chain and proper action for its elimination can be strongly facilitated if working procedures are cut into units, thoroughly described and mechanisms defined to improve their functioning.

This manual is intended to actively guide a quality manager in an (nuclear) analytical laboratory to establish a comprehensive quality system according to ISO/IEC 17025 and ultimately to assist him/her to approach his/her national accreditation body to apply for an internationally accepted accreditation.

1.3. Driving forces bottom up

Poor results provided from an analytical laboratory compromise the reputation to the customers and affect negatively the staff motivation for future work as well. At a certain point, may be enhanced by repeated customer complaints, the involved personnel themselves will ask for a more rigid quality control and quality assurance system which allows them to maintain a higher level of accuracy and precision of their results.

The driving motto applied to all analytical laboratories is: “Confidence and satisfaction of laboratory staff derives in great extent from recognition and not from criticism of their performance”.

A laboratory should always try to keep abreast with latest analytical developments as described in the literature or advertised by manufacturers of equipment. There might be financial constraints to fully meet these terms but a general upgrading of outdated procedures and equipment should be envisaged.

Laboratory specialists are often better informed about new developments in their field than their managers. New methods and procedures need careful validation before they can be fully integrated into daily operation. The procedures and protocols of validated methods and techniques are integral part of the whole QA/QC system. On the other hand documented evidence on staff quality, laboratory tools, trainings and staff development is quite important as well.

Laboratory staff may wish to create their own written instructions for particular procedures frequently encountered in daily work without being imbedded into a formal QA/QC system. Only, it should be noted that such instructions most probably will not be comprehensive as common understanding and agreements between currently involved staff members will rarely be described in sufficient detail. Though such records can be used as a basis to establish a protocol or a Standard Operating Procedure (SOP) the human factor may preclude the comprehensive treatment on such an improvised basis. The call for a more formal approach on QA/QC will definitely come from the experience with such offhand attempts.

In many nuclear (research) establishments facilities are made available to serve third parties and in university research reactors, such services may contribute to sustain the annual budget. International acceptance of results, respect on the performance, all may be enlarged by establishing a QA system. Compliance with the ISO 17025 also assures proper handling of requests, planning and customer services. A well organized and transparent routine to serve customers/users increases the confidence in safe performance of this relatively sophisticated techniques and enhances trust and public perception after all.

Transfer of specific experience is hampered particularly in laboratories with frequently changing personnel, and every new member is trying to create his own working sequences. If results depend on the co-operation of several staff members such a situation can easily cause chaos and heavy friction between the co-workers. Such situations can be greatly resolved, and the demand to do so might emerge from the person(s) affected, to provide them with detailed written procedures to clarify responsibilities and demands to be fulfilled by each individual. As part of a QA/QC system such procedures assure some level of continuity even when personal experience is lost due to shift or exchange of personnel.

There is a lot of relevant literature available and, at the beginning it is not easy to select the most relevant sources. Quality managers will need to read a number of general documents to form the exact picture of what is needed and afterwards select the relevant ones. Attention should be given on the size and number of documents provided to the laboratory staff. Too much literature at once may confuse the staff and will not be effective.

Table I gives some examples of the structure of available quality related literature.

TABLE I. THREE EXAMPLES OF THE AVAILABLE LITERATURE SOURCES CONNECTED TO THE SPECIFIC REQUIREMENTS OF THE ISO 17025 STANDARD

1st level	2nd level	3rd level	4th level
International standards	Guidance documents	Handbooks	Scientific journals/articles
		General	
ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories, ISO, Geneva (1999)	EURACHEM/CITA Guide to Quality in Analytical Chemistry (2001)	Quality in the Analytical Chemistry Laboratory (Prichard, E., Ed., John Wiley & Sons (1995)	Basic Steps Toward a Self-sustainable Quality System and Laboratory Accreditation, P. Bode, et al., Springer, Germany (ISSN 0949-1775), Accredit. and QA **3** (1998) 197–202
		Internal quality control	
ISO 17025 Chapter 5.9	IUPAC Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Labs., Pure & Appl. Chem. **67** (1995) 49–56	ASTM Manual on Presentation of Data and Control Charts Analysis, ASTM Manual Series: MNL 7 (1991)	Quality Control in Activation Analysis, K. Heydorn, J. Radioanal. Nucl. Chem. Articles, **151**, No. 1 (1991) 139–148
		Method validation	
ISO 17025 Chapter 5.4.5	Fitness for Purpose of Analytical Methods — A Laboratory Guide to Method Validation and Related Topics, Eurachem (1998)	Statistics for Analytical Chemistry, Miller and Miller, Ellis Horwood PTR Prentice Hall (1993)	What Exactly is Fitness for Purpose in Analytical Measurement?, M. Thompson and T. Fearn, Analyst, **121** (1996) 275-278

The most appropriate standard for laboratories performing measurements and calibrations is ISO 17025 standard entitled “General Requirements for Competence of Testing and Calibration Laboratories”, ISO, Geneva, 1999. ISO standards or their national equivalents have been accepted in more than 100 countries around the world. A formal accreditation process, including audits of related ISO 17025 quality requirements implementation, is organized at regional and international levels, leading to mutual recognition of accreditation certificates in a most effective way. International Laboratory Accreditation Co-operation (ILAC) is the top body in this respect. Examples from the regions might be European Co-operation for Accreditation (EA) and Asia Pacific Laboratory Accreditation Co-operation (APLAC). It is advisable to obtain information about existing accreditation bodies in the respective country or region as early as possible.

However, ISO 17025 standard is prepared as a general standard for all types of testing and calibration activities. For this reason the requirements described might need some supporting explanation and guidance for specific areas. Also important are technical articles from scientific journals describing a state of the art of the relevant laboratory activity.

2. PLANNING AND PREPARATORY ACTIVITIES

Development and implementation of a management system for quality assurance requires not only human and material resources. Drafting procedures, discussions on harmonization will go at the cost of the normal productivity of the laboratory. Laboratory management may justify this additional cost in the frame of the overall performance cost of the institution. Not only should the benefit and cost of the operation be specified, but also milestones and deadlines must be given so that it becomes clear beforehand how long it will take, e.g. laboratory accreditation may be applied for.

It is recommended to use the principles of project management for implementation of the quality system. Any project starts with an orientation stage, where the laboratory makes a rough assessment of its challenge and prospect. On the basis of this orientation, line management can take a go/no-go decision if implementation of a quality system should be pursued at all, and when an implementation plan should be provided. This approval by line management supports the technical manager with a stated priority for the development of the quality system in view of the laboratory's other activities.

2.1. Orientation: Objectives and needs

The technical manager conducts the orientation stage, to be concluded with a positioning paper. In this orientation it has to become clear why there is a need for a quality system and for what, whereas an indication is needed of the required resources.

2.1.1. Orientation on the need for quality system

The discussion about having a quality system in a laboratory could be initiated by the head of the laboratory ('technical manager' in the jargon of the quality vocabulary) since he may have read about it in literature, may have heard about it from colleagues or received information about it via related activities with other national or international partners. He may realize that implementing a quality system requires a lot of work, and that it will require from the employees to some extent a change in attitude to the conduct of their daily tasks. The first presentation to the staff of the laboratory of implementing a quality system should therefore be strong and convincing. The technical manager needs to be prepared for this.

At first the technical manager has to assess the needs for implementing a quality system in his laboratory and why accreditation should be attained eventually. Table II present examples of driving forces. Is this process externally driven, e.g. since customers demand for it? There may be legal reasons as well, e.g. to keep the laboratory's license for working. Or are there internal considerations, e.g. excessive repetition of work or poor results in proficiency testing? One or two strong reasons may be enough at this stage: an exhaustive list would rather be implausible.

TABLE II. EXAMPLES OF DRIVING FORCES

External driving forces	Internal driving forces
Request from society: reliability, competent partners, international acceptability, value for money	▪ To reduce repetition of work ▪ To reduce miscommunications ▪ To demonstrate quality of work ▪ To support the change from monopoly to market oriented.
Legal considerations: e.g. nuclear regulatory bodies, legislation, forensic services, metrology networks	▪ To differentiate from competitors and to stay in business.

The manager should inform other employees about his ideas, and tell them, at first in a very general way, about the reasons for having a quality system and his expectations on the benefits and consequences for the daily work. The manager should encourage the other employees to envision opportunities of such a change, and to identify current deficiencies in the organization (e.g. miscommunications, poor documentation, poor planning) or in the conduct of work (e.g. contaminations, repetition of work, unstable equipment) that will need attention. Such 'internal driving forces' are very important for the motivation of the people involved.

2.1.2. Scope of coverage

Next, it is important to decide which areas should at least be covered by the quality system. Will it be just those activities dealing with the external services — like counting natural radioactivity in food products, or neutron activation analysis (NAA) on environmental samples? Or will it be more, since the group providing the services could be part of a larger department with many interacting activities. Even if non-routine is carried out, this should not be considered as restricting for implementing quality assurance. Or, on the contrary, will it be restricted to only one type of measurement, like ^{137}Cs measurements in clay, even though the laboratory is capable to analyze other matrices and/or even perform NAA?

α, β, γ-Spectroscopy, NAA and X ray fluorescence (XRF) share the advantage that simultaneously many radionuclides or elements can be determined, and that the matrix effects can accommodated for relatively easily. As such, instead of limiting to 'determination of ^{137}Cs in soil', a laboratory may easily extend its scope towards 'multi-element determinations in geological material', for example. In general it is recommended to consider a broad scope of applications.

When dealing with α- or β-measurements, a new type of matrix or analyte may require development of new chemical separations. If this 'method development' is a regularly returning part of the laboratory's activities it may be considered for inclusion in the quality system since the ISO/IEC 17025 addresses this item specifically (Clauses 5.43 and 5.4.4).

There is also a EURACHEM Guide available with suggestions for quality assurance in research and development and non-routine operations.

There may be regulatory requirements to the measurement, e.g. set due to the need for export certificates of goods or because of (inter) national comparability. It is important to

collect such requirements during this orientation stage since compliance to them may require additional investments in equipment and/or may set demands to the accommodation.

Another aspect to be considered upfront is if sampling, i.e. collecting the material from the field or industrial plant, belongs to the laboratory's regular activities or not. Many laboratories consider that their task starts when receiving the sample from the customer, and that the customer is responsible for collecting a representative sample. One of the reasons is that, otherwise, the laboratory has to demonstrate that the collected sample is representative of the population. There are opportunities here, of course. In many countries national standards exist for routine sampling, e.g. of soil or surface water, and the subsequent sample treatment and sub-sampling. Even if such a standard is not available in the country, the laboratory may take advantage of similar standards existing in other countries; in the quality manual, and to the customers it then should be made clear that sampling is done in compliance with such a standard. As an example, the sampling procedures of the American Environmental Protection Agency (EPA) or Food and Drug Administration (FDA) are often also internationally accepted.

It is also important to decide which employees will work according to the quality system. In general, it is recommended to avoid situations in which only the technicians doing routine measurements have to deal with the rules and requirements of the quality system whereas others, as the academic staff, have opportunities to circumpass them.

The general attitude is that: "Accepted quality assurance procedures are mandatory to everybody involved in the laboratory — irrespective of his/her position or grade".

The ISO/IEC 17025 does not specify any requirements to laboratory safety or — as in the case of nuclear analytical techniques — radiation safety. Conventional and radiation safety standards are different in each country. However, the laboratory may prefer to pay sufficient attention in its operations and documented procedures to aspects of conventional and/or radiation safety. One of the considerations is that the documents to be developed may serve in the future to train newcomers to the laboratory. There is always a risk that there will be no transfer of expertise on these safety aspects if not documented since the current staff may consider it 'common knowledge'.

2.1.3. Self-assessment of current situation

It is important to make an inventory of what is already available. Sometimes there are already (rudimentary) written procedures, even though they are lists of 'one-liner' only. There may be already notebooks and logbooks for equipment (like a notebook with information on the detector calibration — peak positions and energy resolutions), or people may prefer using the manufacturer's equipment manuals (as with the operation of the multi-channel analyzer). At this stage, it is also relevant to assess which equipment is available in the laboratory, a list of instruments with a notification if they are fit for the purpose or not. This list is not restricted to spectrometers, but should also include balances, milling and sieving machines, etc. It is also relevant to list the chemicals (e.g. pure element standards) and reagents.

Certificates of (certified) reference materials are probably already neatly archived in a binder and, similarly, reports with results of participation in intercomparison are readily available.

In many laboratories equipment is already uniquely labeled with a sticker or stamp and an inventory may exist, e.g. with the procurement department. Table III gives an indication of

typical items that may already exist in relation to the various main clauses of the ISO/IEC 17025. Please note that ‘calibration’ here refers to calibration services, e.g. calibration of the emission rate of radioactive sources, and not to the calibration of spectrometers for daily use.

TABLE III. EXAMPLES OF TYPICAL QUALITY ITEMS

ISO/IEC 17025 Clause	Typically available quality item
4.1 Organization	▪ Organogram ▪ Responsibilities defined ▪ Supervision
4.2 Quality system	▪ Mission statement
4.3 Document control	▪ Written instructions ▪ Calibration tables ▪ Description of software
4.4 Review of requests, tenders and controls	▪ Registration of customer’s request, correspondence
4.5 Subcontracting of tests and calibrations	▪ Usually not applicable in nuclear analytical laboratories
4.6 Purchasing services and supplies	▪ Purchasing documents available in procurement department ▪ Procedure for purchase, reception and storage of purchased goods usually existing but not written down
4.7 Service to the client	▪ Current communication with the customers; invitation to visit the laboratories and facilities
4.8 Complaints	▪ Usually done informally
4.9 Control of non-conforming testing and/or calibration work	▪ Usually done informally
4.10 Corrective actions	▪ Usually done but not registered
4.11 Preventive action	▪ Usually done but not registered
4.12 Control of records	▪ Lab journals, notebooks, worksheets, records of detector calibration, calibration certificates; all paperwork that has information on the conduct of work
4.13 Internal audits	▪ Usually not existing
4.14 Management reviews	▪ Usually not existing
5.1 General	▪ Knowledge on parameters that may affect the test is usually available but not documented

ISO/IEC 17025 Clause	Typically available quality item
5.2 Personnel	▪ Job descriptions ▪ Records of courses followed, etc. ▪ Restrictions to the use of equipment or change of settings
5.3 Accommodation and environmental conditions	▪ Registration of temperature, humidity
5.4 Test and calibration methods and method validation	▪ In nuclear analytical spectrometry: mostly in-house developed methods. Often, validation can be demonstrated on the basis of previously analysed certified reference materials, duplicates, work by different employees. ▪ Uncertainty is often not fully evaluated but major sources are taken into account, like counting statistics. Information on uncertainty evaluation is available through the IAEA.
5.5 Equipment	▪ Regular checks of performance of spectrometers (energy and resolution checks) ▪ Equipment manuals ▪ Institute's codes on labels ▪ Trained people to run the equipment ▪ Worksheets
5.6 Measurement traceability	▪ Certificates of reference materials; sometimes calibration certificate of balance
5.7 Sampling	▪ Often not applicable
5.8 Handling of test and calibration items	▪ Registration of incoming samples, registration of sample treatment in lab journals ▪ Unique coding of samples ▪ Storage facility for samples
5.9 Assuring the quality of test and calibration results	▪ Regular analysis of control samples, blanks, neutron flux monitors ▪ Regular measurement of background ▪ Participation in intercomparisons
5.10 Reporting the results	▪ Your current report to the customer

A first estimate of the resources needed has to be made during this orientation stage. The main problem here is the estimate of the amount of time needed. This depends on many factors: the size of the laboratory and its activities to be covered, the number of employees, the amount of time that can be made available, the quality awareness and commitment of the employees, the employees' abilities to draft procedures, etc.

Experience has shown that a quality system may be implemented in two years in a laboratory of about five employees if the employees can dedicate 15% of their time, on the average, to developing the quality system. At this stage, it is wise to overestimate slightly the time needed, and to aim at completion of the project within three years after the real start.

The material investments are not necessarily very large. ISO/IEC 17025 does not set requirements to the state of the art of instruments; only to their condition: they should be 'fit-for-the-purpose' (Clause 5.5.2). There is seldom a need for major investments in new equipment or additional tools. There is a possibility that chemicals for standardization have to be replaced, and that service by a certified calibration body (e.g. for balances or thermometers) will be needed. It is recommended to make a first estimate of the cost of attending additional training courses on the quality items. The national accreditation body and/or private organizations may organize such courses (as an introduction or for extensive training). At the end of the project, the fee of the national accreditation body has to be accounted for.

2.1.4. Conclusion from the orientation stage: The positioning paper

The positioning paper resulting from this orientation stage has to be comprehensive, i.e. it should contain not more than two pages. It should state clearly the reasons why the quality system has to be developed, expected benefits to which activities it will apply, resources needed and the next step to be taken on the road towards implementation (see Table IV). This will be the definition stage of the project, when a detailed implementation plan will be drafted.

TABLE IV. CONTENTS OF A POSITIONING PAPER

Brief introduction on the international trends on quality
Driving forces for the laboratory
Expected benefits
Areas to be covered and scope
Resources needed
Stages in the project

The technical manager has to present this positioning paper to his line management. Line management should give its support to the next stage of the project, i.e. drafting of an implementation plan. It is important that all employees are informed on the support of line management, and it can be of great help to get a memorandum of line management with a written statement of commitment, as well as with an identification of the priority of it all.

It is even better if line management expresses its approval of the initiative and support to proceed with the preparation via a plenary meeting with the employees involved.

2.2. Definition: Implementation plan

A well conceived plan is needed for an effective and efficient implementation of the quality system. Many quality components have to be put in place and/or have to be

developed. The contributions of various employees have to be co-ordinated and tested for mutual consistence and compliance with the requirements of ISO/IEC 17025. In addition, it may be expected that more explanation on the interpretation of these requirements is needed, and interactions with other parts of the organization may raise questions.

It is assumed above that the technical manager (head of the laboratory) has envisioned the quality system. As such, he is the most appropriate person to guide the process or supervise it. There is a fundamental advantage if the head of the laboratory himself takes the lead in this process since he thus demonstrates the importance and his own commitment.

In relatively larger groups, the laboratory head may wish to be assisted by one of the employees, a specialist who probably will have the capabilities to later become the laboratory's quality manager. This person should, obviously, also be well motivated on the quality issue. By preference, it should be one of the technical staff, someone with a thorough understanding of, and experience with the work process. This person should have such a position that the other employees respect him; he should be tactful to guide the discussions and should have some editorial skills for drafting and finalizing the written procedures.

The technical manager or person in charge of quality system (so called quality coordinator) should study the requirements of the ISO/IEC 17025, Chapter 4 and other background information (see Table I). Additional textbooks are useful, e.g. on the principles of statistics for chemical measurements. The national body may also inform him if additional regulations should be considered for the process towards accreditation, and the other employees and/or customers may call his attention to official methods of measurement to be implemented.

The quality coordinator has to complete this definition stage with a plan for implementation. This will include the workload to be divided. It is therefore important to get a good view on which documents are already available and usable. The current (quality) status of the laboratory's instruments (spectrometers and lab-equipment, including e.g. pipettes): stability (spectrometers), calibration status (e.g. balances), degree of accuracy and repeatability (pipettes), availability of manuals has to be known in details. There may already be rudimentary procedures and instructions thus the quality co-ordinator has to evaluate how they may fit in the quality system to be developed. Also, status of standards, reference materials and calibration sources have to be assessed and their certificates retrieved (if still possible, usually the materials 'survive' their documentation).

Many aspects of the organization of institutions are often already documented. Organization charts may exist, as well as a description of the responsibilities and mandates of the various management levels and job descriptions. Some organizations have mission statements and perhaps also already a written quality policy. The quality co-coordinator should collect whatever is available.

One of the cornerstones of quality assurance is insight on the potential sources of error. This means that the monitoring and accounting of errors is important part of quality control system.

All of the above should result in a draft action plan for implementation. The quality coordinator has to list the activities, including the development of (graphical) control charts for monitoring technical variables, starting an administration, identification and labeling of chemicals and equipment, design of a sample custody system and a first indication of the

procedures and instructions to be written. A first suggestion for milestones, i.e. deliverables at a given point in time should be included in this plan. The milestones should be realistic and simple, for example 10% of the drafts would be ready after six months, 30% of the drafts and 10% of the final documents have to be ready after 12 months, etc. This draft action plan has to be presented to the personnel and the workload divided.

TABLE V. EXAMPLES OF CRITICAL TECHNICAL AND ORGANIZATIONAL PARAMETERS

Technical	Organizational
▪ Counting geometry ▪ Neutron flux gradient ▪ Purity of vials ▪ Energy resolution ▪ Differences in matrix composition of sample and standard	▪ Planning of measurements ▪ Record keeping of customer's requirements ▪ Tractability of events ▪ Training of newcomers ▪ Follow-up of complaints

An illustration of the structure for this part of the work is given in Table V. A template/layout for the documents, with guidance on the aims and contents of the various paragraphs to be completed, and a procedure for verification, amendment and approval of the drafts have to be worked out.

It is important to distribute the work in such a way that the available human resources are used in an optimal way. At this stage it must also be discussed and decided which activities can be done in parallel, and which should be done sequentially.

Finally, when a more definitive action plan can be made, it would become clear who will do what and when it should be ready. This action plan can be summarized in a Gantt chart. It is important to note that it will be practically impossible to make the committed time available as a step-function at the start of implementation. Usually commitments from the past have to be completed first, wand the new requests for work should be scheduled for a longer turnaround time than usual. Therefore, the deliverables at the first milestone should not be overestimated. It is even recommended to postpone the actual starting date for a few months, in order to complete current work and to make available the necessary human capacity by slowing down normal productivity.

The employees involved in quality system should agree with the action plan and demonstrate their understanding and commitment to the project implementation. The plan is subsequently presented to line management for a go/no-go decision. By signing the plan, management also gives evidence of its understanding, support and commitment to the work.

3. IMPLEMENTATION OF THE QUALITY SYSTEM (PRACTICAL ASPECTS)

3.1. Awareness building

At the moment when management takes a decision to implement the quality system, however, it is not to be expected that staff involved would be familiar with the selected

standard and its requirements, or even with the background and reasons for implementing the quality system. The process by which necessary information is shared, creating positive perception of the quality system, is called awareness building.

Awareness building has to reach staff at all levels. As the duties and responsibilities for managerial and technical work are assigned so are responsibilities for the quality system components shared between staff members. For example, management will need to perform a management review and on its basis prepare quality improvement plans; internal auditors will perform audits according to the audit plan and prepare internal audit reports; technical staff will need to prepare technical standard operating procedures, instructions, etc., and keep records in a structured way.

For most of the laboratory staff these activities will only be part-time activities. The quality manager will often be the only person engaged full time with quality system**.** Besides overall responsibility for the implementation and maintenance of the quality system, the quality manager is responsible for initiating and undertaking actions necessary to assure appropriate quality awareness.

Experience shows that quality awareness building might take some time. It is actually an ongoing process. Depending on the size of the organization or laboratory, it might be appropriate for the quality manager to create a group of colleagues who support implementation of the quality system. They should help disseminate the necessary information. Care should however be taken not to impose a strong top-down action.

Training of staff in quality control principle and practice plays important role. This may include:

- Information seminars,
- Lectures (internal or external lecturers).
- Visit to a similar institution with already established quality system
- Direct discussion with staff members
- Provision of examples for quality documentation, etc.

Awareness building, confidence and competence creation are coming through organized training and well targeted courses. Enough time needs to be assured to allow staff members to become familiar with the topic. Good communication skills and transparency is crucial for the team work success.

In most cases, the awareness building and related interest for the quality system are stimulated when there are some achievements already present. It might also be appropriate to limit the initial implementation of the quality system to selected activities in the laboratory. Quality system implementation for routine tasks of the laboratory is normally more straightforward than for the research and development, ad hoc or non-routine activities. All of this depends on the size and activities of the organization implementing a quality system and cannot be generalized. Specifics of the organization need to be well recognized and the awareness building structured accordingly.

In conclusion, awareness building is an integral part of the quality system implementation. Exchange of ideas, personal communication and clearly defined aims are prerequisites for its success and effectiveness a good information flow.

3.2. Quality control and validation

3.2.1. Introduction

QC process contains technical core activities within it. The major aim of QC is to maintain technical processes "under statistical control". This is of particular importance for an analytical laboratory because the laboratory's product — analytical results on unknown samples — cannot be directly verified. Hence, the only possibility to generate confidence in the correctness of the laboratory results is providing evidence that the analytical processes were under control during the time of the measurements. QC in particular, addresses those measures that are used to verify the validity of the final results. Validation monitoring aims to prove whether the results are acceptable and no mistakes have been made.

QC ought to be planned, described in the quality documentation, performed in a systematic manner, recorded and reviewed. To reduce the fraction that has to be rejected, QC must be embedded into an overall systematic approach to avoid mistakes *before* they are made, and this is commonly referred to as "quality assurance".

The indispensable complementary measures (with some detailed suggestions) are:

- Planning: identify (what) and define (how):

  – type of quality control
  – frequency of quality control (when)
  – control limits
  – actions if control limits are exceeded
  – periodic review of results.

  Documentation: describe QC and complementary measures, responsibilities (who), control limits, evaluation and review, follow-up, corrective actions.

- Recording:

  – results with attributes (date, procedure, material, operator, instrument, calibration and correction factors, values of relevant environmental parameter, other experimental parameters)
  – association to production work
  – which QC results are relevant for a particular sample
  – which samples are affected by a particular QC result
  – quality control charts
  – corrective actions.

- Review:

  – quality control charts
  – frequency of out-of-control situations
  – trends and drifts
  – irregular patterns
  – statistical evaluation
  – repeatability (short-term fluctuation)
  – intermediate reproducibility (mid- and long-term fluctuation)

- uncertainty components (fluctuation attributable to factors such as operator, instrument, material, calibration, treatment, etc.)
- identification of systematic effects
- bias
- agreement with method validation and customer requirements
- refine control limits
- assess effectiveness of QC system
- non-conforming work "escaping" the controls
- economical balance (why)
- effort commensurate to type, frequency and criticality of work (too much)?
- are measures appropriate to avoid unacceptable risks (too little)?

3.2.2. Statistics and charting

Where a result from a particular measurement process is monitored over a period of time, a great deal of data is generated. These data are normally presented in a table for interpretation. One of the most useful ways is to present the data in a graphic and plot them in a control chart. The user can define warning limits on the chart to act as 'warning bells' when a trend shows that the system might go out of control (a preventive signal) or real 'alarm bells' (action limits) when the system is out of control (corrective actions have to be taken). Examples of control charts are: the FWHM of a peak in a spectrum, the measured activity of a calibration source, background counts, readings from thermometers or humidity monitors, etc.

Successive measurements of a characteristic attribute using a particular method will show a natural variation arising from the method. The set of results or population will have an average or mean value and, most commonly, the individual measurements will be symmetrically distributed around this mean in a normal or Gauss Ian distribution. The distribution of data about the mean value is governed by the standard deviation and, statistically, it is somewhat unlikely (5% probability) for a member of the population to be farther away from the mean than two standard deviations and very unlikely (0.3 % probability) to be further away from the mean than three standard deviations. Thus 95% will always lies within ±2 standard deviation of the average, while 99.7% will always lie within ±3 standard deviation of the average. Further measurements should behave in the same way and lie within those boundaries. If they do not, then it is probable that some changes have occurred to the measurement system, which has significantly altered its performance, thus causing a shift in the mean or an increase in the standard deviation. The purpose of the chart is to make this change evident. The user must decide whether or not this change is significant.

The simplest type of a QC chart is the Shewhart Chart. It is typically used to monitor day-to-day variation of an analytical process. The variation of an established 'standard' or quality control sample is shown in the Chart. Measurement values (activity, FWHM) are plotted on the y axis, while time of successive measurements on x axis (i.e. daily, weekly, etc.). The QC sample could be a radioactive source of different energies where the low energy peaks allow to monitor electronic noise and the higher energy peaks monitor the resolution of the detector. The Shewhart Chart can show the data in daily basis, but if the counting system shows enough stability, measuring time interval could be enlarged to weekly. Background control charts could be performed on a weekly basis during the weekend. As long as variation in the measured results for the QC sample is acceptable, it is reasonable to assume that the measured results for real samples in those batches are correct.

But how could it be determined what is acceptable and what is not?

Statistical analysis of measurements is a reliable tool to check the acceptability of data. First of all, the QC sample is measured a number of times (about 20 times) in the same day. The average or mean value and the associated standard deviation are calculated. The mean value is frequently used as a 'target' value on the Shewhart Chart, i.e. the value to 'aim for' or so called "true value". The standard deviation is used to set the warning limits on the chart. The mean value and its standard deviation is shown in the Chart. Once the chart is set up, day-to-day QC sample results (mean value and standard deviation) are plotted. Recording the mean values and their standard deviations on the chart each day, the precision and accuracy of results become visible. Anomalies or unwanted patterns such as 'drift', or results lying outside the warning or action limits can be detected.

Figs 1–4 show Shewhart Charts for four types of data:

(i) data subject to normal variation,
(ii) data subject to normal variation but displaced,
(iii) gradual drift,
(iv) step change.

It is accepted to fix 'warning limits' at ±2 standard deviations and; action limits at ±3 standard deviations. From the statistical rules is expected that only 3 cases in 1000 measuring data fall outside the action limits, and only 1 case out of 20 measuring data fall between the action and warning limits.

When using control charts and statistical evaluation of data, any point that falls outside the action limits is considered as potential error and points that exceed the warning limits have to be analyses carefully. There are also some other signals, which normally indicate a problem with the measuring system:

(i) two successive points outside action limits,
(ii) four successive points outside warning limits,
(iii) ten successive points on the same side of the mean,
(iv) an upward or downward trend of several points.

Several control charts can be set up in nuclear analytical techniques. For example in the γ spectroscopy the evaluation of the measuring system can be performed as follows:

- FWHM chart provides evaluation of the QC of the resolution of a detector. A degradation (enlargement) of the FWHM at low energies could be caused by electronic noise inside the detector, while at high energy might indicate a vacuum problem.
- FWTM chart allows to monitor tailing due to damage of the detection crystal, bad P/Z ration may indicate a leakage current
- The ratio FWHM/FWTM for different gamma ray energies allows to monitor the quality of the detector
- Activity chart allows to monitor the stability of the whole measurement method
- Background chart might discover contamination
- Peak position chart provide indications for electronic stability and the influence of environmental conditions such as temperature or humidity.

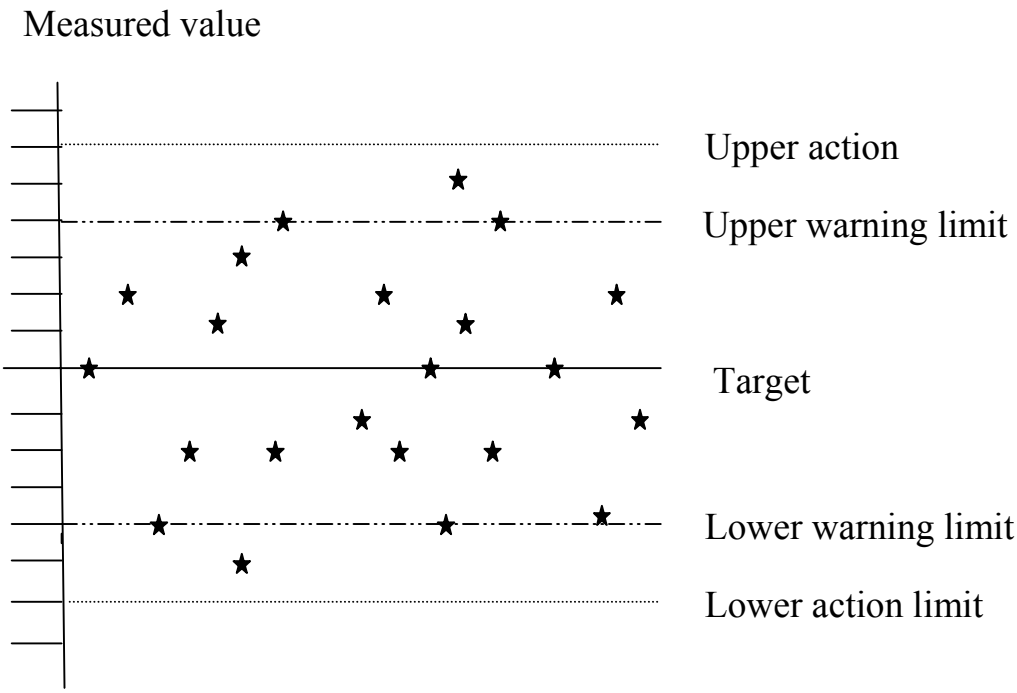


FIG. 1. Shewhart chart showing data in control about the target value.

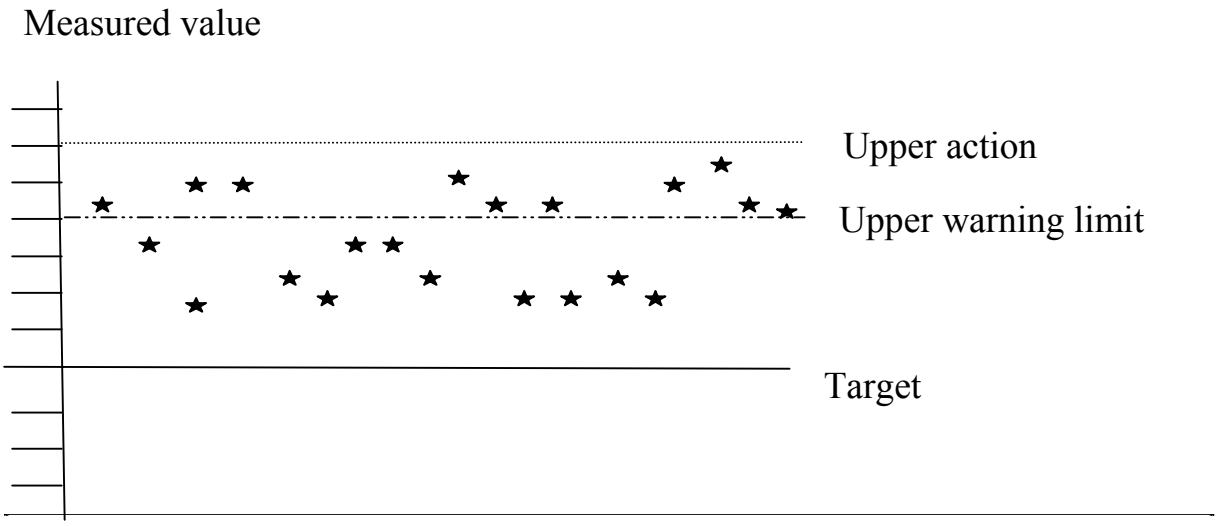


FIG. 2. Shewhart chart showing data offset from the target value.

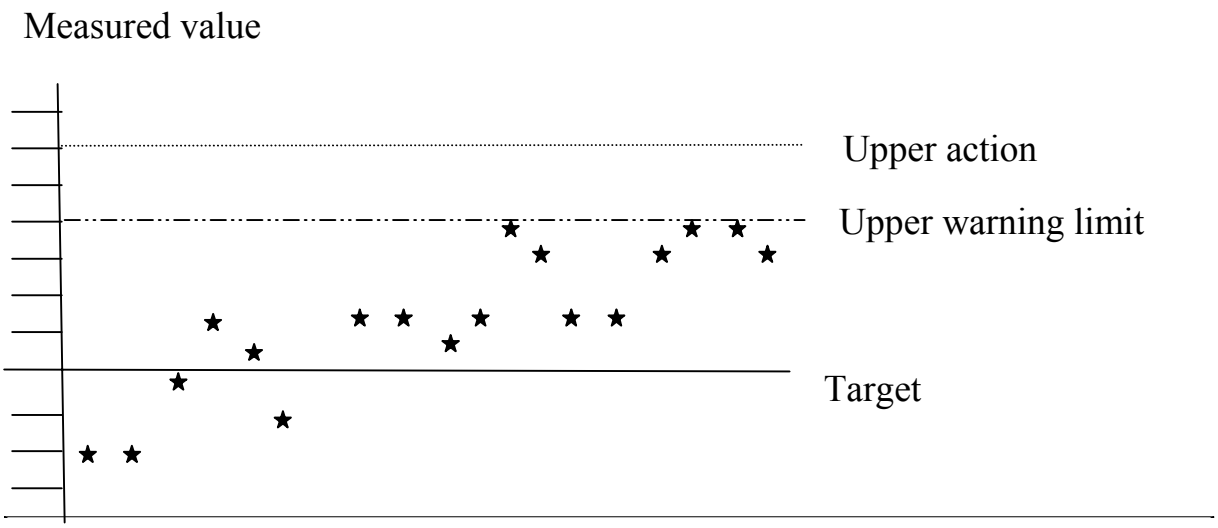


FIG. 3. Shewhart Chart showing drifting data.

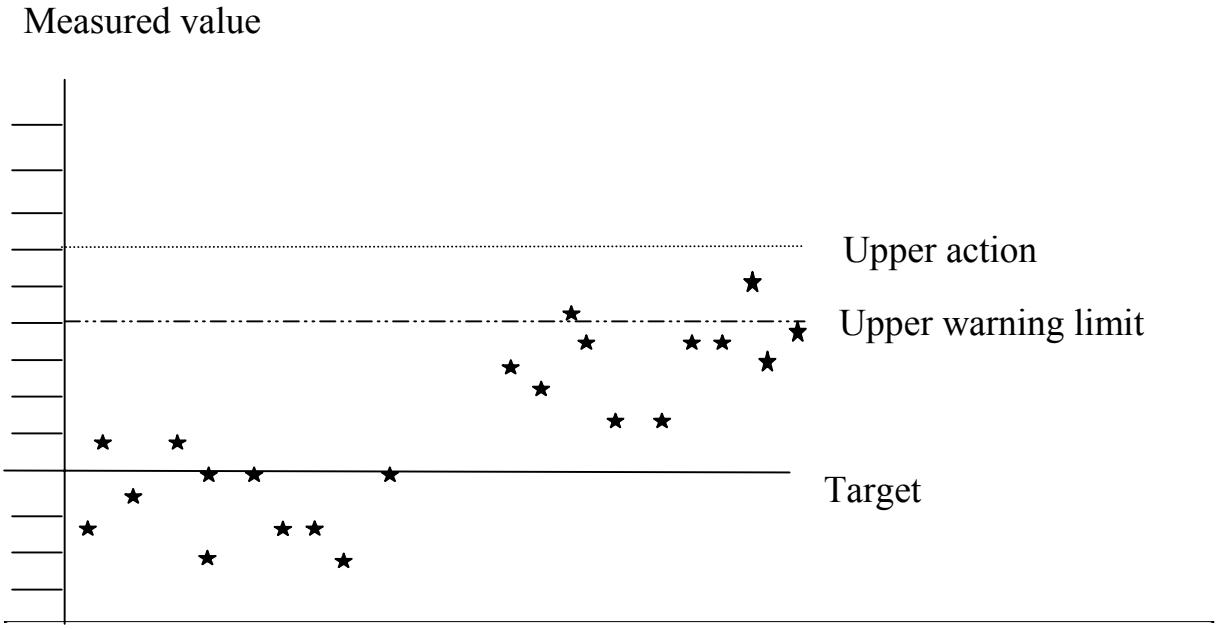


FIG. 4. Shewhart Chart showing data with a step change.

3.2.3. Use of Certified Reference Materials (CRM)

It is a requirement of ISO/IEC 17025 that measurements be traceable to the SI or other appropriate references such as certified reference materials (CRM). Where ISO/IEC 17025 refers to the calibration of equipment, in nuclear analytical techniques, it is more appropriate to consider the calibration of the whole measurement process.

This is done by using CRMs as a transfer standard from NIST, PTB, IRMM, NPL, etc. for the calibration and validation (such as efficiency calibration at different energies). The certificates of these materials provide important information to be included in the evaluation of the measurement uncertainty.

3.2.4. Validation

Method validation is an essential part of good laboratory practice and is a requirement of ISO/IEC 17025 for in-house methods. Because most of nuclear analytical tests are not standard methods, the laboratory should demonstrate that the method is "fit for purpose" before introducing the test in routine. “Fit for purpose” means the method meets client’s requirements or generally accepted international levels (e.g. levels of minimum detectable amount).

For illustration purposes, let construct a scenario where a customer wants to test a laboratory. From this external viewpoint, the most important parameter may be described, as follows:

Repeatability (precision): The customer sends ten identical samples from a homogeneous batch of material at one time to the laboratory; the spread of the ten reported results would then be a representation of the repeatability. The observed fluctuation would essentially stems from short-term (“random”, within-batch, within-run) effects.

Intermediate reproducibility: The customer sends ten identical samples from a homogeneous batch of stable material to the laboratory, but in intervals of two months; the spread of the ten results, reported in distances of two months, would then be a representation of the intermediate reproducibility. The observed fluctuation stems not only from short-term effects, but in addition it arises from factors changing between batches and runs with a longer period of time. Examples for such factors are: operator, instrument, maintenance or repair work, calibration, reagent or reference solution batches, seasonal fluctuations, run effects, etc.

Laboratory bias/accuracy: The customer would probably like to know how accurate are the results, that means how results fit with the reality (true value). The closeness of mean value with the true value (assuming it is known) represents a measure of the accuracy of the laboratory.

Reproducibility: The customer sends ten identical samples from a homogeneous batch of stable material to ten different, independent laboratories; the spread of the ten results reported by the ten different laboratories would then be a representation of the reproducibility. This parameter is not subject to control by laboratory internal QC measures. It is usually assessed during the method validation or during special round-robin experiments and monitored in the frame of external QC (interlaboratory comparisons and proficiency testing programmes) exercise.

Other criteria: There are other quality criteria that might be applied in some cases:

1. *Selectivity and specificity*
   - To which extent is the method sensible to interferences,
   - What measures are in effect to assure the absence of (or correction for) critical interferences?
2. *Decision limit, detection limit, quantification limit, rate of non-detection, and rate of false detection*
   - These are closely related to the sensitivity of the method and to the control of blank, background and cross-contamination.

3. *Background, blank and cross-contamination*
   - *Background* means the measurement signal observed in the absence of analyte
   - *Blank* means role of impurities in the reagents; in principle it has the same effect as *background*
   - *Cross-contamination* has a similar effect as *blank*, but refers more to an accidental occurrence
   - *Background* and/or *blank* can usually be measured and corrected for, but their level and fluctuation pose also limitations on the achievable accuracy and contribute to the overall uncertainty
   - Especially for low level type of analysis, careful control of *background* and/or *blank* is essential; evaluation with the help of QC charts is highly recommended for early detection of adverse trends and drifts arising.
   - Laboratories dealing with samples with a wide variation of analyte levels will certainly have a need to implement appropriate process control measures to avoid cross-contamination, such as cleaning, monitoring or separate equipment.

4. *Matrix effects*
   - There may frequently be problems in obtaining appropriate and representative reference standards and control samples for a wide variation of sample materials
   - Development of standard addition techniques (spiking) and characterization of (secondary) in-house working standards are perhaps some of the few workarounds to that problem.

5. *Selection of QC measures*

From a practical viewpoint, QC is usually implemented at two different levels: final measurement and analytical procedure, as schematically shown in Fig. 5.

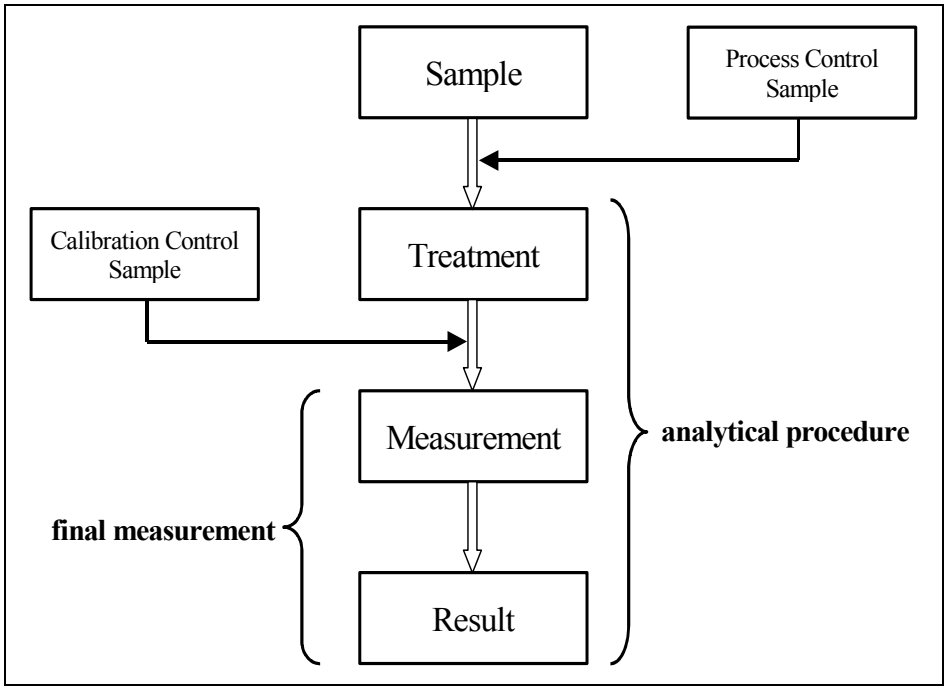


FIG. 5. Scope of QC measures (schematic).

Table VI lists potential QC measures corresponding to the target parameter and the scope of the QC measure.

TABLE VI. LIST OF QC MEASURES

Parameter to check	Final measurement	Whole procedure
Repeatability	Replicate measurements on preparations of routine and/or QC samples	Replicate analysis of routine and/or QC samples
Intermediate reproducibility	▪ Long term observation of QC measurement results ▪ Variation of calibration factors ▪ Re-measurement of retained sample preparations	▪ Long term observation of QC sample results ▪ Re-testing (full analysis) of retained sample material
Accuracy/bias	▪ Measurement of validated preparations of certified reference materials ▪ Measurement of primary or secondary reference standards ▪ Comparison with an alternate, independent measurement method on the same preparation	▪ Participation in proficiency testing ▪ Participation in interlaboratory comparison ▪ Customer supplied blind controls
Background	Background measurements	
Blank		Analysis of blank control samples or control samples with analyte levels at the lower end of the working range
Matrix effects	Measurement of reference standard with comparable matrix	▪ Analysis of control samples with comparable matrix ▪ Standard addition (spiking) techniques
Selectivity/specificity	▪ Comparison with an alternate, independent measurement method ▪ Application of spiking/tracer techniques ▪ Scientific reasoning	▪ Analysis of control samples and/or blanks spiked with potential interferences ▪ Scientific reasoning
Cross-contamination	▪ Measurement blanks ▪ May be covered by other QC measures above	▪ Room and/or workplace blanks ▪ Cleaning/monitoring protocols ▪ Identification of equipment and records of usage
Overall confirmation		Representative and regular proficiency testing

6. Organizing review

As a general experience it happens that many laboratories do not include QC samples with each batch of samples processed. Frequently cost effective laboratory-developed QC samples are not available and CRMs are considered too precious to be consumed on a regular basis. As a result QC is often limited to the analysis of blanks.

It should make clear that even though the method may be validated on the basis of CRMs, this does not guarantee absence of systematic errors during analysis. In neutron activation analysis (NAA), QC can be extended to verification of neutron flux gradients, controls on the correctness of the irradiation date, time and duration, decay period, and correctness of the dead-time correction. All of these can be accomplished by analyzing a material of known composition together with the real samples. Use of control charts of course is indispensable to inspect for trends and decide if a deviating result is just the odd-one out, or really implies that the measurement reflects a systematic error. If a laboratory operates using sample changers, they need QC to assure that the samples are placed in the right order in the sample changer. This may be accomplished by putting the QC samples in between the normal samples in such a way that the entire batches are divided in asymmetric portions.

We should also refer here to the opportunities of developing laboratory control materials on the basis of locally available goods, in combination with control charts.

3.2.5. Measurement uncertainty

Measurement uncertainty estimates should cover all values that can be reasonably attributed to the measurand.

The following requirements of ISO/IEC 17025 are particularly noted:

- Significant components of measurement uncertainty should be identified (components less than 1/5 of the total measurement uncertainty will not usually have much impact on the total measurement uncertainty);
- A ‘reasonable estimate’ of the (total) measurement uncertainty needs to be made;
- Consideration of reproducibility (ISO 5725) alone may not be sufficient and additional effects need to be considered such as curve fitting, geometry and density of the sample, coincidence summing;
- When existing data are used as basis for estimating measurement uncertainty, then additional studies may still be needed to evaluate some components. However, the new work should not involve major new research and development (R&D).
- Strategies for evaluating measurement uncertainty are available in ISO GUM and the IAEA-TECDOC “The IAEA guide on quantifying uncertainty in nuclear analytical measurements” (in preparation). But adherence to these strategies is not an ISO/IEC 17025 requirement.

3.3. Instrument and laboratory management

Quality assurance not just aims towards prevention of unacceptable results. It also serves to improve the performance and efficiency at the laboratory by reducing the number of repetitions due to, e.g. careless preparation or unqualified errors. It implies extensive attention

to fitness for the purpose of measuring equipment and all associated tools, such as pipettes, chemicals and reagents, and their actual use.

In many laboratories it is already a good practice to check, e.g. the energy resolution of gamma ray spectrometers, but such fitness tests are not always systematically done for other equipment and supporting materials. Using control charts helps to identify possible systematic deviation from regular performance in time.

3.3.1. Management of equipment

Errors, duplication of work, repetition are often due to confusion in design, execution, management and responsibility during the practice. The responsibility implies certain duties as well as given rights. A staff member should take over the responsibility for the instrument(s), chemicals, reference materials, etc. Several types of instruments can be identified requiring a different rigidity of management (Table VII).

TABLE VII. CLASSIFICATION OF BASIC INSTRUMENTS

General service equipment	Not used for making measurements or with minimal influence on measurements, such as hot plates, non-volumetric glassware, etc.
Measuring equipment	Gamma ray spectrometers, hydrometers, spectrometers, timers, balances
Volumetric equipment	Flasks, pipettes
Measurement standards	Masses, reference thermometers
Computers	

Examples of tasks and rights of a manager of instruments and material are given in Table VIII. Obviously, the equipment managers, depending on the size of the laboratory can manage group of instruments based on their experience and profile.

'Single point of failure' is a potential risk when only one equipment manager is taking over all the responsibilities. Therefore, it is essential (and required, refer to ISO/IEC17025 Clause 4.1.4j) that other laboratory specialists should be able to perform the most essential tasks, such as fitness tests, and have an understanding of the critical parameters, acceptance specifications and about what to do in case of non-conformance.

Making staff responsible for instruments helps to ensure that the instrumentation and laboratory tools have the required quality status when the experiments commence. A poor human performance may have detrimental (and sometimes even harmful) effects on the quality of work of other people as well, and may even damage the instrumentation or quality of standards and reference materials. Continuous training of staff is important in this respect. The training includes all aspects of instrumental analysis with a final goal to qualify respective staff as authorized users.

TABLE VIII. EXAMPLES OF TASKS AND POWERS OF PERSONS, RESPONSIBLE FOR INSTRUMENTS, LABORATORY TOOLS AND LABORATORY PREMISES

Tasks	Rights
▪ Receipt control of new instruments, etc. ▪ Fitness (performance) test of instruments, etc. ▪ Drafting standard operating procedures for the use of the instruments, etc. ▪ Verification of rules for good housekeeping (especially for laboratories), and rules for safety, good order and discipline ▪ Training and qualification of other employees ▪ Management of keys (if relevant) ▪ Maintenance of the logbooks ▪ Preparation of a maintenance and calibration scheme (if relevant) ▪ Inspection of user lists ▪ Follow-up actions after non-conformance registration ▪ Contacts with suppliers and experts, if relevant For persons, responsible for the laboratory's premises: ▪ All management tasks for instruments and tools in the respective premises for which no special responsible person has been appointed.	▪ Putting instruments into service and /or taking them out of service ▪ Fitness for the purpose of instruments. ▪ Qualification of employees Extension/ending of qualifications ▪ Selection of spare parts, new instruments, etc.

3.3.2. Labelling, identification and administrative aspects

Quality management of instruments aims to know the current status and performance of equipment. There are several administrative options to identify the performance of instrument and material, varying from the use of red and green stickers to the use of small notification sheets with the instruments. Also, such identifications 'fit' and 'not fit for the purpose' have to be applied to chemicals, reagents, standard solutions, and reference materials. This may sound reasonable for self-made reagents. It is also recommended to first analyze, e.g. in 6–10 fold, a newly procured (certified) reference material or laboratory control material before making it generally available for quality control.

Instruments that are significant to the conduct of analysis should be uniquely identified (ISO/IEC 17025 Clause 5.5.4). In many laboratories an inventory system already exists, and durable instruments often have a sticker or stamp with a number, or identification is otherwise applied. A laboratory is entirely free in selecting its coding system. It may prefer to identify in the code the year of procurement and the type of instrument. The coding can be directly linked to an administration in which, as a minimum, the relevant information from ISO/IEC 17025 Clause 5.5.5 is kept).

Improper standardization is one of the main causes of error in the analytical laboratory. It is often due to improper stock solutions of the standard. Typical mistakes made are miscalculations, use of standard and sample at different temperatures, changing concentrations due to solvent loss, deterioration due to interaction with CO_2, etc. It should be prohibited to pour unused portions back into the reagent bottle. This is often done to save costs, but it may result in general deterioration of the entire reagent.

Chemicals, reagents and stock solutions should be labelled with typical information such as identification number, composition and, if relevant, concentration, toxicity, inflammability, pH, solvent, date of purchase/production date, expiration date. All information on preparing stock solution should be kept in a notebook. It should remembered that reference solutions often expire within one year (or even shorter) after opening or preparation.

The location of chemicals should be carefully selected. Whereas conventional safety sets requirements to the storage of chemicals the laboratory may decide to separate certain chemicals in order to avoid cross-contamination. Typically, chemicals used for standardization should be stored and/or handled separately from reagents. The same applies to glassware, spoons, etc.

3.3.3. Use of equipment

It is important for the laboratory to keep track of the history of use and tests of its instruments from the moment they were procured until they become obsolete. If use of instruments is recorded new users have an immediate insight into the current activity and use in the past. For instance, a users list with a balance can identify not only the date of use and user but also the substance weighted to prevent a potential risk of contamination.

The use of standard or reference solutions may be recorded together with information on the mass of the total bottle before and after its usage. Especially if such a solution is not regularly used, such a record makes it possible to detect evaporation and transpiration losses of the solvent, and thus changes in the concentration.

Quality management of instruments has its implications if a laboratory has to share them with other users. The laboratory may demand that at least a record of the use of instruments is kept, and that the fitness for the purpose is being demonstrated upon return. The ISO/IEC 17025 addresses this subject explicitly in Clause 5.5.9.

As with equipment, the risks of loaning chemicals to outsiders should be well considered. Can the outsider demonstrate that after the loan the chemical is of unchanged quality?

Part of the information on the use of instruments consists of the records of non-conformance. This can be non-conformance due to inability to meet specifications, malfunctioning (breakdowns, faults, interferences). This non-conformance registration may be kept in a logbook of the instruments or combined as a special chapter in the entire register of non-conformances. The non-conformance may be observed by any employee of the laboratory, and should be addressed to the person who is responsible for the instruments for cause analysis and remedial and/or corrective actions. The responsible person may consider ordering the non-conformances in such a way that trends and/or correlations may be found (like malfunctioning as a function of the day of the week, the person who used it, temperature, etc.).

Sometimes equipment is left running outside office hours, e.g. overnight or in the weekends (like for drying of samples, or simply for measurement of radioactivity). Situations may occur where the equipment may have to be switched off, but this may have disastrous consequences if done wrongly by a non-authorized person. Therefore, it is recommended to put a simple notification with such equipment about what to do if a dangerous situation (e.g. onset of a fire) is suspected.

3.3.4. Fitness tests

The laboratory has to check that instruments are 'fit for the intended purpose' (Clause 5.5.2). This is achieved via a fitness or performance test. Such an inspection should be a planned activity that can be specified in the quality manual, e.g. each time before the instrument will be used or every Monday morning between 8 and 9 a.m.

The laboratory is free to choose the interval of such performance tests as long as it can demonstrate that it is highly unlikely that the instrument was not fit for the purpose in between tests. The quality indicators, forthcoming from the tests (such as energy resolution, peak position, background levels, pipette repeatability) should be verified to unambiguous quantified criteria, also specified in the quality manual. The choice of these criteria depend on the status of the instrument, mission of the laboratory and type of tests, impact of the measured parameter to the quality of the test, effect of non-conformance to the operation of the laboratory, etc. It is recommended to plot the value of the measured parameter in a (control) chart to inspect for trends and, if relevant, to justify that the interval between tests could be safely extended. It is even better to register more than one parameter this way, such as the peak position and FWHM on 122 keV and on 1332 keV; the reading of the balance at a 10 mg calibration mass and at a 100 mg calibration mass, etc.

The performance test is carried out by either the staff responsible for the given instrument (as with gamma ray spectrometers), or by each qualified employee of the laboratory (as with pipettes). In either case, documented procedures are needed for these performance tests in which the criteria are specified and reference is given on the powers in acting on non-conformance. Fitness or performance tests may result in adjustments of the equipment and sometimes in corrective maintenance so as to bring the equipment in a state of fitness.

Calibration is a special type of fitness test. Calibration is well defined as 'the set of operations which establishes, under conditions, the relationship between values indicated by a measuring instrument, or values represented by a material or reference material, and the corresponding values of a quantity realized by a reference standard'.

The relationship between channel number of the multichannel analyzer and the gamma ray energy is therefore a calibration. The relationship between the emission rate of a radioactive source and the detector's efficiency is a calibration. The determination of the repeatability of a pipette is a calibration. Calibration does not imply a change in the setting of an instrument. For some instruments (as with balances, thermometers, hydrometers) the service of a certified calibration body may be considered to attain the link to the reference standard. The calibration body also assesses the value of the 'calibration mass', built-in in many of the balances nowadays available. Also sets of calibrated masses have to be re-calibrated regularly.

Calibration differs therefore from adjustment, defined as 'the operation intended to bring a measuring instrument into a state of performance and freedom from bias, suitable for its use'. Adjustment is an action, which implies a change in the setting of an instrument; it often follows the calibration step. The zeroing of balances is an example of this.

3.3.5. Preventive maintenance of equipment

Instruments in a neutron activation analysis/gamma ray spectrometry laboratory need relatively little maintenance. One might consider the LN2 filling of the Dewars as a type of

maintenance. It is recommended to consider a schedule for preventive maintenance of some instruments such as high voltage supply units (removal of electrostatically attracted dust), vacuum pumps (as with a freeze dryer, refreshment of the vacuum oil), balances, sample-size reduction machines, clean benches, glove boxes (replacement of filters). Maintenance should be announced on time to the employees of the laboratory (Clause 5.5.6).

The laboratory may also consider using labels on the equipment that identify the maintenance day. A non-conformance sheet should be filled in if maintenance cannot be performed on the planned day.

There is hardly a need for an extensive stock of spare parts for this type of instruments. Vacuum oil should be in stock if vacuum pumps are intensively used. Observations during maintenance, adjustment and/or repair (and also LN2 filling) should be registered. Typically, maintenance is done by the person responsible for the instrument; however, maintenance may also be contracted out to an external body in which case a maintenance report should be provided.

3.3.6. Good housekeeping

Rules for 'good housekeeping' (Clause 5.3.5) are a quality management tool to control and to manage the conditions under which work has to be carried out in the premises (laboratories and offices) — such as conventional safety, radiation safety, waste management, stock and supply control, cleaning, etc. In many laboratories 'good housekeeping' is often erroneously addressed as 'good laboratory practices'. Some of these rules, especially those related to safety, will be based on legal country rules. Others are based upon consensus between the users of the premises. Typically, it is not easy to lay down these rules into the format of procedure (or work instructions). Therefore, a document with some descriptive text is often preferred. Some items that may be considered as 'good housekeeping' are listed in Table IX.

TABLE IX. SOME TYPICAL EXAMPLES OF GOOD HOUSEKEEPING

Cleaning up, taking care of the look of laboratories and offices
Management of laboratory white coats
Cleaning and storage of glassware
Waste management (normal, radioactive, glass, liquid, paper, animal)
Smoking/eating regulations
Use of photocopy machine
How to act in case of emergency (first aid, who to warn, etc.)

Nonetheless, some aspects of good housekeeping, like stock control, are preferably described in a procedure. The laboratory has to implement a system that ensures that work will not be halted by a shortage of appropriate reagents, reference standards, standard solutions, glassware, pipette tips, tissues, forms, gases, spare parts and other important consumables. An inventory should be made of the minimum and maximum amounts needed for undisturbed continuation of daily operations. The minimum amounts needed in stock depend on the rate of consumption and the time needed within the organization to replenish the stock (ordering, purchasing, delivery time). The maximum amounts also depend on safety aspects (chemicals), stability, storage capacity and budget. An example of a stock control list

is given in Table X. For all consumables, an inventory should be made, specifying e.g. the vendor, catalogue numbers, container size, grade, and all other important specifications. It must be clear who is the responsible person for the stock management. This person should be empowered to order the replenishment. The procedure has also to describe how to address the responsible person if the minimum supply is about to be used.

TABLE X. EXAMPLE OF STOCK CONTROL

Item	Minimum amount	Maximum amount
Wipers	2 bags	3 bags
Kleenex tissues	4 boxes	40 boxes
Gloves	1 box	3 boxes
Pipette tips	1 box in use	1 box of 100 pcs.
Capsules	100	300
Rabbits	4	14
Filter paper	3 boxes	10 boxes of 1000 pcs.
Ethanol 96%	1 L	2 L
Acetone	0.5 L in bottle	1 L
LSC vials	1 box in use	2 boxes of 125 pcs.
Sealing foil	1 piece in use	1 new pc.

Safety instructions are often already available. If not, the laboratory may wish to develop a concise booklet with a systematic overview of the rules for conventional and radiation safety.

Sometimes organizations try to have all of the quality documents available through the organization's intranet. For safety instructions internet is not recommended; hard copy documents are preferred. Laboratory staff should be provided with detailed information on the use, safety, (radio) toxicity, hygroscopic behavior, flammability, etc. of chemicals and reagents. Merck index can be used in this case. In addition the laboratory may desire to have at least one person trained for providing first aid, who eventually may be a voluntary fire fighter.

3.3.7. Computers

Computers are often not taken into account if it comes to management of instrumentation, whereas they often form an integrated part of gamma ray spectrometers, or are even being used to control miniature neutron source reactor (MNSR). Laboratories are developing their own software to control and interpret their measurement data. Therefore, ISO/IEC:17025 contains general clauses related to the use of computers and software in the laboratory (4.12.1.4; 5.4.7.2). The laboratory has to document and validate properly home-made software while it is implicitly assumed that commercially available software fit for the purpose. However, it should be noted that in rare cases software routines do not perform as they are supposed to. It is worthwhile to mistrust and validate these products at least once before use.

Backup procedures are important not only to archive measurement data after they have been processed, but also to safeguard data if the computers would be affected by a virus or by electromagnetic shocks.

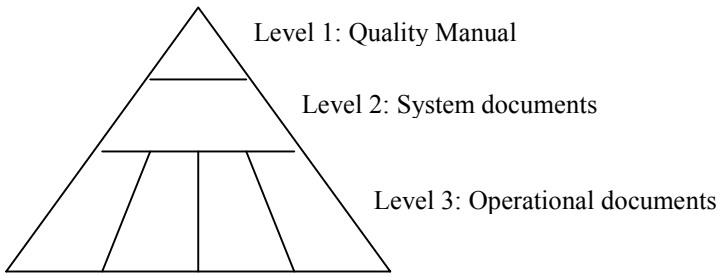
4. IMPLEMENTATION OF THE QUALITY SYSTEM (DOCUMENTATION)

Documenting analytical methods on which the work of the laboratory is based is a part of the larger documentation system that is required by the quality standard

4.1. Overview of documentation system

4.1.1. Introduction

It is convenient, for the introduction of this topic, to classify all the documents and forms that comprise the quality system into three "levels", as shown in the diagram below.



Level 1 describes the purpose and organization of the laboratory, its policy and commitment to quality, and how it complies generally with the requirements of ISO 17025. It typically contains only one relatively short document, the "quality manual" that can be handed to clients for their information.

Level 2 documents describe in detail the procedures and systems that the laboratory maintains to ensure that ISO 17025 is satisfied in every relevant aspect. There are no prescriptions on how the material should be divided and how many documents are required for this purpose, but many laboratories find it convenient to structure the material in such a way that each document is focused on one of the major issues in the standard: personnel, documentation, deviations, etc.

Level 3 contains the bulk of the quality documents: instructions and validation for new analytical methods, internal QC procedures, forms for recording intermediate data and final results, etc. Some laboratories structure their system in such a manner that some of the Level 2 documents are supported by groups of documents in Level 3, while others prefer no direct relationship between Levels 2 and 3.

4.1.2. Requirements of ISO 17025

Each document and form in the quality system must comply with certain formal requirements in the quality standard: unique identification code, numbered pages, formal authorization.

These requirements and ways to satisfy them will not be discussed here because: (i) they are available from the standard, (ii) there is little motivation for recommending a particular format, and (iii) many organizations already have instructions on the layout of documents.

The person, who designs the template that will be used for writing all the other documents, is cautioned against too much non-essential detail that may obscure crucial information. One should also be aware of pages containing little information, and which only fill the files and cause resentment with operational staff.

4.1.3. Proposed structure for documentation system

4.1.3.1. Identification codes for documents and forms

It is a requirement of the standard that every document and form shall have a unique identification code. The following format is proposed for this purpose:

LAB-ABC-123

where

- LAB identifies this laboratory within the larger organizational structure of the institute,
- ABC indicates a particular type of document or form,
- 1 2 3 are a series of numbers typically assigned in sequence.

The way all the documents are coded in different "types" depends entirely on the needs of the laboratory and cannot be prescribed. The quality manager is advised, however, to formalize this issue as early as possible. Once the process of composing the different documents has started, it becomes exceedingly difficult to revise the codes. It is also important to involve management and laboratory staff in this process to ensure better coverage of the activities and get early agreement. A straightforward codification system is suggested in the following paragraphs to assist the quality manager in this process.

4.1.3.2. Documents relating to quality management

- Documents describing the quality system itself, i.e. the quality manual and all the Level 2 documents (e.g. LAB-SYS-123).
- Work instructions or standard operating procedures (SOP) that give detailed instructions on how to collect and prepare samples, separate and/or measure the analyte, calibrate the instruments, carry out tests, etc. (e.g. LAB-SOP-123).
- Documents containing data that are essential for operating the quality system and for carrying out the laboratory work: inventories of system documents and forms, lists of counting instruments and testing equipment, responsibility and authority of personnel, codification system, etc. (e.g. LAB-DAT-123).
- Technical reports on the validation of new methods (e.g. LAB-VAL-123).
- Reports from ad hoc tests on the performance of routine methods and instruments that are in use: to expand scope of method to another matrix or lower concentration, to find and eliminate cause of unexpected behaviour, to compile data from proficiency test or inter-comparison study, etc. (format: LAB-PFT-123).

– Reports of ad hoc tests on the performance of routine methods and instruments that are in use: to expand scope of method to another matrix or lower concentration, to find and eliminate cause of unexpected behaviour, to compile data from proficiency test or inter-comparison study, etc. (format: LAB-PFT-123).
– Documents recording the findings and recommendations from management and other reviews, action plans to correct deficiencies found during external assessment, reports on audit performed on a supplier or subcontractor, etc. (e.g. LAB-AUD-123).
– Formulation of the same type of work that will be done for a particular client until further notice, and according to schedules and arrangements that have been agreed to in advance, i.e. quality plan for a standing order (e.g. LAB-CLN-123).

4.1.3.3. Documents relating to job management

There are certain types of documents and files that must be identifiable for reference purposes, but which will be in active use only for a brief period. It is useful to indicate the year (either calendar or financial) in which it was opened by using the last two digits, and to re-start the serial number from "001" every year. The following are suggested examples:

– Quotes and tenders submitted to prospective clients in 2001 (e.g. LAB-TD-01/123).
– Analytical jobs or tasks that were opened in 2002 (e.g. LAB-JB-02/123)

4.1.3.4. Forms

Forms are used for various purposes in the laboratory and for management. The following examples are provided to stimulate the quality manager during the design of a system for documentation that will meet the needs of the laboratory.

– Data sheets with raw and intermediate laboratory data (e.g. LAB-FDS-123).
– Test report with measured concentration or activity values (e.g. LAB-FTR-123).
– Forms used for recording information relating to quality management: complaints from clients, non-conformance, internal QC, internal audits, etc. (e.g. LAB-FSY-123).

4.1.4. Scope of the documentation system

Laboratories often share a similar experience during and after introduction of a management system for quality. Their initial intention is usually just to add a quality system to the other systems that already exist for financial, personnel, safety and other matters. But as a culture of quality is established, they find their administration becomes structured by the quality system, and more and more of their other activities are incorporated in it. This has been referred to as a maturity model for quality.

The quality manager is therefore advised to consider at a very early stage the possibility that the quality system will ultimately cover more issues than was envisaged in the early planning phases. It may be prudent to consider making provision for the following topics and more:

– Job descriptions for individual staff members (e.g. LAB-JDS-123)
– Individual files with training and qualification records of personnel (e.g. LAB-XPR-123)
– Files with comprehensive internal QC records on each counting facility, including information on configuration, parameter settings, calibration, performance tests,

maintenance, operating manuals, non-conformance, internal audit reports, etc. (e.g. LAB-XCF-123).
- Files with internal QC records on methods or groups of related methods, containing SOPs, validation reports, results for test samples, performance test reports, non-conformance, audit reports, training material, etc. (e.g. LAB-XOP-123).
- Files with internal QC data on measuring and testing equipment such as balances, automatic pipettes, pH meters, heating equipment, etc. (e.g. LAB-XMT-123).

4.2. Writing Level 1 and Level 2 documents

4.2.1. How to structure the information

A major decision to be taken by the quality manager is how to organize and present all the required information in these documents. The one option is to follow the structure of the quality standard, and address every group of clauses in ISO 17025 with a corresponding paragraph or section in the quality documents. This will be easy for the assessors to follow later, but the laboratory personnel often find it difficult to relate to such a presentation. The other option is to start from the logic that the personnel are accustomed to and can understand, and then build the documentation around this basic framework. This will not correspond directly to the standard, and therefore more difficult for the assessor to follow. But the task of the assessor can be simplified by preparing a table (typically in a LAB-DAT document) with all the clauses of ISO 17025 listed in one column, and indicate in the next column either where this issue is addressed in the Levels 1 and 2 documents, or why this point is not relevant to this quality system. Many laboratories seem to prefer the latter option.

The next problem is to decide what information should appear in the quality manual and what can be moved to the Level 2 documents. There is no simple answer to this question. One useful criterion is the fact that the quality manual is often provided to clients, and should therefore not contain too much information that makes it hard to read.

4.2.2. Preparing the quality manual

The activities and needs of the laboratory have a major impact on the scope and structure of this document. If the laboratory is part of a larger organization that has achieved accreditation under a quality standard (e.g. ISO 9000 series), a number of issues are covered already and cannot be handled independently. If not, every clause of the standard must be covered directly or by implication. A laboratory doing mainly routine work will find it necessary to focus on issues that may be less important for a laboratory that is primarily occupied with ad hoc requests and research projects.

The quality manager is advised to study the quality standard carefully, trying to understand and apply every clause from the perspective of work being done in the laboratory. Once this has been completed, a first mental framework can be made of how the laboratory may attempt to satisfy all the requirements of the standard. During a second reading one can start to assign groups of clauses in the standard to the quality manual, and others to Level 2 documents.

The next step can be the first draft of a table of contents for the quality manual (e.g. in the document LAB-SYS-001), followed by keywords and notes on what information (i.e. clauses in ISO 17025 should appear in each section. It is advisable to continue in this vein, and abstain from writing the actual document, until similar frameworks are available for all

the Level 2 documents. An example of a possible table of contents is presented as an appendix.

It is important that this initial design and composition of documents be carried out in close co-operation with laboratory management and personnel. It might be a daunting task because the staff is not yet familiar with the concepts and terminology of the standard, and the quality awareness programme may be in an early stage. The quality manager is, however, strongly advised to persist with these efforts, because the ultimate success of the quality system depends on their understanding of its principles and how it affects their work.

It is, however, recommended that the quality manager be responsible for writing the final version of all these documents. This will ensure a consistent style, and will reduce possible overlap between documents on the one hand, and omission of important issues on the other. It will also assist in drawing up the list of cross-references to clauses in ISO 17025.

4.2.3. Preparing the Level 2 documents

The manner in which clauses of the standard are assigned to the quality manual and Level 2 documents and how many of these lower level documents are required to cover all the important issues depend largely on the needs and preferences of the laboratory. The following examples are suggested to assist the quality manager in the identification of supporting systems.

- System to manage routine and non-routine analytical tasks in the laboratory: service design, job planning, job identification, laboratory and client reports, data storage, invoicing, etc.
- System to control the design of new documents and forms, review and revision of existing ones, and release of these to the users, etc.
- System for sample management: receipt, identification codes, chain of custody, safe and secure storage, handling of radiation and contamination, disposal, etc.
- System to handle deviations: complaints from clients, non-conformance, necessary deviations from instructions or arrangements, client communication, etc.
- System for the training and qualification of personnel
- System for internal QC on methods and instruments: formulation of performance checks and limits, action in case of non-compliance, processing and recording of data, internal audits, etc.
- Procurement, subcontractors and other supporting services
- Sales and marketing
- Radiological management programme: classification of work areas, access control, radiation workers, control over radioactive material, control over liquid and gaseous effluent, handling and transport of radioactivity, safety evaluation of new methods, etc.
- Radiological surveillance programme: regular contamination and radiation surveys, personnel monitoring and dosimetry, monitoring of liquid and gaseous release, safety audits, surveys on environment, etc.
-

The last two issues are not directly relevant to ISO 17025 and can be omitted. They are included here in order to illustrate how the quality system can conveniently accommodate other issues that are important to the management of the laboratory.

4.3. Writing Level 3 documents

4.3.1. General remarks

The level of such a document has a direct effect on how consistently the analytical method can be applied by laboratory personnel, thus on the reproducibility of its results. It is also the primary contact point or interface of the majority of personnel with the quality system, and its impact on the attitude and culture of the staff is very important. Designing and writing these documents require a lot of planning and attention.

A common trap is to assume that every reader will have a similar background or operational experience, and will understand the mechanics of the analytical method to the same extent as the person who developed and documented it in the first place. Such assumed knowledge can be dangerous for future users.

Once a laboratory has established its quality system, the person who develops a new method will also be responsible for designing the necessary forms and formulating the method. But during the initial phases of implementing the system, this work will have to be delegated to operational personnel who are already doing the work. This requires another strategy (because some may lack the writing skills), and pose a different set of problems (because the writer is so familiar with the method that important details are often forgotten).

The following process is suggested for developing the writing skills required for documenting Level 3 documents that will comply with the quality standard:

- The quality manager writes one document using the template that is provided in the Appendix, and discusses this with operational personnel to explain the purpose and requirements of every section of the document.
- Each person that is involved in the writing process is then requested to write down for one method only: (i) a list of everything that is used when doing the work, and (ii) all the steps that are taken from start to finish to complete the procedure. The focus at this stage should be on a complete coverage, and not on style, language or compliance with the standard.

The quality manager casts this information in the style that is preferred by the laboratory, adds the missing sections (with the help of laboratory management where necessary), and ensures the document complies with the relevant Level 2 document(s).

These are again discussed with the personnel involved with the purpose of empowering them to write Level 3 documents and identifying the persons who can (as well as those who can not) contribute to the programme of documenting the quality system.

4.3.2. Strategies for writing Level 3 documents

The best starting point is to take inventory of the analytical work that is being done in the laboratory by listing all the analytical methods in use. The quality manager is advised at this stage to focus primarily on complete coverage and not on structure. It is usually easy to delete items from such a list, but difficult to add new ones once writing the document is under way. The next step is to divide this material, which can be substantial even for a medium sized laboratory, into a number of logical units or building blocks. Each of these units will then be documented and implemented as a separate SOP.

This is a demanding task that must be carried out with the co-operation of operational personnel. The quality manager will usually have to go through a number of iterations before the product is acceptable to all involved. Two strategies can be considered.

(a) The cookbook strategy

This provides a complete recipe for the full analytical process from start to finish: sample preparation through measurement to data processing and production of a test report. The product is easy to use, but will involve a frequent repetition of large sections of text. A method on activation analysis and one on the measurement of fission products in food, for example, will both contain the same sections on the energy and efficiency calibration of the gamma spectrometer and on its operation for the capture and processing of data.

(b) The modular strategy

This approach in based on functional units or modules that can be combined as required to build a suitable method. The same module can be used to build different methods. A method for measuring ^{210}Po in fish can be composed from a general method on the drying and homogenizing of animal tissue, one on the digestion of biological material, one on the separation of polonium from aqueous samples, and one on quantitative α spectrometry. Some laboratories prefer the term "work instruction" for the basic modules and "procedures" for their logical combinations, but this distinction is often vague and not essential.

Many laboratories seem to prefer the latter strategy because: (i) it reduces the volume of paper that is involved in documenting the system; (ii) it makes it easier for operational personnel to learn and move on to new methods; and (iii) basic changes often require revision of one module only. It is easier to document a modular system and maintain it in the long run, but it requires rigorous planning at the early stages. Once the laboratory is committed to a certain structure, it will require a lot of hard work to change direction. This is a serious obstacle because the staff is still inexperienced when the structure is designed. The quality manager is advised to have the structure of the documentation system accepted by personnel before embarking on the actual writing of the documents.

4.3.3. Composing an SOP document

It is not easy to write these documents. The following guidelines and suggestions may assist the writer in producing a better product.

- Information should appear in roughly the same order that the user will need it when performing the analyses. It is rather confusing to read at a certain point about things that should have been done a few steps previously.
- This document is not intended as a short checklist to assist the memory of the analyst. It is primarily a training material that can guide a newcomer through the process, giving advice at stages where problems or uncertainty frequently arise.
- Divide the operation into a number of logical units with breaks where the work can be interrupted, if necessary, e.g. sample receipt, digestion of material, analyte separation, calibration, measurement, data collection and processing, reporting, etc.
- Write down every step in the form of an instruction (e.g. "Filter solution through a 0,45 μm type X and wash three times with 20 mℓ distilled water"). Refrain from describing the outcome of this operation (e.g. "The suspended material must now be removed").

– Avoid acronyms and jargon not used outside the laboratory, and abstain from using conversational language. Personnel might initially find the correct use of the ISO terminology as somewhat strange. It is, however, essential to establish the accepted vocabulary if the laboratory wishes to communicate with colleagues and market its services and products internationally.

A template for writing Level 3 documents is provided in the Annexes.

4.3.4. Composing a form

Forms are typically used for recording raw and intermediate data collected during the execution of a method — as test reports with intermediate and final results, and for the administration of the quality system. ISO 17025 only requires that these forms be uniquely identified, but there are a number of practical issues to consider:

– A standard layout is preferred so that the forms used by this laboratory can be easily recognized: presence and position of company logo, position of identification code and revision number, fonts and style used, division into sections, etc.
– Control over forms and trackability of data are enhanced if only one page is used for every form, using reverse side printing where necessary to collect a large amount of information.
– A common problem with forms is that the available space is either too little or too much for the typical amount of information it is intended for. Some frustration (even resentment) of users can be eliminated if the designer of a form first tries to complete a printed copy, and rectifies deficiencies before it is released for general use.
– Data are often copied directly from a data sheet into a spreadsheet for processing and printing of a test report. This process can be simplified and errors reduced if the two formats correspond with respect to the sequence in which the different fields are listed, the names by which these fields are identified, the units of measure that are used, etc.

4.4. Document control

4.4.1. Management of the document control function

(a) Scope of material to be covered

It is a requirement of ISO 17025 that all Levels 1, 2 and 3 documents be stored in a safe and secure location. The laboratory will usually have to provide additional facilities and make arrangements to comply with this. It may be more cost effective if these are also utilized to satisfy other needs of the laboratory. The quality manager is therefore advised to collaborate with management to provide an integrated service that also covers records for completed analytical tasks, manuals of all instruments, personnel records, safety, finance and administration, policy and planning, etc.

(b) Controlled copies of documents

It is also a requirement that only the current revision of a document or form be used. This implies that the laboratory must have a system to keep track of all the copies that are in circulation (known as “controlled copies”), so that these can be withdrawn and replaced with revised copies when required.

It also implies control measures that will prevent personnel from making uncontrolled copies, because these will not be recorded and therefore difficult to withdraw. Control can be achieved either by printing controlled copies on marked paper or by marking the copies themselves — as long as these marks are in a color that will not be copied.

Identifying documents that cannot be revised, and which therefore need not be replaced can reduce the amount of work involved in these control measures. Most "reports" relates to work that is done only once, and falls in this class: validation, performance tests and audits. If a related document is written in the future, it can be released under a different identification code.

(c) Backup of pulse height spectra

Methods that are based on pulse height spectra (e.g. α and γ spectrometry) rely on sophisticated software to process these spectra and produce decay corrected activity values. The printouts are stored with the rest of the analytical results, but may not be sufficient to ensure trackability of data. The quality manager is therefore encouraged to make provision for: (i) copies of these spectra to be archived in a secure and readable format (e.g. as CD copies), and (ii) a codification system that will make it easy in the future to track method validation, performance test and analytical task of its supporting spectra.

4.4.2. Physical and organizational requirements

The availability of computer technology has started a move away from paper based management. If one considers the advantages of computer based information systems, it is clear that this trend can only grow in the future. The quality accreditation bodies may currently still focus on paper work, but the quality manager is advised to prepare for the future and design a documentation system that can be operated with the minimum of paper.

One of the advantages of such a system is the immediate access to the current edition of documents and forms. There are, however, also a number of potential problems caused by this easy access. These requires attention during design and implementation:

- The documents and forms must be protected against unauthorized modifications.
- Personnel can draw documents and forms from the system as and when required, and without the need to inform anyone about it. They will then use these to perform their duties. There is a need for a mechanism to: (i) inform all the unidentified users about documents and forms that have been revised, and (ii) ensure that the obsolete version is withdrawn and replaced with a current one.
- The documentation relies entirely on one storage system, which can be corrupted or even destroyed by computer defects. There is a need for alternative storage that is secure, and from which all the data can be retrieved and used again.

4.4.3. Release a new document

The same elements are usually present in the process of writing and releasing new documents. The following steps are suggested for this purpose, and to assist the quality manager in developing a strategy that will satisfy the needs of the laboratory:

- A person nominated according to the policy of the laboratory writes the first draft of the document (e.g. revision "00/A"). This manuscript, together with a page (or a system

form) for written feedback, is distributed for peer review to colleagues who can make contributions, and to the quality manager.
- The author incorporates this feedback in revising the manuscript; taking into consideration that a certain comment may be either a "suggestion" (which is left to the judgment of the writer to use) or an "objection" (which can not be ignored).
- The author can treat the revised manuscript as the final version (revision "00") or send it out for a second time for comments (as revision "00/B") if the revision included fundamental changes to the original manuscript.
- The author, one or more persons nominated to check the document, and another person authorized to approve its release — they all sign the final version. *(A different approach would be required if the laboratory relies on a computer based documentation system.)*
- Copies of this approved document are made available to persons on the distribution list. Names can be added to this list if copies are supplied later to other staff.

The laboratory can have a policy that a document representing the outcome of investigations carried out by one person, and which contains conclusions and recommendations only, need not be authorized formally before release. Typical examples are validation, performance tests and audit reports. Such a policy would be in line with the principle of project management that final responsibility should be assigned to the project leader. The final document is issued under the authority of the author only, but management must approve its recommendations before they can be implemented.

4.4.4. Review and revise existing documents

It is required that each of the Level 1 and 2 documents, as well as the standard operating procedures, be reviewed by a competent person at regular intervals, typically every one to two years. The purpose of such a review is to ascertain whether: (i) the action described in this document is a reliable reflection of what the personnel are actually doing, and (ii) this is the most effective and efficient way of reaching the stated objective. If there is a difference, the document may need revision. There may also be other reasons why management would decide to modify a particular document.

The revision of an existing document follows essentially the same steps as the process to compose a new one: distribution of the revised draft (as revision "01/A") for peer review, incorporation of the feedback received, authorization and release of final document (as revision "01"). The main difference is that all the controlled copies of the obsolete version must be withdrawn and destroyed.

5. IMPLEMENTATION OF THE QUALITY SYSTEM (ORGANIZATIONAL ASPECTS)

5.1. Receipt of samples

Samples may enter the laboratory via various ways: by (package) mail, by courier or by personal delivery.

The laboratory has to develop a procedure for control of the samples upon receipt, and it has to set its criteria for acceptance. This receipt control should be done immediately upon

delivery, and it is recommended to have many employees qualified for this receipt control. In Table XI an overview is given of the checks that may be considered in such a receipt control.

TABLE XI. TYPICAL CHECK LIST UPON RECEIPT OF SAMPLES

Name of customer
Date of receipt
Number of samples
Unambiguous identification or coding
Damage during transport
Accompanying letters
Reference to order number

The samples will be transferred to a storage facility prior to analysis if the delivery meets the laboratory's acceptance criteria.

5.2. Trackability/sample custody

The laboratory has to provide the results of the analysis for each sample according to the sample code used by the customer. The laboratory has to develop quality assurance practices by which the samples can be followed throughout its way in the laboratory (storage, sample size reduction, weighing, irradiation, measurement and reports). This so called *Trackability* enables the ready retrieval of the different elements of a record to allow unambiguous correlation with a uniquely identified sample (Fleming, J., et al., ACQUAL **1,** 1996, 41–43; 233–234).

Trackability of samples, test portions, results, reports and remainders can be accomplished by applying a unique code to the sample as soon as it is accepted by the laboratory, and by careful registration of all events related to this sample and its derivatives with reference to the sample code: on receipt registration forms, method selection forms, weighing forms, laboratory journals, etc. The choice of this code should be easily identifiable for the laboratory staff. The code may include, alphanumerically: a key to the customer and to the specialist performing the analysis, irradiation and counting facility, measurement geometry, date, etc. It is important to assess which conditions should be made 'visible' in the code so as to allow for easy retrieval. Codes should be as compact as possible; if relevant, it is better to make reference to the blank by using the character 'B' than to use the full reference 'blank' as part of the code (and filename). The laboratory has of course to make a reference table between the customer's code and the laboratory's code.

It is important to have a flowchart of the analytical process, especially if different techniques have to be applied on the same or on different test portions of the sample. A flowchart makes the work process more transparent and may also contribute to design coding systems by which all steps can be linked to one another.

Well organized facilities (cabinets, rooms) for storage of the material are a must. It may be considered to have separate storage facilities for samples prior to analysis, for remainders of samples of which the test portion is under investigation, for remainders of samples of

which the analysis has been completed and for the test portions prior their final disposal (e.g. after sufficient decay of the induced radioactivity).

All of these facilities should be secured to limit access by unauthorized personnel. Also, the contents of these facilities should be complete and kept up to date.

Bookkeeping (simple paperwork, a notebook or forms will suffice) has to be set up in these facilities to link the stored material to its position on the shelves, the date of arrival, related job number, person in charge of the analyses, date of removal/disposal.

Exceptions to the requirements on limited access may be inevitable if samples have to be stored in centralized freezers or other facilities with controlled environmental conditions. Still, the laboratory has to take adequate actions so as to assure the customer that his samples are well taken care of. A special problem may occur if the customer sets requirements to confidentiality and/or secrecy and if a central storage facility with access by various people is needed. In such a case the laboratory — and the customer — have to take measures to prevent identification of the customer and/or the type of samples.

5.3. Preparation of the test portion

The laboratory most likely has procedures for sample size reduction, homogenization, drying, moisture determination and sub-sampling. Quality assurance can easily be implemented for these steps. The laboratory may draft a general procedure describing which subsequent actions are usually taken for different sample categories (such as sediment, plant material, biological material), e.g. using a matrix table. Next, a form may be developed where the authorization for the preparation plan is provided in case of a new sample type, and where the actual conditions are registered. A pre-printed form (e.g. listing: "Oven drying at __^{o}C during m/h/d") could be used. In this way, full trackability of the sample preparation steps is assured.

There are situations where the laboratory may decide to validate a sample preparation step before applying it. The homogenization step is an example of this. Such a validation is at the scrutiny and scientific responsibility of the laboratory. Note 3 in Clause 5.4.5.3 of the ISO/IEC 17025 applies here: "Validation is always a balance between costs, risks and technical possibilities".

5.4. Procurement

Laboratories may be empowered by their mother organization to select their instruments, chemicals, consumables, etc., but a separate department generally does the procurement. There may be cases, especially when it comes to procurement of consumables and chemicals, for example, where the procurement department orders products that are different from what the laboratory specifically ordered. This is often due to a lack of communication between the laboratory and the procurement department on the importance of certain goods to assure quality of the work. It is therefore recommended to communicate timely with the procurement department to find out a quality-cost compromise.

The laboratory has to identify its preferred suppliers (Clause 4.6.1). This choice may be based on the supplier's reputation. Procurement of goods from suppliers with an ISO 9001 or ISO 9002 certification may be preferred, not only to ensure product quality but also because such companies at least have procedures implemented for complaint management.

When procuring instruments, there may be a preference for companies with demonstrated after sell maintenance. The number of suppliers may be limited, but should not be so that would make the laboratory entirely dependent on one or two companies only. There has to be an evaluation process of these suppliers (Clause 4.6.4). This may be done by the procurement department taking account the technical opinion about the quality of products.

5.5. Management of internal improvement

Implementation of a quality system is a continuous process.

5.5.1. Management review

5.5.1.1. Introduction

Management review is a regular event. Reviewing covers:

(i) progress towards the tactical and strategic objectives of the laboratory,
(ii) effectiveness of the current policies and systems in attaining these goals,
(iii) alignment of the laboratory with the wider objectives of the organization as a whole.

The outcome of such a reviewing is typically a series of findings or conclusions about the current situation, and a number of recommendations or decisions on how it can be improved.

5.5.1.2. Practical aspects

Clause 4.14 of the ISO/17025 clearly specifies that the laboratory's executive management should do the management review. However, the laboratory's executive management is at least one level higher than the laboratory manager is. Preferably, the person who is formally responsible for the quality system should take the lead.

Participation in the review process depends largely on circumstances. A smaller laboratory may invite all the staff that can contribute to the discussion, while a larger one may involve only the leaders or supervisors of different operational groups. The laboratory manager and quality manager must be present. It is recommended that the next higher level of management also attend these review meetings. This can resolve questions that may arise at the meeting about the vision, plans or policies of the larger organization.

If review meetings are held too often, there may be little to discuss and staff will consider it a waste of time. If they are too far apart, it may be too late for effective action. Clause (4.14) suggests at least every 12 months but a time interval of about three months seems to be a reasonable compromise. It is prudent to schedule these meetings to correspond to events on the calendar of the organization where relevant information is required or becomes available: the annual budget, annual reports, board meetings, compilation and release of strategic or business plan, etc.

Success of a management review depends largely on the availability of reliable information about the current situation inside the laboratory, inside the larger organization, and about the market where the laboratory operates. Such information is often not readily

available, and staff is asked to collect, process and present specific information as part of their regular duties.

Clause 4.14 specifies which information should be available, as a minimum, for management review. The laboratory's own ideas about the improvement for the next period, with milestones and deliverables have to be developed further in this meeting.

The following topics are suggested to assist in this process:

- Summary of salient information from the (monthly) financial statements of the laboratory; which can be supported by trends from previous periods
- Volume of analytical work (e.g. number of tasks or samples) that were completed during this and corresponding periods in the past
- Number of complaints and cases of non-conformance registered during this and preceding periods
- State of implementation of the quality system, using criteria that were formulated in advance (e.g. number of Levels 2 and 3 documents completed, instruments under QC, state of sample receipt and custody, fraction of work covered by codification system, number of staff qualified, internal audits)
- Progress with the development and validation of new analytical methods
- Action taken to promote a culture of quality awareness in the laboratory.

5.5.1.3. Administration

The strategic or business plan of the laboratory provides the essential foundation for the management review. It is recommended that this document:

(i) is integrated into the quality documentation, e.g. as a LAB-SYS-document,
(ii) is compiled just ahead of the annual budget to collect and present information for that purpose,
(iii) is composed by the same team that will do the management reviews.

An effective plan should cover the following issues:

- A description of the vision and mission of the laboratory; and a critical analysis of its current situation: any technique can be used, e.g. SWOT (strengths, weaknesses, opportunities and threads) or driving forces can be used, provided the personnel are accustomed to it.
- Strategies that are planned to boost strong points and opportunities of the laboratory, and counter its weak points and external threats. If, for example, the technical background and competence of the personnel have been identified as strength, the laboratory may decide on a strategy of additional training to sustain this asset. A laboratory that is focussed on activation analysis may see a new opportunity in the measurement of radioactivity in the environment and decides to enter this field. Implementation of a new strategy involves everyone, and is expected to last a few years.
- Specific objectives to be reached during the next (financial) year; formulated as separate projects with definite start and end dates, clear deliverables, action plans, and a person nominated to take responsibility for each.
- Budget for the next year: manpower, consumables, services, overheads, capital items, etc.

Participants to the management review meeting are required to objectively evaluate the recent performance of the laboratory and compare it with the planned strategies, short term objectives and budget of the laboratory.

They must decide whether the current policies and procedures of the laboratory are effective in realizing its stated mission and objectives and, if not, what should be done to bring it in line with what is expected.

It is important that all the findings and recommendations of the management review meeting be fully documented, e.g. as a LAB-AUD document. This document is a valuable tool for keeping both personnel and management informed about the progress made.

5.5.2. Internal audit

5.5.2.1. Introduction

This work is carried out to verify that the planned arrangements in the laboratory have in fact been implemented as agreed. Such an audit consists of two components. The first is a list of the essential elements of the plan. The second part is the physical verification, based on objective evidence only, that each of these elements is in place.

5.5.2.2. Practical aspects

The auditor draws up the "audit plan", which is the list of essential elements, in collaboration with the quality manager. The audit is impartial and fair. The inspection is carried out by a specialist that understand the situation, and its outcome does not depend on the preference of the auditor.

It is important to draft an effective audit plan. Each point to be verified is formulated as a statement. The list must be short in order to keep the cost of the audit and the input from operational staff within reasonable bounds, but it must be comprehensive enough to cover every essential element of the plan. Audit should include only issues that can be verified by objective evidence.

The word "plan" as used above, can have different meanings. The following are examples of plans that can be the objects of internal audits. It is clear that in some cases the audit plan will be applied once, while in other examples it can be applied repeatedly on the same plan — or even for different plans,

- Routine application of analytical methods: The same audit plan may be applied repeatedly to most of the methods in use, if the elements on the list are carefully selected and formulated.
- Performance tests on counting facilities: It is possible to formulate the performance specification in such a way that the same audit plan can be applied to a γ-spectrometer, gross αβ counter, liquid scintillation counter, etc.
- Development and validation of a new analytical method
- A large contract carried out by the laboratory on behalf of a client
- Implementation and maintenance of the different management systems as described in Level 2 documents; the diverse nature of these systems may require a separate audit plan for each case

– External groups that provide services and products, e.g. subcontractors for analytical work, service providers, manufacturers of equipment, suppliers of special chemicals, etc.

Laboratory management can carry out initial audits on the quality manager regarding implementation of Level 2 documents. This will improve their understanding and mutual agreement on the design of the quality system. It is suggested, however, that the quality manager be trained as soon as possible, perhaps by the ISO accreditation body, to carry out audits on the implementation of the Level 3 documents. A larger laboratory may consider training additional personnel for specific areas.

The prime task of the auditor is to seek for compliance of the operations with the laboratory's quality manual and with the ISO/17025, rather than searching for mistakes, non-compliances and hair splitting. If non-compliance is found, it is important to investigate if this is an incidental case or not. If not, the laboratory may have a justifiable reason for it.

5.5.2.3. Administration

It is recommended to include in the quality system documents with prepared audit plans, e.g. LAB-FSY documents. They can then be revised as the quality manager gains more experience, and operational personnel become convinced about the benefits to be gained from regular audits.

Such a form should make provision for follow-up, so that attention can be given to deficiencies that have been identified during the previous audit(s). It is also suggested that a copy of each completed audit plan be kept with the records of the activity that is being evaluated. This information is useful for the identification and elimination of persistent problems.

5.5.3. Non-conformances and corrective actions

5.5.3.1. Introduction

This is the most important reactive tool at management's disposal; one that allows the laboratory to react positively and benefit from the discovery that something is not as it is supposed to be.

The process starts from an observation that does not conform to the expectations of the quality system. This initial step is followed by an investigation that involves different role players in order to identify the root cause of the deviation, followed by a plan of action:

(i) to correct the consequences of the error, and
(ii) to prevent its occurrence in the future.

A procedure for handling non-conformance, if properly applied, can be of immense help in building a quality culture. Non-conformance is registered in a neutral manner, which can reduce confrontation, and encourage personnel to air their problems. The emphasis on preventive action supports a value system based on continuous improvement.

The success of this programme depends largely on the management's ability to investigate and resolve an occurrence without blaming somebody for it. Staff will start

supporting the system as they observe how their own performance improves, and the laboratory as a whole is reaping the benefits from it.

5.5.3.2. Practical aspects

It is often difficult to identify the root cause of the non-conformance, and to decide on cost-effective preventive action. The quality manager is advised to constitute a small team of persons with the technical background and experience to help in this task. This group should meet as soon as possible with the quality manager as co-ordinator. It can include experts from outside the laboratory if it is considered necessary. The findings of the team and the action steps (activity, responsibility and target date) must be documented.

One of the first tasks of this team is to collect more information on the event, and to supplement the scanty description that is required for registration. This might even involve taking some measurements so that discussions can be based as far as possible on facts rather than on personal opinion.

It is inefficient to treat defective equipment as non-conformance because there can be no doubt about the best course of action (the equipment must be repaired), the process takes far too long, and such a breakdown does not necessarily constitute a weakness in the quality system. It is therefore suggested that a record of defects and a description of the repairs are kept with the operational file of each instrument. The information in these files can be studied annually with the aim of identifying patterns or trends that may indicate a common problem.

There are cases where the search for the root cause and the implementation of preventive steps may require some development work, or where non-conformance directs the attention to an interesting technical or scientific point that should be investigated. Such a project often requires an extended period to be completed, and may delay closure of the non-conformance for months. The laboratory can consider moving the control of this project from their con-conformance to their project management systems.

5.5.3.3. Administration

A special form is required for registering non-conformance and recording subsequent action. Control of records is improved if this form is printed on one sheet of paper.

The quality manager should keep a register of every non-conformance that typically contains the date of registration, identification code, short description, and date of closure. These data are useful for management review, and the record will ensure that some items are not left open for an indefinite period.

It is recommended that the entries in this register be studied at least once every year in search of trends or patterns. It could be found that a new type of deviation is developing, or is confined to a group of methods or to a particular technique. These findings can assist during the annual review of the Strategic or Business Plan of the laboratory.

5.5.4. Preventive actions

Whereas corrective actions are actions undertaken to eliminate a problem and prevent reoccurrence, preventive actions are pro-active and are undertaken to reduce the likelihood of potential sources of problems. In this way, preventive actions are more to be interpreted as

opportunities for improvements and are no reaction to the identification of problems or complaints.

There are several ways to implement preventive actions:

- On a continuous basis, the lab personnel can introduce opportunities for improvements. This could be an ad hoc proposal for improvement or an idea generated during a root cause analysis of a problem, where the specific corrective action for a problem generates other ideas to improve similar potential problems.
- On a fixed basis, e.g. during a management review. At a management review meeting a trend or general root cause analysis could be made of all the complaints, internal audits, non-conformances, results from interlaboratory comparisons and internal quality control runs received or made during the last year. From this general overview, a specific agenda point could then involve preventive actions where the management decides on specific actions it is willing to undertake to improve the situation.
- On-line analysis of data, e.g. using a trend analysis in control charts, could lead to preventive actions to avoid future problems.

5.5.5. Complaints

5.5.5.1. Introduction

Problems that are left unattended could be aggravated. Complaints must be attended to quickly and effectively, because they provide the quality manager with a free survey of how the market feels about the services and products that are supplied by the laboratory.

Very few of the dissatisfied clients take the trouble of complaining. The rest (some studies indicate more than 90 %) simply stay away and never come back. The discontent spreads because those who did not complain to the laboratory in the first instance are sure to tell other colleagues about the poor service they received. It is, however, also true that the good news about a complaint that was resolved promptly and openly, is also spread in the market.

5.5.5.2. Practical aspects

The negative perceptions associated with complaints make it hard for the quality manager to obtain effective support for handling it. It is therefore suggested that this topic be covered in one combined section for “Client’s comments”, which covers both the negative elements (claims) and the positive ones (praise). All cases can be handled in the same manner and personnel should be made aware of both complaints and compliments.

A procedure similar to non-conformance can be applied to complaints (or client comments): i.e. registration of incident, preliminary investigation of the situation, identification of the root cause, formulation of effective corrective and preventive action, and a plan of action (with persons responsible and target dates).

5.5.5.3. Administration

Effective administration of complaints also requires a form to register a new event and record its resolution and closure, as well as a register or inventory list of all complaints.

5.5.6. Review of documentation

5.5.6.1. Introduction

The quality documentation system of the laboratory is not an archive. It contains only a full and clear description of the current state or position of the laboratory on its way of continuous improvement. However, this process of evolution is never completed, and the quality manager will never be able to consider these documents as "finished".

There are many driving forces behind these changes. One force is the discovery or identification of defects in the current system by means of tools presented in the preceding sections. The elimination of a defect usually implies a change to the documentation. Another force is the push of technology. The laboratory cannot ignore the development of methods and equipment that are more sensitive, selective, efficient, or reliable, etc. There is also the pull of the market where clients are asking for lower detection limits or quicker results or higher accuracy, etc. The laboratory must adapt to these new circumstances in order to survive. These changes affect the quality documentation too.

5.5.6.2. Practical aspects

Two actions that are relevant to changes in the documentation system, are sometimes not clearly understood by the laboratory staff that are involved in these changes:

- *Review:* This is the work done by a competent person to evaluate the contents of a document with respect to (i) the current state of affairs it is supposed to be describing, (ii) all the requirements that it must satisfy, e.g. ISO 17025 or the client; and (iii) its effectiveness in reaching its objectives.
- *Revise:* This is the work done when changing the contents of a document to bring it in line with all the known requirements.

A document that has been reviewed need not necessarily be revised. Different people may be involved in the review of a document, e.g. one competent analyst for a laboratory method, laboratory management and the client with a quality plan, or selected senior personnel with Level 2 documents. However, it is recommended that only one person be nominated to do the revision.

The quality manager will have to strike a balance between the need for accurate descriptions, and the work involved in revising and releasing documents. Omission of a crucial element or statement that can cause serious errors must be rectified immediately. However, there are trivial problems (e.g. with grammar or layout) that can be postponed until the next scheduled revision of this document. It is often difficult to decide where to draw the line.

Some laboratories find it necessary to implement a system that allows minor handwritten changes to a document This will be acceptable provided that: (i) it can be proven that the master document and all its controlled copies have been changed, and (ii) all the changes are legible, dated and signed by the designated person. It is suggested that the quality manager should encourage laboratory staff to submit notes about the practical problems they encounter with particular clauses, or with suggestions on how to improve it. These can be filed with the master document, and incorporated during its next scheduled revision.

5.6. Customers

There is an old saying that should be posted on the doors of every laboratory that aims at services to third parties: “It takes months to find a customer, and seconds to loose him again”. The International Standard ISO/IEC 17025 includes requirements for the interaction between customers and the laboratory (e.g. Clauses 4.4, 4.7 and 4.8). The interaction is based upon the principle of proper planning upfront (i.e. quality assurance) to assure that the customer’s question is well understood and can be answered in a satisfying way.

The laboratory will have to develop and to document procedures for the handling of requests from customers and the planning of work. It is best to start with the development of some checklists to get a feeling which items need to be available before a sound decision can be made if the request of the customer can be answered satisfactory. Once experience has been gathered on this part of management, documented procedures can be made.

5.6.1. The service request

When requests for services come in, who should answer them? Normally a person in charge of marketing is responsible for communication with customers. It is important to have at hand a checklist for information required from the customer before an answer may be given. Suggestions for questions to be included in this checklist are given in Table XII.

TABLE XII. EXAMPLES OF ITEMS ON A CHECKLIST FOR REQUESTS OF ANALYTICAL SERVICES

Name, address, phone number of customer
Date of discussion
Outline of request (e.g. determination of A in B)
Sample matrix
Number of samples
Expected concentration
Relevant information on other components of the sample,
so as to assess potential interferences
Information on sample treatment or storage
Information of amount of material available for analysis
Required degree of accuracy
Required degree of precision
Required limit of detection
Reporting date (turnaround time)
Special wished on reporting (e.g. electronic versions)
Date of delivery of samples at laboratory
Remainders back to customer or to be destroyed by laboratory
Special wishes on confidentiality or secrecy
Contract required

The customer will nearly always ask about the costs of the services. An indicative pricelist should therefore be readily available. Since the request has not yet been evaluated on its feasibility, some care has to be taken with providing immediately a price, and it should be made clear that at this stage it can only be an indication.

Next, the customer should be informed that his request will be immediately evaluated and that he will be informed with a reasonable time frame –which should be quantified, e.g. within 15 m, 2 h, 1 d etc.- on the feasibility.

It is important to keep a good record of all communication with customers, also in this preparatory stage. Here again comes the advantage of having communications by the same person so as to keep all communication records in e.g. one laboratory journal. An alternative might be to prepare a file-system that can be accessed by several people (including, e.g. a secretary).

5.6.2. Review and planning

The laboratory should take ample time for a proper review (evaluation) of the new request. Is the requested analysis been done before? Is the available analytical technique capable to answer the request? Are equipment and human resources available within the slot that is set by the delivery date and reporting date?

An archive with examples of analyses of typical samples is assumed to be prepared in the frame of the quality system.. Such an archive may suit and speed-up future evaluations if material of similar type has to be analyzed or if typical detection limits are searched for. The archive can be composed of copies of analysis reports and these should include details on the experimental conditions (e.g. irradiation duration and neutron flux, decay and counting times, counting geometry, sample mass etc.).

In the planning also ample time should be included for interpretation of the measured spectra and calculation of concentrations, uncertainties and detection limits. The review and planning may result in a confirmation to the customer on the feasibility of the analysis meeting the customer's requirements, or in the need for extra consultation, e.g. if the required deadline cannot be met, or if more information is needed on the samples. Perhaps firstly test-analyses should be done (at no charge). It is also possible that the laboratory would like to give the customer advises for packing or coding of the samples.

The note 3 with Clause 4.4.1 of the ISO/IEC 17025 states " A contract may be any written or oral agreement to provide a client with testing and/or calibration services". Written confirmation is preferred for better record.

Some customers may require an extensive contract for the services, e.g. if a large number of samples has to be analyzed during several months, or if during e.g. 1 or 2 years there will be a regular (daily, weekly or monthly) supply of samples. The laboratory may also take the initiative to such a contract that may serve to protect not only the customer's interests but also the interests of the laboratory itself. Examples of Clauses that may be included in a contract are given in Table XIII. A written procedure for drafting contracts may include, but should not be limited to, these examples as potential paragraphs to be considered.

TABLE XIII. EXAMPLES OF CLAUSES FOR AN EXTENDED CONTRACT FOR ANALYTICAL SERVICES

Names and affiliations of customer and contractor (laboratory)
Title of contract
Aim of the work (e.g. determination of A in B during months x-y, 200n
What will be reported
Where and when to deliver samples (day of week, time)
Batch size
Milestones (e.g. X% will be completed by day Y)
Preferable minimum/maximum sample mass
Quality control applied
Total running time: Reporting dates (e.g. day of week)
Information included in report to customer
Format of reports
Storage and storage time of remainders
Technical manager at laboratory
Costs
Method of payment
Proprietary rights
Statements on confidentiality and/or secrecy
Liability statement

5.6.3. Service to the customer

It is a good practice to offer customers the opportunity to visit the laboratory's premises and facilities, to meet the people performing the analyses and, if desired, to witness the conduct of the analysis or measurement. The laboratory may wish to prepare some simple but attractive informative material about the technique and its opportunities since often customers may ask for it. A photocopy of a contribution to a scientific journal may be interesting for peers or academics but it is less recommendable for customers from industry or the government, for example. Customers supplying large quantities of samples may even be asked to include blind duplicates that are randomly placed, which will be analyzed at no charge. The only thing the customer has to do in such a case, upon reporting by the laboratory, is to feed back the codes of the duplicates.

The laboratory can draft its service policy to the customer with a simple statement in the quality manual, which may include an indication that when visited by a customer, it will take all measures to assure the confidentiality of its other customers.

5.6.4. Sampling

Thiers (1957) claimed that "Unless the complete history of a sample is known with certainty, the analyst should not spend his time analyzing it….". This statement, whether justifiable or not, at least indicates the importance of primary sampling of the population to be studied. However, the representativeness of the sample presented to analysis is often unknown to the analyst and most analytical laboratories leave this aspect to remain the customer's own responsibility.

If the laboratory has to participate in the sampling, timely discussions with the client are needed to elucidate the analytical request and prepare a sampling plan, as well as prepare for sample storage, sample preparation and archiving. Moreover, it is important to elaborate upfront on the information that has to be registered on sampling, environmental and instrumental/process conditions. Table XIV lists some typical questions that have to be answered.

TABLE XIV. QUESTIONS RELATED TO SAMPLING

Availability of directives, norms or standard methods
Representativeness and appropriateness of samples to the analytical request
Selection of measurand, accuracy and precision
Selection of sampling plan; registration of critical parameters
Selection of tools, containers, analytical portion preparation; all related to contamination and/or loss of measurand
Quality of homogenisation procedure
Size and/or masses of the analytical portion to be derived from the sample taken
Are all employees involved in sampling aware of the critical parameters
Are statistical practices being used; selection of laboratory control materials, etc.

In all cases, the laboratory should assure that the sample taken are properly labeled/coded and that measures are taken and facilities are available to store the samples without affecting their integrity and/or quality. It may be a challenge to participate in a project in which a large number of biological samples have to be analyzed but only if there is sufficient storage capacity at, e.g. low temperature.

It should be noted that the Clause 5.7 'Sampling' in ISO/IEC 17025 applies to the primary sampling only, i.e. sampling from the population under study. The requirements in this clause are not relevant if a laboratory derives its analytical (test) portion from a sample delivered by the customer; in which a case the sampling is considered the responsibility of the customer. The laboratory is recommended to emphasize in its tenders, confirmations to customers and/or contracts that if the primary sampling is done by the customer the analytical results apply to the analytical (test) portion taken and that the results can not be interpreted as being representative for the population under study.

5.7. Reporting

At the end of the formal analytical process, a formal test report or calibration certificate will usually be issued to the customer. All information requested by the customer must be included and consequently some form of checking is necessary prior to release to the customer. As all work until this point has been controlled internally, it is essential that someone who is familiar with the processes used to provide data for the final report should cross-check such report.

Even in the best managed laboratory and with the best trained and motivated staff, people will (occasionally) make mistakes. This costs time and, if not remedied before transmission to the customer, leads to the reporting of erroneous results, embarrassment and possible loss of confidence in the work of the laboratory. Therefore, before formal issue of a test report or calibration certificate it is essential to thoroughly check the data. The following points should be borne in mind:

Validation of calculations:
Usually such data have been checked at the point of origin, but it is advisable to recheck them prior to formal issue of the report. In the case of calculations that have been carried out automatically by computer, the software should have been validated and the configuration controlled.

Check for typing and systematic errors:
Reports, etc. will often follow a standard format. With the use of 'cut & paste' software tools it is advisable to check each report for typing and systematic errors.

Criteria for rejection:
Prior to internal reporting, the relevant quality control parameters should have been assessed, however, it is necessary to perform a final check prior to the release of data.

Measurement reports (internal) and reports to customers (external):
Be aware that an internal report from one laboratory to another, or to a central reporting unit, will not be as 'formal' as a report to an external customer. It may be that external reports have to be authorized for release by a person properly trained and authorized to do so.

Formats, contents:
Many reports will follow a standard format. But it may be necessary to include: statements about environmental conditions at the time of measurement; uncertainties associated with the result; whether the results of the analyses are in compliance with the standard being applied etc. Other contents may include: deviations from the test method; uncertainty statement; opinions & interpretations; sampling related data; traceability.

Verification with customer contract/requirements:
The report should be checked to ensure that it complies with the relevant customer requirements.

Applicability statement:
It may be that the authorization for release off the data is accompanied by a caveat that relates the data to the test portion and/or sample as received.

Legibility (responsibility) statement:

Finally, someone has to take responsibility for the report. This may be a simple signature authorizing release of the data, or may be accompanied by a more weighty statement.

Three additional suggestions:

- The report to the customer may deviate from the formal final report of the test, as prescribed in Clause 5.10.2–5.10.3 of ISO 17025. This is also foreseen in clause 5.10.1.
- The laboratory should be well aware that, in the customer's opinion, the date of reporting is the moment he has the report on his desk whereas in the analyst's opinion, the date of reporting corresponds to the date of printing the report. The customer's opinion is most important, hence the laboratory should include a safety margin in its planning so that printing is done a few days before the customers expects the report.
- The laboratory should be well aware that its customers are not familiar with scientific notations (like the E-format), abbreviations and other jargon. At least a legend to the report should be considered. In addition, care should be taken to limit the number of significant digits (generally 3).

5.8. Human resources management

Application of ISO 17025 is a management tool, and as such has the full support of the laboratory management. As it is reasonable to make the assumption that staff costs relate to between 30 and 50% of a laboratory's total budget, it is clear that optimization of Human Resources Management is an essential part of implementation of a quality system. In effect, in order to assure a well motivated, trained and efficient workforce, there are three main areas that can be addressed: training (in particular, on-the-job training), quality awareness and education, and internal audit.

5.8.1. On the job training

A new member of staff starting work in a nuclear analytical laboratory will arrive with a certain amount of prior experience and training. Nevertheless, for that person to become an effective member of the team, he/she will have to undergo on-the-job training. On-the-job training schemes may be centrally managed, in the case of large organizations, or more locally run in smaller organizations, but in any case, the training received should be assessed for its effectiveness and be documented.

In general, on-the-job training can be split into generic training and task specific training, e.g. generic training may comprise such items as safety related training, and task specific training relate to the operation of an instrument. Within the training needs, refresher training should be considered, e.g. for task specific training as staff move within the laboratory; and for generic training routine refreshment of safety related issues. Working within any laboratory environment requires a certain amount of safety related training, and often this training is carried out centrally.

In general, the following areas should be considered when applying an on-the-job training system:

- Provide general introduction to the house rules and quality system
- List items that should not be done without appropriate training/explanation/supervision

– Collect evidence of educational background of all staff
– Declare present staff members who are, in principle, qualified at the start of the quality system implementation for relevant items; this may be done by the responsible officer, i.e. head of the laboratory or department — the so-called 'Grandfather' principle)
– Be aware of the need for backup for staff in critical positions such as deputies; multi-skilling may be required.

It is important to realize that even experienced visiting scientists require some on-the-job training in order to assure that these guests become aware of the laboratory's rules for registration, acceptance criteria, non-conformance reporting, etc. In addition, the laboratory has to decide on the qualifications of their trained personnel once and forever or if they prefer regular re-qualification on the basis of demonstrated performance. The latter may be needed if jobs, or parts thereof, have not been carried out for several months, for instance.

5.8.2. Training of internal auditors

As the quality system develops, it will become necessary to train staff members to function as internal auditors. This has two main positive effects:

(1) the staff members themselves become more aware of the quality system;
(2) the organization commences a process of continuous improvement, facilitated by managing its own internal quality audits.

The standard requires that internal quality audits are carried out by, 'trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.' In the case of small laboratories it may not be possible to remain completely independent; nevertheless, it is advised that the internal audit process be started as soon as possible within a laboratory seeking accreditation. The very mechanism of carrying out internal audits will increase awareness of quality systems among the workforce.

Regarding the training of internal auditors, it is likely that contact will need to be made outside the organization — perhaps to the accreditation body — to organize training for internal auditors. Once the first internal auditors are trained, and as the quality system develops, the organization may wish to consider training its own internal auditors.

6. OUTLOOK

Much has been said about the practical aspects, the write-ups, and the organizational aspects of implementing a quality system that complies with the ISO 17025 recommendations. QA/QC is a concept of gradual improvement of performance, documentation and validation needs, and, however, is also a constant awareness about progress and scientific development. This is not a static system, it is supposed to be a dynamic, structured and evolutionary process for the benefit of analytical laboratories. The human factor, amongst others, implies a certain bias in the assessment of material properties, but, in fact, it induces as well the opportunity for development and improvement.

The quality system, including formal accreditation, should therefore be seen as a challenge for constant improvement of reliability and it is not only an intellectual exercise but has profound practical implication. As stated before, the move from "faster, cheaper and more" towards "more reliable, more valuable, and more quality oriented" is strongly effecting

the daily life and economic prospects. It is hoped that a general quality culture will emerge and support a sustainable development in a broad sense. This needs active engagement and responsibility. Establishment of the QC system is a long way to go but no other choice, the today competitiveness in the world market calls for QC and accreditation. In a shrinking world of globalization and merging of culture analytical laboratories will stay competitive only if quality prevails quantity and adheres to a common standard. The ISO 17025 is currently the most widely accepted standard.

It is hoped that this guidebook can contribute to a better understanding of the basic ideas behind ISO/IEC 17025, the international standard for "General requirements for the competence of testing and calibration laboratories". This technical document provides basic information and detailed explanation about the establishment of the QC system in analytical and nuclear analytical laboratories. It is a proper training material for training of trainers and making familiar managers with QC management and implementation. It could encourage nuclear analytical laboratory to go for a quality system and for formal accreditation. It will assist the developing countries MS to understand the QC system and facilitate the implementation process. Nuclear analytical techniques, together with other advantages, could strongly benefit from a well documented QA/QC system and increase its market value and acceptance in the public perception.

ANNEXES

Annex 1
TEMPLATES

A number of examples are provided here to assist the quality manager in composing some of the documents and forms that will be required by the quality system. The focus here is on the contents. Little attention is given to the appearance of documents because this is a matter of personal preference, and the layout is often defined for the whole organization. The quality manager is advised, however, to keep the graphic design of forms and the title page of documents as simple as possible, and to confine the information to whatever is required by ISO 17025 and by the policy of the organization.

The following examples are available:

6.1. Quality manual

The quality manual example is structured in the way analysts usually operate, and a laboratory is often organized. It differs somewhat from the structure of ISO/17025, but the relevant clauses in the quality standard are listed in each section to assist the writer.

The writer must find a balance between statements that are too vague or too restrictive. From this perspective it is also advisable to complete the text of the quality manual after the other Level 2 documents have been written, and when the quality manager is more experienced.

The quality manual is designed to be a very practical document. It gives a concise description of how the laboratory really operates on a daily basis. This document should describe a picture that is somewhat better than the present situation, to provide a challenge to the staff.

6.2. Analytical method

The example presents a general framework for a Standard Operating Procedure or Work Instruction that is used to describe a particular analytical method. The contents of each paragraph are described as an indication of what information is typically expected. The title and index pages are not included in this example.

Presentation of format and contents of this document is often a matter of laboratory or its institution policy. The example given is provided to help the quality manager where such guidelines are not available.

6.3. Job description

The first example gives an example for a job description.

The second example is suitable for a laboratory that applies a system of performance appraisal and management. The document is used first as a “contract” between the incumbent and her/his direct supervisor for the next period (typically six months), and forms the basis for

the performance appraisal at the end of this period. During this appraisal and subsequent interview, the performance of the worker is evaluated according to whatever system is used by that organization, and the job description is revised for the coming period. This process is repeated according to the cycle that is used in the organization.

6.4. Non-conformance

The proposed form is suitable for the registration of non-conformance, for describing its root cause, for formulating corrective and preventive action, and for recording the plan of action to deal with it. It is assumed that somebody in the laboratory will register the problem, and hand the form to the quality manager to initiate and manage further action.

Some of the elements of this form can be retained for the management of complaints. It will need a section with information on the client, but less space is required for the action plan.

6.5. Internal audit plan

6.5.1. Counting facility

The example is composed in general terms and can be used for most of the large counting systems in the laboratory (α- and γ-spectrometry, gross αβ-counting, liquid scintillation counting *etc.*). The quality manager may, however, find the audits to be more effective when using a separate plan for each technique, with elements that are specific for each type of instrument.

The auditor ticks off the items that comply with the expected performance, or indicates the aspect(s) in which the current situation does not satisfy the description for that item. The responsible person and auditor often discus such a deficiency; and find a way to deal with it during the audit. It is advisable to record suggestions on how to resolve a particular deficiency.

6.5.2. Radiochemical method

The use of this form is similar to the audits on instruments.

6.5.3. Method validation

Audits on method validation and the use of non-routine methods are difficult to apply because of a lack of analysts with a scientific background to understand the methodology, and who are skilled in auditing too. The example tries to reduce this problem by focusing on a number of quality criteria that are relevant to this type of work, and which are accepted internationally. Scientific and technical questions are left to the professional competence and integrity of the analyst.

6.6. Test report

The diversity of analytical work carried out by laboratories can not be covered by a single document. The example therefore only demonstrates how the important requirements of the quality standard can be satisfied.

6.7. Qualification

According to item 5.2.5 of ISO 17025, qualification of laboratory staff has to be developed. The template in the example gives some ideas how to do this.

2. TEMPLATE 1: QUALITY MANUAL

2.1. Quality system policy and objectives

Start the quality manual with a statement on the vision of the laboratory, and the commitment of its management to quality. Include a summary of the corporate quality policy (if available).

$ 4.2.2 (a)

2.2. References

List the documents in the format that is accepted for the laboratory or for the organization.

2.2.1. Normative references

These documents describe the external policies and regulations that must be satisfied by this quality system. Examples are ISO 17025 and the quality manual of the organization (if available).

2.2.2. Internal documents

This will typically include all the Level 2 documents. A small laboratory can list all its methods or Level 2 documents (perhaps as an appendix), while a larger one with many methods may find it more convenient to have a separate inventory of documents and forms.

2.3. Quality management systems

2.3.1. Organizational aspects

Describe the scope of the analytical products and services provided by the laboratory, and the general strategy it applies to assure the quality of this work. Describe the position of the laboratory in the larger organization with respect to lines of authority (an organogram may be helpful) and the technical support it provides and receives.

$ 4.1 and 4.2

2.3.2. Accountability

Define relationships inside the laboratory, and show position of quality manager. Identify persons with special responsibilities (e.g. sample receipt and custody, document control, standards custodian *etc.*) and authority (e.g. approval of Level 2 documents, work instructions, test reports *etc.*). Describe arrangements when the responsible person is not available.

$ 4.1 and 4.2

2.3.3. Documentation and records

Describe the structure of the system documentation, and the handling of operational records (raw and processed analytical data) and technical information (manuals *etc*). Explain how the safety and security of these are maintained. Describe (here, or use a separate Level 2 document) how documents are written, reviewed, revised, authorized and released. Backup of raw data (e.g. pulse height spectra), intermediate results and reports should be addressed.

$ 4.3 and 4.12

2.4. Quality support systems

2.4.1. Personnel

Define the systems and procedures that are used by the laboratory to ensure that analytical work is carried out, without supervision, only by competent persons who have been trained and authorized to do so. Describe how the records for this system are maintained. A larger laboratory may find it convenient to present details in a separate Level 2 document.

$ 5.2

2.4.2. Client liaison and marketing

Describe how the needs of the client are translated into a service brief, the submission of tenders, the handling of orders and deviation, and after-sales service. Quality plans for non-routine projects and for standing orders can be addressed.

$ 4.4 and 4.7

2.4.3. Accommodation and environment

Describe how environmental conditions that may affect the quality of results are monitored and maintained, and how radioactive contamination between rooms is prevented. Radiological management and surveillance can be introduced here.

$ 5.3

2.4.4. External quality control

Confirm that the laboratory is committed to external verification of its own quality control measures through analyses of reference materials, inter-laboratory studies and proficiency testing. Describe the procedures that are applied for this purpose.

$ 5.9

2.5. Technical management systems

This section forms the core of the document as far as operational staff is concerned. It will be easier for them to relate to its contents, if it is structured is a way that they can recognize from their typical laboratory experience. This example presents one possibility, but the quality manager might find another structure better suited to the needs of that laboratory.

2.5.1. Sampling and sample management

Describe the way in which a sampling plan is formulated and carried out (if the laboratory is involved in sampling), and how the integrity and identity of the sample is maintained during transport and storage. Indicate how problems like sample degradation, contamination and tampering are reduced. One can dedicate a Level 2 document to this issue. State the policy of the laboratory on the period after completion of analyses that samples are kept.

$ 5.7 and 5.8

2.5.2. Internal quality control/equipment

Describe the systems and procedures to produce objective evidence that equipment and facilities are always under control while they are being used for analytical work, and the action taken when they are outside specification. A laboratory operating a variety of instruments and methods, can consider using a separate Level 2 to cover all the details.

$ 5.5/5.9

2.5.3. Calibration and traceability

Describe the steps that are taken to ensure that all calibration standards used for instruments and methods are traceable to national standards. Provide proof that methods and instruments are calibrated before they are used for analysis.

$ 5.6

2.5.4. Routine analyses using validated methods

Describe the typical flow of a task in the laboratory: receipt and registration, breakdown of client's needs and service design, allocation of jobs to different groups/persons, analyses using documented methods, compilation of laboratory reports into one client report, and the handling of records. List special arrangements for standing orders.

$ 5.4.1 and 5.4.2

2.5.5. Non-routine methods and method validation

Describe the typical quality criteria that are applied (selectivity, precision, trueness, detection limit *etc.*) to decide whether the method is fit for purpose. Show how the uncertainty of measurement is determined (or estimated) and reported. Describe steps to validate in-house computer programs to capture and process data.

$ 5.4.3 to 5.4.7

2.5.6. Reporting of results

Describe how intermediate and final results are verified and reported to the client, and who are authorized to release test reports. Handling of special requirements (e.g. electronic data transfer), follow-up work and amendments must be addressed.

$ 5.10

2.6. Management of internal improvement

2.6.1. Management review

Describe the reviews that are carried out periodically to ensure that the practices and procedures of the laboratory are suitable and effective.

$ 4.14

2.6.2. Internal audits

Describe the systems and procedures that are regularly used to verify that the agreed arrangements are in fact carried out in practice.

$ 4.13

2.6.3. Non-conformance, corrective actions and preventive actions

Describe the systems and procedures that are used to identify and register non-conformance, to search for and find the root cause of the problem, to apply corrections to take effective steps to prevent its occurrence in the future (corrective actions). Preventive actions are actions undertaken as improvement or to prevent potential sources of non-conformances.

$ 4.9 to 4.11

2.6.4. Complaints

Describe the systems and procedures that are used to register client complaints, and to ensure that corrective and preventive action has been taken.

$ 4.8

2.7. External support

2.7.1. Purchasing services and supplies

Describe steps to ensure a reliable supply of material and equipment, to verify that the received goods are fit for purpose, and to maintain their integrity.

$ 4.6

2.7.2. Subcontracting of tests and calibrations

Describe the procedure when some of the analyses are performed outside the laboratory, and steps to ensure that the client is kept informed and satisfied.

$ 4.5

3. TEMPLATE 2: ANALYTICAL METHOD

3.1. Purpose

Give a brief description of what will be achieved when the instructions are carried out; with the focus on the product that will be delivered. *Example: To obtain the activity concentration of ^{131}I in fresh milk samples.*

3.2. Scope

Summarize the field of application of the method that is described in this document, in order to inform the reader specifically of what is included and what is excluded. The scope defines the limits within which the method has been validated, and where the user can expect it to produce reliable results. The following aspects are essential:

– Type of material to be analyzed: A method that has been tested for tap water, will not necessarily be fit for sea water or the liquid from a sewage plant.

– Working range: Define both lower (detection limit) and upper limit.

– Reliability of the results: Quantify the precision and accuracy; and at different concentration regions if applicable.

– Other restrictions that have been identified during validation.

These limits should be sufficient to allow the analyst to decide whether the method is fit for the intended purpose. The application of a method outside its proven scope, is probably the most important single cause of poor analytical results.

3.3. References

3.3.1. Normative references

List the documents and regulations that provide guidelines or requirements for the product of this method. Revision of these will automatically lead to revision of this document. *Example: Document describing the client requirements for a dedicated analytical method.*

3.3.2. External documents

This category covers relevant publications in technical journals, books, reports, operating manuals for instruments and software, and other sources of information.

3.4. Quality system documents

This will include the validation report on this method, performance test reports, other supporting methods, and sources of the information that is required by this method.

3.4.1. General

This section is included to have space for issues that the writer considers to be relevant to the method, but which does not fit elsewhere. The topics are optional, and are listed only to assist the writer.

3.4.2. Health and safety

Stipulate all the factors that must be considered for the safe execution of this set of instructions: the protective clothing or equipment, interlocks on facilities, radiation and contamination hazards, toxic or dangerous chemical substances, regulatory requirements, and the support or supervision by safety personnel. All the facilities and procedures to control and minimize the effects of hazardous waste (if any) should be addressed.

3.4.3. Outline of the method

Give a general overview of the logic of the method, the principles involved and the flow of the process. Discuss the application where the results will be used. This should help the reader to understand the process before encountering the detailed instructions.

3.4.4. Definitions and abbreviations

Define concepts, phrases or abbreviations that are not generally used in the analytical field, or which may be applied in a novel context in this document. Note: The laboratory may consider a glossary of technical terms, including the ISO definitions, with explanations that operational staff can understand. This can be a LAB-DAT-document — see 4.4.1.3 (b).

3.4.5. Typography

This may be necessary where computer communication is required. Discuss the conventions that were used by the author, in order to explain some unfamiliar concepts to the reader. Examples: The code “File”|”Open”|”Read only” may mean options from a menu with drop-down or pop-up menus. Input by the operator may be identified using a different font.

3.5. Requirements

In this section all the facilities, equipment and consumables are listed. Sufficient detail is provided so that the analyst can replace items or replenish stock in the future. The sub-division that is suggested here can be adjusted to suit the needs of the laboratory and the characteristics of the method.

3.5.1. Apparatus

List all the larger instruments and counting facilities:

– Gamma-spectrometer: Use facility LAB-XCF-123
– Liquid scintillation counter: Use facility LAB-XCF-234 or –237

List and describe all the tools, equipment and apparatus that are required for carrying out these instructions; including items that have been designed and manufactured specially for this method:

– Automatic pipette: Adjustable between 100 and 500 µl, with disposable plastic tips.
– Special handling tool: Manufactured to drawing LAB-DRW-012-456

3.5.2. Material

List all the consumables that will be required, and specify any parameter that may affect the quality of the results (e.g. a particular brand that is known to be reliable). List standard laboratory ware.

– Membrane filter, cellulose nitrate. 47 mm diameter, 0.45 µm pore: Use brand XYX.
– Petri dish: Glass, 100 mm diameter, with lid, 15 required.
– Planchette, stainless steel, 47 mm diameter with 3 mm rim: Use standard item LAB-CS-345.

3.5.3. Chemicals

List all the chemicals to be used, including radioactive standards and certified reference materials that are required. Specify issues that may affect the quality of the data, e.g. grade of chemical of specific brand that is preferred.

– Ion exchange resin: Use ABC type 1X8, 200 mesh, chloride form.
– Hydrochloric acid, analytical grade, about 35% HCl: Use brand XYZ item A-123.
– IAEA-321: Milk powder.

3.5.4. . Reagent and test solutions

Describe the solutions to be prepared prior to execution of the analyses, including test solutions that will be used for internal QC. The reagent containers must be labeled with at least the following information: description, nominal concentration, expiry date, and name of the analyst. In addition, the test samples must have a unique identification code that will allow trackability of data.

– Dilute HCl: Dilute 40 ml (measuring cylinder) concentrated hydrochloric acid (analytical grade, about 35 % HCl) to 1 l (volumetric flask) with distilled water. Shake and transfer to a labeled plastic bottle.

– Test solution RA-01/$$ (ca 200 mBq.ml-1 of 226Ra): Pipette about 2 ml of standard radioactive solution LAB-CRS-345/12 in a plastic container and weigh accurately. Add 1 ml of conc. HCl, fill to the neck and weigh. Shake to mix solution. Calculate correct 226Ra concentration, and add the next number to the identification code (= $$). Label the container.

3.5.5. Quality system elements

List all the elements from the quality system that will be used in this method, including software that has been developed in-house for data processing:

– Data Sheet LAB-FDS-123: (Radium in water) Raw data.
– Test Report LAB-FTR-234: (Radium in water) Measured activity concentration.
– Spreadsheet LAB-QSS-345: (Data processing) Radium in water samples

3.6. Instructions

Divide the process that is used in this method, in a number of logical groupings or sections. Use points where the process can be interrupted as break points. The following are examples:

- *Sample receipt and verification*
- *Chemical separation of the analyte*
- *Calibration of measuring instrument*
- *Measurement of prepared samples*
- *Data processing and reporting*
- *Records and closure*

Now describe each of these sections as a number of separate and consecutive steps to be carried out by the analyst. Number each of these instructions:

- List the instructions in the same sequence it must be performed.

- Use the commanding style (*i.e.* the imperative) to formulate an instruction. *Example: "Stir the solution for about 5 minutes" (and not: "The solution should be stirred for 5 minutes").*

- Focus on the action that the analyst must take *("Activate F1 to evacuate")* and not on the expected outcome or result of that action *("The system must be evacuated").*

- Assume that the reader is capable of operating the equipment (if not, the need for on the job training can be specified in paragraph 4) and skilled in the basic techniques that are required (e.g. weighing, titration, calculations *etc*). Assume, however, that this is the reader's first contact with this method.

- Give the analyst an indication of the errors on measurement that can be tolerated so that time is not wasted on being over-meticulous on the one hand, or that repeatability suffers from carelessness on the other.

  The following are examples:

- Weigh *out (4,0 ± 0,5) g of ……*
- *Use 100 ml (measuring cylinder) of ……*
- Boil *solution for 5 to 10 minutes before ……*

There are a number of shortcomings that many writers seem to overlook when documenting a method. They are, however, rarely missed by technicians — and usually discovered during an assessment. The quality manager is encouraged to verify if the following uncertainties are covered in the formulation:

- How is the analyst informed that this is the correct method to apply in this case?

- How does the analyst know that this is the correct batch of samples he/she is supposed to analyze?

- What is the analyst supposed to do if something is not clear or different from what is expected: numbers that are not legible, too little sample material, difference between sample list and what is written on containers, and more of those problems that crop up in the laboratory?
- How does the analyst know where and when and how to hand in the laboratory report?

4. TEMPLATE 3: EXAMPLE OF JOB DESCRIPTION

Version 2.1

Job title: Lab Technician

Purpose: Ensuring the good execution of the lab experiments and the day-to-day operation of the laboratory

Reporting: The Lab Technician reports directly to the Laboratory Head

Job requirements:

- Qualified candidates must have at least 2 years of college chemistry or 3-5 years of chemical/lab experience
- An internal training on radiochemistry
- Being familiar with computer sciences such as word processing, spread sheet, databases
- Working individually as well as in a group
- Being aware of quality and safety regulations

Tasks:

- Preparing and realizing all actions correctly in conformity with the valid instructions and procedures (experiments, measurements and analyses,…)
- Collecting, processing of data and preparing the analysis report
- Keeping the existing lab infrastructure operational and controlling it in conformity with the valid safety and quality principles
- Managing the consumables, radio-chemicals and samples in the laboratory
- Reporting all irregularities noticed during the analysis procedure, as well as every defect or bad operation of the analysis apparatus to the Laboratory Head
- Strictly observing the safety regulations concerning working with dangerous and/or radioactive substances

Working environment:

- Working with chemicals, radioactive substances and cryogenic liquids

	Approved by: Lab Head	**Signed by:** Lab Technician
Name:	J. Daniels	D. Brown
Date:	2001-06-18	2001-06-18
Signature:		

Company Logo

Performance Management

Reference: ***LAB-JDS-123***	Revision: ***01***	Start date: ***2001-01-01***

Name of incumbent	
Designation	
Personnel number	
Management	

Objective of the post:

Give a short description of the expected output of this post. The focus is on the products and services to be delivered, not on the responsibilities of the person. Try to provide answers to the following questions: What is the contribution of this post to the overall mission and activities of the laboratory? Why is this post necessary? What would be missing should this post be left vacant.

A convenient approach is to apply a formulation one would use when this post is advertised.

Contracting	Incumbent:	Date:
	For laboratory:	Date:

Performance Appraisal	Scheduled date for next appraisal meeting	Date:
	Incumbent:	Date:
	For laboratory:	Date:
Main tasks of this post (key performance areas and specific objectives) with quantitative standards and performance indicators.		Performance appraisal and comments
List two to four most important items in each of the following categories: *Key performance area (KPA): Responsibilities/duties permanently assigned to this person. Work that is a fixed feature of laboratory, and goes automatically to this person without discussion. Routine tasks.* *Specific objective (SO): Project with definite starting date, and expected to be completed at target date. Work that is done only once; and according to action plan .* *List the criteria that are accepted by the person and his/her supervisor, and that will be used to measure performance at the end of evaluation period. Use quantitative measures when possible.* *This example applies to the quality manager of a young quality system.*		*Leave space for notes and comments made in performance appraisal.* *List aspects where this person performs above and below the standard agreed upon.*

KPA 1: Cultivate quality awareness among personnel To plan and implement effective action to train operational personnel in the principles of QA/QC, the requirements of ISO17025, and the policy and plans of the laboratory to become a quality organization. a) Discussions held with staff: topics, attendance, feed-back b) Personal evaluation of progress by laboratory manager c) Proven skills of staff to formulate basics of analytical methods	*As the system matures, KPAs relating to audit on internal QC, review of documents, handling of non-conformance and complaints can be added.*
SO 1: Level 2 documents To finish revision 00 of all the documents that are identified in the list LAB-DAT-001but have not been completed yet, and provide controlled copies of these to personnel. a) These documents satisfy all the requirements of ISO17025 b) The documents have been approved by the laboratory manager c) Feed-back from operational staff on state of consultation d) Target date for completion: 31 July	
SO 2: Storage for documentation To furnish Room 205 so that it can be used as a safe and secure store for all originals of the system documents and forms, job files with laboratory data on analyses, records of method validation, application software, computer backups, instrument manuals and documentation on reference material. a) The space for job files is sufficient for three year's data b) The changes are approved by the Safety Committee c) Feedback from Document Control function d) Target date for completion: 30 April	
SO 3: Audit plans for internal QC To design generic audit plans that can be used to verify the effectiveness and the state of implementation of the quality system regarding: (i) internal QC on large counting facilities, (ii) analytical methods based on instrumental techniques, and (iii) the validation of new analytical methods. a) Feedback on consultation with operational staff b) Evaluation by laboratory manager c) Uniform layout and coverage of essential elements d) Target date for completion: 30 June	

5. TEMPLATE 4: NON-CONFORMANCE

Laboratory Name Serial number ***LAB-NC-***

Part A: Registration of non-conformance		
Requirement or expectation:	Performance or observation	
	Complaint from customer Food control	
	Results are approximately 1000 times too high	
Signature: *Lab Head*	ID: *CMM*	Date: *2002-02-17*

Use Part E if more space is required

Part B: Root cause analysis		
Investigating team: *Lab Head*		
Findings: *An error in the formula for the calculation of the result in the Excel Spreadsheet lead to an overestimation of 1000.*		
Signature: *Lab Head*	ID: *CMM*	Date: *2002-02-18*

Part C: Correction/Corrective/Preventive action	
Correction: *1. A new corrected report has been sent on 2002-02-18*	
2. An analysis of previous measurements shows that no previous measurements have the same error. (2002-02-18)	
Corrective action: *1. Adapt the formula in the Excel sheet.*	
Name: *Lab Technician DLa*	Target date: *2002-01-18*
Closure (Name): *Lab Head CMM*	Date: *2002-01-19*
Preventive action:	
1. Check the other spreadsheets to check if the same error exists there also.	
Name: *Lab Technician DLa*	Target date: *2002-01-19*
Closure (Name): *Lab Head CMM*	Date: *2002-01-19*

Form: LAB-FSY-234 (rev 02)

Part D: Non-conformance follow-up	
The Spreadsheets and all other related Spreadsheets were checked and the error was corrected. The problem should not occur any more.	
Closure (Quality Manager): *QAM PVe*	Date: *2002-03-30*

Part E: Notes

Laboratory Name Instrument identification code: ***LAB-XCF-***

6. TEMPLATE 5: INTERNAL AUDIT PLAN 2002

Group	Audit team	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
1 Techniques													
LSC				x									
γ- spectrometry							x						
NAA									x				
α- spectrometry											x		
TLD-dosimetry													x
2 Organization													
Quality manager		x											
Documents and Archiving		x											
Personnel department		x											

Made up by:	Quality Manager	Date:
Approved by:	Director	Date:

Audit Plan: Counting Facility

Action or requirement	Observations on compliance
ID code is clearly displayed on instrument, files, documents and forms	
Performance tests are described in numbered and authorized document(s)	
This includes test samples to use, frequency of tests and action if out of control	
Operating instructions and/or manuals are readily available to analysts	
Current settings of operating parameters are documented	
Performance tests carried out and results documented according to schedule	
Prescribed action taken when instrument performance was outside limits	
Defects, breakdown and corrective action recorded in instrument log-sheet	
Defective equipment formally re-commissioned after repairs & maintenance	
Samples to be counted are stored in safe and secure location	
Completed samples are removed (as waste or in secure store)	
Sample lists are verified, and traceability could be demonstrated	
Data in e-format are labelled and saved according to laboratory policy	
Deficiencies from previous audit have been rectified	

Auditor:	Signed:	Date:

Form: LAB-FSY-346 (rev 04)

Laboratory Name Method identification code: ***LAB-XOP-***

Audit Plan: Radiochemical Method

Action or requirement	Observations on compliance
Performance tests are described in numbered and authorized document(s)	
This includes test samples to use, frequency of tests and action if out of control	
Preparation of current test sample(s) is fully documented and signed	
Performance tests carried out and results documented according to schedule	
Prescribed action taken when results for test sample(s) are outside limits	
Only controlled copies of analytical procedures are used in laboratory	
Reagent containers are clearly marked according to laboratory policy	
Chemicals stored in	
Traceability of test and tracer solutions to standards can be demonstrated	
Calibration of balance(s) and adjustable pipette(s) can be demonstrated	
Demonstrated that method is carried out as described in written procedure	
Trackability of values on test report to raw data can be demonstrated	
Samples to be analysed are stored in a safe and secure location	
Toxic substances and hazardous waste handled according to policy	
Protective clothing, equipment and safety procedures applied as required	

Auditor:	Signed:	Date:

Form: LAB-FSY-361 (rev 02)

Laboratory Name

Validation report: ***LAB-VAL-***

Audit Plan: Method Validation

Action or requirement		Observations on compliance
Client requirements are described in quantitative and measurable terms		
Action plan for project is formulated according to policy and approved		
Capability is demonstrated[1)] for performance criteria: Sampling		
Selectivity		
Detection limit		
Working range		
Precision		
Accuracy or trueness		
Safety, health and environment		
Feedback and reporting to client according to action plan can be demonstrated		
Changes to action plan and client specification are documented and approved		
Experiments, results, calculations and conclusions are documented		
Source documents are arranged and available for archiving		
Trackability of results to source documents can be demonstrated		

1) Grade as A (criteria is satisfied) or B (criteria has been adjusted) or C (still busy with this aspect)

Auditor:	Signed:	Date:

Form: LAB-FSY-321 (rev 02)

7. TEMPLATE 6: EXAMPLE OF A TEST REPORT

Mol 2004-06-14

OLEOTEST n.v.
Attn. M. Ph. Bastijns
Lage weg 427

2660 Antwerp

47911-00

Nuclear spectrometry
Building GKD Room 70
Boeretang 200
2400 Mol
Tel. +32-14-33 28 28
Fax +32-14-32 10 56

Analysis report

Description : Rough Soya-oil
ref.: 52.7537

Your reference : 00/010

Our reference : SN 3716

Reference date : 2000-04-02
Date of receipt : 2000-04-02

Results : On date of 2002-04-02: 12.00

^{131}I < 1.5 Bq/l
^{134}Cs < 1.6 Bq/l
^{137}Cs < 1.6 Bq/l

Method : High resolution gamma-spectrometry
Ge-detector : according to method MT.KB.001
Geometry : 250ml
Measurement time: 900 minutes

Operator Spectrometry R. Van Ammel	Service Head M. Bruggeman

This report shall not be reproduced except in full, without written approval of laboratory.
The results in this report relate only to the items tested or calibrated

Analysis report

Laboratory for Environmental Radioactivity
SCK-CEN, Boerentang 200, 2400 MOL
Tel.: +32-14-33 28 32 Fax.: +32-14-32 10 56

To: Jef Cenens
SCK-CEN
Boerentang 200 GKD
2400 Mol
Belgium

Reference no.:	**102.81.5324**			
Description:	Month June. Tritium control in atmospheric humidity, rooms 111 and 109.			
Date of receipt sample:	**2002-07-01**			

Our identification	Your identification	Analysis	Date of performance	Result (2s uncertainty)	Unit	Method of analysis	Opera-tor
21846.30629	1/06/111	H-3	2002-07-01	17 ± 5	Bq / l	MT.LRM.600	RJa
21847.30630	3/06/111	H-3	2002-07-01	15 ± 5	Bq / l	MT.LRM.600	RJa
21848.30631	4/06/111	H-3	2002-07-01	13 ± 5	Bq / l	MT.LRM.600	RJa
21849.30632	7/06/111	H-3	2002-07-01	16 ± 5	Bq / l	MT.LRM.600	RJa
21850.30633	8/06/111	H-3	2002-07-01	16 ± 5	Bq / l	MT.LRM.600	RJa
21851.30634	10/06/111	H-3	2002-07-01	21 ± 6	Bq / l	MT.LRM.600	RJa
21852.30635	11/06/111	H-3	2002-07-01	36 ± 6	Bq / l	MT.LRM.600	RJa
21853.30636	14/06/111	H-3	2002-07-01	18 ± 5	Bq / l	MT.LRM.600	RJa
21854.30637	1/06/2250	H-3	2002-07-04	22 ± 6	Bq / l	MT.LRM.600	FVe
21855.30638	4/06/2250	H-3	2002-07-04	24 ± 6	Bq / l	MT.LRM.600	FVe
21856.30639	7/06/2250	H-3	2002-07-04	18 ± 5	Bq / l	MT.LRM.600	FVe
21857.30640	8/06/2250	H-3	2002-07-04	21 ± 5	Bq / l	MT.LRM.600	FVe
21858.30641	10/06/2250	H-3	2002-07-04	23 ± 6	Bq / l	MT.LRM.600	FVe
21859.30642	14/06/2250	H-3	2002-07-04	< 10	Bq / l	MT.LRM.600	FVe

Method: Liquid Scintillation Counting of Beta emitters in water

Report approved by: F. Verrezen (Laboratory Head)	**Date of signature**	**Date print out**
		2002-03-12

This report shall not be reproduced except in full, without written approval of laboratory.
The results in this report relate only to the items tested or calibrated.

8. TEMPLATE 7: QUALIFICATION TABLE - LOW LEVEL MEASUREMENTS

	LVe	**ABe**	**LMa**	**Emr**	**DHe**	**...**
Sampling	2002-01-04					
Dispatching	2002-01-04					
LSC		2001-10-03				
α-spectroscopy				2001-03-04	In training (by EMr)	
Total α-β				2001-03-04	In training	
Equipment calibration		2001-10-03			(by EMr)	
Equipment maintenance		2001-10-03				
Issuing test reports			2000-06-03			

* The mentioned date is the date on which authorization was granted.

Made: 2002-02-02

Approved by Lab Head: 2002-02-03

Annex 2
EXAMPLES OF QUANTIFIED CRITERIA

Principles of quantified criteria

- Frequency of calibrations / adjustments :
  - once a week
  - before every individual use

- Comparison with previously obtained results:
  - successive results within 10 %

- Specifications by manufacturer
  - accuracy of pipette better than 0.6 %

Criteria for the quality of results (general)

- Qualitative analysis:
  - element assignment is unambiguous

- Quantitative analysis:
  - results will only be reported if, for the relevant element, the result for that element in the analysis of the control sample differs less than 3 standard deviations from the assigned value differs less than 10 % from the assigned value

Criteria for receipt of samples

- All samples should be accompanied by at least all of the following information:
  - name and address of customer
  - unambiguous coding by customer
  - (if relevant for composed samples) unambiguous identification of part to be analyses
  - number of samples
  - requested analysis, degree of accuracy and precision

- Check also
  - quality of packaging / encapsulation
  - (visually) degree of homogeneity
  - storage conditions, processing conditions, etc.

Criteria for sample drying

- dry samples similar as the control sample (or as prescribed in the certificate of a similar reference material)
- dry samples 17 h in an oven at approximately 105 ^{o} C
- freeze-dry samples until the vacuum is < 0.05 mbar

Criteria for sample sizes

- test portion mass typically less than 300 mg
- test portions should fit in capsules of 10 mm high and 10 mm diameter
- total mass of sample provided by customer should be at least twice as large as the mass of the test portion

Criteria for spectrometer performance

- spectrometer performance at least once a week, (or …..at least every other week), (or …at a given day)
- control source and blank measured at ….cm geometry
- performance measurements during …h
- calculated source activity should be less than 3σ from group mean value or assigned value
- FWHM should differ < 10 % from last observed value (unless the spectrometer has been modified after the last measurement)
- FWHM/FW(0.1M) < 2.9
- Channel number of calibration peak with highest energy should be within 5 channels of last measurement, unless…
- Slope of energy calibration line should differ less than 0.01 from last calculated value, unless….

Criteria for pipette and balance

- Pipette
  - calculate the mean and standard deviation of 10 gravimetric replicates; standard deviation < … % (e.g. as specified by vendor)
- Balance
  - calibration to be done by certified calibration body, once per year
  - verification of calibration once per week using calibrated masses (or built-in calibration mass): observed masses should differ less than … % from assigned values slope of calibration curve should differ less than … % from unity

Criteria for control (and reference samples)

- Accept results if

$$| x_{observed} — x_{assigned} | \leq 2. \sqrt{(\sigma^2_{observed} + \sigma^2_{assigned})}$$

Inspect results, and possibly accept results if

$$2. \sqrt{(\sigma^2_{observed} + \sigma^2_{assigned})} < | x_{observed} — x_{assigned} | \leq 3. \sqrt{(\sigma^2_{observed} + \sigma^2_{assigned})}$$

Do not accept results if

$$| x_{observed} — x_{assigned} | > 3. \sqrt{(\sigma^2_{observed} + \sigma^2_{assigned})}$$

- Example of extra criterion:

$$100 \text{ x } | x_{observed} — x_{assigned} | / x_{assigned} \leq 10$$

Criteria for reagents, stock solutions, environmental conditions

- Reagents and stock solutions:
    - expiration date 1 year after preparation
    - transpiration and evaporation losses during storage < 1 % per month
- Environmental conditions:
    - temperature stability e.g. + /- 2 degrees Celsius
    - relative humidity < 80 %
    - radioactivity background: define a ‘standard’ background spectrum, and compare current situation using similar criteria as e.g. for analysis of control (reference) samples

Annex 3
FORMS

INSTRUCTION FOR TAKING OUT-OF-ORDER EQUIPMENT, OPERATING OUTSIDE OFFICE HOURS

Name experimentalist: Tel. during office hours Tel. outside office hours
Stop experiment if.....
Experiment/ Equipment: - water: - vacuum: - pressurized air : - elektronic supplies: - chemicals: - temperature: - gas supplies :
Danger: fire, explosion, toxic gases - liquids - dust Fire fighting/extinguishing :
Remarks at problems:
Switching-off of equipment :
Applicable from.... until ...
Date: Initials:

Spectrometer:
Name: Date: No.:

NIM-rack
manuf. :
type no. :
serial no. :

HV-supply
manuf. : Polarity :
type no. : HV. setting :
serial no. :

Main Ampl.
manuf. : Polarity : Coarse gain :
type no. : BLR setting : Fine gain :
serial no. : PUR on/of : Shaping time :
internal amplification (*0.1/*1.0) :

ADC
manuf. : Group size : ULD :
type no. : Course gain : LLD :
serial no. : Threshold : Coinc./Anti :
Analyse/Off : BLR/D :
Zero level : 0-100/0-10% :

Offset (U/D)	64	128	256	512	1024	2048	4096
U							
D							

Buffer
manuf. :
type no. :
ident. no. :

Pulse generator
manuf.:
type no.:
ident. no.: Normalize:
Polarity:
Pulse/sec.:
Pulseheight:

Switch setting (R/L):

R													
L													
R													
L													

Attenuation (U/D):

U D	*1.2	*1.4	*2	*2	*2	*5	*10						

Detector
manuf. : HV. setting:
type no. :
serial no. :

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