2 SECTION 2: ORGANIZATION OF THE WORK AND ADOPTION OF GOOD PRACTICES

This section will provide you some general recommendations for the organization of your work in the XRF Laboratory. Depending on the extent of your experience in XRF practice, some of the advised actions might be already implemented in your work. Other issues, apparently trivial, might force you to reconsider whether what you have or what you do really complies with the general concepts we got acquainted in the previous section.

We encourage you to review the provided contents and draw your own conclusions!

2.1 THE MAIN TASKS

The organization of the work in the XRF Laboratory requires of a set of actions, aimed to:

- Identifying the key products that are intended to be provided to the customers, in order to objectively focus in planning the work.

  > Hereinafter the term *service* may be used instead of *product*, to correspond to the specifics of the work of most of the XRF practice this module is addressed to.

- Ensuring the availability of the resources and information necessary to support the provision of services.
- Determining the optimal sequence of processes required for the provision of such services.
- Monitoring the workflow of the contracted services and the quality of the achieved results.
- Implementing actions aimed to seek continual improvement of the system.

2.2 ALL STARTS FROM THE MISSION...

The portfolio of services depends on the organization mission, which can be of different nature:

- **A social mission defined by a parent organization:** Your XRF laboratory or group may be part of an institute, agency, ministry or other non-commercial type of organization, to which you are supposed to provide some analytical services. For example, a XRF laboratory is established to provide analytical support to research/service mission of the Organization.
- **Specific services as part of an enterprise or consortium:** Your unit may be responsible for the qualitative and quantitative characterization of materials produced by a given manufacturer.
- **More versatile tasks, in the case of independent organizations:** Your XRF laboratory might be looking for insertion in the market of analytical services. In that case, your tasks will vary depending on the customer's needs, and you will have regular and occasional customers. The definition of your mission shall reflect that versatility as one of your strengths!

2.3 ORGANIZATION OF THE WORK

Keystones for a successful organization of the work in the XRF Laboratory are:

- The staff competence, capabilities and commitment.
- A reliable technical infrastructure and adequate working conditions.
- Availability in sufficient amount and quality of the needed resources (equipment, components and information).
- Documented technical procedures for all the analysis performed in your XRF practice, ensuring that a sound work can be carried out and that the risks of scraps are minimized, if not excluded.
- A quality control practice to avoid the release of non-conforming work or defects.

2.3.1 RELYING IN A COMPETENT STAFF

The staff of the XRF Laboratory must be selected on the basis that its qualifications and skills are adequate for the intended work.

Strong interpersonal skills and abilities to take initiatives are additional qualities contributing to the consolidation of a team spirit.

Commitment of the staff at all levels constitutes a basic premise to ensure continual improvement of the organization.

2.3.2 CONTINUAL QUALIFICATION OF THE STAFF

The management shall ensure a continual enhancement of the XRF personnel qualifications, to ensure their capability to fulfil the requirements of future tasks.

Management shall establish a plan for training of the personnel, and maintain records on the achieved results (CVs, diplomas, certificates).

Organizational training plan:

- social skills (communication, languages, time management, etc.);
- technical skills as needed in the processes (training specific to the XRF practice);
- computer skills;
- knowledge of markets and of customer needs;
- future demands related to strategic and operational plans and objectives of the organization;
- quality management knowledge.

Among the actions addressed to keep a continual technical improvement are the subscription to specialized journals, participation in scientific and technical conferences, workshops, revision, etc.

2.3.3 Internal communication with the staff

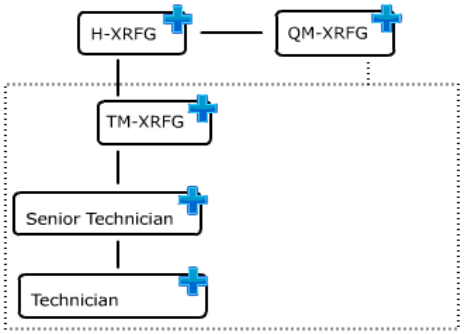
The management shall ensure a permanent and efficient communication with the XRF staff, in order to avoid misunderstandings or delays affecting the work performance.

Clear definitions in work assignments, as well as transparency in management decisions and staff expectations ensure a mutual understanding and commitment!

The communication is of utmost importance when defining the tasks for a given service, terms of delivery and expected requirements.

2.4 Defining responsibilities and authority

A hierarchical scheme of authorities avoids conflicts of responsibilities in taking decisions.



- Responsibilities must be defined as to correspond to the performance of the required processes.
- Substitutes must be nominated to ensure continuity of work in the event of unexpected absences.

Responsibilities shall be defined as not to create evident conflicts with the expected outputs. For example, the person defining the control samples for internal quality control is aware of the identity of the duplicate and control samples, and therefore, shall not be allowed to perform analytical work.

Lists of substitutions shall be prepared as to cover the main tasks required for the steady work of the laboratory, but not necessarily all of the functions. Some functions, which do not require of highly specific competence, can be performed indistinctively by more than one person.

2.5 Working conditions

The locals and workplaces in the XRF Laboratory must comply with the international or national regulations. Attention must be paid to:

- Workload
- Stress factors
- Social structure within the organization
- Internal communication
- Personnel protection
- Ergonomics
- Lighting
- Ventilation, air-conditioning/heating and many other factors
- De-humidification is a typical requirement in tropical and subtropical regions!
- Reliable mains supply and earthing.

All can be combined to enhance the overall effectiveness of the organization in achieving its quality objectives.

2.6 SAFETY REGULATIONS

While regulating safety is a national responsibility, international standards and harmonized approaches to safety promote consistency and help to provide assurance that nuclear and radiation related technologies are used safely.

For over 50 years the International Atomic Energy Agency (IAEA) has had a safety standards programme. More than 200 safety standards have been published which reflect an international consensus on what constitutes a high level of safety for protecting people and the environment.

The standards provide a robust framework of fundamental principles, requirements and guidance to ensure safety. They are applicable, as relevant, throughout the entire lifetime of facilities and activities.

2.6.1 SAFETY REGULATIONS IN YOUR XRF LABORATORY

XRF laboratory must comply with international and/or National safety regulations.

XRF laboratory's safety is everyone's responsibility. The laboratory supervisor shall monitor the safe operation of the physical facilities, laboratory apparatus, chemicals, and laboratory procedures.

Fire extinguishers must be accessible and free from obstruction.

First aid tools shall be available at hand reach.

2.6.2 SAFETY REGULATIONS IN RADIATION PRACTICE

A license often must be obtained prior to working with an X-ray machine and radioactive materials. Once obtained it must be kept current.

Area Requirements

The local components of an analytical X-ray system shall be located and arranged and shall include sufficient shielding, or access control such that no radiation levels associated to the analytical assay exist in any surrounding local area.

Each area or room containing analytical X-ray equipment shall be posted with a sign bearing the radiation symbol and the words "Controlled Zone". This includes labelling equipment.

2.6.2.1 RADIATION PRACTICE. PERSONNEL REQUIREMENTS.

Individuals must complete X-ray training prior to operating or/and maintaining analytical X-ray equipment. This training will include identification of radiation hazards associated with the use of the equipment; significance of various radiation warning, safety devices and interlocks incorporated into the equipment; proper operating procedures for the equipment; recognition of symptoms of an acute localized exposure; and proper procedures for reporting an actual or suspected exposure.

If the X-ray device contains a radioactive source, replacement, leak testing, or other maintenance or repair procedures shall be conducted only by individuals specifically authorized.

Persons exposed to radiations must have personal dosimeter. Control of the received doses must be carry out by qualify services.

2.6.3 LABORATORY AND CHEMICALS SAFETY REGULATIONS

XRF laboratories and chemical storage rooms shall be maintained in a clean and orderly condition at all times.

Local exhaust ventilation (such as laboratory fume hoods, glove boxes or industrial ventilation) is required when handling chemicals in a manner that can produce an airborne hazard.

Personnel handling chemicals shall use personal protective equipment (chemically resistant gloves, eye wear, footwear, lab coats, aprons, coveralls and respiratory protection).

Laboratories and chemical storage rooms shall not be used as eating or food preparation places.

Laboratory refrigerators shall not be used for food storage.

2.6.4 ELECTRICAL SAFETY REGULATIONS

All electrical installations shall be in compliance with National Standards.

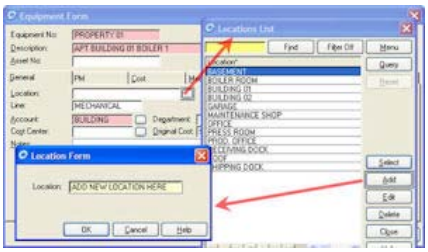
Electrical receptacles used in laboratories and chemical storage rooms subject to wetting, caustic, or acid atmospheres shall be in moisture proof enclosures.

Electrical Apparatus must be equipped with ground plugs and properly grounded.

USE OF EXTENSION CORDS IS NOT RECOMMENDED.

2.7 STOCKS

Technical documentation (certification of quality, catalogues, Material Safety Data Sheets, sample information), and materials (samples, Certified Reference Materials, sealed source and chemicals) shall be stored in well organized places, adequate for each kind of material.



An inventory (hard copy or electronic format) of all the materials must be keep updated. Some commercial software are designed for this purpose.

IF SUITABLE SOFTWARE IS NOT AVAILABLE TO KEEP HIGHLY AUTOMATED ELECTRONIC RECORDS, AT LEAST HARD COPIES SHALL BE MAINTAINED. IT IS BETTER TO HAVE A SIMPLE SYSTEM FOR CONTROL THAN HAVING NONE!

2.7.1 THE USEFULNESS OF STOCK RECORDS

Stock records are useful...

- to prevent loses of service manuals or difficulties in their prompt allocation, thus avoiding delays in work assignment realization.
- as a warning on the availability of spare parts and components stock, thus allowing a more efficient planning on purchase.
- to keep a track on consumables and spare parts consumption.

2.7.2 SAMPLES AND CERTIFIED REFERENCE MATERIALS

- Certified Reference Materials (CRMs) must be provided by suppliers supported by a QMS.
- CRMs must have the corresponding certification emitted by the QMS of the manufacturer.
- CRMs must be preserved in the correct environmental conditions (temperature, humidity, light) ensuring its stability.
- After receiving samples, check them for integrity and conformity with costumer's specifications on delivery.
- Store samples in proper conditions and clearly identified. If possible, keep portions of samples as witness for further analysis in case of complaints.

2.7.3 CHEMICALS

Chemicals should be stored in areas approved for their use. Appropriate hazard warning label shall be placed on doors of chemical storage areas.

- An inventory of all chemicals shall be maintained with the Material Safety Data Sheet (MSDS) and be readily available.
- Chemicals shall be stored in chemically compatible families.
- Workers shall be instructed on safe chemical handling and safe chemical storage procedures shall be enforced.

2.7.4 RADIOACTIVE MATERIALS

- Possession and use of radioactive sources requires a laboratory license, obtained from the national regulatory body. Radioactive sources must comply ISO 2919 requirements.
- Radioactive materials should be stored only in areas properly marked and approved for their use.
- Manipulation of radioactive material must be done by properly trained personnel.

2.8 RELIABLE INSTRUMENTATION

The instrumentation shall comply with the technical specifications required for the performance of the intended work. Instrument requires **proper operating conditions** for long term fault free operation:

- Adequate environment operating conditions.
- Protected against external electrical disturbance, electromagnetic interference or electrostatic discharges.
- Properly wired and grounded.

Instrumentation shall be periodically verified and calibrated. Records containing the calibration results (certificates) must be maintained. Measuring instruments shall be unequivocally labelled to ensure traceability of the measurements.

Faulty instruments or suspected to give erroneous results must be keep apart and signed "OUT OF SERVICE". After reparation, the instrument must be calibrated before use.

2.8.1 SEMICONDUCTOR DETECTOR CONDITIONING

Care should be taken not to exceed the ambient temperature range specified by the manufacturer. Moisture, corrosive gases and liquids, and conductive dust can all have adverse effects on a system that is not adequately protected.

Incorrect voltage or frequency may cause a malfunction or damage to device. Detector can be affected by rapid changes of voltage. Follow manufacturer's specifications about the rate of rising the voltage. Detector could be damaged if polarity of applied voltages is incorrect.

Electrical noise must be maintained as low as possible. Precautions should be taken to avoid electrical disturbances (electrical noise, electrostatic discharges, sags, swells, surges, short and long term outages). Proper wiring practice will minimize the influence of electrical noise, which may cause malfunction of equipment.

The cooling of the semiconductor detector (electrical or using liquid nitrogen) should be guaranteed all the time during detector operation. In the specific case of liquid nitrogen, the accompanied dewar should be refilled periodically. It is necessary to take into consideration that the lack (or unexpected absence) of liquid nitrogen can cause abnormal functioning (or damage) to the semiconductor detector.

Everyone operating these devices should be educated in its capabilities and limitations.

2.8.2 Ensuring stability in method performance

It is a good laboratory praxis to a have "log-book" documenting the sample preparation (date of preparation, unique sample preparation number, calibration standard / sample description, weight for pressing, etc.) and the spectrum acquisition (date of measurement, spectrum file name, acquisition time, live time, dead time, tube current when applicable).

Also any essential changes (replacement of protective foils, changes in geometry, other high voltage setting, etc.) and problems (instabilities, repair of equipment, etc.) must be noted.

Several parameters shall remain constant during sample's measurements for an established calibration:

Geometry

- vacuum conditions (if applicable)
- thickness and kind of protective foils

In the case of x-ray tube excitation:

- anode material
- kind of secondary target
- high voltage

The sample position must be reproducible and the irradiated area must be in the centre and smaller than the sample area itself. For tube excitation the detector must be electrically insulated from the rest of the spectrometer, because grounding problems will result in deteriorated energy resolution and other spectrum deformations.

2.8.2.1 Ensuring stability in method performance. Parameters under control.

Linearity as a function of the tube current

The tube current for secondary target and direct X ray tube excitation spectrometers will have linear influence on the spectrum emitted by the sample. Do measurements of a calibration standard with increasing tube current as parameter, from the lowest to the highest setting. Keep record on the stability of these parameters, and make corrections whenever the observed variations exceed the maximum acceptable boundaries.

Detector resolution (FWHM) as a function of time.

The better the resolution the better overlapping peaks can be apportioned. A value for the FWHM close to specification of the detector should be reached. The manufacturer have specified his detector under certain conditions, usually at a low count rate for a Mn Ka peak (originating from an Fe-55 source).

Stability of the energy calibration

For the Rh secondary target spectrometer a drift of the energy calibration is observed. In order to control the situation you can measure a Zr foil repeatedly.

Calibration.
For *each* element under consideration (in principle for each element found in an unknown sample) an adequate number of very well defined standards, with *a range of concentration values bracketing the sample concentration*, must be prepared. For calibration, use measurements of either pure standards with 100 % concentration (metals), or simple compounds (of highest possible concentration) pressed as pellets.

2.9 OUTSOURCING

Where the XRF Laboratory procures outside tests and calibrations, it shall use only outside support services that are of adequate quality to sustain confidence in its technical activities.

The choice of suppliers of services shall be based on the quality of the provided items, the promptest delivery term and the lowest cost/benefit ratio.

Every year the quality of the provided services and products shall be evaluated during the management revisions.

2.10 DOCUMENTED TECHNICAL PROCEDURES

An efficient and sustainable organization of the technical work carried out in the XRF Laboratory requires of the definition and establishment of documented technical analytical instructions. Such procedures allow ensuring the harmonization in XRF work practice and fulfilment of minimal requirements for ensuring a commensurable quality of the analytical results. The technical analytical instructions shall include:

- a clear definition of the scope of the procedure.
- a technical description of the theoretical (or implicit) principles on which the instruction is based on.
- the main steps to be followed and the sequence of tasks to be performed.
- the format to report the achieved results.
- a clear statement of the criteria of quality performance achieved.

For example, a procedure can be prepared for a characterization of a material using XRF.
The scope, principles and relevant interferences of the method shall be declared. For some kind of samples, a treatment step must be explained, including the chemicals, solutions and apparatus involved in the process The main relevant characteristics of the instrument and the process followed to set up the measurement conditions must be described, as well as the data acquisition, processing and quantification. Finally, a report with information of measurement values and interpretation of the results is delivery.

2.11 HANDLING ASSIGNMENTS AND ENSURING CUSTOMER SATISFACTION

Costumer needs vary in a wide range of possibilities and the scope of an established procedure could not cover the customer requirements.

Therefore, a constant communication with the customer becomes an unavoidable need to ensure a smooth flow of the work and to pursue agreement with the customer in regard to any modifications or changes arising from unexpected issues revealed during the realization of a given work assignment.

The general work flow chart provided below is an effort to comprise any possible scenario.

2.11.1 A GENERAL ASSIGNMENT FLOWCHART