GOOD FOOD LABORATORY PRACTICES

सत्यमेव जयते

FOOD SAFETY AND STANDARDS AUTHORITY OF INDIA
MINISTRY OF HEALTH AND FAMILY WELFARE
GOVERNMENT OF INDIA
NEW DELHI
2018

ACKNOWLEDGEMENT

The contents of this GFLP document are sourced from the following documents of FAO-

1. De Jonge LH & Jackson FS (2013). The feed analysis laboratory: Establishment and quality control Setting up a feed analysis laboratory and implementing a quality assurance system compliant with ISO/IEC 17025. In: Makkar HPS (Ed.). Animal Production and Health Guidelines No. 15. FAO, Rome, 89p
2. FAO (1993). Manual of food quality control: Quality assurance in the food control chemical laboratory. Food and Nutrition Paper 14/14. FAO, Rome, 126p
3. Weatherwax J & Martin PG (1986). Manuals of Food Quality Control: 1. The Food Control Laboratory (2nd edition). FAO, Rome, 67p

GOOD FOOD LABORATORY PRACTICES (GFLPs)

TABLE OF CONTENTS

1.0 SCOPE:

1.1 These Guidelines specify the general requirements for the competence to carry out systematic sampling of food samples, conduct chemical, microbiological tests and testing of packaging materials to ascertain the quality of food. It covers the tests performed using standard methods, non-standard methods, and laboratory-developed methods.

1.2 These Guidelines are applicable to all organizations performing tests to ascertain the quality of food material including packaging material. These include, for example, first-, second- and third-party laboratories, and laboratories where testing forms part of inspection and product certification.

These Guidelines are applicable to all laboratories regardless of the number of personnel or the extent of the scope of testing and/or calibration activities. When a laboratory does not undertake one or more of the activities covered by this Standard, such as sampling and the design/development of new methods, the requirements of those clauses do not apply.

1.3 The notes given provide clarification/guidance of the text and examples. They do not contain requirements and do not form an integral part of these Guidelines.

1.4 These Guidelines are for use by laboratories in developing their management system for quality, administrative and technical operations. Laboratory customers, regulatory authorities and accreditation bodies may also use it in confirming or recognizing the competence of laboratories. This International Standard is not intended to be used as the basis for certification of laboratories.

NOTE 1 The term 'management system' in these Guidelines means the quality, administrative and technical systems that govern the operations of a laboratory.

NOTE 2 Certification of a management system is sometimes also called registration.

1.5 Compliance with regulatory, calibration of equipment/glassware and safety requirements on the operation of laboratories is not covered by these Guidelines. These Guidelines are over and above ISO/IEC 17025:2005 framed specially for those testing laboratories engaged in sampling, conducting chemical and/ or microbiology tests and testing of packaging materials to ascertain the quality of food. Laboratories seeking accreditation as per ISO/IEC 17025:2005 should comply with the requirements of ISO/IEC 17025:2005.

2.0 STRUCTURE OF FOOD LAB:

2.1 Personnel:

Personnel need to clearly understand the nature of the foods they are testing and reasons for testing when undertaking contract review and method selection.

2.2 The Management Structure:

An up-to-date chart showing the organisational structure and lines of responsibility of the laboratory is an important feature of the quality assurance programme and should appear in the Quality Assurance Manual. When the laboratory is part of a larger organisation it may also be desirable to have a chart showing the management and operational relationships which control the input of work requested and the output of results from the laboratory, the overall picture of the laboratory and the resources available for it. The general structure of staff is shown in Figure 2.1.

The office of Head of Laboratory (other titles such as "Chief" or "Director" are often used) may include a Deputy if the laboratory staff is sufficiently large. Usually, however, the duties of the Head, in his or her absence, are assumed by a senior supervisor of the analytical staff. The analytical and support staffs are discussed below in Sections 2.5 and 2.6, respectively. The administrative staffs includes all administrative assistance such as a secretary, typing and filing clerks, a management assistant and a librarian (if the laboratory library is of a size to need one). Basically the administrative staffs are those persons generally involved in "office" or "paperwork" functions. These staffs are very important to the smooth operation of a laboratory. It is false economy to understaff the administrative group because their work often must then be done in part by the analytical or support staff.

Figure 2.1 The staffing structure of a typical food testing laboratory is as follows:

Head of Laboratory			
Officer –in-Charge (**Chemistry Section**)	Officer –in- charge (**Microbiological Section**)	Officer –in-charge (**Biotech. Section**)	Officer –in-charge **(Administrative Section)**
Team Leaders Technical Staff/Analyst Supporting Staff	Team Leaders Technical Staff/Analyst Supporting Staff	Team Leaders Technical Staff/Analyst Supporting Staff	Secretarial Staff Supporting Staff

2.2 Head of the Laboratory:

Although the duties of the head of the laboratory are many, some may be delegated and others undertaken by other parts of the food control administration. The laboratory Head may have to give evidence in court or write documents used in court, in which case he must have a thorough understanding of food and related law and court procedure. There will also be involvement in committee work and relations with other organizations. The laboratory Head is usually the spokesman for the laboratory in many instances.

Note: In case of FSSAI labs, the Head must prepare work plans with the food safety officers (FSO) and overall food control authorities. Sampling plans agreed with the FSO should aim at areas of concern and major abuses.

2.3 Supervisors:

The supervisor is the on-site manager of the laboratory. Having supervisors assigned to specific units or areas of work permits the Head to more effectively plan for execution the total workload of the laboratory.

Supervisors can be expected to do analytical work in addition to their supervisory duties. However, if their group exceeds five professional analysts, it is best not to require additional analytical work except for occasional problem solving and trouble-shooting. A reasonable maximum number of analysts for one person to supervise is 10 to 12. This can be more if nonprofessional support staff is added.

A supervisor's duties can include the following:

1. Assisting the Head in overall laboratory work planning and planning the work of the group supervised.
2. Receiving and assigning samples for analysis, within the group.
3. Answering questions and assisting in solving analytical problems posed by individual analysts.
4. Reviewing the reports of completed work and making appropriate recommendations.
5. Ensuring that the group has the necessary supplies and equipment to do the work.
6. Ensuring that proper laboratory safety and housekeeping practices are followed by the group.
7. Recommending to the Head new instruments or equipment needed, and training needs of individual analysts.
8. Recommending appropriate disciplinary action when needed to enforce laboratory rules or regulations.

9. Supposed to manage the entire laboratory in the absence of the Head.

Supervisors should train one or more analysts in their group to serve as backups, to supervise the group in the supervisor's absence. The back-ups should be given some formal classroom training in supervision in addition to on-the job experience.

2.4 Team Leaders:

Another important, and often overlooked, position is Team Leader. A team leader is a senior analyst who has been assigned a small group, usually not more than 4, to do a specific task or type of analysis. The leader has no supervisory functions as such, but is the coordinator of the group's activities and is the contact point for the supervisor.

Team leaders are most useful when a large number of a repetitive type of analysis is to be done in a specified period of time. This could be a specific analytical survey or an emergency public health problem requiring screening analysis. The leader usually works along with the group in addition to the coordinative function. Such experience is often useful to determine if the assigned leader has potential as a future supervisor.

2.5 Analytical Staff:

The basic job of the analytical staff is to analyze the samples received and to issue a report. They may also be required to appear in court as fact or expert witnesses to give evidence in relation to a report. They may also be called onto offer advice to industry and trade, to assist in improvement of food quality, or advice on conformity with standards or other legal requirements. This can involve the laboratory staff in factory visits and even requests to carry out experimental work. Whether or not the laboratory undertakes such work will be a matter of organizational policy. The decision will depend on a number of factors, including the availability of alternative facilities, the nature of individual ownership, etc. The integrity of the analyst is paramount, and superiors must be informed of any conflict of interest that arises. As in the case of food inspectors, it is proper for the analysts to have no vested interest in regulated industries. This requirement is mandatory in many countries.

2.6 Support Staff

The support staffs of a laboratory is those persons working in and for the laboratory who are not conducting analyses or are not involved in administrative duties. Some examples of duties include:

1. Glassware washing.
2. Cleaning and housekeeping maintenance.
3. Disposal of sample reserves (when no longer required).

4. Pest control.
5. Heavy lifting and moving.

Support staffs typically have little or no educational qualifications beyond the ability to read and write. However, they must be willing and able to learn not only their duties, but also laboratory safety procedures. It is most important that sufficient persons are hired as support. The work they do must be done by someone and this is usually an analyst or technician when there is insufficient support staff. There is no fixed module for numbers of support workers, but 15-20% of the number of analytical staff is often sufficient.

3.0 INFRASTRUCTURE AND ACCOMMODATION AND RELATED REQUIREMENTS:

3.1 General Principles:

Facilities must allow the laboratory's work to proceed both effectively and safely.

Laboratory design should reflect the general features of the work programme anticipated in the long-term (10-20 years) rather than the specific pattern of current work.

3.2 Design of the Laboratory:

Even though the final design of the laboratory is made by architects and engineers, the analytical staff should be involved in some of the decisions that will ultimately affect their working environment and conditions. The food control laboratory have several functions such as chemical analysis of foods for proximate composition, trace metals, additives, GM testing, nutrients and toxicants, some basic food microbiology analysis and product organoleptic evaluation .

3.3 General Considerations:

Laboratory layout should be devised with efficiency in mind. For example, the distances staff have to walk for the different steps of the analytical processes they undertake should be as short as possible, though bearing in mind that some procedures may have to be segregated from others for analytical and/or safety reasons.

There is often a 5 year period from the decision in principle to build a new laboratory to when it is accepted and operational. Also there will usually be an expectation that it will not require major alteration for a further 10 years. Given that the work load may change in this time span there are real disadvantages in designing a laboratory just to reflect

the detail of the currently anticipated work load. Even within a given work volume, events may demand that the relative emphasis given to the different types of analyses may change. Additionally, advances in instrumentation and analytical methodology may alter the space and environmental requirements for a particular analysis. There is an argument for designing a laboratory in terms of "generic" activities and "specialised" activities.

Generic activities can be categorized as "wet chemistry" which will require extensive provision of fixed benches with water, power, sinks, fume cupboards, reagent shelves, glassware cleaning and storage, as compared to "instrument rooms" where less extensive servicing (though with additional piped gas supplies and perhaps stabilised power supplies) and flexible arrangements of movable tables/benches may be adequate.

Specialised rooms may be required for "clean air" work (e.g. on some environmental contaminants) or for work with substances which need to be handled with special care either for safety or for cross-contamination reasons, e.g. radioactive materials and some particularly toxic substances or for storage and dispensing of standards of pure compounds which are being analysed at trace levels elsewhere in the laboratory. A specialised room for large-scale and/or dusty sample preparation activities, e.g. grinding, blending, mixing, stirring will be invaluable, particularly if work is envisaged on heterogeneous analytes (e.g. aflatoxins in nuts or figs where primary samples of 30 kg are sometimes needed). With this approach the important design parameters are those concerned with correctly identifying the needs for specialised activities and with estimating the relative needs for the generic activities of "wet chemistry", "instrument room" and - for that matter - "food microbiology" if, as is often the case, that is to be carried out in the same premises.

Offices are needed for management and for clerical staff. There must be toilet and washing facilities for all staff. Eating, drinking, and smoking are always discouraged, and should be prohibited, in the laboratory proper. It is the responsibility of management to provide an appropriate alternative area for these activities. A separate staff room, however small, deserves consideration since it not only provides a greater degree of safety to laboratory personnel but also helps to ensure sample integrity. To provide for a prompt exit in the event of fire or other emergency, at least two entrances/exits must be provided for each laboratory whenever possible.

3.4 The Chemical Laboratory:

From a quality assurance standpoint, the design features which are important are those which can lead to erroneous results or to "lost" work, leading to missed deadlines and cost overruns. Erroneous results can arise from test materials becoming contaminated (e.g. by dust) or by cross-contamination from another sample or from a standard. Whilst

good working practices will usually control most situations satisfactorily, a design which provides complete segregation of trace analyses from highly concentrated formulations and from pure substances used in preparing analytical standards is virtually essential: the segregation must apply to all facilities for washing/cleaning equipment, washing and storage of glassware, use of protective clothing and even transfer of notebooks and records.

Design features which avoid dust, whether from environmental sources or from other samples are highly desirable from the quality assurance standpoint. Dust contamination of test materials is essentially sporadic and uneven; as such it is likely it will often be missed by the normal quality control checks. Design should aim for dust avoidance by using glass-fronted reagent shelves, keeping work-tops clear of unnecessary "static" items, regular cleaning of work surfaces with absorbent cloths, floor and furniture designed so that they can be cleaned with vacuum cleaners with suitable exhaust filters or absorbent mops. Designs which involve cleaning by the traditional "duster and brush" approach which simply spread contamination more widely should be avoided. Ventilation intakes and fume cupboard exhausts must be sited carefully so as to avoid re-circulation of laboratory air and the associated risk of contamination of test materials and hazard to laboratory staff.

3.4.1 Equipment and Instruments:

The complexity of equipping a laboratory and the consequent delay in production of useful results should not be underestimated. In the early stages, the requirements for equipment may seem large and complex but once the laboratory is established, the running costs are relatively low. It is sometimes not appreciated by the non-technical administrator that an analysis may require 10 or 20 individual items and that if even one is not available the analysis cannot be carried out. On the other hand, many items are common to different analyses so that, once the many hundreds of items required in a food control laboratory have been provided, there comes a point at which productivity can rise sharply and investment decrease. The logistical problems of maintenance, repair and replacement of equipment are also considerable. Adequate provision must be made for obtaining spares and replacement parts and for their storage. It is false economy if staff are being paid but cannot do an important part of their work due to a lack of relatively inexpensive equipment. Some of the instruments and equipment needed for chemical analysis by a modern food control laboratory are: (for purposes of this listing, 'instruments' are measuring devices and 'equipment' are processing devices. Apparatus made primarily of glass are not included).

Some of the instruments and equipment needed for chemical analysis by a modern food control laboratory are: (for purposes of this listing, 'instruments' are measuring devices and 'equipment' are processing devices. Apparatus made primarily of glass are not included).repeated.

Instruments:

1. Analytical Balance
2. pH meter
3. Spectrophotometer, UV-visible, double-beam
4. Spectrophotometer, atomic absorption
5. High Performance Liquid Chromatograph (with UV and differential refractive index detectors)
6. Gas Chromatograph (with flame ionization and electron capture detectors)

Equipment:

1. Blender
2. Grinder
3. Pulverizing hammer mill
4. Air oven, forced draft
5. Vacuum oven, with pump
6. Muffle furnace
7. Centrifuge
8. Refrigerator
9. Freezer
10. Heaters and hot plates
11. Steam and water baths
12. Water distillation still or deionizer

All of the above equipments and instruments are moveable, although the larger or more sensitive units are generally not moved, once placed. The major items of fixed equipment constructed in place are the fume hoods. The extensive use of solvents, ashing and noxious chemicals in food analysis, requires more fume hoods than other types of laboratory work. In fact, to experienced food analysts, there never seem to be enough hoods, even in a well-equipped laboratory. Fume hoods may be purchased pre-fabricated with outlets for services. The material of construction is most important, especially if the hood has to withstand acid fumes in general and perchloric acid in particular. The supplier must be given full details of the use to which the fume hood will be put. Hoods can be constructed out of local materials such as wood, preferably hard woods, coated with epoxy resins. Such should never be used for acid digestions, but only for solvent extraction work.

3.4.3 Utilities:

Electricity must either be a stable supply, or the voltage must be stabilized by either one large stabilizer for the whole laboratory, or by a unit for each of the instruments requiring it. The lab should have sufficient number of electrical sockets. There must be several cold water taps per bench to allow for rinsing, condensers, etc., but hot water can be restricted to those sinks where apparatus is washed. In a larger laboratory a distribution system for distilled or deionized water would be advantageous. Fume hoods should have adequate provision for water taps, compressed air valves, electrical sockets etc.

Special methods, such as trace analysis, usually require distillation from glass apparatus of water initially partially purified by distillation or deionization. The initial purification produces water very low in salts, but if the original supply contains organic matter this may not be removed, and traces of resin material may be present. A steady supply of compressed air is required for an atomic absorption spectrophotometer (AAS) and is very useful to have available at the bench. A compressor is suitable for use with the instrument but if used for other purposes at the same time it must be capable of supplying those needs without affecting the AAS supply. Apart from the inaccuracy that will result from a change in the flame characteristics, sudden failure of the air may result in a flashback, which is expensive if the mixing chamber is destroyed, and could be dangerous. However, it must be emphasized that manufacturers design this part of the instrument to be as safe as possible under flashback conditions. Therefore, in many ways it is probably better to have a separate air supply to the AAS. This instrument also has to be provided with a ventilation hood to remove gases formed during operation, particularly if nitrous oxide is used as the fumes are very toxic.

Utility services require a large space but need to be concealed for aesthetic reasons, yet require an easy access for repair purposes. To satisfy these conflicting demands, the main runs may be in voids above false ceilings and in floor ducts. Secondary services are then run to outlet points on benches taken from floor level along the wall behind benches in voids especially incorporated in the design of the bench fittings. Frequent access points are provided for maintenance purposes.

Drain pipes should be of high density polythene or copolymer polypropylene with screwed joints. These show good resistance to most organic and inorganic chemicals. The drainage lines may be embedded in the flooring. As it is not acceptable to discharge laboratory wastes directly into the sewerage system, all waste from laboratory sinks and other waste fittings should be led first into dilution pots (about 5 litre capacity) before being released into the main sewers. Buildings can be designed to include a large dilution tank where all laboratory sink waste is directed before entering the sewerage system.

4. ENVIRONMENT CONDITIONS, SAFETY AND RELATED REQUIREMENTS:

4.1 Environmental Control:

Adequate control of temperature, humidity and dust is important to staff comfort, instrumental performance and safe working (e.g. with flammable solvents). If they are to perform properly optical instruments often require stable temperature conditions. Electronic equipment may have prescribed operating ranges for environmental temperature and humidity. Computers may need to be protected from strong magnetic fields from other equipment; any staff or visitors with heart pace-makers must avoid such fields. Cooling water, either from mains supplies or localised refrigeration may be necessary for the proper functioning of some equipment. Test materials, reagents, standards may need to be stored under controlled conditions. Some substances are affected by sunlight or fluorescent lights and must be protected from it. Delicate balances and optical instruments may need to be protected from vibration (e.g. from blenders, shakers and centrifuges) or may even need stabilised supports. All these needs have to be identified and documented so that proper procedures for monitoring them and taking necessary action can be included in the quality assurance system.

Records will be needed which show that: samples are received, stored, handled and analysed under environmental conditions that will not adversely affect analyses; temperature, humidity and light controls are adequate in sensitive areas to protect samples, extracts from them, personnel and equipment; the results of environmental sampling in laboratory areas are recorded; these should include records of air-flow rates across fume cupboard apertures.

4.2 Housekeeping Control:

As with any other aspect of the laboratory's activities, the responsibility for housekeeping activities must be clearly defined. Cleaning staff and laboratory staff must each have clear instructions as to their respective duties in relation to:

1. cleaning of floors, vertical surfaces (e.g. cupboards, walls, windows and doors),
2. horizontal surfaces (e.g. work surfaces, shelves), equipment, interiors of refrigerators, freezers, fume cupboards, controlled environment stores
3. control of the contents of refrigerators, freezers, fume cupboards, controlled environment stores

4. checking the performance of air-conditioning and dust extraction equipment and fume-cupboards
5. pest control

The quality assurance programme will include work schedules, records of observations and of action required/taken covering housekeeping activities of this nature.

4.3 Safety Features:

The building and laboratory design should include a number of safety features including:

1. The fire areas of corridors should be formed of concrete blocks.
2. Services should include a shower sprinkler system near each doorway so that a worker can take an immediate shower, clothes and all, in the case of accidental general contact with corrosive or poisonous liquids or fire.
3. There should be built-in eye wash fountains or at least portable eyewash stations (obtainable from most chemical supply firms).
4. The traffic flow, the egress pattern and the proportions of the laboratory are all safety considerations. It must always be possible to leave the laboratory safely irrespective of the initial site of a fire. Serious thought must be given to the number and location of fire extinguishers and stand pipe systems, and to the availability of sprinkler systems.
5. Laboratories should be well-lit so that the operator does not have to peer too closely over potentially hazardous material in order to see what he is doing. There should be ample working space and bench tops and other surfaces should be kept clear of all material except that in current use.
6. Benches are best without shelves, only services, these being operated from the front so that the operator does not have to stretch across the bench. It is still common to see reagents on shelving at the back of benches (or above the centre of double-width benches) but it is probably safer if such reagents can be kept on side - shelve or in trays which are brought to the bench as required.
7. Flooring needs to be of a non – slip material, resistant to acids and solvents, but not so hard as to be tiring to stand on for a few hours at a time. No material is entirely satisfactory. Well-laid linoleum and a filled epoxy resin on top of concrete are among the best available. It is advisable not to polish laboratory floors.
8. Pollutants generated within the laboratory must be removed safely, quickly and efficiently. In particular, toxic or noxious gases must be removed expeditiously through a duct system that does not exhaust near the building air conditioning intake.
9. The building must be planned for security. Restriction of access is of considerable importance because of the extremely valuable and sensitive equipment used in the laboratory work as well as to protect the integrity of official samples.

10. It is very advisable to have an efficient fire and smoke detection system with appropriate alarms. Common fire detection equipment is usually either rate-of-temperature-rise or fixed-temperature detector using a substance of known melting point. There are advantages (and disadvantages) to each type of detector and the laboratory Head should select the one he feels best fits his laboratory.

Designing a laboratory to afford protection against every kind of hazard should be aimed at, but, the level of safety for the most general applications and to provide supplementary systems in areas of higher hazard has to be achieved.

A safe solvent storage area is ideally separate from the laboratory building in a stand-alone structure. It can be a small building of one room and some possible design features are: (reasons are given in parenthesis)

1. Construction of cement blocks or bricks. (Only non-flammable materials surround the solvents.)
2. For a stand-alone building, double walls with insulation between. The exterior wall can be material other than block or brick. (Provides insulation from the sun and makes air conditioning more effective.)
3. An epoxy film to cover the entire floor plus 10 cm up the base of the walls. (Any solvent spillage will pool and evaporate, rather than soak through the floors or walls.)
4. A copper pipe (about 25 mm) inside the room, which goes through the floor and is embedded about 2 m in earth. (A ground pipe to bleed off any static electricity charges - which often build up when solvents are poured).All metal objects in the room are to be attached to the pipe using heavy guage single strand copper wire. Also, attach a short wire with an alligator clip. (This grounds all metal. The clip is used to ground any metal cans used for solvent transfer.)
5. Storage shelves of metal and connected by wire to each other and the grounding pipe.
6. Air conditioning is external, with the entrance duct at the top of one corner of the room and the exit duct at the base of the opposite corner.(The room must be cooled as many solvents will boil at hot outside temperatures. The air entrance on top and exit on the bottom diagonally across the room, will cool the room and will also serve to sweep and remove any solvent fumes on the floor - solvent fumes are generally heavier than air and will pool on the floor.)
7. The door is of metal and fire-rated for at least one hour, with a positive closure. It must seal well when closed. The door sill is at least 10cm high. (Fire doors are metal sheathed around cement. The closure, the sea land the high sill all act to prevent escape of solvent, either floor spillage or fumes.)
8. Air conditioner exits duct with a fire baffle (to prevent flashback) and ducted to exit in the outside air at building roof height. (Fumeshave a better chance of being carried away by breezes and someone smoking nearby will not present a fire risk.)

9. An extinguisher system, which should be carbon dioxide or Freon type and not water sprinklers.

5.0 PERSONNEL RELATED REQUIREMENTS:

1. The personnel should be technically competent to perform their duties as allotted to them whether operating on specific equipments/ performing tests /evaluating results/signing the reports.

2. Qualification for doing specific tasks shall be judged on the basis of their education, training, specific experience and demonstrated skill.

3. Regular and refresher training should be organized to keep the personnel update in their domain of activity.

4. Specific job description for each personnel should be defined with their role and responsibility.

5. Personnel should wear proper uniform and protective clothing's, etc as required depending upon the test method.

6. While doing test no phone calls/ cell calls should be attended to avoid any type hazards and carelessness while performing the test.

7. Normally blank determination along with the known-standards must be carried out in duplicate/ replicate to check the accuracy of the results obtained and human error etc.

8. All the analysis records must be documented either through hardcopy or through soft copy to demonstrate that the tests are really been carried out.

9. Random checking of the result should be done inter-laboratory and intra-laboratory to check the proficiency of the personnel.

10. In case of hazardous analysis, special precautions as provided in the methods should be taken for self and surroundings.

11. While opening and closing the laboratory room, safety precaution should be taken care of depending upon the nature of the laboratory, equipment and test method. Special care should be taken for microbiological lab. Instructions in this regard must be displayed in the lab.

12. In case of contractual appointment, technical competency of the personnel should be judged and they should be put on job only after they are trained and their competency in the respective field is established.

13. Alternative arrangement of personnel should exist in case one is not available but not at the cost of their technical competency.

14. Personnel should be medically fit depending upon the test method he is deployed to avoid any hazards.

15. Special precaution should be taken by the personnel during break time to ensure that tests are carried out as per prescribed method and no relaxation is given in the test method.

16. Calculation should be rechecked on random basis by the supervisor.

17. Daily wages should not be put to job.

18. The personnel at the time of working in the laboratory should be alert and concentrate on their work only.

19. Supervisory officer should randomly watch the analysis activity and guide from time to time to increase the competency of analyst.

20. Eating habits should be avoided in the laboratory.

21. First Aid box should be available in the lab. along with emergency Telephone no. of hospital/doctors/contact person.

22. During odd times person should avoid working lonely.

23. Fuming chamber must be used for test requiring ash, protein determination, evaporation of solvents etc.

24. While pouring down acids etc in the basin, water taps should be kept on slowly.

25. Electrical equipments should be handled with great care.

26. Poisonous and hazardous chemicals must be kept under safe custody.

27. Manual sucking from mouth of liquid should be done with bulb type pipette.

28. Competency of the personnel should be judged regularly by giving unknown samples.

29. No external or internal pressure should be put on analyst.

30. Output should not be linked with quantum of work. More emphasis should be on quality output or results.

6.0 TEST METHODS:

1. The laboratory shall use only official methods depending on the requirement of the test, its sensitivity and nature of the commodity which is being tested and quality/safety factors to be determined.

2. In case of non-official method, validation of the methods as per set norms is a must and their range of detection/quantification, L.O.D./L.O.Q. limitations etc. must be established.

3. Selection of method is very important depending upon the requirement of the test and customer requirement.

4. Estimation of uncertainty of measurement should be available for each method in context of the food commodity and test to be done.

5. External calibration of the equipment is a must annually or depending upon its use. However in case of any equipment being used very frequently, internal calibration facility should be available and done regularly with a record thereof.

6. Glass apparatus should be calibrated.

7. In case of standard chemicals required in testing, whose purity can alter the result should be certified reference material with proper traceability.

8. In case of recovery and PPM level extraction from a food commodity, percentage recovery must be established for each food and the contaminant/constituent which have to be determined and the calculation should take care of such recovery.

9. Sometimes official methods do not prescribe the interfering material in the test method, limitations ,its sensitivity, range of detection and qualification, capability of the equipments being used, due to change of the sophisticated equipment as prescribed in the method for a particular model/ technology. Hence it is necessary to establish the suitability of such methods for their

particular test and equipment, etc before giving the results. Obviously the method needs to be validated internally for its particular use using particular equipment.

10. Standard solution/CRM Solution should be stored at required temperature and condition and its strength should be checked regularly and record thereof should be maintained.

11. Calculation should be done and rounded off while reporting the results to the required level of standard.

12. SOP as far as possible should be available for test method along with the protocol.

13. Method should be available while performing any test to follow exactly the test method prescribed. No short cuts should be followed and tests should not be done on a memory basis alone.

14. Purity of the solvents, water being used and other chemicals should be checked regularly and a record thereof should be maintained.

15. In case of any controversy or marginal results, only reference methods should be used.

16. In case of micro biological analysis standard culture must be available to establish the confirmation of the microbes. SWAB testing must be done for inoculation room and media preparation room regularly to ensure that it is not contaminated.

17. The results should be recorded commensurating with the calibration of the glass apparatus etc e.g. in case of a burette, the result should be reported only to the displayed capabilities of the burette.

18. Special precaution should be taken for pipetting and ejecting the solution from the pipette. The solution should not be blown by air through mouth.

19. All the apparatuses specially glass should be contamination free and should be cleaned and rinsed thoroughly before use. No chemicals should be used after its expiry or otherwise if it looks like deteriorated or decomposed.

7.0 EQUIPMENTS:

1. All the equipments being used should be under permanent control of the laboratory and should be capable of in context of the test method.

2. The equipment must be calibrated depending upon the requirements by an outside accredited lab and/or internally as the case may be.

3. In case the sophisticated instruments are shifted from one place to another the same should be re-calibrated.

4. Depending upon the uses, the equipments should be internally calibrated either daily or at a periodically interval as the case may be.

5. Instruction manual, operation manual and other details of the equipments like calibration, due date of calibration, safety precaution, etc must be available at the side of the equipment.

6. Each equipment should be uniquely identifiable.

7. The equipment should be placed and test must be performed under a proper environmental condition as prescribed. Normally the room should be dust-free, air conditioned with controlled humidity. Special condition needs to be followed in case of equipment being used in case of micro biological analysis like Air handling unit, etc.

8. Each sophisticated equipment should have IQ, OQ and PQ Certificate from the manufacturer.

9. LOD/LOQ/ Range of detection/ range of quantification must be established for each equipment in context of the test method, nature of the food commodity, constituent to be determined. The reason being that normally in official methods, the model of the equipment being used along with its accessories becomes old whereas due to technological advancement a model of the equipments are upgraded along with accessories and software, hence the LOD, LOQ, etc must be established and should be checked as claimed by the manufacturer which may not commensurate with the limits given in the official methods. SOP must be available for operation.

10. Equipments not working should be placed under a tag " out of order"

11. Software being used in the equipment must be validated and a record thereof should be available.

12. Maintenance plan of the equipment should be available and should be done under annual maintenance contract.

13. Equipments should not be subjected to overloading or mishandling which could give erroneous results.

14. In case the equipment send outside the laboratory for repair, etc. proper procedure of packing and transportation as prescribed by the manufacturer should be followed.

15. Intermediate checks of the equipments must be done through known and certified standards regularly. The equipment should be handled by technically competent and trained personnel only. Such personnel should be trained on routine maintenance and minor repair of the equipments.

16. Proper procedure as prescribed by the manufacturer should be followed for cleaning of the equipments and its accessories before and after use.

17. The SOP for safe handling, transportation, storage, use and plant maintenance of the equipments must be available to ensure proper functioning and to prevent deterioration /contamination.

18. Do and don'ts regarding important instruction should be available along with side of the equipments and should be visible all the time.

19. Due care should be taken to ensure constant voltage supply of electricity as required for the equipment to avoid fluctuation and thus variation in results.

20. After return of the equipment from repair, the same procedure should be followed as that for new equipment to ensure that the results rendered by the equipments are as per capability of the equipment. In such cases the instruments needs to be recalibrated before put to use.

21. Equipments where gases are being used, the purity of the gas should be as per requirement of the equipment/test method.

22. Gas cylinders should be put outside the laboratory room at a well secured and approachable place.

23. Temperature and humidity of the room where the equipments are placed must be recorded daily. In case of micro biological laboratory, special precaution should be taken as per requirement of the test method for environmental conditions especially in case of isolation and determination of pathogens.

24. In case of a mobile food testing laboratory a separate SOP should be available and the equipments used in such laboratory should be technologically sturdy to avoid variation in results. Calibration of such equipments needs to be done very frequently preferable daily before being put to use.

25. Software being used in the equipment should be capable of achieving the accuracy required and should be complied with the specification related to the test method.

26. Software should be upgraded and validated from time to time.

27. Obsolete equipments giving erroneous results in context of the requirement of the test method should not be put to use.

28. The equipment should be placed on a vibration free platform.

29. Daily cleaning of the equipment should be done by trained personnel as per SOP

30. Proper safety precautions should be taken for equipments running round the clock in the absence of the personnel.

8.0 CERTIFIED REFERENCE MATERIALS / STANDARDS AND REFERENCE CULTURES:

Testing, validation/calibration, standardization & reference materials are inter-related due to dependent on each other. Without proper reference materials, it is not possible to make up any idealized and reliable measurement system. As per the lab quality assurance procedure reference materials are required for all types of testing and validation/calibration. These are widely used for validation/calibration of an apparatus and testing procedure, assessing the true value.

The reference materials are generally used for, to develop and validate accurate method of analysis ensuring traceable measurement results at the working level, to calibrate measurement system and to demonstrate the accuracy of results, assure the long term adequacy and integrity of measurement quality assurance programme and monitor the lab performance, use in inter laboratory comparison and proficiency testing programme.

The laboratory shall ensure to maintain the reference standards, which are certified by the competent body having traceability to a national/international system like NIST etc.

The certificate provided by the supplier/manufacturer shall be maintained in the laboratory for records.

The reference standards having high purity, critical characteristics and require to store in special condition and hence its, to be stored in appropriate special condition as per the requirements. The substances are to be kept in sealed vial and shall be stored in dry place, away from heat, sunlight & moisture.

The reference material of various parameters such as metals, pesticides, antibiotics, volumetric standards etc. may be received from standard brand like Sigma, Aldrich, Fluka, Riedel-de-Haen, Dr. Ehrenstrofer Gmbh, Merck, Supelco etc. in regular intervals accompanying with certificates with proper label. The certificates shall include the name of the standard, the purity, uncertainty at a stated level of confidence, expiry/ validity/ shelf life, QC release, chemical formula / structure, assay/potency level of confidence / chromatogram, storage condition etc. The same shall be verified for the label, certificate & condition during receiving of the standards.

The reference standard solutions are required for sample analysis, quantification and QC checks. The laboratory shall be prepared the standard solution as needed like stock / primary, intermediate & working solution and wherever applicable the purity shall be considered during preparation. The standard solutions shall be kept in screw capped glass vials, standard volumetric flasks/stoppered conical flask (transparent/amber coloured) in air-conditioned room / refrigerate /deep freezer depending upon storage condition & requirements.

The standards shall be prepared from bulk reference standard materials received from the market as A grade material. The selection criteria for the bulk material intended to accept as working standard in assay and purity of substances. For accepting the material to be taken as working standard the molecule must be subjected to chemical characterization. First the standard stock solution to be prepared from which different working standard is made. The preparation of standards is generally carried out in regular interval as per the requirement / laboratory protocol and the records of those are to be maintained and labelled with concentration & date of preparation.

The preparation of working standard is generally carried out during analysis/ whenever necessary and records of these are to be maintained.

The intermediate checks of the standards shall be checked in regular interval to ensure the performance, stability & integrity of the standards and records of those are maintained with Quality Control Chart / Levey-Jennings Chart etc.

The shelf life / expiration date declared by the reference standards providing organization is generally applied to unopened condition that have to store at

recommended temperature. Hence it is the responsibility of the laboratory to maintain the critical characterization, performance, stability & integrity of the standards through proper handling, storage etc & same shall be ensured by the intermediate checks in regular interval / as per the laboratory protocol.

For some reference standards the shelf life / expiration date may not declared by the reference standards providing organization, in those cases the following shelf life may be considered when the standards are stored un opened at recommended temperature

1. Room temperature items, which are not temperature sensitive and usually are stabled for five years from the date of receipt.
2. Refrigerated items usually are stabled for two years from the date of receipt.
3. Freezer items usually are stabled for one year from the date of receipt

However it is the primary responsibilities of the laboratory to ensure the performance, stability & integrity of the standards through intermediate checks in regular interval / as per the laboratory protocol.

Reagent solution/standard solutions shall be prepared in established manner, for preparation of reagents the testing personnel refers to be relevant reference. After their preparation, those are to be stored in appropriate storage condition i.e. protected from light, tightly stoppered, refrigerated etc. Wherever, it is recommended reagents are to be prepared freshly. All the reagents/solutions bottles shall be properly labelled with name, date of preparation, concentration etc.

All reference standards shall be kept under responsible person to maintain proper storage, transport, security, integrity, mishandling etc and the relevant records are also to be maintained. The utmost care & protection shall be taken during handling & preparation of standards to avoid cross contamination & health hazard.

The reference culture/microbial pure cultures are used establishing acceptable performance of media, performance of the kits, validation of methods and assessing/evaluating the laboratory performance. The reference microbial strains are directly collected by laboratory from recognized national or international collection (ATCC, MTCC, NCIM etc) with traceability. Generally the reference strains are received in lyophilized stage or deep-frozen stage. If the reference strain has been thawed they shall not be refrozen.

The reference microbial stains are used for Quality control; internal quality control and performance of culture media in terms of productivity, selectivity, performance evaluation and interpretation of result. The reference cultures are received either on slant form or in lyophilized forms in vials.

On receipt the reference cultures, requires to revive in the laboratory. The active cultures shall be sub-cultured on to recommended medium and incubated at temperature specified. For lyophilized culture the outer surface of the vials is disinfected, wrapped with thick cotton wool and neck of the culture vials is broken. The contents transfer into 3 to 5 ml of recommended broth medium and mixed properly. The suspension is to streak on the recommended agar plate and incubates at specified temperature. Reference cultures to be checked for its purity, homogeneity, and typical morphology. Subsequently they have to check for characteristic reaction in selective medium and biochemical reactions. Whenever necessary, serological test as per analytical procedure is also to be carried out to check the pure culture.

Sub-culturing from original stock in regular intervals as working culture for routine use and records to be maintained. The intermediate checks on the purity and biochemical characterization also to be checked. All the working cultures are properly locate with name, date etc. & to be kept under proper storage condition.

All reference standards / pure culture stains are to be kept under responsible person to maintain proper storage, transport, security, integrity, mishandling etc and the relevant records are also to be maintained. The utmost care & protection shall be taken during handling of microbial pure cultures for to avoid cross contamination & health hazard. The laboratory has to maintain procedures / instruction for all.

[Sources of reference materials are at Annexure 8.1]

9. CALIBRATION AND PERFORMANCE ASSESSMENT RELATED REQUIREMENTS:

For accurate test results, lab shall be ensured that the equipments which are suitable for intended purpose and capable of providing valid results, such instruments would be regularly inspected, checked & calibrated accordingly. So laboratory should establish a schedule for the calibration and performance verification of equipments/instruments, which will be direct influence on the test results.

The calibrations to be done by in-house (internal)/external agencies/competent body having traceability to a national / international standard (NABL accredited lab) depending upon the type of equipment / instruments.

Laboratory management has to first segregate and classify the instrument, require external and internal calibration. The interval / frequency of calibration has to decide by the laboratory considering the equipment/instruments type, uses, experience and need base, previous performance of the equipment etc.

Regarding external calibration laboratory has to collect the information from the calibration agencies / laboratories to ascertain the facility / capability to fulfil the

laboratory requirements, status of accreditation, charges etc and based on the same the competent calibration agencies / laboratories can offer for the calibration job. The calibration for the instruments like balance etc is recommended to perform on site and others may be sent to workshop/workplace. The laboratory shall ensure calibration status / performance status of the instruments / equipments goes outside of the laboratory control after return / put into service.

It is the laboratory responsibility to verify / check the calibration certificate in terms of the lab requirements, traceability to the primary standard, ensure the capability / calibration range, uncertainty, due date of next calibration (if require) etc and laboratory has to evaluate the services. All records are to be maintained.

The sophisticated instruments such as GC / GC-MS, HPLC/ LC MS MS, AAS/ AES, ICP-MS/ OES, UV-VIS Spectrophotometer etc. are recommended to check the performance verification & operational qualification (OQPV) al least once in a year / depending upon criticality of the uses of the instruments through the service providers / OEM. It is the responsibility of the laboratory to verify / calibrate the instruments to ensure the performance in regular basis / before put into use / analysis by use reference standards etc.

The equipments like thermometer, pressure gauge, humidity meter, laboratory may calibrate through external calibration agencies with proper traceability in regular intervals / as per the lab protocol and the laboratory may use the same equipments as standards for verification.

Where the certain criteria e.g. temperature, humidity etc has a direct effect in the result of the test, the measuring device should be appropriate in quality to achieve the perfect accuracy and those devices should be calibrated (internally/externally) traceable to national/international standards.

In case of incubators, water baths, ovens, furnace etc. the stability of temperature, uniformity of temperature distribution and time required to achieve the equilibrium are to establish initially by experienced personnel. The documented monitoring system of operating temperature is also be maintained.

Other equipments/instruments like conductivity meter, pH meter, refractometer and other similar devices are to be verified in regular interval and prior to use with reference standard.

Laboratory also ensures that the performance of the lab autoclave is also capable to meet the specified time and temperature, pressure. Devices used for controlling/monitoring of operating cycles are verified as well as calibrated. Laboratory is also ensuring to maintain records of autoclave operation including temperature/pressure and time for every cycle. In addition to monitoring the effectiveness of the autoclave operation during its cycle also be checked by use of

chemical/biological indicator for monitoring sterilization/decontamination purpose when a load has been processed.

The weight and balance are also to be calibrated traceably at regular interval / as per the lab protocol. The performance of the weighing balance to be checked in regular interval / every time before use.

The volumetric equipment such as dispenser/diluters, pipettes, volumetric flasks etc. used in lab are to be checked to ensure the performance of the equipment. The equipments shall be checked for the accuracy of the delivered volume against the set volume and the precision of the repeat deliveries also be measured. Laboratory shall obtain the certified specific tolerance supplies from manufacturer/companies and calibrate through external agencies with traceability. The laboratory may also follow internal verification and intermediate checks on accuracy. The lab also ensures to provide a dedicated balance as reference to carry out the in-house calibration verification of glass wares etc.

The status of calibration (internal / external) of all the equipments/instruments including frequency of calibration, date of last calibration, due date of next calibration, plan and procedures shall be maintained by the laboratory

10. PURCHASE OF CONSUMABLES/ EQUIPMENTS :

The laboratory should be maintained a proper system on purchase service & supplies of all media, chemical, reagents & other requirements/appliance, consumables to avoid undesirable, unconfirmed supplies of them and also ensure there should not be any effect on the of test analysis / result.

Requirements like name of the chemicals, appliances, glassware's, consumables, brand name, quantity, Management, rate contract/ comparative quotation, quantity available in stock shall be well documented by the laboratory.

Purchase shall be made in a systematic manner through a proper purchase / laboratory protocol considering different aspect like quarter/half year/annual requirements, pattern of previous consuming, approximate cost etc. Regarding purchase of equipments / instruments laboratory shall be need based considering the laboratory requirements, indented for use, accuracy / sensitivity, sophistication / latest version, future plan / work load etc.

On arrival of all the purchase materials, the laboratory shall be received & verified with reference to the order placed and the relevant criteria like quantity, brand, code, certificate of analysis, date of expiry , guarantee / warranty, condition of the items on arrival etc. The necessary entries / documents shall be maintained.

All instructions related to the purchase like storage, handling, inventory, responsibility etc shall be maintained & documented by the laboratory and followed in a systematic way.

The items/ supplies will effect on test result and the laboratory shall be evaluated the same to ensure the quality. Records are also to be maintained.

It is the responsibility of the laboratory to verify the certificates, reports etc related to services opt by the laboratory from the calibration agencies, equipment / instrument manufacturer / service providers etc..

Laboratory shall evaluate the performance of the supplier & service providers in regular intervals and approved list supplier & service providers with all details shall be maintained.

The laboratory must be maintained procedures / instruction & documents / records for all.

11. SAMPLING & SAMPLE HANDLING:

Sampling for testing or analysis is a process of taking a representative portion from a material or product to test (e.g. by physical measurements, chemical analysis, microbiological examination), typically for the purposes of identification, quality control, or regulatory assessment. The sampling is a significant role in testing activities as it reflects the ultimate test results.

It is not mandatory that all the laboratories shall be involved in sampling activities. However the laboratory involves in sampling shall maintain at least the following

The laboratory policy & declaration on sampling.

The laboratory should have authorized personnel / sampler with adequate knowledge, training etc on sampling.

The laboratory shall maintain the sampling plan & procedure in respects of the products / materials that shall include selection, withdrawn & preparation of samples during sampling. The same shall be based on appropriate statistical method / regulatory guidelines / references.

Work instruction shall be maintained for the personnel involve in sampling activities.

The laboratory should have all facilities like tools, equipments / instruments etc requires for various sampling.

The laboratory shall maintain the relevant data & operation related to sampling, procedure use, location, date / time of sampling, identification of sampler, other specific requirements like environmental conditions, transportation, statistics the sampling procedures are based upon etc and documents shall be maintained.

All incoming samples shall receive through the receiving section maintained and supervised by laboratory responsible person. On receiving section the laboratory responsible personnel initially checked the relevant overall criteria like sample identity/labelling, mode of transportation, condition of the sample including packaging, sample quantity, verification of fees (whenever necessary), parameter to be tested etc against the customer declaration / requirements. Any abnormalities / deviation / doubts from the normal condition, suitability of the sample for tests etc , the same shall be clarified from the customer / laboratory responsible personnel before accepting the samples for registration / testing. In case microbiological test samples, the same shall be received in the sterilized container/sample box etc. The laboratory documentation system shall includes all relevant information such as customer details, date of receipt, condition of the sample on receipt, sample quantity, transportation , parameters to be tested ,observation/remark (if any) etc.

The laboratory shall maintain a system on traceability of all accepted samples and the same shall be maintained throughout the retention of the sample in the laboratory without any confusion.

The laboratory must have all infrastructures, facilities for storage and preserve the samples depending on the physical, chemical and microbiological properties to maintain the sample integrity, security, avoid loss / damage, deterioration etc. The laboratory must have proper documented system on retention & disposal of the tested / remnant / reference samples. The retained samples may also use for retesting and the internal quality assurance purpose. Wherever necessary the specific storage like deep freezer, refrigerator etc shall be provided and during storage the environmental conditions shall be maintained, monitored & documented.

The laboratory must have documented system, procedures, instructions & facilities for conditioning and preparation of sample according to the standard method or laboratory protocol to maintain the homogeneity, avoid loss / damage of the test sample.

The laboratory should ensure to maintain a proper documented system procedure for handling of test items including sample receiving, storage, transportation, retention / disposal, integrity, avoid and prevent loss/damage of the test samples.

11.1 General Principles:

The identity, homogeneity and integrity of the materials being handled by the laboratory must be ensured throughout the time they are under the control of the

laboratory e.g. from sample receipt to data report and authorized disposal of the surplus material.

The analytical data report must reflect the composition of the received material as a whole.

11.2 General:

Sampling is a defined procedure whereby a part of the substance material or product is taken to provide for testing or calibration of a representative sample of the whole. Sampling may also be required by the appropriate specification for which the substance material or product is to be tested or calibrated. In certain cases (e.g. forensic analysis) the sample may not be representative but is determined by availability.

The sampling procedure should describe the selection, sampling plan, withdrawal or preparation of sample from a substance, material or product to yield the required information. If the customer requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with appropriate sampling data and shall be included in all documents containing test and /or calibration results.

The laboratory shall have the procedure for recording relevant data and operations relating to sampling that forms part of the testing and calibrations that is undertaken. These records shall include the sampling procedure used, the Identification of the sampler, environmental conditions (if relevant) and diagrams and other equivalent means to identify the sampling location as necessary.

11.3 Samples may be conveniently classified under two broad divisions:

I. Formal samples – These are samples taken to determine if the food complies with national or local laws or regulations and
II. Informal Samples – These are samples taken for the purpose of monitoring or as part of survey work.

Formal follow-up samples can be taken if informal samples receive adverse laboratory reports. Formal or informal sample are also taken under others such as follow-up to a consumer complaint.

11.4 Sample Collection:

Work scheduling is greatly facilitated by arranging a sampling programme for routine monitoring with the inspectorate. As part of a general food control programme there is need to:

1. Regularly inspect foods at different stages in the manufacture and distribution chains, using planned surveillance programme.
2. carry out general surveys of the quality of the food supply through random sampling and analysis.
3. monitor certain specific problem areas with regards to food safety – specific potential risks, e.g. level of metallic contaminants pesticide residues, mycotoxins etc.
4. inspect foods for export, for certification of quality (if needed)
5. inspect food import. This is best done on all imported consignments by formal sampling carried out systematically in a manner representative of the lot.
6. formal sampling should also be done on locally produced food products based on the food inspector's observations or because a random or investigatory samples under the regular programme was unsatisfactory or the product is one that requires thorough surveillance. Analysis of formal or informal samples is also necessary in an emergency such as an outbreak of food poisoning.

11.5 Quantity of Samples to be collected, product wise is shown at Annex 11.1

(Ref. Annex II of FSSAI Manual on General Guidelines on Sampling)

11.6 Sample Receipt and Assignment:

Receipt and identification of a sample have to be clear and unambiguous for the quality assurance to be maintained. The laboratory register of test materials should be of a type where papers are numbered and cannot be removed. Entries on computer-based registers must be protected against deletion and /or alteration. A back-up copy must be produced and stored separately from the original.

When a sample is received for analysis, there must be a system to track the sample throughout its initial stage, analysis and later reserve storage. This is usually embodied in a record-keeping system, which is keyed to a unique identification number assigned to the sample at the time of sampling. This number can be sequential (i.e., 00001 to 9999) or can be devised to give information (i.e., 024-95-07) the 24th sample taken in 1995 under sampling programme No7) The record must show the movement of a sample, its receipt, assignment to a laboratory person for analysis, return to the sample and eventual dispersal. One of the administrative staff should be given this record – keeping function and closely supervised by the laboratory Head. It is preferable to use a card system rather than a logbook as cards are more flexibly handled. There are certain items of information, which should be on each card:

1. Sample number

2. Product name
3. Date Sampled
4. Date received at the laboratory
5. Type of sample (Survey, Complaints etc.)
6. Method of storage (dry, refrigeration, freezing etc.)
7. Storage location (coded for easy finding)
8. Date assigned for analysis
9. To whom assigned (the analyst should initial to show receipt)
10. Date retuned (from analysis)
11. From whom returned (maybe different from the original analyst)
12. Reserve storage method and location
13. Final disposal of samples, method and date.

Example of a typical sample record is given at Annex 11.2 (ref. Appendix 5.1 of FAO 14/14)

11.7 Control and Storage:

The storage of test materials is of major importance if the analytical data produced is to reflect and be traceable to the original sample. Deterioration of test materials invalidates any results. Therefore; test materials must be stored so as to ensure their integrity, safety, legality and stability. The laboratory must guard against deterioration, contamination and loss of identity. Special care will be needed where trace analysis is involved in order to ensure that extraneous materials do not contaminate the test materials and equipment.

There are three basic forms of storage - room temperature (dry room), refrigeration and freezing. The QA programme should specify the conditions to be used (Annex 11.3 - reference Appendix 5.3 of FAO 14/14). There are also problems associated with the type of container in which food can be stored. Foods that contain fats and oils should not be stored in copper or metallic vessels and foods that easily desiccate such as fruits need to be stored in ways, which avoid loss of water.

Perishable, unfrozen food must be maintained at 0C – 4C and frozen food kept preferably at -18 C or below. All perishable items should be examined within 36 hrs. Of collection .Perishable samples that have been examined within 36 hrs after being sampled should be frozen, canned. Dry non-perishable foods maybe stored at room temperature before analysis.

The test material could also be dried and stored pending analysis, if analysis will not be affected by drying.

Special conditions apply to test materials, which are to undergo microbiological examination as well as chemical analysis. If a test material is to be stored frozen and a number of separate analyses are to be performed, it is preferable to sub-sample before freezing.

All test materials when stored must be properly and indelibly labelled so that identification is not lost .The most effective method of labelling maybe to place the label in its own plastic bag, inside the test material container, but separated from the food by a suitable layer.

The sample is then stored until it can be matched with a suitable test note containing all the above information and any other relevant information required for analysis and interpretation of the results. The test note should preferably be of the type that incorporates enough space for the test results and observations. The sample and the test note should (when matched if they arrive at different times) be clearly and indelibly marked with a registration number and passed to the analyst. From this point onwards, the analyst will identify everything pertaining to that sample with the laboratory number.

11.8 The Analytical Sample (Test Portion):

Before removing the test portion (s) for analysis, the analyst must be certain that all records are in order, integrity has been maintained containers are intact and sealed (if any) ,unbroken.

Any ambiguity in the analytical requirement must be resolved, e.g. with canned pickle in oil, is the analysis to be done on the pickle, oil or the whole contents of the can.

For analysis, the analyst first removes a test portion. If the test material comprises more than one item (fruit, vegetable etc.) the test portion should contain material from each item – usually achieved by comminuting a number of items and removing a portion. After the test portion has been removed, the remaining test material is returned to the storage.

11.9: Referral of the Test Material:

On occasions it may be necessary to pass a test material to another laboratory for some specialized analysis or because of some analytical facility not being available with the laboratory or because of overload of work. Unless the other laboratory is a part of the same QA programme or the two laboratories are accredited by the same (or equivalent schemes), this referral would mean that the test portion sent for that analysis ceases to

be quality assured by the parent laboratory. This should be made clear in the analysis report to the customer.

11.10 Test Material Disposal:

Sample disposal is relatively a simple matter. The only problem arrives when there is a hazard involved in the destruction or the sample remains must have specific treatment e.g. a sample of groundnut heavily contaminated with alfatoxin. Any residual material if valuable such as flavouring concentrate maybe required to be returned to the originator. The register should therefore have a column in it for details of when, how and where the test material was disposed.

11.11 Documentation for QA Programme:

1. Register for sample receipt: Test material identification
2. Flow chart of the sample submitted for laboratory examination
3. Storage conditions for food test materials

12.0 QUALITY ASSURANCE MEASURES:

12.1 General Principles:

The QA programme for a laboratory covers all the policies and activities, which can affect the quality of its output.

12.2 Definition and Scope:

A QA programme maybe defined as a mechanism used to ensure that the data are fully reliable, suitable for the intended purpose, presented on time and at an acceptable cost. It is a formal, planned activity whose purpose is to provide assurance that the quality control programme is actually affected and is designed to fit the needs of the laboratory.

The scope of the quality assurance system has to be developed in such a way that there is confidence that whenever data are reported

1. the identity and integrity has not been compromised
2. the analysis has been conducted by member of the staff who is competent for the task
3. the equipments and the methods are appropriate and are working properly
4. the laboratory can demonstrate its current capability to produce valid data.

The format adopted in meeting these requirements may vary from laboratory to laboratory.

Each laboratory (or group of laboratories) meets different requirements, operates within a different organizational environment, experiences different constraints and should have a QA programme, which takes account of these factors.

There are certain factors, which are common

1. The use of validated methods
2. The use of standard operating procedure (SOP) in the laboratory
3. Calibration and traceability of measurement (including use of certified reference material)
4. External assessment of performance

While facilities exist for accreditation of laboratories for particular types of work, it is usual to find requirements for these features within this scope of accreditation process along with the requirements for topics which may include organization and management, laboratory accommodation and environment, equipment maintenance, handling of test materials, test methods and quality control procedures (and method performance characteristics), staff training and performance, security, records and reports, sub-contracting of work , outside support services , handling of complaints , quality audit and system review.

12.3 Preparation:

QA Programme is concerned with everything that goes on in the laboratory, which may affect “Quality”. Each member of the staff involved in the QA programme must be

1. clear about what they are expected to do
2. know how to do it and
3. be able to show that they had done it properly

Documentation is a major feature of QA programme. Formulation of a QA programme should contain three essential components

a) Prevention, which requires an orderly programme of planning and positive actions before or during analysis to ensure that all analytical systems are performing appropriately, easy calibration and maintenance of instruments use of reference materials and training.

b) Assessment, a form of control that includes periodic checks on the analyst performance e.g. analysis of check samples and validation of methodology.

c) Correction, an action taken to determine cause (s) of quality defects and to re-establish proper functioning of analytical operations e.g. trouble

shooting to correct malfunctioning equipment, re-evaluation of methodology and re-training.

12.4 External Proficiency Testing:

Proficiency testing is the part of QA programme, which looks at the accuracy (correctness') of the results actually being reported by the laboratory on real test materials. An independent external assessment of the correctness of the typical result provides an impartial test of analytical quality: this is done by proficiency testing scheme, i.e. methods of checking laboratory testing performance by means of inter laboratory tests. This includes comparison of a laboratory's results at intervals with those of other laboratories. Procedures used when analyzing test materials for such a scheme are those normally used by the laboratory.

The proficiency testing is likely to grow in importance because of regional and international developments which require laboratory data to be mutually acceptable between nations for many regulatory purposes .A harmonized protocol for the proficiency testing of the chemical laboratories has been prepared by joint ISO /AOAC International/IUPAC Working Group (November 1992, ISO /REMCO, N263). This identifies and explains the major features recommended for proficiency testing scheme. The major features of the protocol are listed in Annex 12.3 (Ref. Appendix 10.1 of FAO Manual14/14)

12.5 Internal Quality Control Checks:

Blank Analysis:

Blanks are to included in analytical methods .A blank is characterized as a sample included in the analytical processes, which has all the properties of the actual sample except that it does not contain the substance of interests.

Duplicate Analysis:

Duplicate sample analysis is the analysis of the same sample twice in order to determine the precision of the analysis.

Spike Analysis:

A sample is split into two sub samples in the laboratory. One is analyzed according to the specified procedure. The other is treated by adding a known amount and concentration of the indicator being measured, running this specified procedures. This should increase the concentration in the spiked sample relative to the unsp sample, by a predictable amount. Usually 10 percent of the sample are split and spiked. They are used to test the accuracy of the laboratory method.

12.6 External Performance Evaluation:

In order to verify that a laboratory possesses the capability to provide accurate and reliable test data in its day to day operations and to maintain high standards of performance, a competent, disinterested third party is necessary to evaluate laboratories based on personnel, physical facility, instrumentation and quality assurance / quality control programme, and the laboratory's performance. For this purpose, an organization should participate in inter laboratory comparisons of the proficiency testing programs.

12.7 QA Manual:

Each laboratory with a QA programme should have a manual that documents the operations of the laboratory. A typical manual might consist of the following:

a. Title page with signatures of all approving officials and date of issue (Annex 12.1)-Ref: Appendix 2.2 of FAO manual 14/14
b. Table of contents
c. Organizational structure and exactly where the laboratory fits into this structure
d. Objective of the quality assurance programme
e. Essential elements of the QA Programme (Annex 12.2)-Ref Appendix 2.1 of FAO manual 14/14.
f. Documentation forms
g. Performance and frequency of Audit
h. Corrective and follow-up action

A Statement of the QA Policy both general and specific is needed in the QA Manual; the objective of the laboratory should be clearly defined. The principal objective of the laboratory, for example, is to produce reliable results

12.8 Implementation:

Actual implementation of the QA programme is a co-operative effort of the management, and member of QA unit, section leaders and analyst. Management decides the amount of resources to be allocated to the QA Programme. This decision determines the nature and the size of the QA unit. In formulating the QA Programme, this unit receives technical input from the analyst. Once formulated by the QA Unit and approved by the management the QA programme is ready for introduction.

Analysts are responsible for day-to-day maintenance of the programme. The QA Unit periodically monitors this adherence and makes its report and recommendations to the management, which then decides on the action to be taken so as to achieve compliance with the programme.

12.9 Revision of the QA Manual:

The QA Manual must be designed so that the change is easily accommodated. It is essential that organizational pattern emerge, workload shifts and methodology develops. The QA Manual can react rapidly to these changes in the work of the laboratory.

12.10 Documentation Required or the QA Programme:

- Laboratory elements to be considered in the QA Manual
- QA Manual cover and contents pages (Annexe 12.1)
- A statement of QA Policy

13.0 LABORATORY RECORDS:

13.1 General Principles:

All the information that has any particular relevance to the materials and the analysis performed on them must be documented in a systematic fashion at any point in its passage though the laboratory. Records must allow a test material to be traced back to its arrival and any information that arrive with it.

Records should be such that if the need for reanalysis arises, it could be done under the same conditions and in the same way as before. Records must be retained and protected from misuse, loss or deterioration for an agreed time.

13.2 Sample Collection Records:

Responsibility for sample collection generally falls outside the food-control laboratory; hence it is not covered here.

13.3 The Analysts Worksheets:

The analyst worksheet provides a written account of the laboratory analytical results. Certain requirements apply to all worksheets.

1. All the basic information must be recorded directly on the worksheet before analysis has begun.
2. As soon as the worksheet is obtained it should be initialled.
3. All entries should be clearly legible and made in permanent ink.

4. No entries should be erased or over -written if an incorrect entry is made. The analyst should draw a line through the incorrect entry; write above it the correct figure or word and then date and initial the corrected entry.
5. Data should not be discarded without explanation.
6. The exact analytical method should be referenced clearly and completely. If the method has not been published or is not covered by SOP it should be written in full on the worksheet or as an attachment to the worksheet.
7. If the analysis has been made in duplicate or triplicate etc. the result of each analysis as well as the summary of all result must be recorded.
8. If more than one analyst is involved in analysis the worksheet must indicate which analyst broke this seal and which analyst performed each segment of the analysis.
9. Any continuation sheets that accompany the analyst worksheets should be numbered in a consecutive series e.g. 1 of 8, 2of 8, 8 of 8 pages. An example of ex analyst worksheet is shown at Annex 13.1(Ref: Appendix 11.1 of FAO manual 14/14)
10. Worksheets are check for accuracy, completeness and compatibility with other documents by the supervisor or the designated representative.
11. The date on which the analyst submitted the worksheet to the supervisor is indicated.
12. The exact method used is referenced. Any modification to the referenced method is stated and the reason for the modification is given.
13. All calculations are clearly shown with the proper number of significant figures used.
14. The use of controls and their results are specified.

13.4 Analyst relies increasingly on instruments, which produce a hard copy record of the instrumental readings. Taking chromatographic charts as an example, this must be clearly labelled (test material number, analyst, date and any other necessary identifiers) and stored in a logical sequence. Chromatograms of Standards, recoveries and sample extracts must be cross- referenced to each other and to the responsible analyst laboratory notebook to allow for easy checking of results. The full chromatographic conditions employed must be stated on the chromatogram or must be readily obtainable from the analyst's notebook.

14 LABORATORY REPORTS:

14.1 General :

The laboratory report is a condensed version of the data appearing in worksheets and laboratory notebooks. It must contain all the information normally necessary for the customer to utilize the result it contains

14.2 The **format of laboratory report** typically include the following

- Name, Address of the Laboratory
- Name, Address of the customer
- Certificate/Report Number
- Page Identification (Page X of Y)
- Sample received details (Dates, Names of deliverable, receiver)
- Unambiguous identification of sample / test material (Description, Laboratory Number etc.)
- Analysis conducted, Methods, Procedures any deviation from standard practices.
- Preparation of test material, taking of test portions
- Results
- Uncertainty of measurements
- Comments on significant of findings (if expected by the customer)
- Date of report
- Authorizing signature

14.3 Retention of Laboratory Record:

The sequence of records should form a continuity of documentation to produce a clear, accurate and in-disputable history of the test material with all aspects of documentation in agreement.

All sample registers worksheets, reports and associated documents must be retained for a period, which is determined by the management in consultation with the customers and is documented.

Storage of such material should follow the normal rules of archiving in terms of indexation, traceability, security, appropriate levels of protection against fraud and tampering, from fire, flood etc. Backup copies must be held of any records stored as electronic signal on magnetic media. This should be renewed at appropriate intervals.

Dates and signature of individuals who withdraw and return documents in storage must be recorded.

14.4 Documentation for QA programme:

- Analyst worksheet
- Laboratory report
- Procedures for checking of results
- Procedures for authorization for report
- Period for retention of documents
- Procedures for archiving and disposal of documents.

Annexure 8.1

A. Sources for Biological Reference Material:

1. Within India reference strain can be obtained from IMTECH, Chandigarh (www.imtech.res.in);
2. National Chemical Laboratory, Pune (www.ncl-india.org);
3. Christian Medical College, Vellore (www.cmch-vellore.edu);
4. Central Research Institute, Kasauli, HP (http://mohfw.nic.in);
5. National Institute of Communicable Diseases, Delhi (www.nicd.ac.za)

[Ref: - NABL 102]

B. Sources for Chemical Reference Material:

NIST – Trace Certified Reference Materials (CRMs) may be used

C. Sources for Physical Reference Material:

National Physical Laboratory, Delhi (www.nplindia.org)