UNIT 13 REQUIREMENTS SPECIFIC TO FOOD TESTING LABORATORIES - PHYSICAL AND CHEMICAL PARAMETERS

Structure

13.0 OBJECTIVES

After reading this unit, we shall be able to:

- understand the Quality and Safety requirements of food products; and

- identify the Chemical and Physical testing requirements of food products; and
- apply Laboratory Quality Management System requirements to a laboratory testing chemical and physical parameters in food products.

13.1 INTRODUCTION

Safety of food is a basic requirement governing the quality of food found anywhere along the food chain. "Food safety" implies absence or acceptable and safe levels of contaminants, adulterants, naturally occurring toxins, micro-organisms, pathogens, etc. or any other substance that may make food injurious to health on an acute or chronic basis. Food quality can be considered as a complex characteristic of food that determines its value or acceptability to consumers. Besides safety, quality attributes include: nutritional value; organoleptic properties such as appearance, colour, texture, taste; and functional properties. Taking into consideration the importance of food in the lives of human beings, the quality of food is governed by law in every country of use. Every user country controls the Quality of food, mainly the safety aspects, both produced within the country and that imported, through institutions of food laws and regulations. The Prevention of Food Adulteration Act rules and Regulation under the aegis of Ministry of Health and Family Welfare, Government of India, is the main law governing food safety and quality requirements in India, on the lines of the Food and Drug Administration of USA or the European Union's law on food.

Food safety which is the most important quality parameter contributing to food quality is generally related to presence/absence of food borne hazards in food at the point of consumption. Food reaches a consumer via supply chains that may link many different types of organisations like primary producer, manufacturer, transport and storage contractors, distributors and retailers and caterers, etc. Food safety hazards can occur in the food chain at any stage, hence adequate control is required to be exercised throughout the supply chain. The confirmation of the adequacy of controls is invariably through laboratory testing. Hence the reliability and accuracy of testing plays a very important role in ensuring food quality.

13.2 QUALITY AND SAFETY REQUIREMENTS OF FOOD PRODUCTS

The quality (including safety) parameters of different types of food products, that are encountered at various stages of food chain have been listed below. Most of these would be sold to common consumers, through grocery shops, super bazaars, food chains, etc. The requirements have been given in generic terms for various broad categories of food. These will have to be applied as applicable (as per the food laws).

13.2.1 Different Categories of Food and Related Products and their Quality Parameters

1. **Agricultural products – raw and processed (which would include food grains and seeds, spices, fresh fruits and vegetables, as well as processed like flours, oils and fats, ground spices, tea, coffee, sugar, dry fruits, masala mixes, etc.)**

a) Physical/visual Examination, Grading, Organoleptic (sensory) tests, etc.

b) Proximate/ Nutritional Analysis.

c) Miscellaneous Parameters like pH, moisture, ash, acidity, fat content, etc.

d) Micronutrients including vitamins.

e) Filth.

f) Adulterants.

g) Residue content (as applicable)
   i) Pesticide residues;
   ii) Mycotoxins/Naturally occurring toxin;
   iii) Residual radioactive materials;
   iv) Heavy or toxic metals and other adulterants or trace elements;
   v) Enzymes and hormones;
   vi) Allergens;
   vii) Dioxins, acrylamide, PAH, PCB's, Furans and environmental and other organic chemical contaminants; and
   viii) Genetically modified foods.

h) Microbiological parameters (where applicable).

Food safety parameters are - e) to h) as stated above.

2. Foods of Animal Origin – Raw and Processed (Meat, Fish and Poultry as well as Dairy Products

Physical/visual Examination, Grading, Organoleptic tests, etc.

a) Proximate/ Nutritional Analysis.

b) Micronutrients including vitamins.

c) Adulterants.

d) Residue Content (as applicable)
   i) Pesticide residues,
   ii) Heavy or toxic metals and other adulterants or trace elements,
   iii) Veterinary Drugs/Antibiotic residues,
   iv) Enzymes and hormones, and
   v) Biotoxins and Allergens.

e) Quality Indicators – Histamines, etc.

f) Microbiological Parameters (as applicable).

Food safety parameters - d) to f) above

3. Ready to eat Food Products (Jams, Jellies and Juices, Pickles and Sauces, Ready to Eat Packaged foods Farsan's, Biscuits, etc.)

a) Physical / visual Examination, etc.

b) Proximate / Nutritional Analysis.

c) Compositional and other miscellaneous parameters like pH, water activity, moisture, etc.

d) Sensory evaluation.
e) Micronutrients including vitamins (as applicable).
f) Filth.
g) Adulterants.
h) Residue Content (as applicable).
    i) Pesticide residues,
    ii) Mycotoxins/Naturally occurring toxins,
    iii) Residual radioactive matters,
    iv) Heavy or toxic metals and other adulterants or trace elements,
    v) Enzymes and hormones,
    vi) Allergens,
    vii) Added Additives,
    viii) Dioxins, acrylamide, PAH, PCB's, Furans and environmental and other organic chemical contaminants, and
    ix) Genetically modified foods.
i) Microbiological parameters testing (as applicable).

Food safety parameters – f) to i) above

4. Miscellaneous (Sold through super bazaars and similar outlets)

a) Food additives – food colours, Food additives like baking powder, salt, etc. – Both Quality and safety requirements.
b) Water Testing – Mostly safety requirements.
c) Organic food testing - Both quality and safety requirements.

Although the list distinguishes between the parameters contributing to food safety aspects and others. The testing requirements generally would include for both these types of parameters as applicable to various categories of food products.

Testing for most of the quality attributes would be at percentage levels and would not require a specialized laboratory. For the purpose of this discussion paper, these group of tests are categorized as **Group 1**.

Certain other parameters like micronutrient (vitamins, etc.), organoleptic tests would require certain degree of specialization. Then again tests for detection, identification and estimation of adulterants and additives in processed and ready to eat foods would also require greater amount of specialization and investigative capabilities. Further, the microbiological parameters for determining food safety aspects would invariably involve testing for pathogens, which would call for additional skill and training requirements. For the purpose of this discussion paper these group of tests are categorized as **Group 2.**

While chemical hazards, the most important from chemical safety point of view, would invariably involve testing at residue {sub-ppm (mg/kg) and ppb (µg/kg)} levels and hence would require the most specialized test setup involving sophisticated instrumentation and highly skilled and trained manpower. For the purpose of this discussion paper these group of tests are categorized as **Group 3.**

13.3 CHEMICAL AND PHYSICAL TESTING REQUIREMENTS OF FOOD PRODUCTS

From the point of view of testing the Physical and Chemical requirements of food products can be divided into following categories:

a) Physical/visual Examination, Grading,

b) Organoleptic (Sensory) tests,

c) Miscellaneous chemical parameters like pH, moisture, Ash, Acidity, Fat content, filth content, proximate/Nutritional analysis, higher level adulterants, etc. These are generally the parameters which are present in higher levels, and

d) Residue analysis – Both desirable like micronutrients including vitamins, and undesirable like Pesticide residues, mycotoxin residues, drug residues, etc.

13.4 LABORATORY QUALITY MANAGEMENT SYSTEM

Compliance to the requirements of ISO/ IEC 17025: 2005 by any laboratory carrying out testing and/or calibration activities provides assurance regarding their competence to carry out on a consistence basis the specific tests and/or to which these requirements are applied. However, ISO/IEC 17025 covers general requirements which are generic in nature. Hence specific guidance is generally required for applying various clauses of ISO 17025, specially concerning the technical requirements to different technical areas of testing like Chemical and Biological testing of Food products. This Unit makes us familiar with application of the generic requirements specified in ISO 17025 for Chemical (including physical) testing of food products.

13.5 MANAGEMENT REQUIREMENTS (CLAUSE 4 OF ISO 17025)

While applying the various Management System elements as specified in Clause 4 of ISO 17025:2005 to a laboratory engaged in Chemical and Physical parameters in Food Products, specific guidelines are required to be kept in mind as applicable to specific applications. While describing these, the numbering of the clauses have been aligned with the numbering of ISO/ IEC 17025.

The numbering of the clauses below refers to the numbering of ISO/ IEC 17025. Where clause numbers from that standard are omitted no further clarification is felt necessary for food testing laboratories.

13.5.1 Organisation and Management

The requirements prescribed in this clause are generic in nature and are applicable to all laboratories engaged in all types of testing and/or calibration activities including the Food testing. As per the requirements of Clause 4.1.5 (h) the laboratory is required to have Technical management which has overall responsibility for technical operations. A laboratory engaged in testing of chemical parameters of Food testing is required to provide person (s) with

appropriate competence commensurate with types of chemical parameters of food related testing carried out, which would constitute the technical management. In case laboratory is engaged in diverse testing activities like general chemical parameters and say residue analysis, the laboratory may be required to consider more than one persons as constituting the technical management.

13.5.2 Document Control

All the requirements of document control as specified under this clause are applicable. In case if the laboratory is using any sophisticated, computer controlled equipment like Inductively Coupled Plasma (ICP) Spectrometer, Gas Liquid Chromatograph – Mass detector or Liquid – Mass detector, for residue analysis in food products; or Automatic protein analysers, etc., which make use of proprietary software's, then the document control procedure may require to be extended to these softwares also. In case the laboratory makes use of Laboratory Information Management System (LIMS) for data acquisition from various analytical instruments and for the purpose of calculations and reporting, then again the software used will require to be suitably addressed for its control and validation.

13.5.3 Review of Requests, Tenders and Contracts

Each laboratory is required to draw out the testing scope which will denote its range of activities the range of tests and/or analyses. The scope of a laboratory engaged in testing of food products for chemical parameters can be defined in terms of:

a) The type of food product tested/analysed.

b) The types of tests carried out – Individual tests under a broad category.

c) The test method(s) used – reference to a specification, inhouse method number along with technique if relevant.

d) The range of analysis normally defined as range of concentration and the accuracy/precision. In cases of residue analysis it is desirable to include information on the limits of detection/quantification.

The above scope is required to be carefully arrived at after considering the laboratories capabilities in terms of equipment and infrastructure available, level of competence of manpower at any given point of time. When ever a request for conduct of tests is received it needs to be reviewed in terms of capability of the laboratory. It is important at this point to understand precisely the requirements of the customer, especially in terms of specification or expected range of different parameters because this would generally govern the type of test method used. For example, this becomes very relevant in case of residue testing and also in cases where laboratory has at its disposal the test facilities using different techniques (test methods) for a given parameter. The laboratory is required to make appropriate choice of test method based on the type of food product received for testing and the range of parameter value expected. This information also needs to be communicated appropriately among all the testing sections involved in the analysis of product received for testing – General parameter testing section, residue testing section, sensory evaluation testing section, etc.

13.5.4 Subcontracting of Tests

In case, for few of the parameters in a range of tests required to be carried out for a food product, the laboratory does not have testing facilities or its testing

facility is temporarily out of order, then a provision exists that these tests may be sub-contracted to a competent sub-contractor. As per ISO 17025, a competent sub-contractor is the one who complies with the requirements of ISO 17025. When a food testing laboratory decides to sub-contract, it will also needs to ensure, besides the above, that the sub-contracted laboratory also has the testing capability for the tests and range and accuracy/precision equivalent to its own for the food product under question, before taking the sub-contracting decision.

13.5.5 Purchasing of Services and Supplies

In a chemical laboratory the most important items of procurement are consumables like glassware and Chemicals besides the equipment used for testing purposes. While deciding about the quality of these for procurement purposes, following aspects should be kept in mind:

Glassware

a) The glassware made from borosilicate glass is always preferred because of their property of heat and chemical resistance. However, in case of certain types of chemical analysis like Hydrofluoric acid and in Borate estimations or when used for the purpose of storage of low concentration metal Certified Reference Material (CRM), the Glass is not preferred. The laboratory may require to use plastic bottles and equipment. Hence depending on the type of chemicals required to be used a proper choice should be made and an appropriate specification drawn up for glass, plastic or any other material chosen.

b) When volumetric measurements are required, Class A glassware should generally be used, which is more accurate (has narrower tolerances than the Class B glasswares) and comes with a manufacturers certificate of individual volume.

Chemical, Reagents, standards

a) The general chemicals used in chemical analysis are commercially available for purchase from various sources– dealers, importers, manufacturers, etc. These are also available in different grades depending on their purities– Pure grade, Laboratory reagent, Analytical reagent, etc. Depending upon their use or as specified in the Test method standard, an appropriate choice is required to be made. Whatever the grade claimed, the actual quality of these chemicals and reagent is very much subject to who the manufacturer is. Since it is not possible to check all the chemicals on receipt, through testing, making an appropriate choice of chemical manufacturer through suitable vendor evaluation/reevaluation process is very important.

b) Certified Reference Materials (CRM) play a very important role in present day analytical test laboratory since most of the modern analytical equipments used, require the use CRM for generation of test results. These also have a direct bearing on accuracy of test results obtained. Reference Materials (RM) used for calibration and calibration verification of equipment should invariably be purchased from sources which provide traceability to SI units. However such sources are generally not available for Chemical CRM’s. In such cases, the RM’s should be procured from known competent suppliers or make use of consensus standards. CRM’s must be accompanied by reference value certificate.

c) The inventory of all chemicals is required to be maintained. The information concerning the inventory, typically should include chemical name, purity, manufacturer, lot number (where relevant), amount received and used, storage location, date of receipt and date of expiry. In case the date of expiry is not indicated by the chemical supplier, the same should be defined by the laboratory based on past experience or literature information, etc.

Equipment: The equipment characteristics like range, capacity, sensitivity, least count, accuracy, etc., play a very important role in determining the Test Method performance characteristics. Hence great care should be exercised, while defining the equipment specification and short listing of equipment manufacturers. These should be commensurate with the testing scope of the laboratory. While buying costly and sophisticated analytical equipment like GC – MS, LC-MS, ICP, AAS, GC –ECD/NPD, etc, it is a good idea to insist on a demonstration of testing/equipment capability for the desired testing activity.

13.5.6 Internal Audits

The Internal audit system of a Food laboratory engaged in Chemical testing of food products is required to cover both Management requirements (Clause 4) and Technical requirements (Clause 5) of ISO 17025. A broad checklist for technical requirements which are likely to specific to the laboratory are given below:

a) Personnel: Competence requirements for various levels in the laboratory which would affect quality have been identified commensurate with the scope of testing of the laboratory**.** Staff available are competent to do the work they are assigned. They are properly trained and up-to-date training records are being maintained. Tests are only carried out by authorised analysts. The authorization criteria is defined and is commensurate with the criticality of the tests general chemical requirements of food products and residue requirements.

b) Equipment: The equipment in use are suitable for the testing activities carried out – at least in terms of range and limits. Major equipment are correctly maintained and records of this maintenance are kept. Calibration plan is available and followed for calibration and verification of measurement equipment from competent calibration agencies where relevant records of calibration (certificates) demonstrating traceability to national standards are available. Based on predefined acceptance criteria these calibration results are verified for suitability. Calibrated equipment is appropriately labelled or otherwise identified. Appropriate instructions for use of equipment are available. Instrument performance checks show that performance is within specification.

c) Test Methods and procedures: Preferably standard methods are used with no deviations. In-house methods/deviated standard methods are appropriately validated. Alterations to methods are appropriately authorised. The most up-to-date version of the method is available to the analyst. Analysts are following the methods specified.

d) Standards, calibrants and certified reference materials: The Reference materials and standards actually required for the tests are available and stored appropriately. These are certified or are the 'best' available. The

procedure for preparation of working standards, their labelling, storage, usage, etc., are documented and followed in actual practice. New batches of standards are compared against old before use. Where reference materials are certified, copies of the certificate are available.

e) **Quality Control (QC):** CRM's are regularly used as unknown samples for the purpose of Internal QC. There is an appropriate degree of calibration for each test. QC check samples are being tested by the defined procedures, at the required frequency as per predefined plan and there is an up-to-date record of the results and actions taken where results have exceeded action limits. Results from the random re-analysis of samples show an acceptable measure of agreement with the original analyses. Where appropriate, performance in proficiency testing schemes and/or interlaboratory comparisons is satisfactory and has not highlighted any problems or potential problems. Where performance has been unsatisfactory, corrective action has been taken.

f) **Sampling and Handling:** In case the laboratory is involved in onsite sampling activities, adequately defined documented procedures, which are based on statistically sound methods are available. Sampling personnel are trained and follow these methods onsite. There is an effective documented system for receiving samples, identifying samples against requests for analysis and showing progress of analysis and fate of sample. Samples are properly labelled and stored under appropriate conditions. Sample integrity is maintained through out the progress of the sample in the laboratory.

g) **Records:** Notebooks/worksheets include the date of test, analyst, analyte, sample details, observations related to all stages of analysis of sample including sample preparation, extraction, equipment observations (graphs and chromatograms, etc.) and outputs, test observations, all rough calculations, any relevant instrument traces, and relevant calibration data. Notebooks/worksheets are completed in ink, mistakes are crossed out and not erased, and the records are signed by the analysts. Where a mistake is corrected the alteration is signed by the person making the correction are maintained. The laboratory's procedures for checking data transfers and calculations are being complied with. In case of data available on electronic media like chromatograms, calculation details, appropriate procedures exist for their retrieval, retention and safety.

h) **Test reports:** The report meets all the relevant requirements of ISO 17025 and the test method.

i) **Miscellaneous:** There are documented procedures in operation for handling queries and complaints, obtaining feedback, system failures, Internal audit and management review, control of non-conforming testing, correction and corrective action and preventive action, etc. are kept. The laboratory documentation is kept up-to-date and is accessible to all relevant staff.

13.6 TECHNICAL REQUIREMENTS (CLAUSE 4 OF ISO 17025)

13.6.1 General

All the relevant provisions, as specified in ISO 17025 for this general element are applicable. No additional Guidance felt necessary.

13.6.2 Personnel

The Food Testing Laboratory Management is required to define its staff competence requirements for different levels of operation and activities within the laboratory. These typically cover all the activities that directly or indirectly affect the quality of testing operations. In a chemical laboratory these would include the laboratory personnel involved in following activities:

a) Sampling, if applicable,
b) Sample preparation activities,
c) Operation of equipment,
d) Sample analysis activities including all stages – Sample extraction, operation of equipment (if separately allocated), actual testing, etc.,
e) Test method development and validation (specially relevant in residue analysis of food products), Interpretation of data, supervision of testing and other operations, authorised signatory for test reports and Technical management,
f) Subsidiary functions like maintenance and calibration of equipment, sample entry, purchase, etc.
g) Quality assurance functions, if separately provided for, and
h) Laboratory Management.

Laboratory needs to deploy competent manpower for all the functions/activities as stated above. Competence is defined in terms of capability to do an assigned job competently. However in terms of assigning, it should be defined in terms of combination of educational qualification, experience, skill and training. Hence the laboratory needs to define these competence criteria for various functions as above. For a chemical testing section of a food testing laboratory, these need to be defined in suitable terms. In terms of educational qualification for functions specified at c), d), e), g) and h) (could be excluded in case the lab management functions are purely administrative) should at least be qualified to degree level in Chemistry.

For certain specialized testing activities plus supervisory and other activities, it may be desirable to prescribe a higher level of qualification. The experience component would vary depending upon the activities carried out. Certainly a person involved in method development, Supervisory or Technical Manager functions role would be expected to have about 5 years of relevant experience and may be even higher degree in qualification Post graduate or Doctorate degree. In some cases of complex testing activities (residue testing) specific experience in that area may be necessary. For persons engaged in testing of Physical parameters like grading, sensory evaluation etc., the tests for which are some what subjective in nature, competence of persons performing the test established through experience and skill is very important. Specific training to develop skill, for making judgment based on visual examination is considered very essential.

At any point of time (entry level or subsequently because of technological advancement in testing activities, etc.) if a gap is created, the same can be bridged up through training. The laboratory is expected to have an on going system for identifying such gaps and providing suitable training. The need to periodically retrain staff should be considered where a method or technique is not in regular use or if one person is shifted from one activity to other, say from general testing section to specialized one like sensory evaluation, residue

testing, etc. In each case the critical interval should be established and documented.

The training is expected to be considered effective only when it is suitably evidenced through demonstration. Analysts may only perform tests on samples if they are either recognised as competent to do so, or if they do so under adequate supervision. Continued competence should be monitored, for example, through using quality control techniques or participation in proficiency testing schemes, etc.

13.6.3 Accommodation and Environmental Conditions

Depending on the type of testing carried out in a food (chemical) testing laboratory the laboratory environment and accommodation may play a significant role on the accuracy and precision of test results. It can affect the test method performance, it may affect the equipment performance or may even cause contamination in the sample itself.

The first factor to be considered is the laboratory **layout and design**. Following important factors need to be kept in mind while designing a Food testing laboratory.

a) Different types of testing activities taking place – These would determine the types of separation in activities that is required for avoiding cross contamination. In case a food testing laboratory is planning to test food products for pesticide, antibiotic and other residues, then the residue testing areas need to be segregated from the general test areas. Even within the residue testing area there may be a need to segregate sample preparation area, CRM storage and equipment area, etc. Hence adequate care should be taken of these aspects, after careful consideration at the time of designing of the laboratory itself.

b) Other than the testing areas, there should be clearly demarcated areas for activities like various administration departments of the laboratory, sample receipt and storage areas; sample preparation areas, chemical and other consumable storage areas, washing area, tested sample storage area, disposal area, canteen and toilet blocks, etc.

c) Layout should be conducive to restricting access only to authorized personnel, to the controlled analytical areas, depending on the type of testing being carried out.

d) Layout should be such that it ensures adequate work space to each employee to accomplish assigned tasks. Very important for general chemical testing areas, where lack of adequate working space may become a safety hazard. Sufficient space also must be available for storage of supplies, equipment and accessories. Space should be available for writing reports and other clerical works, storage of records and other documents. Sufficient space must be available for each instrument to facilitate its operation and maintenance. Very relevant for sophisticated equipment like GC-MS, LC-MS, ICP, AAS, where access to instrument back side is essential for maintenance purposes.

e) For a chemical laboratory the Bench tops, sinks, floors, ceilings and wall coverings should be so selected that they provide reasonable levels of resistance to chemicals, corrosive fumes, and other types of specific atmosphere that may be prevalent in a chemical testing laboratory. Where

applicable, it shall provide ease of cleaning, strength, abrasion resistance, heat resistance, thermal shock resistance, stain resistance, bacteria and fungus resistance and corrosion resistance.

f) Chemical testing laboratory must be provided with adequate Fume Hoods of appropriate exhaust specifications and material of construction. The performance of these hoods in terms of air circulation and exhaustion capacity should be periodically monitored.

g) There should be separate demarcated areas for housing gases used in a chemical laboratory for use with equipment like GC, AAS, ICP, etc., and the stabilizers and UPS's essential for use with all sophisticated analytical equipment.

Atmospheric Conditions: The laboratory should provide appropriate environmental conditions and controls necessary for particular tests as per the requirements stated in the test method or for operation of particular equipment including temperature, humidity, freedom from vibration, freedom from airborne and dust borne contamination, special lighting, radiation screening. Critical environmental conditions must be monitored and kept within predetermined limits.

Safety: Chemical testing laboratory must be provided with all types of relevant safety equipment and fire extinguishers; face wash and emergency shower, protective clothing -gowns, coats, gloves, goggles, etc.

The chemical testing laboratory should also be provided with where appropriate, procedure for handling chemical spills; evaluation procedures including a plan of the facility showing the location of safety equipments and fire extinguishers; policy on the use of protective clothing - gowns, coats, gloves, goggles etc.; policy on eating, drinking in the laboratory; waste disposal procedures; routine cleaning and disinfection procedures for work benches, floors, centrifuges, refrigerators, etc., special procedures for handling hazardous substances, etc.

13.6.4 Test and Calibration Methods, Method Validation Test Method Selection and Validation

a) Principle: As per the requirements specified in ISO 17025, Clause 5.4, the Analytical laboratories are free to use any test method – Standard (Published in International, Regional or National standards) Method, Laboratory developed method or Non-standard method as long as it is appropriate for required application to meet the customers requirement of range and accuracy. However, it is preferable that a laboratory uses standard methods. In case the laboratory is engaged in testing on behalf of a regulatory body then it is imperative that it must use the test method referred in the relevant regulation. Even while using standard methods, the laboratories should verify their own ability to achieve satisfactory performance *vis-à-vis* the documented performance characteristics of the method, before any samples are analysed. In all other cases however, the laboratory must ascertain that the test method used is appropriate and fit for the purpose. The most common way of ascertaining the same is through the process of Method Validation. The details of Method validation are covered in Unit 15. **Most important consideration** of all is, that it is the laboratory's responsibility to ascertain from customer about the testing requirements and use a method out of its defined scope which matches the

requirement and is suitable for the purpose intended, be adequately validated where required and be documented and provide results that are traceable to stated references at an appropriate level of uncertainty.

b) Use of Standard Method: The method prescribed by the standard development organisation is expected to be pre-validated mainly through collaborative studies and the method performance characteristics like Limits of detection/quantification, reproducibility, repeatability, uncertainty of measurement are well defined. No further validation would be required if these methods are followed by the test laboratory without any deviations. In case any deviations (not short cuts) are required to be done in terms of using different sample preparation/ sample extraction technique, different reagent/solvent, alternate technique (replacing GC with HPLC); then it is expected that the user laboratory validates all such changes before use.

c) Validation of Test Methods: Laboratory developed methods, non-standard methods, standard methods used outside their intended scope, standard methods amplified or modified and established method revised to incorporate improvements or extended to a new problem, all need to be validated before use. Details of all such methods are required to be maintained. These should include validation data, limitations of applicability, procedures for quality control, calibration and documentation requirements, clearly documented validated test method for internal use by the testing sections. Further details are covered in Unit 15.

13.6.5 Chemical Analysis

i) **General Category**: Quality Parameters (Moisture, Ash, Solubility, Acidity, etc.) and Composition or nutritional value related (like Fat, Protein, Fibre content, etc.). The limits for these tests, as prescribed in product standards, are most of the times at percentage levels. Generally for these tests, well defined predefined standard test methods are available published by all standard formulation bodies, regulatory bodies and independent bodies like AOAC, GAFTA, CODEX, etc. These can be directly adopted by the laboratories without making any changes. Under those circumstances, other than initial and periodic evaluation of laboratory performance checks to verify that the laboratory performance matches with that specified in the published method the laboratory does not need to do full fledged method validation. Periodic Participation in Proficiency testing/Inter lab comparison testing is one of the best way establishing the laboratory performance checks.

ii) **Residue Category**: Generally for all the parameters governing safety aspects of food products, maximum residue/contamination levels are prescribed under regulations. Details of the residues, adulterants/ contaminants for which residue limits may be prescribed under regulations for various categories of food products are given at 13.2 above. For estimation of various types of residues in food products. Test methods are available prescribed in various types of documents – AOAC, EPA, PAM and in variety of Analytical Chemistry Journals. However, these methods may not be utilizable straight away for residue estimations by any laboratory due to following reasons:

a) Residue analysis is generally carried out to estimate undesirable material in food products for which maximum limits are set by standard

formulating bodies, regulators, etc. These limits are generally in the range of mg/kg (ppm), µg/kg (ppb). Hence it is essential that laboratory establishes its method performance characteristics like Method Detection limit, recovery factor, etc., to match with the limits prescribed. Generally for residue analysis the Method performance characteristics are a combination of Men (testing and other personnel), Method (method of analysis which includes extraction procedure) and machine (Equipment/technique used for analysis, which are generally sophisticated equipment like GC, HPLC, GC-MS, LC-MS, AAS, ICP, etc.). The published methods would normally have prescribed methods based on the experiments carried out on a particular set of equipment, based on a particular generation of technology using an extraction technique on few matrices, using personnel possessing a specific skill level. However when a laboratory tries the same method, it may have different generation of equipment, the matrix it tries on may be different, the testing personnel would definitely be different hence the skill level. Sometimes it may have to develop its own methods – Extraction techniques, analysis method and parameters based on information contained in published methods. Hence the laboratory needs to establish its own method performance characteristics for the method decided. This process of establishing method performance characteristics is called Method Validation.

13.6.6 Estimation of Uncertainty of Measurement

Laboratories are required to estimate uncertainty of measurement for the tests being carried out. In cases where the nature of test method precludes rigorous, metrologically and statistically valid, calculation of uncertainty of measurement then it is expected that the laboratory may at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the results does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data. When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis. This is generally applicable to test methods used for residue analysis. The details of Measurement of uncertainty are covered in Unit 15.

13.6.7 Control of Data/Data Management

In a present day chemical laboratory, utilizing sophisticated Equipment like GC's, HPLC's, GC-MS, LC-MS, ICP, AAS, etc., computers may be utilized in a number of ways to control these equipment two most important being i) for controlling the equipment operation and ii) for Data handling, processing and integration. Since these processes directly affect the output of these equipment, it is very important that the laboratory understands the processing system and has a system in place for validating and verifying the same. These are described briefly as follows:

a) **Computer controlled automated system:** In this case outputs of many related equipments are controlled through a single computer system which is also responsible for processing the output and presenting a consolidated

report after necessary computation, etc. These may be operated either simultaneously or in controlled time sequence. System validation is required to be carried out by validation of individual components plus an overall check on the dialogue between individual components and the controlling computer. An initial check that the computer's, interfaces and connecting cabling have sufficient capacity for the required tasks is necessary. Besides this, the systems may require to be validated by checking for satisfactory operation (including performance under extreme circumstances) and establishing the reliability of the system before it is allowed to run unattended. The use of quality control samples and standards run at intervals in the sample batches is subsequently required to monitor correct performance on a day-to-day basis. Calculation routines can be checked by testing with known parameter values. Electronic transfer of data should be checked to ensure that no corruption has occurred during transmission through the use of 'verification files' and backed-up hard copy of the data.

b) **Data handling or processing systems, integration systems:** When using computer controlled equipment like Chromatographs and Spectrometers, etc., during the initial step leading to data processing, the output from analytical instrument will usually need to be converted to a digital signal using an analogue/digital converter. The digitized data is then translated into a recognisable signal (numbers, peaks, spectra according to the technique) by the software algorithm. The algorithm makes various decisions (such as deciding where peaks start and finish, what sort of base line/background correction is required or whether a number should be rounded up or down) according to programmed instructions. The algorithm is a common source of strange performance and validation should test the logic behind the decisions made by the algorithm. It may be difficult to validate these systems in isolation from the analytical instrument producing the original signal. Usually the whole system is validated in one go, by using chemical standards or reference materials. Such validation is normally acceptable.

13.6.8 Equipment

A food testing laboratory engaged in testing of different chemical and physical parameters is required to be furnished with all the items of equipment commensurate with the scope of testing of the laboratory covering as applicable different types of tests listed at 13.2 above. These should also include equipment required for activities like sampling (if applicable), preparation of test items, processing and analysis of tests and data.

The choice of equipment in terms of capacity, range, least count, sensitivity, etc. should be made based on the scope of testing of the laboratory defined in terms of range, accuracy and method detection limit. For a residue testing laboratory, depending upon the residue limits specified and tested, the choice of technique and equipment will have to be made. In short, all equipment used in food testing laboratory should be of a specification sufficient for the intended purpose, and kept in a state of maintenance and calibration consistent with its use.

Equipment normally found in the chemical laboratory can be categorised as:

i) General equipment like hotplates, heating mantles, stirrers, water baths, centrifuges, non-volumetric glass wares or glass wares like measuring

cylinders used for making rough volume measurements, etc. These are equipment, which are not used for making measurements or those which have minimal influence on measurements.

ii) Equipment which are used for making actual measurements: Volumetric glass wares like volumetric flasks, pipettes, burettes, etc. and measuring instruments/equipment like analytical balances, Thermometers, Hydrometers, Protein Analysers, spectrophotometers (UV, IR, etc.), Chromatographs (GLC, HPLC, GC-MS, LC-MS, etc.), Spectrometers (ICP, AAS, etc.), etc.

For General equipment, rudimentary maintenance through cleaning and safety checks as necessary are sufficient. Calibrations or performance checks will be necessary where the setting can significantly affect the test or analytical result (e.g. the temperature of an oven, a muffle furnace or constant temperature bath).

For measurement equipment, the correct use of the equipment is critical to analysis and therefore it must be correctly used, maintained and calibrated in line with the equipment specification/test method, for a given application, as may be applicable. For example, for equipment's like Spectrophotometer in addition to general calibration procedures used for calibration of wavelength and absorbance using say a solution of standard potassium dichromate, it will need to be calibrated using CRM relevant to the analysis being carried out at the time analysis itself.

In the case of volumetric glass ware, in addition to calibration to establish accuracy, cleaning procedures, storage and segregation of volumetric equipment may be critical, particularly for trace analyses where leaching and adsorption can be significant.

Correct use combined with periodic servicing, cleaning and calibration will necessarily ensure an instrument is performing adequately. Where appropriate, periodic performance checks (also called as calibration in general terms) should be carried out. Some examples of these are - the response of the equipment; stability and linearity of sources; sensors and detectors; the separating efficiency of chromatographic systems; the resolution, alignment and wavelength accuracy of spectrometers; etc. Frequency of such performance checks (calibration) needs to be specified by every lab based on it's experience and on frequency of utilization and various sources of information as may be specified in instrument manuals. General guidelines as available in various accreditation documents may also be used. It is also possible to build performance checks – system suitability checks – into test methods. These may be based on the levels of expected detector or sensor response to calibrants, the resolution of calibrants in separating systems, the spectral characteristics of calibrants, etc. These checks should be satisfactorily completed before the equipment is used.

13.7 TRACEABILITY OF MEASUREMENT

The calibration program in a chemical laboratory should be designed to ensure that, where applicable, all measurements are traceable through certificates held by the laboratory, either to a national or international standard or to a certified reference material. Where no such reference standard or certified reference

material is available, a material with suitable properties and stability should be selected or prepared by the laboratory and used as a laboratory reference.

The required properties of this material should be characterised by repeat testing, preferably by more than one laboratory and using a variety of methods. This concept is as defined in ISO Guide 35:1989, "Certification of reference materials - General and statistical principles".

Depending on the types of calibration required, Analytical (Chemical) tests can be divided into following categories:

a) Some analytical tests depend critically on the measurement of physical properties, such as weight measurement in gravimetry and volume measurement in titrimetry. Since these measurements have a significant effect on the results of the test, a suitable calibration Programme for these quantities is essential.

b) Where a test is used to measure an empirical property of a sample, such as flashpoint, equipment is often defined in a national or international standard method and traceable reference materials should be used for calibration (verification) purposes where available. New or newly acquired equipment must be checked by the laboratory before use to ensure conformity with specified design, performance and dimension requirements.

c) Instruments such as chromatographs and spectrometers, which require calibration as part of their normal operation, should be calibrated using reference materials of known composition (may be solutions of pure chemicals).

d) In some cases, calibration of the entire analytical process can be carried out by comparing the measurement output from a sample with the output produced by a suitable reference material that has been subjected to the same full analytical process as the sample. The reference material may be either a synthetic mixture prepared in the laboratory from materials of known (and preferably certified) purity, or a purchased certified matrix reference material. However, in such cases, a close match between the test sample and the matrix reference material, in terms of the nature of the matrix, and the concentration of the analyte has to be assured.

Frequently in a chemical analysis it is not possible to calibrate individual parameters within a method. In such cases, traceable calibration of the entire method may be possible using a Certified Reference Material (CRM). The CRM is subjected to the same processes as the samples. The analysed degree of agreement between the analysed value for the CRM and its certified value may be used to determine the accuracy of the analysed values obtained for the samples. This concept is very useful in residue analysis.

Individual calibration programmes are required to be established depending on the specific requirements of the analysis. Procedures for performing calibrations are required to be documented either as part of specific analytical methods or as a general calibration document.

13.8 SAMPLING

In case of Food products, analytical (Chemical and Physical) tests are generally required to establish quality/safety parameter of a lot of agri-

products, a batch of processed food, etc., to verify if the lot conforms to the product specific requirements. However in a laboratory only a small quantity is required for conduct of various types of tests, say 50 grams for pesticide residue analysis, 5 -10 grams for moisture content or protein content. Based on the tests on such a small sample portion a decision is required to be taken regarding the lot quantity which may be anything from few kgs to few tons. If the test portion is not representative of the original material, it will not be possible to relate the analytical parameter measured, to that in the original material, no matter how good the analytical method is or how carefully the analysis is performed. Hence it is very essential that the samples drawn for laboratory testing should be representative of the lot/batch.

All food testing laboratories are not required to carry out the sampling activities prior to testing. In most cases organisations/individuals outside of the test laboratory are responsible for sampling and as far as the laboratory is concerned it's responsibility is restricted to the sample received.

However if the laboratory is responsible for drawal of sample (sampling), then selection of an appropriate sample or samples, from a larger amount of material is a very important stage in chemical analysis process carried out by the laboratory. It is generally a very complex operation although seeming to be very simple. To make it effective the sampling stage should be carried out by, or under the direction/supervision of an experienced person, with an understanding of distribution of the analyte under question within the lot of the food product. The laboratory is required to define a system for drawing samples on a consistent basis, which is generally known as sampling plan. Many of the standard writing bodies establish and publish validated methods for sampling of various food products and commodities. These methods are based on established statistical methods, which also incorporate knowledge input based on experience. Where specific standard methods are not available the laboratory can make use of a method adapted from similar applications. Further while dealing with analysis of Chemical contaminants and residues in food products special precautions are required to be ensured to avoid external contamination at the sampling stage, on account of sampling instruments used or packaging used for keeping the drawn sample, etc.

Sub-sampling within the laboratory – Even if a laboratory is not responsible for sampling from a lot into laboratory sample, once the sample is received in a laboratory, the process of drawal of portion of the sample into a portion for performing the test, may require further treatment like sub-division into test portion and/or milling and grinding prior to analysis. In these cases, it is required to ensure that the test portion taken for analysis should be representative of the laboratory sample. To ensure that the test portion is homogeneous it may be at times be necessary to reduce the particle size by grinding or milling. At this stage, it is necessary that appropriate care is taken to ensure that segregation does not occur during sub-division. In some cases, it may be necessary to crush or coarsely grind the sample prior to sub-division into test samples. The sample may be sub-divided by a variety of mechanisms, including the process of coning and quartering, or through use of a rotating sample divider or a centrifugal divider. The particle size reduction step may be performed either manually (mortar and pestle) or mechanically using crushers or mills. At this stage care should also be taken to avoid cross contamination of samples and to ensure that the equipment does not contaminate the sample (e.g. metals) or that the composition of the sample is not altered (e.g. loss of

moisture) through excessive heat generation during milling or grinding operations. Many standard methods of analysis contain a section that details the preparation of the laboratory sample prior to the withdrawal of the test portion for analysis. In absence of these, the laboratory should develop and document suitable methods based on their experience and literature data, etc. In cases where these aspects are very important sub-sampling should also be included in uncertainty estimations.

13.9 HANDLING TEST AND CALIBRATION ITEMS

Samples of food products may be received in a laboratory in a variety of containers. These may be plastic or glass bottles, plastic bags, paper or cloth bags, corrugated boxes, tin containers, etc. On receipt of a sample in the laboratory, these should be first verified to ensure that no leakage has taken place, container is intact or there are no other evidences to indicate that any contamination would have taken place during transport. And that these were maintained within set temperature or other environmental tolerances during transfer to the laboratory and prior to testing. Only after ensuring all this they should be accepted for registration.

a) Sample registration: On receipt, a sample must be registered into the laboratory records. The form of registration may vary. In most laboratories, a sample register will be used, but in some cases, the sample details may be written directly on worksheets or into workbooks or may be directly entered into the LIMS. Simultaneously the Unique identification number allotted should also be indicated on the sample container.

Identification labels on the sample container should be secure and legible. The writing should be such that it should not get obliterated or misplaced when placed in the laboratory racks or benches during testing process. It could be in the form of a separate attached label or written on the container itself with permanent marker. Labelling should be firmly attached to the sample packaging and where appropriate, be resistant to fading, autoclaving, sample or reagent spillage, and reasonable extremes of temperature and humidity. It should also be ensured that the identity is not lost during the process of further division or sub-sampling when required to be tested in two testing sections of the laboratory – say general testing section, residue testing section, microbiology lab, etc.

b) Sample retention and storage: The sample retention criteria need to be established based on the type of samples and the tests being carried out and the shelf life of the food product. From receipt to the time of disposal the samples should be stored in appropriate atmosphere to preserve integrity of the sample and should be in line with the manufacturers instructions if any. Extremes of environmental conditions should generally be avoided, especially for food products, which might change the composition of the sample, for example, causing loss of analyte through degradation or adsorption. Laboratory may require to be equipped with suitable Deep freezers or cold storages if it is involved in testing of perishable food products like fruits and vegetables, fish and other meet products, etc. In such cases environmental monitoring of storage conditions also may become necessary.

13.10 ASSURING THE QUALITY OF TEST AND CALIBRATION RESULTS

As per the requirement specified in ISO 17025, the laboratories engaged in testing of food products are required to plan and operate a system for internal quality control (QC) checks, in order to monitor day-to-day and test method batch-to-batch analytical performance. In addition to this they are also required to participate as often as possible in Proficiency testing schemes (external QC). These measures are required to be done as per a periodically drawn out plan. The frequency and the type of QC would depend upon the nature and frequency of analysis, criticality of testing and test variability, test difficulty and uncertainty in measurement, etc. For example, the need for internal QC would be much higher for a residue analysis involving a difficult matrix where the test method is based on an internally validated method than say for a routine analysis like moisture content which is carried out strictly as per a standard method specified in AOAC.

Some of the quality control measures available to a chemical testing laboratory for monitoring of analytical performance are:

a) Use of Certified reference materials / Internally generated reference material, as QC samples,

b) Replicate testing and repeat testing by other analysts/examiners,

c) Positive and negative controls,

d) Spiked samples, standard additions and internal standards,

e) Alternate methods,

f) Retesting of retained items,

g) Correlation of results for different characteristics of an item, and

h) Control Charts, Trend analysis, etc.

Depending upon the type of tests performed and stability and availability of samples, one or more of the above measures may be adopted for different chemical tests performed by the laboratory as per a well defined plan. Results of QC test should be assessed against a predefined acceptance criteria and in case the results are not within the acceptance criteria, system must exist for analyzing the problem and taking appropriate corrective action.

While planning for Internal Quality Control measures it should be ensured that the level adopted should be demonstrably sufficient to ensure the validity of the results. A level of about 5% of the total samples tested is considered adequate for routine analysis. That is one in every twenty samples tested is expected to be QC sample. For more complex analysis involving many different steps, involving complex matrices, like that for residue (pesticide, antibiotic, mycotoxins, etc.) analysis in food products the quality control level can be as high as 20% to 50%. For complex analysis, which may be performed infrequently, a full method performance validation using spiked samples may be required to be performed every time the test is performed.

Proficiency testing (external QC)

Regular participation in Proficiency testing is one of the best ways through which an analytical laboratory can monitor its performance against its own

standards and in comparison with other similar laboratories. Proficiency testing helps to identify performance parameters in terms of repeatability and reproducibility and also bias (systematic error) if any. The details of Proficiency testing are covered in Unit 15.

13.10.1 Reporting of Results

All the requirements, as specified in ISO 17025 Clause 5.10, as relevant to a laboratory engaged in sampling and/or testing are applicable. In addition, the food testing laboratories engaged in residue analysis while reporting results must always report the Limit of detection/Limit of quantification as relevant, along with each residue result reported. In case a particular residue is not detected by the analysis method employed, then the laboratory must report the result as "Not Detected" along with the Detection limit. The result must never be reported as "Absent" or in "Traces".

Check Your Progress Exercise 1

Note: a) Use the space below for your answers.
b) Compare your answers with those given at the end of the unit.

1) What aspects should be kept in mind while purchasing the consumables in laboratory?

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2) What are the important factors to be kept in mind during planning the layout and design of laboratory?

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3) Explain sample registration?

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4) Which are the quality control measures available to a chemical laboratory for monitoring of analytical performance.

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13.11 LET US SUM UP

The ISO/ IEC 17025:2005 specify the generic requirements for laboratory carrying out testing or calibration activities including food testing. However, a laboratory engaged in testing of chemical and physical parameters of food is required to provide person(s) with appropriate competence commensurate with type of physical and chemical parameters of food related testing carried out, which would constitute the technical management. Further, if the laboratory is using any sophisticated computer controlled equipments like GG-MS, LC-MS, for residue analysis or ICP-MS for heavy metals which make use of proprietary software's then the documents control procedure may be required to be extended to these software's also. While making purchase of services and supplies it must be kept in mind that the instruments being purchased must be capable of determining the residue or heavy metals well below the maximum limits prescribed for different food products.

With respect to technical requirements the food testing laboratory management is required to define its staff competence for different levels of operations and activities within the laboratory. Chemical parameters in food products can broadly be divided into general category and residue analysis for general category well defined standard test methods are available in the literature published by standards formulating bodies like AOAC, GAFTA, CODEX etc. which can be directly adopted by the laboratory. For residue analysis it is essential that a laboratory should establish its method performance characteristics like method detection limit, recovery factor etc. to match with the prescribed limit, since residue limits are generally in ppm or ppb. Generally for residue analysis the method performance characteristics are a combination of Men (testing and other personals), Method of analysis (which include extraction procedure) and machine (equipments/techniques used for analysis, which are generally sophisticated like GC-MS, LC-MS ICP-Ms etc.). Equipments for food testing laboratory engaged in physical and chemical parameters are required to be furnished with all the items of equipments commensurate with the scope of testing. The choice of equipments in terms of capacity, range, least count, sensitivity etc. should be made based on the scope of testing.

13.12 KEY WORDS

AAS : Atomic Absorption Spectrophotometer.

AOAC : Association of Official Analytical Chemists.

Calibration : Calibration is a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding values realized by standards.

Certified Reference Material (CRM) : Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure, which establishes its traceability to an accurate realisation of the units in which the property values are expressed, and

for which each certified value is accompanied by an uncertainty at a stated level of confidence.

EPA : Environmental Protection Agency (USA). Methods are available free on the NET.

FOSFA : Federation of Oils, Seeds and Fats Association.

GAFTA : The Grain and Feed Trade Association.

GC – MS : Gas chromatograph with Mass Spectrometer.

GC –ECD/NPD : Gas chromatograph with Electron capture/ Nitrogen Phosphorous detector.

ICP : Inductively Coupled Plasma detector.

LC-MS : Liquid Chromatograph with Mass detector.

Limit of Detection (LoD) : The lowest content that can be measured with reasonable statistical certainty. It is also defined as the lowest concentration of analyte in a sample that can be detected, but not necessarily quantified under the stated conditions of the test.

Limit of Quantitation (Loqo) : It is the lowest concentration of analyte that can be determined with an acceptable level of repeatability precision and trueness under the stated conditions of the test. It is also defined as the content equal to or greater than the lowest concentration point on the calibration curve. Also sometimes known as ‘limit of determination’, ‘limit of Reporting’

LIMS : Laboratory Information Management System (LIMS).

Measurement Uncertainty : A parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand.

Method Validation : It is the confirmation by examination and through provisions of objective evidence that the particular requirements for a specific intended use are fulfilled.

PAM : Pesticide Analytical Manual, Published by United Stated Department of Agriculture (IUSDA), Test Method Manual is downloadable from the NET.

Proficiency Testing (Inter Laboratory Comparisons) : A recognised way for a laboratory to monitor its performance against both its own requirements and the requirements of Accreditation bodies

Quality Control (QC) : The operational techniques and activities that are used to fulfill requirements for quality. Examples of QC: Control charting; blank determinations; spiked samples; repeat determinations; blind samples.

Range (Measuring-Working) : Set of values of measurands for which the error of a measuring instrument is intended to lie within specified limits.

Recovery : The fraction of analyte added to a test sample (fortified or spiked sample) prior to analysis, the unfortified and fortified samples, percentage recovery (%R) is calculated as follows:

$$\%R = [(CF-CU)/CA] \times 100$$

Where CF is the concentration of analyte measured in the fortified sample; CU is the concentration of analyte measured in the unfortified sample;

CA is the concentration of analyte added (measured value, not determined by method) in fortified sample.

Reference Material (RM) : Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Repeatability : It is the smallest expected precision. It gives an idea of the sort of variability to be expected when a method is performed by a single analyst on one piece of equipment over a short timescale, i.e. the sort of variability to be expected between results when a sample is analysed in duplicate.

Reproducibility : It is the largest measure of precision normally encountered. Reproducibility gives an idea of the sort of variability to be expected when a method is performed by different laboratories – different analyst, different equipment, different conditions, over a long timescale, i.e. the sort of variability to be expected during inter laboratory comparisons.

Sample : A portion of material selected to represent a larger body of material.

Sample Handling : This refers to the manipulation to which samples are exposed during the sampling process, from the selection from the original material through to the disposal of all samples and test portions.

Sample Preparation : This describes the procedures followed to select the test portion from the sample (or sub-sample) and includes: in-laboratory processing; mixing; reducing; coning and quartering; riffling; and milling and grinding.

Sensitivity : This is effectively the gradient of the response curve, i.e. the change in instrument response which corresponds to a change in analyte concentration, where the response has been established as linear with respect to concentration

(within the linear range of the method) and the intercept of the response curve has been determined.

Sub-sample : This refers to a portion of the sample obtained by selection or division; an individual unit of the lot taken as part of the sample or; the final unit of multistage sampling.

Test Portion : This refers to the actual material weighed or measured for the analysis.

Traceability : 'Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties

Note: Most of the Definitions included above are based on Standard definitions as defined in Publications like VIM, EURACHEM, ISO, IUPAC, etc.

13.13 ANSWERS TO CHECK YOUR PROGRESS EXERCISE

Your answer should include following points:

Check Your Progress Exercise 1

1) a) The glass ware made from borosilicate glass is always preferred because of their property of heat and chemical resistance. However, in case of certain types of chemical analysis like Hydrofluoric acid and in Borate estimations or when used for the purpose of storage of low concentration metal Certified Reference Material (CRM), the Glass is not preferred. The laboratory may require to use plastic bottles.

   b) When volumetric measurements are required, Class A glass ware should generally be used, which is more accurate (has narrower tolerances than the Class B glass wares) and comes with a manufacturers certificate of individual volume.

   c) The general chemicals used in chemical analysis are available in different grades depending on their purities– Pure grade, Laboratory reagent, Analytical reagent, etc. Since it is not possible to check all the chemicals on receipt, through testing, making an appropriate choice of chemical manufacturer through suitable vendor evaluation/reevaluation process is very important.

   d) Certified Reference Materials (CRM) play a very important role in present day analytical test laboratory since these have a direct bearing on accuracy of test results obtained. Reference Materials (RM) used for calibration and calibration verification of equipment should invariably be purchased from sources which provide traceability to SI units. RM's should be procured from known competent suppliers or make use of consensus standards. CRM's must be accompanied by reference value certificate.

e) During purchase of instruments, equipment characteristics like range, capacity, sensitivity, least count, accuracy, etc., play a very important role in determining the Test Method performance characteristics. Hence great care should be exercised, while defining the equipment specification and short listing of equipment manufacturers. These should be commensurate with the testing scope of the laboratory. While buying costly and sophisticated analytical equipment like GC – MS, LC-MS, ICP, AAS, GC –ECD/NPD, etc., it is a good idea to insist on a demonstration of testing/equipment capability for the desired testing activity.

2) a) In case a food testing laboratory is planning to also test food products for pesticide, antibiotic and other residues, then the residue testing areas need to be segregated from the general test areas. Even within the residue testing area there may be a need to segregate sample preparation area, CRM storage and preparation area, equipment area, etc.

b) Other than the testing areas, there should be clearly demarcated areas for activities like various administration departments of the laboratory, sample receipt and storage areas; sample preparation areas, chemical and other consumable storage areas, washing area, tested sample storage area, disposal area, canteen and toilet blocks, etc.

c) Layout should be conducive to restricting access only to authorized personnel, to the controlled analytical areas, depending on the type of testing being carried out.

d) Layout should be such that it ensures adequate work space to each employee to accomplish assigned tasks. Very important for general chemical testing areas, where lack of adequate working space may become a safety hazard. Sufficient space also must be available for storage of supplies, equipment and accessories. Space should be available for writing reports and other clerical works, storage of records and other documents. Sufficient space must be available for each instrument to facilitate its operation and maintenance. Very relevant for sophisticated equipment like GC-MS, LC-MS, ICP, AAS, where access to instrument back side is essential for maintenance purposes.

e) For a chemical laboratory the Bench tops, sinks, floors, ceilings and wall coverings should be so selected that they provide reasonable levels of resistance to chemicals, corrosive fumes, and other types of specific atmosphere that may be prevalent in a chemical testing laboratory. Where applicable, it shall provide ease of cleaning, strength, abrasion resistance, heat resistance, thermal shock resistance, stain resistance, bacteria and fungus resistance and corrosion resistance.

f) Chemical testing laboratory must be provided with adequate Fume Hoods of appropriate exhaust specifications and material of construction. The performance of these hoods in terms of air circulation and exhaustion capacity should be periodically monitored.

g) There should be separate demarcated areas for housing gases used in a chemical laboratory for use with equipment like GC, AAS, ICP, etc., and the stabilizers and UPS’s essential for use with all sophisticated analytical equipment.

3) On receipt, a sample must be registered into the laboratory records. The form of registration may vary. In most laboratories, a sample register will be used, but in some cases, the sample details may be written directly on worksheets or into workbooks or may be directly entered into the LIMS. Simultaneously the Unique identification number allotted should also be indicated on the sample container.

   Identification labels on the sample container should be secure and legible. The writing should be such that it should not get obliterated or misplaced when placed in the laboratory racks or benches during testing process. It could be in the form of a separate attached label or written on the container itself with permanent marker. Labelling should be firmly attached to the sample packaging and where appropriate, be resistant to fading, autoclaving, sample or reagent spillage, and reasonable extremes of temperature and humidity. It should also be ensured that the identity is not lost during the process of further division or sub-sampling when required to be tested in two testing sections of the laboratory – say general testing section, residue testing section, microbiology lab, etc.

4) Some of the quality control measures available to a chemical testing laboratory for monitoring of analytical performance are:

   a) Use of Certified reference materials/Internally generated reference material, as QC samples,
   b) Replicate testing and repeat testing by other analysts/examiners,
   c) Positive and negative controls,
   d) Spiked samples, standard additions and internal standards,
   e) Alternate methods,
   f) Retesting of retained items,
   g) Correlation of results for different characteristics of an item, and
   h) Control Charts, trend analysis, etc.

Regular participation in Proficiency testing is one of the best ways through which an analytical laboratory can monitor its performance against its own standards and in comparison with other similar laboratories. Proficiency testing helps to identify performance parameters in terms of repeatability and reproducibility and also bias (systematic error) if any.

13.14 SUGGESTED READING

Accreditation for Chemical Laboratories, UKAS Publication ref: LAB 27, Edition 1, 2000.

CITAC- EURACHEM *Guide to Quality in Analytical Chemistry* - An Aid to Accreditation, Edition 2002.

ISO/IEC 17025:2005, *General Requirements for the Competence of Testing and Calibration Laboratories*.

ISO Guide 30, *Terms and Definitions used in Connection with Reference Materials*.

ISO 9000, *Quality Management Systems - Fundamentals and Vocabulary*.

NABL 103 - *Specific Guidelines for Chemical Testing Laboratories.*

Technical note: c&b-002, February 2005 *"Quality Assurance of Equipment Commonly used in Chemical and Biological Testing Laboratories"*, Issued by Singapore Accreditation Council - Singapore Laboratory Accreditation Scheme (sac-singlas).

VIM: 1993, ISO *International Vocabulary of Basic and General Terms in Metrology*.

Note: Considerable assistance has been taken from the above publication in drafting this Unit.