## CENTER FOR ADVANCED FACULTY TRAINING IN HOME SCIENCE XXVIII TRAINING PROGRAMME ON

## "New Dimensions in Food Quality Analysis towards achieving Comprehensive Food Safety"

From 3<sup>rd</sup> September to 23<sup>rd</sup> September 2015



**Organized By** 

Dr. Mahalakshmi V. Reddy Director - CAFT H.Sc Dr .K. Manorama Course Director

*Co – Directors* 

Dr. Anurag Chaturvedi Dean of Home Science Dr. M. Sreedhar Sr. Scientist, QC Lab,



Quality Control Laboratory and

Center of Advanced Faculty Training in Home Science Professor Jayashankar Telangana State Agricultural University Rajendranagar, Hyderabad 500 030

## Acknowledgement

The Director, CAFT (Home Science), PJTSAU, Dr. Mahalakshmi V. Reddy, Course Director, Dr. K. Manorama (Principal Scientist and Head, Quality Control Laboratory, PJTSAU), Course coordinators, Dr. Anurag Chaturvedi (Dean, Faculty of Home Science, PJTSAU) and Dr. M. Sreedhar (Principal Scientist, Quality Control Laboratory, PJTSAU) gratefully acknowledge the financial support provided by the Indian Council for Agricultural Research (ICAR) Education Division, for conducting the 21 days training program entitled "New Dimensions in Food Quality Analysis for Achieving Comprehensive Food Safety" held from 3<sup>rd</sup> September 2015 to 23<sup>rd</sup> September, under Center for Advanced Faculty Training in Home Science.

Our special thanks to Dr. V. Praveen Rao, Special Officer of the Professor Jayashankar Telangana State Agricultural University (PJTSAU) the newly created university after Telangana State Division for fully extending cooperation to conduct CAFT – H.Sc activities under the Faculty of Home Science. We express our sincere thanks to Dr. Anurag Chaturvedi, Dean of Home Science for providing total support and valuable contributions to the training.

We express our sincere thanks to Keynote speakers Padmashree Dr. V. Prakash, Distinguished Scientist CSIR and Ex-Director, CFTRI, Mysore, for gracing the inaugural function and delivering the Keynote address on "<u>Role of Chemistry and Biology in Food Safety Net; for a</u> <u>sustainable dimension of Food Quality"</u>. Dr. D. Rama Rao, Director NAARM, had kindly provided accommodation for Dr. V. Prakash during his stay at Hyderabad for the Inaugural function, and also for Dr. Shailaja Naik, Reviewer appointed by ICAR to oversee the performance of the training programme.

We gratefully acknowledge other eminent speakers including our Special Officer, Dr. V. Praveen Rao and Dr. D. Raji Reddy, Director of Research, PJTSAU and the Dean, Faculty of Home Science, Dr. Anurag Chaturvedi, during the Inaugural and Valedictory Sessions of the training.

We acknowledge the guest speakers who came from PJTSAU, NIPHM, NIN, First Source Laboratories, St. Ann's College Mehdipatnam, Pesticide Residue Laboratory and PJTSAU. Rice Research Station, ARI, PJTSAU, Rajendranagar and College of Food Science and Technology, Rudrur. We also acknowledge the institutes which allowed for field visits to all our CAFT trainees – State Food Laboratories, Nacharam, Hyderabad and First Source Laboratories, Nacharam, Hyderabad. We thank the Director of the Extension Education Institute for providing comfortable accommodation and mess facilities to the participants.

We acknowledge the help rendered by Dr. V. Sashibhushan, Principal Scientist and Head, Pesticide Residue Laboratory, All India Network project on pesticide residues, PJTSAU for providing comfortable classroom and facilities including LCD projector for the entire period of the training. We thank the Non teaching staff of CAFT and Quality Control Laboratory, PJTSAU, for the support and help rendered all through the training period.

Dr. K. Manorama Course Director Dr. Mahalakshmi. V. Reddy CAFT Director

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#### **EXECUTIVE SUMMARY**

By

## Dr. K. Manorama Course Director

Food analysis is the discipline dealing with the development, application and study of analytical procedures for characterizing the properties of foods and their constituents. Analytical procedures are used to provide information about a wide variety of different characteristics of foods, including their composition, structure, physicochemical properties and sensory attributes. This information is critical to our rational understanding of the factors that determine the properties of foods, as well as to our ability to economically produce foods that are consistently safe, nutritious and desirable and for consumers to make informed choices about their diet. The objective of this course is to review the basic principles of the analytical procedures commonly used to analyze foods and to discuss their application to specific food components, e.g. lipids, proteins, water, carbohydrates minerals, vitamins, pesticide residues, microbial contaminants, fatty acids, and also review food safety and its implications for human health.

#### **Reasons for Analyzing Foods**

Foods are analyzed by scientists working in all of the major sectors of the food industry including food manufacturers, ingredient suppliers, analytical service laboratories, government laboratories, and University research laboratories. The various purposes that foods are analyzed listed below.

- **&** Government Regulations and Recommendations
- Standards- Mandatory Standards: Standards of Identity, Standards of Quality, Standards of Fill-of-Container, Standards of Grade, and Voluntary Standards.
- ✤ Nutritional Labeling
- \* Authenticity
- Food Inspection and Grading
- ✤ Food Safety
- Quality control
- \* Research and Development

In recent years, there have been significant changes in the preferences of consumers for foods that are healthier, higher quality, lower cost and more exotic. Individual food manufacturers must respond rapidly to these changes in order to remain competitive within the food industry. To meet these demands food manufacturers often employ a number of scientists whose primary objective is to carry out research that will lead to the development of new products, the improvement of existing products and the reduction of manufacturing costs.

Many scientists working in universities, government research laboratories and large food companies carry out basic research. Experiments are designed to provide information that leads to a better understanding of the role that different ingredients and processing operations play in determining the overall properties of foods. Research is mainly directed towards investigating the structure and interaction of food ingredients, and how they are affected by changes in environment, such as temperature, pressure and mechanical agitation. Basic research tends to be carried out on simple model systems with well-defined compositions and properties, rather than real foods with complex compositions and structures, so that the researchers can focus on particular aspects of the system. Scientists working for food companies or ingredient suppliers usually carry out product development. Food Scientists working in this area use their knowledge of food ingredients and processing operations to improve the properties of existing products or to develop new products. In practice, there is a great deal of overlap between basic research and product development, with the basic researchers providing information that can be used by the product developmers to rationally optimize food composition and properties.

In both fundamental research and product development analytical techniques are needed to characterize the overall properties of foods (e.g., composition, color, texture, flavor, shelf-life etc.), to ascertain the role that each ingredient plays in determining the overall properties of foods, and to determine how the properties of foods are affected by various processing conditions (e.g., storage, heating, mixing, freezing).

With this background in mind the current training programme on "New Dimensions of Food Quality Analysis towards Achieving Comprehensive Food Safety" was conceptualized with the primary objective of training faculty on newer methods of Food Analysis, Food Safety and its importance, importance of up gradation of laboratories to meet with International ISO/IEC 17025-2005 standards, process of accrediting the laboratory with NABL requirements, and other related topics. Hence the training was proposed as a 21 days training program and sanction was accorded by ICAR for the financial year 2015-16. All the Vice- Chancellors of agriculture universities, Deans of Home science and Directors of Foods and Nutrition, training coordinators of at least 40 KVKs, Associate Deans of Colleges within PJTSAU, were sent the training brochure and nomination form by Post and E-mail asking for deputation of at least two eligible faculty members for the training.

Initially there was a lot of response from faculty members from all over India and they also uploaded an advance copy of the nomination form. There were 6 outstation participants and one nominee from PJTSAU, Hyderabad, who dropped out in the last moment, due to official and personal reasons. Eight participants were from out station Universities like Assam Agricultural University, Gujarat Agricultural University, Navsari, UAS, Dharwad, UAS, Haasan, and the remaining were from Hyderabad, including PJTSAU, Konda Laxman Telangana State Horticultural University and ANGRAU. Hence the training program was offered to twenty participants by accepting the nominations and adding a four Research Associates who are currently working in related projects in the Quality Control Laboratory and three PhD students. Knowledge level of the participants regarding the training was assessed through pre-evaluation, before the commencement of the training.

The program was inaugurated at the Extension Education Institute (EEI) Auditorium, with in the campus in which the Quality Control Laboratory is situated. The Chief Guest was Dr. V. Prakash, Distinguished Scientist, CSIR and Ex-Director, CFTRI, Mysore, who also delivered the keynote address. Dr. Anurag Chaturvedi, Dean, Faculty of Home Science, Dr. Mahalakshmi V. Reddy, CAFT Director and Dr. K. Manorama, Course Director were on the Dias. The highlights of his speech are on the effect of climate change on food production, and the relevance of the climatic changes on food safety and food security. He emphasized on avoidance of wastage of food and the importance of value addition of agricultural commodities to overcome economic problems.

He stated that growth of the companies/industry increases with the inclusion of R&D in the system and also indicated the regulatory angle of food quality and its importance. He said that awareness of diet regulation for rural and urban population should be structuralized and that chemistry and biology training is imperative for all professionals involved in food production, testing and safety. The participants and audience were very impressed with the lecture.

Dr. D. Raji Reddy, Director of Research, PJTSAU, delivered a lecture on Climate change and crop quality-Applications of Food testing facilities for crop quality analysis. Details on projected climate change, potential impact of climate change on Food Safety, health, bio-diversity, environmental contaminants, and the impact of chemical residues and Mycotoxin on quality of life and health safety hazards were addressed. Surveillance monitoring and development of mitigation measures were discussed. He concluded by stating that measures exist to achieve the substantial emission reductions required to limit likely warming to 2°C.

Dr. Bhaskara Chary, Scientist D, National Institute of Nutrition spoke about Food Compositional Analysis-A long Experience in analysis and documentation of Food Composition Tables. The detailed protocols followed for edition and preparation of Food Composition Tables supplied by the National Institute of Nutrition in the form of the book on "Nutritive value of Indian Foods", which is to be revised in November 2015, was elaborated upon.

Dr. Anurag Chaturvedi, I/C Dean, Home Science gave a lecture on Food Safety and Quality Assurance. The paradigm shift from an importing country to an exporting country with respect to food production has emphasized the need for quality food production as per export demands. Different categories of food contaminants like physical, chemical and microbial sources of contamination were elaborated upon and methods to control this kind of contamination by quality management were emphasized. ISO 22000 Food Safety Management System was discussed in detail.

Dr. K. Manorama, Principal Scientist, Quality Control Lab, PJTSAU gave an introductory lecture on Food Quality Testing. Need for Food Analysis, reasons for testing of foods, properties to be measured, appropriate analytical techniques for different nutrient parameters, latest research and development with respect to methods used for nutrient analysis were all covered in detail. Standardization of methods, need for validation of new methods or modified methods, were elaborated. Government regulations and recommendations, mandatory standards and nutrition labeling, and importance of accuracy in food analysis with respect to the regulations and labeling, were also discussed.

Dr. K. Aparna, Scientist, Quality Control Laboratory, PJTSAU enlightened the participants about Proximate Analysis with Accuracy and Precision-Theory and Practical. An overview of proximate analysis starting from sample preparation, use of suitable calibrated equipment, adoption standard published methods for analysis of moisture, protein, ash, fat and crude fiber content was elaborated upon. The lecture was followed by hands-on-training and practical on proximate analysis for 2 days.

Dr. K. Manorama, Principal Scientist, QC Lab, PJTSAU explained about Uncertainty of measurement in analysis. Methods of calculation of uncertainty of measurement for proximate principles were taught with detailed calculations that are involved. Identification of sources of uncertainty, terminology used, quantifying uncertainty components, Calculation of probability distribution of variables for uncertainty measurement was demonstrated, with examples.

Dr. Abhay Ekbote, Director, Pesticide Residue Laboratory, National Institute of Plant Health Management gave a detailed lecture on Implementation of ISO-17025 in Food Testing Labs. The lecture covered all aspects of the ISO/IEC 17025-2005 international standard for testing laboratories. The Scope of this International Standard specifies the general requirements for the competence to carry out tests and/or calibrations, including sampling. This International Standard is applicable to all organizations performing tests and/or calibrations. Details of all 25 clauses defined under Management and Technical requirements were elaborated upon.

Dr. V. Sashibhushan, Principal Scientist, All India Network project on Pesticide residue analysis, PJTSAU, delivered a lecture on Gas Chromatography (GC), Principles of Operation and use of GC for pesticide analysis. Sanitary and phytosanitary issues related to Food Safety, export norms with respect to pesticide residues, need for food safety to supplement food security were emphasized. Detailed principle of Gas chromatography and method used for pesticide analysis was explained theoretically and also practically demonstrated.

Under practical sample processing was done with hands-on training to the participants. GC, HPLC, GC-MS-MS and LC-MS-Ms instruments were demonstrated. Basic information on Quadrupole LC-MS-MS was given. Operation of the instruments, mobile and stationary phases, column-oven and temperatures were used, pumps, auto-injectors, communication modules, flow change valves, ESI and APCI sources were all shown and explained in detail.

Dr. M. Sreedhar, Principal Scientist and Mr. Ch. Jagan, Research Associate, QC Lab, PJTSAU ICP-OES elaborated upon Principles of operation-Theory and Practical. Principle of operation of inductively coupled plasma Optical Emission Spectrometry was elaborately discussed after emphasizing on the need for accurate and precise estimations of minerals and heavy metals in raw and processed foods. Complete details of the equipment were explained and method development for mineral and heavy metal analysis was elaborated upon. Software operation in the instrument was demonstrated to smaller groups of participants. Actual analysis was done and results were calculated.

Mr. M. Poshadri, Assistant Professor, Food Science and Technology, College of Food Science and Technology PJTSAU, spoke on the importance of Food Quality and Nutrient Analysis for the Food Processing Industry. The main points of discussion in this lecture were on Quality which was defined as the totality of features relevant to the ability of a product to fulfil its requirements.

Dr. K. Manorama, Principal Scientist and Mr. Ch. Jagan, Research Associate, QC Lab, PJTSAU explained about HPLC-Principles of Operation-Theory and Practical High-performance liquid chromatography (HPLC; formerly referred to as high-pressure liquid chromatography), is a technique in analytical chemistry used to separate, identify, and quantify each component in a mixture.

Use of HPLC for B-complex vitamin analysis with detailed methods were explained theoretically and also demonstrated in the laboratory. Thiamine, riboflavin and niacin were estimated and hands-on experience was given to participants on B-complex vitamin analysis.

Dr. I. Sreenivasa Rao, Professor and Head, Department of Extension Education, PJTSAU took a class on Pedagogy-Non-verbal Communication. Pedagogy training is mandatory for every 21-day training programme.

Dr. Veeranjaneyulu, University Librarian, PJTSAU, enlightened the participants about available E- Resources for Agriculture and Allied Sciences. Different websites, login i.ds, and methods of accessing websites for journal articles, books and other web based learning User i.ds and gateways for accessing various websites were shared with participants.

Dr. M. Sreedhar, Principal Scientist, Qc Lab, PJTSAU, spoke about the use of Gamma Irradiation chamber for improving shelf life of foods and seed viability for germination and Practical included demonstration of the chamber.

The speaker explained about GC 5000, and its current dose rate 1.76KGY/hr and its design. The gamma irradiation chamber was made by BRIT (Board of Radiation Isotopic Technology).

Dr. Sneha Gogte, Professor and HOD, St. Ann's College for Women, gave a lecture on Microbial Quality Analysis in Food grains and processed food testing. The Scheme of Presentation of the lecture was as follows: Biometrics, Food from Microbiologist's point of view, Constant interaction of plants, animals and microbes, Food Categories as Microbiologist view, some lessons from nature and facts and figures of microbes-man-food- associations, methods of testing including conventional and modern methods.

Mr. Y. Sudhakar, Vice President, First Source Laboratories, Hyderabad, during the field visit to their laboratory, spoke on the role of Laboratories in Food Safety Systems. The laboratory is accredited as per ISO/IEC: 17025:2005 and complies with the good laboratory practices to meet the current Global regulatory requirements (NABL, APEDA, FSSAI, and GAFTA). He also shared his experiences in food safety monitoring systems in India.

Dr. K. Manorama explained on the functioning of gas chromatography for the detection of GC is F1D for fatty acids. Organic compounds on flame ionization detector were detected as fatty acid methyl esters. The organic compounds which are volatile were the fatty acid content was measured based on area normalization.

Dr. Satish Yadav, Sr. Scientist, NIPHM, gave a lecture on NABL Accreditation-Process and requirements. He explained about Accreditation Process, Preparation of Laboratory for Accreditation which includes Application to Accreditation, Maintenance of Accreditation like Conformance to applicable standards & NABL Requirements, NABL Terms and Conditions, Modification to Accreditation criteria, Adverse decision against the laboratories Surveillance & Renewal of Accreditation and Appeals & Complaints.

Dr. C. Damodar Raju, Sr. Scientist, Rice Research Centre, PJTSAU, delivered a lecture on Application of Food Quality Testing in Crop quality Improvement-Rice. The speaker described the research work on breeding for crop improvement being conducted at Rice Research Station, PJTSAU. His lecture was an eye opener to all food scientists that any varieties produced by scientist will not be accepted by people until they meet the food quality requirements.

Dr. S. Vanisree, Pr. Scientist, Institute of Biotechnology, PJTSAU, continued the above topic on Application of Food Quality Testing in Crop quality Improvement-Rice. The speaker explained about the appearance of rice grain, layers of rice grains, physical characteristic of rice grains and chemical properties of rice grain which are important to determine before submitting release proposals for any newly developed varieties of food grains. Different properties were described, including Gelatinization, gel consistency, chalkiness and translucency, water uptake ratio etc. She also explained about bio-fortification of rice giving example of "Golden rice".

Dr. C. V. Rao, Senior Scientist, NIPHM, Hyderabad, gave a lecture on Documentation required for NABL assessment. He spoke about the requirements for accreditation laid down in the International Standard ISO/IEC17025:2005, documents to be maintained, quality and technical requirements like personnel, environment and accommodation conditions, space, design, health and safety, validation, equipment, laboratory information management systems, sampling, sample preparation, sample retention and storage, reagents, physical standards, calibration and measurement of traceability.

Dr. G. Nageswara Rao, Professor of statistics, College of Home Science, PJTSAU, Hyderabad, explained about the statistical methods and sampling methods.

#### Visit of ICAR expert nominee:

The ICAR nominee, Dr. Shailaja Naik, Dean, Faculty of Home Science, Dharwad, visited the CAFT training programme for two days on 21<sup>st</sup> and 22<sup>nd</sup> September 2015. During the session, discussion was carried out with the participants and the final outputs of the discussion were noted under the categories like strengths, weaknesses, and opportunities for improvement.

### **Evaluation of the Training:**

Evaluation of participants was conducted before and after the conclusion of the training programme. Participants were provided with the pre and post evaluation schedule, to assess the existing knowledge and knowledge gained through the 21 days training on "New dimensions of Food Quality Analysis towards achieving Comprehensive Food Safety". Clearly there was substantial difference in the test scores of the participants between the pre and post evaluation. Participant feedback on the training program too was obtained and most sessions were rated as either excellent or very good. They also stated that the topics covered were very useful to all the participants. Few suggestions such as inclusion of more group discussion and project proposal preparation, were suggested by few participants.

### Valedictory:

The training program was concluded with the valedictory function on 23<sup>rd</sup> September 2015, at the University Library Committee Hall of PJTSAU. The chief guest was Dr. V. Praveen Rao, Special Officer, Professor Jayashankar Telangana State Agricultural University. Dr. D. Raji Reddy, Director of Research, presided over the function. Dr. Anurag Chaturvedi, Dean of Home Science was also present Dr. Mahalakshmi V. Reddy, CAFT-Home Science, Director welcomed the gathering. Dr K. Manorama, Course Director gave a brief report on all the activities carried out during the training program. Dr. V. Praveen Rao, Special Officer, PJTSAU, released the CDs of 21 days training program and the CAFT Home Science Newsletter. Dr. Anurag Chaturvedi, Dean, Faculty of Home Science, Hyderabad addressed the gathering about the importance of Food Safety and Quality Assurance. Dr. Raji Reddy spoke about the various activities of the training programme and commended the organizers. The chief guest spoke about the need for group discussions, preparation of project proposals in the training. He focused on the participants stating that they are the ambassadors in their respective universities to promote this kind of training. After the speech, certificates were distributed by the chief guest to all the participants.

#### About the CAFT – Home Science Training programme -2015-16

**Title:** "New Dimensions in Food Quality Analysis towards Achieving Comprehensive Food Safety" **Date:** 03-09-2015 to 23-09-2015

## **Training Concept:**

The paradigm shift in Indian agriculture witnessed in case of food commodities from quantity to quality is a welcome phenomenon in the context of present World order. The rapid growth in allied sectors like diary, poultry and fisheries has further consolidated India's position as a nation with marketable surplus. Consequently our country has transitioned from production economy to market economy encompassing growth, productivity, diversity, profitability and quality assurance.

This transformation has led to a spurt in processing activities of food commodities at various levels leading to free availability of processed food at affordable cost. The pivotal role of "Testing Laboratories" in ensuring high quality standards of food material has become much more relevant than ever before.

Accuracy and precision in food analysis using ISO/IEC 17025:2005 system is mandatory in today's changing scenario of food processing. Information on safety standards of food is crucial, especially when people are consuming varieties of foods like ready-to-eat, processed, fortified, functional foods, dietary supplements and Nutraceuticals for improving export potential. Consumers need information on ingredients, nutritional value and safety.

Training in newer methods of precision based food testing for faculty in Agricultural Universities involved in food production and processing is imperative. In pursuance with the vision for establishment of efficient Food Quality Control System, the PJTSAU had prioritized setting up of state-of-art, accredited Food Quality Testing Facility to assist institutions and entrepreneurs in fulfilling the requirements for implementation of international standards.

A well equipped Laboratory with high end analytical instruments functioning on internationally accepted protocols is under operation at the University's Head Quarter. Essential infrastructural facilities are placed over the work space of 6000S.ft. with well furnished Instrumentation lab rooms, uninterrupted power supply to support all the equipment. Technical capabilities of the Lab include state – of – the art Liquid and Gas Chromatography, Optical Emission Spectrometry, microbiological clean room facility, validated proximate profiling, done using Certified Reference Materials with highest traceability standards. Besides this, fully qualified and trained testing staffs are manning the Lab activities under the expert supervision of Scientists.

## **Objectives:**

- To provide training to faculty in GLP, food quality control with special reference to HACCP, SPS and general certification procedures, quality assurance, qualitative and quantitative methods of analysis.
- To impart training to faculty of Agricultural Universities to develop and upgrade skills pertaining to total quality management in agriculture and food industry.

## **Course Content:**

The main topics that were planned to be covered by theory and practical sessions are:

- ✤ Introduction to ISO/IEC 17025:2005
- Food Safety and Quality Assurance
- Climate change and crop quality-Applications of Food testing facilities for crop quality analysis
- ♦ Implementation of ISO/IEC 17025:2005 in Food Testing Labs.
- GLP and Familiarization and practice with instrumentation and operation of instruments at QC lab.
- Gas Chromatography (GC), Principles of operation-Use of GC for pesticide analysis and fatty acid estimation
- ♦ HPLC-Principles of operation, Use of HPLC for B-complex vitamin Analysis.
- Repeatability, Reproducibility and Measure of uncertainty in food testing.
- Use of Gamma Irradiation chamber for improving shelf life of foods and seed viability for germination
- Microbial Quality Analysis in Food grains and processed food testing
- Food Compositional Analysis analysis and documentation of Food Composition Tables
- ✤ ICP-OES-Principles of operation
- ✤ Application of Food Quality Testing in Crop quality Improvement.
- Privatization of Food testing Labs-experiences of private lab owners.

## Methodology:

The faculty will have interactive sessions with the teaching faculty of the course. The training will be in the form of lectures by experts followed by intensive laboratory training in all novel methods of food analysis including handling and use of ICP, HPLC, GC, and all equipment for

proximate analysis. Group discussions and projects involving planning of laboratory experiments as well as converting the existing laboratories to ISO 17025-2005 standards will be planned.

## Benefits for the participating organizations:

The participants will be able to get on-hands experience in latest analytical tools for estimation of various parameters like proximate principles, vitamins, minerals, pesticide residues, using ISO/IEC-17025-2005 system based protocols. They will be given knowledge regarding establishment of Food Testing Laboratories having ISO/IEC-17025-2005 standards, which can be used for upgrading the testing facilities in their laboratories as well as establishment of new laboratories with external funding. GLP will be inculcated in them for good quality control with respect to food/crop analysis.

## LIST OF PARTICIPANTS

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## LIST OF DROP-OUTS

S. No.	Name of Designation and Name		Contact details	Reasons for Drop out	
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1.	Mrs. Renu Arya	SMS, KrishiVigyan Kendra near Kisan Degree College, Bahraich	09415046343 renupau@gmail.com	No permission from University	
2.	Dr. Pratiksha Singh	SMS, Gurujan Vihar, Gandhi Vidyamandir, Sardarshahr, Churu- 331403	09982597404 pratifrm@gmail.com	No permission from University	
3.	Mr. Ashish Khandelwal	Assistant Professor, Soil Science and Agricultural Chemistry, Bihar Agricultural College, BAU, Sabour. PIN 813210	07050633009 ashish.iasbhu@gmail.co m	Applied too late-No permission from University on time.	
4.	Ms.Varsha Ramachndra Gaikwad	Assistant Professor Soil Science & Agril. Chemistry College of Agriculture, Pune- 411005 (M.S.)	09890561254 vrgaikwad27@rediffmail. com	No permission from University	
5.	Mr. Srinivasa Rao Pidugu	Others, Food Science & Technology, ICAR- Directorate of Poultry Research (DPR), Hyderabad	08106035384 srinivasaraopidugu105@ gmail.com	No information	
6.	Dr. G I Hassan	Assistant Professor, Horticultural-Fruit Science, Sher-e- Kashmir University of Agricultural Sciences & Technology of Kashmir (SKUASTK), Srinagar	09596314268 rasibhassan123@yahoo.c om	No permission from University	
7.	Mrs. P. Jayamma	Assistant Professor, Food Microbiology, College of Food Science and Technology, Pulivendala	-	Personal reason	
8.	Dr. Charith Kumar,	Department of Agri process and Food Engineering, College of Agricultural Engineering & Technology, Aswaraopet, PJTSAU	-	No permission from Associate Dean due to shortage of staff	



28<sup>TH</sup> CAFT-HOME SCIENCE TRAINING ON NEW DIMENSIONS IN FOOD QUALITY ANALYSIS TOWARDS ACHIEVING COMPREHENSIVE FOOD SAFETY FROM 3<sup>RD</sup> -23<sup>RD</sup> SEPTEMBER 2015

## ORGANIZING COMMITTEE

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2.	Dr. K. Manorama Course Director	+91 – 92461 12225 makanuri@gmail.com	Course Director
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4.	Dr. Mulinti. Sreedhar Course Co Director	+91- 99494 37035 Mulisree1969@gmailcom	Technical
5.	Dr. K. Aparna Scientist QC Lab	+91- 98490 19823 aparnakuna@gmail.com	Technical, Food & accommodation
7.	Mrs. Bhavani M Paragi Research Associate	+91 - 70329 02006 bhvn0620@gmail.com	Transport & documents Management
8.	Mrs. Chakilam Sunitha Computer operator	+91 - 77025 96555 ch.kalpesh@gmail.com	Finance & coordination
9.	Mr. Laxman Technical Assistant	+91 - 7793958903	Class room arrangements

## LIST OF RESOURCE PERSONS

Sl.No	Name and Designation of	Phone/Mobile	E-mail I.D.
	Speakers	No.	
1	Dr. V. Prakash,	9845048854	prakashvish@gmail.com
	Ex-Director, CFTRI,		
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2	Dr. Mahalakshmi V. Reddy,	9849047906	Mahalakshmi.v.reddy@gmail.com
2	CAFI (Home Science) Director	0090625220	da aitaan Quahaa aan
3	Dr. D. Raji Reddy, Director of	9989625220	dr.pjtsau@yanoo.com
1	Dr. Bhaskara Chary	0080602767	bhaskarkc@hotmail.com
-	Scientist D National Institute of	JJ8J0J2707	bhaskarke @ notman.com
	Nutrition		
5	Dr. Anurag Chaturvedi, I/C	9989625202	anuragchaturvedi1955@gmail.com
	Dean, Home Science		
6	Dr. K. Manorama	9246112225	makanuri@gmail.com
	Pr. Scientist and Head, QC Lab		
7	Dr. K. Aparna	9849019823	aparnakuna@gmail.com
	Scientist, QC Lab		
8	Dr. Abhay Ekbote,	9493983991	dirpmniphm-ap@nic.in
	Pr. Scientist, NIPHM;		
9	Mr. Ch. Jagan; Research	9866425908	charlapallyjagan@gmail.com
10	Associate, QCL		
10	Mrs. N. Prathima; ; Research	9391167588	prathima.namburi@gmail.com
	Associate, QCL		
11	Dr. I. Sreenivasa Rao, Professor	9492927422	amala_puthota@yahoo.com
	and Head, Department of		
	Extension Education, College of		
	Agriculture, PJTSAU, Hyd.		
12	Dr. V. Sashibhushan, Pr.	7702688891	sash_3156@yahoo.co.in
10	Scientist and Head, PRL	9849928831	
13	Dr. M. Sreedhar, Pr. Scientist,	9949437035	mulisree1969@gmail.com
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14	Dr. A. Poshadri,	9492828965	poshadri_fst@yahoo.co.in
	Assistant Professor, Food		
	of Food Science and Technology		
	PITSAU		
15	Dr. Sneha Gogte, Head.	9346743021	gogtesneha@gmail.com
10	Microbiology Department, St.	2010710021	So Browner of Browner of the
	Ann's College, Hyderabad		
16	Mr. Y. Sudhakar, Vice President,	040-27174037	sudhakar@firstsourcels.com
	First Source Laboratories,		
	Nacharam, Hyderabad.		
17	Dr. C. Damodar Raju, Pr.	9440225385	cdraju2008@gmail.com
	Scientist, RRC, PJTSAU		

18	Dr. S. Vanisree, Sr. Scientist, Institute of Biotechnology, PJTSAU	9491161067	vanisree_dhar@yahoo.com
19	Dr. Satish Yadav; Sr. Scientist, NIPHM;	9704514614	satish1208@rediffmail.com
20	Mr. C.V. Rao; Sr. Scientist, NIPHM;	9490380048	cvrao25@gmail.com
21	Dr. G. Nageswara Rao Retd. Professor of Statistics, College of Home Science, PJTSAU, Hyderabad	9908183794	gadirajur@gmail.com

## TRAINING SCHEDULE

## Schedule of daily lectures/practical topics and Speakers

Day	Date	Time	Topic of lecture	<b>Designation of Speaker</b>
1	03-09-2015	9.30 a.m.	Registration	
		11.30 a.m. to 1.00 p.m.	Inauguration and Keynote address	Dr. V. Prakash, Ex-Director, CFTRI
		1.00 p.m. to 3.00	Lunch break and interaction with Chief Guest	
		3.00 p.m. to 4.00 p.m.	Introduction about CAFT (Home Science)	Dr. Mahalakshmi V. Reddy, CAFT (Home Science) Director
2	04-09-2015	9.30 a.m.	Climate change and crop quality-Applications of Food testing facilities for crop quality analysis.	Dr. D. Raji Reddy, Director of Research, PJTSAU
		11.30 a.m.	Food Compositional Analysis-A long Experience in analysis and documentation of Food Composition Tables	Dr. Bhaskara Chary Scientist D, National Institute of Nutrition
		2.00 p.m.	Pre-testing and evaluation of trainees	Dr. K. Aparna, Assistant Professor, PGRC, PJTSAU
		4.00 p.m.	Visit to Millet Processing and Incubation Centre	
3	05-09-2015	9.30 a.m.	Food Safety and Quality Assurance	Dr. Anurag Chaturvedi, I/C Dean, Home Science
		11.30 a.m.	Introduction to Food Quality Testing	Dr. K. Manorama Pr. Scientist, QC Lab
		2.00 p.m.	Proximate Analysis with Accuracy and Precision	Dr. K. Aparna Assistant Professor, PGRC
4	06-09-2015	SUNDAY-HOLIDAY		ł
5	07-09-2015	9.30 a.m.	Uncertainty of measurement	Dr. K. Manorama
		11.30 a.m. to 4.30 p.m.	Practical on proximate analysis	Mr. Ch. Jagan and Mrs. N. Pratima.
6	08-09-2015	9.30 a.m. to 1.00 p.m.	Implementation of ISO-17025 in Food Testing Labs	Dr. Abhay Ekbote, Pr. Scientist, NIPHM;
		2.00 p.m.	Visit to State Food Laboratories	Coordinators

	(FSSAI) Nacharam				
7	09-09-2015	9.30 a.m. to	Gas Chromatography (GC),	Dr. V. Sashibhushan, Pr.	
		1.00 p.m.	Principles of Operation and use	Scientist and Head, PRL	
		1	of GC for pesticide analysis.	<i>,</i>	
			1 7		
		2.00 p.m.	Practical on use of GC for	Mr. Ch. Jagan, Research	
		1	pesticide residue analysis	Associate, QCL	
8	10-09-2015	9.30 a.m. to	Practical on use of GC for	Staff of Pesticide Residue	
		4.00 p.m.	pesticide residue analysis	analysis Laboratory	
9	11-09-2015	9.30 a.m.	ICP-OES-Principles of operation	Dr. M. Sreedhar, Sr.	
				Scientist, QCL	
		11.30 a.m.	Practical on use of ICP-OES for	Mr. Ch. Jagan, Research	
		to 4.30 p.m.	mineral and heavy metal	Associate, QCL	
			analysis.		
			•		
10	12-09-2015	9.30 a.m. to	Introduction to use of GC for	Dr. K. Manorama, Pr.	
		10.00 a.m.	Fatty Acid Analysis	Scientist, QCL	
		10.00 a.m.	Practical on use of GC for fatty	Mr. Ch. Jagan, Research	
		to 4.00 p.m.	Acid analysis	Associate, QCL	
11	13-09-2015	0.00	SUNDAY-HOLIDAY		
12	14-09-2015	9.30 a.m. to	Importance of Food Quality and	Dr. A. Poshadri,	
		11.00 a.m.	Nutrient Analysis for the Food	Assistant Professor, Food	
			Processing Industry	Science and Technology,	
				College of Food Science	
		11.20	UDLC Drinsiples of energyion	and Technology, PJISAU	
		11.30 a.m.	HPLC-Principles of operation	Dr. V. Monoromo, Dr.	
		10 12.50		Dr. K. Manorama, Pr.	
		p.m.		Scientist, QCL	
		2.00  to  4.00	Practical on Use of HPLC for B-		
		2.00 to 4.00	complex vitamin Analysis	Mr Ch Iagan Research	
		p.m.	complex vitamin / marysis	Associate OCL	
13	15-09-2015	930 a m to	Hands-on experience on use of	Mr. Ch. Jagan	
15	10 09 2010	1.00 p.m.	HPLC for B-Complex Vitamin	ivii. Cii. sugui	
		1.00 p	Analysis		
		2.00 to 4.00	Pedagogy-Non-verbal	Dr. I. Sreenivasa Rao.	
		p.m.	Communication	Professor and Head,	
		1		Department of Extension	
				Education, College of	
				Agriculture, PJTSAU	
14	16-09-2015	9.30 a.m.	E- Resources for Agriculture and	Dr. K. Veeranjaneyulu,	
			Allied Sciences	University Librarian,	
		11.30 a.m.	Use of Gamma Irradiation	Dr. M. Sreedhar, Sr.	
			chamber for improving shelf life	Scientist, QCL	
			of foods and seed viability for		

			germination.	
		2.00 p.m.	Demonstration of Gamma Irradiation chamber.	Dr. M. Sreedhar and Ms. Sandhya Rani
15	17-09-2015	9.30 a.m. to 4.00 p.m.	Project work on planning of set Lab.	tting up of a Food Testing
16	18-09-2015	9.30 a.m.	Microbial Quality Analysis in Food grains and processed food testing	Dr. Sneha Gogte, Head, Microbiology Dept, St. Ann's College, Hyderabad
		11.30 a.m. to 4.00 p.m.	Visit to First Source Laboratories, Nacharam, Hyderabad Role of Laboratories in Food Safety	Mr. Y. Sudhakar, First Source Laboratories, Nacharam
17	19-09-2015	9.30.a.m. to	Practical on Microbial Quality	Mrs. N. Prathima
18	20.09.2015	4.00 p.m.	SUNDAY HOLIDAN	V
10	20-09-2015	9 30 a m	NABL Accreditation-Process	Dr. Satish Yaday:
17	21 09 2015	7.50 a.m.	and requirements	Sr. Scientist, NIPHM;
		11.30 a.m.	Application of Food Quality Testing in Crop quality Improvement-Rice-I	Dr. C. Damodar Raju, Sr. Scientist, Rice Research Centre, PJTSAU
		2.00 p.m.	Application of Food Quality Testing in Crop quality Improvement-Rice-II	Dr. S. Vanisree, Sr. Scientist, Institute of Biotechnology, PJTSAU
20	22-09-2015	9.30 a.m.	Interaction with ICAR Expert Reviewer	Dr. Shailaja Naik, Dean, Faculty of Home
		11.30 a.m.	Documentation for NABL Accreditation	Mr. C.V. Rao, Sr. Scientist, NIPHM;
		2.00 p.m.	Statistical Methods and Sampling	Dr. G. Nageswara Rao, Retd. Professor of Statistics, College of Home Science, PJTSAU
		4.30 p.m.	Post-Evaluation	Dr. K. Aparna, Scientist, QC LAB, PJTSAU
21	23-09-2015	11.00 a.m.	Valedictory Function	Special Officer, PJTSAU and Director of Research, PJTSAU
		2.00 p.m.	Payment of TA bills etc.	

## DAY-TO-DAY REPORT ON TRAINING

## (RAPPORTEUR'S REPORT)

# A. Keynote address at Inaugural Function by Dr. V. Prakash, Distinguished Scientist, CFTRI; 03-09-2015

### **Key points:**

## <u>Title: Role of Chemistry and Biology in Food Safety Net; For a sustainable dimension of Food</u> <u>Quality.</u>

## He covered the following points in his address-

The highlights of his speech are summarized as below:

- 1. Effect of climate change on food production.
- 2. The relevance of the climatic changes on food safety and food security.
- 3. Farmer's role in deciding to market their produce.
- 4. Effect of climatic change is not only affecting the agriculture and it also affects the health of the people.
- 5. Emphasis on Avoidance of wastage of food.
- 6. The value addition of agricultural commodities to overcome the economic problem.
- 7. Growth of the companies/industry increases with the inclusion of R&D in the system.
- 8. Regulatory angle of food quality is very important.
- 9. Awareness of diet regulation for rural and urban population should be structuralized.
- 10. Chemistry and Biology training is imperative for all professionals involved in food production, testing and safety.

He concluded address with discussion on issues related to child mortality, nutritional deficiency, food insecurity, in a country where self sufficiency and stability in food production has been reached but a lack of awareness and improper channelizing of the food system is lacking. He suggested popularization of the quote "Banana a day keeps health good", rather than an apple a day.

Other speakers at the inaugural session were Dr. Anurag Chaturvedi, Dean, Faculty of Home Science, Dr. Mahalakshmi V. Reddy, CAFT Director and Dr. K. Manorama, Course Director. Dr. M. Sreedhar proposed the Vote of Thanks.



CAFT-HOME SCIENCE 21 DAYS TRANING INAUGURATION BY PADMASHREE DR V PRAKASH

## Climate change and crop quality-Applications of Food testing facilities for crop quality analysis by Dr. D. Raji Reddy, Director of Research, PJTSAU 04-09-2015

A very in-depth talk was delivered by the Director of Research on Climate change and its impact on Food Safety. Details on projected climate change, potential impact of climate change on Food Safety, health, bio-diversity, environmental contaminants, and the impact of chemical residues and Mycotoxin on quality of life and health safety hazards were addressed. Surveillance monitoring and development of mitigation measures were discussed. He concluded by stating that measures exist to achieve the substantial emission reductions required to limit likely warming to  $2^{\circ}$ C (40-70% reduction in GHGs globally by 2050 and near zero GHGs in 2100), and that a combination of adaptation and substantial, sustained reductions in greenhouse gas emissions can limit climate change risks. Implementing reductions in greenhouse gas emissions poses substantial technological, economic, social, and institutional challenges, and delaying mitigation will substantially increase the challenges associated with limiting warming to  $2^{\circ}$ C.



Food Compositional Analysis-A long Experience in analysis and documentation of Food Composition Tables by Dr. Bhaskara Chary, Scientist D, National Institute of Nutrition

During this lecture, important nutritional aspects of foods were covered, elaborating upon the various nutrients and their role in maintenance of health and well being and taking care of lifestyle changes. The detailed protocols followed for edition and preparation of Food Composition Tables supplied by the National Institute of Nutrition in the form of the book on "Nutritive value of Indian Foods", which is to be revised in November 2015, was elaborated upon. He conveyed the information that Completely new database of all foods including cooked and processed foods was being prepared for 86 nutrients and bioactive substances of all foods. Database on the antioxidant activities of foods were also being included in the new edition of the book. Modern validated

methods of analysis are being used to create the database and the launch of Indian food composition database website was also being planned by the end of the year 2015. This lecture emphasized the importance of Food Analysis in the nation's Food and Nutrition Security.



Food Safety and Quality Assurance by Dr. Anurag Chaturvedi, I/C Dean, Home Science; 05-09-2015

A detailed lecture was delivered on Food Safety and assurance, starting with a background on Food Safety as per the consumer's demand for quality foods. Food sufficiency should be accompanied by quality food production. The paradigm shift from an importing country to an exporting country with respect to food production has emphasized the need for quality food production as per export demands. Different categories of food contaminants like physical, chemical and microbial sources of contamination were elaborated upon and method to control these kinds of contamination by quality management was emphasized. ISO 22000 Food Safety Management System was discussed in detail. **ISO 22000** is a Food Safety Management System that can be applied to any organization in the food chain, farm to fork. Becoming certified to ISO 22000 allows a company to show their customers that they have a food safety management system in place. This provides customer confidence in the product. This is becoming more and more important as customers demand safe food and food processors require that ingredients obtained from their suppliers to be safe. ISO and its member countries used the Quality Management System approach, and tailored it to apply to Food Safety, incorporating the widely used and proven HACCP principles and Good Manufacturing Principles (addressed by Prerequisite Programs in ISO 22000. Enhancement of Food Safety norms as per Codex standards for Food Additives, contaminants, methods of analysis, sampling methods, export-import system, HACCP, FSSAI and its objectives, were all covered in detail.

Introduction to Food Quality Testing by Dr. K. Manorama, Principal Scientist, Quality Control Lab, PJTSAU; 05-09-2015



Need for Food Analysis, reasons for testing of foods, properties to be measured, appropriate analytical techniques for different nutrient parameters, latest research and development with respect to methods used for nutrient analysis were all covered in detail. Standardization of methods, validation of methods, need for validation of new methods or modified methods, were elaborated. Government regulations and recommendations, mandatory standards and nutrition labeling, and importance of accuracy in food analysis with respect to the regulations and labeling, were also discussed. Quality control at various stages of food production from raw material to processing, and the need for analysis at these different stages was emphasized. One of the most important reasons for analyzing foods from both the consumers and the manufacturer's standpoint is to ensure that they are safe. It would be economically disastrous, as well as being rather unpleasant to consumers, if a food manufacturer sold a product that was harmful or toxic.

Food analysis is the discipline dealing with the development, application and study of analytical procedures for characterizing the properties of foods and their constituents. These analytical procedures are used to provide information about a wide variety of different characteristics of foods, including their composition, structure, physicochemical properties and sensory attributes. This information is critical to our rational understanding of the factors that determine the properties of foods, as well as to our ability to economically produce foods that are consistently safe, nutritious and desirable and for consumers to make informed choices about their diet. The objective of this course is to review the basic principles of the analytical procedures commonly used to analyze foods and to discuss their application to specific food components, e.g. lipids, proteins, water, carbohydrates and minerals.

Proximate Analysis with Accuracy and Precision-Theory and Practical by Dr. K. Aparna, Scientist, Quality Control Laboratory, PJTSAU; 05-09-2015.



An overview of proximate analysis starting from sample preparation, use of suitable calibrated equipment, adoption standard published methods for analysis of moisture, protein, ash, fat and crude fiber content was elaborated upon. Detailed methodology to be adopted, precautions to be taken, methodology followed for equipment calibration and intermediate checks, quality control plans for assuring accurate results, documentation required including developing of a Standard Operating Procedure (SOP), detailed Working Instructions for each equipment, Flow charts for recording actual analysis for each sample, were all discussed in detail.

The lecture was followed by hands-on-training on practical on proximate analysis for 2 days.

# Uncertainty of measurement by Dr. K. Manorama, Principal Scientist, QC Lab, PJTSAU; 07-09-2015. Practical on Proximate analysis was conducted the remaining part of the day.

Methods of calculation of uncertainty of measurement for proximate principles were taught with detailed calculations that are involved. Identification of sources of uncertainty, terminology used, quantifying uncertainty components and calculation of probability distribution of variables for uncertainty measurement were demonstrated, with examples.

# Implementation of ISO-17025 in Food Testing Labs by Dr. Abhay Ekbote, Director, Pesticide Residue Laboratory, National Institute of Plant Health Management, Hyderabad; 08-09-2015

The lecture covered all aspects of the ISO/IEC 17025-2005 international standard for testing laboratories. The Scope of this International Standard specifies the general requirements for the

competence to carry out tests and/or calibrations, including sampling. It covers testing and calibration performed using standard methods, non-standard methods, and laboratory-developed methods. This International Standard is applicable to all organizations performing tests and/or calibrations. These include, first, second and third-party laboratories, and laboratories where testing and/or calibration forms part of inspection and product certification. This Interactional Standard is 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 International Standard, such as sampling and the design/development of new methods, the requirements of those clauses do not apply. The Standard is 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. Compliance with regulatory and safety requirements on the operation of laboratories is not covered by this International Standard. If testing and calibration laboratories comply with the requirements of this International Standard, they will operate a quality management system for their testing and calibration activities that also meets the principles of ISO 9001. This Standard covers technical competence requirements that are not covered by ISO9001.

Details of all 25 clauses defined under Management and Technical requirements were elaborated upon.



Gas Chromatography (GC), Principles of Operation and use of GC for pesticide analysis, by Dr. V. Sashibhushan, Principal Scientist, All India Network project on Pesticide residue analysis, PJTSAU, Hyderabad; 09-09-2015



Sanitary and phytosanitary issues related to Food Safety, export norms with respect to pesticide residues, need for food safety to supplement food security, were emphasized. Technical and regulatory aspects of pesticide residues in the light of modern globalization era, pesticide consumption and the ground reality of pesticide residue problem that exists in India elaborated upon. The need for a wholesome approach involving all stakeholders directly or indirectly involved in food production, implementation of acceptable practices like GAP, GMP, to reduce contamination with pesticide residues was emphasized. Involvement and steps taken by ICAR and GOI in reducing pesticide usage was discussed. Decontamination procedures for pesticide removal were given. The need to synchronize food production, storage and distribution to prevent residue contamination was discussed.

Detailed principle of Gas chromatography and method used for pesticide analysis was explained theoretically and also practically demonstrated. Sample processing was done with handson training to the participants. GC, HPLC, GC-MS-MS and LC-MS-Ms instruments were demonstrated. Basic information on Quadrupole LC-MS-MS was given. Operation of the instruments, mobile and stationary phases, column-oven and temperatures used pumps, autoinjectors, communication modules, flow change valves, ESI and APCI sources were all shown and explained in detail. All activities from sample preparation, injection to detection, separation and quantification of residues were demonstrated. Sample preparation for GC-MS-MS was done group wise. ICP-OES-Principles of operation-Theory and Practical by Dr. M. Sreedhar, Principal Scientist and Mr. Ch. Jagan, Research Associate, QC Lab, PJTSAU, Hyderabad; 11-09-2015



Principle of operation of inductively coupled plasma Optical Emission Spectrometry was elaborately discussed after emphasizing on the need for accurate and precise estimations of minerals and heavy metals in raw and processed foods, also in the light of the "Maggi" controversy. Heated atoms absorb and emit light which is specific to the characteristics of each atom. The plasma is produced by the interaction of an intense magnetic field (produced by radio frequency [rf] passing through a copper coil on a tangential flow of gas (normally argon), at about 15 L/min flowing through a concentric quartz tube (torch). This ionizes the gas and, when seeded with a source of electrons from a high-voltage spark, forms a very high temperature plasma discharge (~10,000 K) at the open end of the tube. In ICP-OES, the plasma, usually oriented vertically, is used to generate photons of light by the excitation of electrons of a ground-state atom to a higher energy level. When the electrons "fall" back to ground state, wavelength-specific photons are emitted that are characteristic of the element of interest. In ICP-MS, the plasma torch is positioned horizontally, and is used to generate positively charged ions rather than photons. Complete details of the equipment were explained and method development for mineral and heavy metal analysis was elaborated upon. Software operation in the instrument was demonstrated to smaller groups of participants. Actual analysis was done and results were calculated.

Importance of Food Quality and Nutrient Analysis for the Food Processing Industry by Dr. M. Poshadri, Assistant Professor, Food Science and Technology, College of Food Science and Technology PJTSAU, Hyderabad; 14-09-2015.

The main points of discussion in this lecture were on Quality which was defined as the totality of features relevant to the ability of a product to fulfil its requirements. **Food quality** is the

quality characteristics of food that is acceptable to consumers The concept of food quality should be consider the different demands of the manufacturer & consumer, the surveillance and the legislative bodies and the economic and ecological & issues associated with food quality. Different factors contributing to food quality were discussed in detail. The benefits of nutrient analysis to the Food Industry were elaborated upon as providing better quality check, increasing Product consistency, easy processing and formulations, better preservation, providing ease of marketing and distribution tasks, increasing yearly availability of many foods, enables transportation of foods across long distances, reduces the incidence of food borne disease, improving the quality of life for people with allergies, diabetics, and other people who cannot consume some common food elements and enabling better food fortification. Each nutrient parameter and its importance in each food matrix was elaborately discussed. Private enterprise recognizes that success is measured in terms of its profitability and this in turn is determined by consumer satisfaction. This is again dependent upon uniformity of products and maintenance of standards. It is therefore necessary to establish controls that ensure product uniformity and quality. A number of food control issues are currently being debated upon at the National and International level with respect to pathogenic microorganisms, allergens, GMOs, contaminants, irradiation and nutrition labelling. Finally, marketing challenges that contribute to the need for quality food and assessment of quality in terms of nutrients were listed out as Globalisation, New distribution strategies, New Processing Technologies, New Materials, New Risks, Climate Change, Energy Efficiency, Water Supply, Greed- Fraud.



HPLC-Principles of Operation-Theory and Practical by Dr. K. Manorama, Principal Scientist and Mr. Ch. Jagan, Research Associate, QC Lab, PJTSAU, Hyderabad; 14-09-2015.

I

High-performance liquid chromatography (HPLC; formerly referred to as high-pressure liquid chromatography), is a technique in analytical chemistry used to separate, identify, and
quantify each component in a mixture. It relies on pumps to pass a pressurized liquid solvent containing the sample mixture through a column filled with a solid adsorbent material. Each component in the sample interacts slightly differently with the adsorbent material, causing different flow rates for the different components and leading to the separation of the components as they flow out the column. HPLC has been used for medical (e.g. detecting vitamin D levels in blood serum), legal (e.g. detecting performance enhancement drugs in urine), research (e.g. separating the components of a complex biological sample, or of similar synthetic chemicals from each other), and manufacturing (e.g. during the production process of pharmaceutical and biological products) purposes. Chromatography can be described as a mass transfer process involving adsorption. HPLC relies on pumps to pass a pressurized liquid and a sample mixture through a column filled with a sorbent, leading to the separation of the sample components. The active component of the column, the sorbent, is typically a granular material made of solid particles (e.g. silica, polymers, etc.), 2–50 micrometers in size.

The components of the sample mixture are separated from each other due to their different degrees of interaction with the sorbent particles. The pressurized liquid is typically a mixture of solvents (e.g. water, acetonitrile and/or methanol) and is referred to as a "mobile phase". Its composition and temperature play a major role in the separation process by influencing the interactions taking place between sample components and sorbent. These interactions are physical in nature, such as hydrophobic (dispersive), dipole-dipole and ionic, most often a combination. Normal and reversed phase flow can be used, where either polar or non-polar mobile phase is combined with polar or non-polar column material. HPLC is distinguished from traditional ("low pressure") liquid chromatography because operational pressures are significantly higher (50–350 bar), while ordinary liquid chromatography typically relies on the force of gravity to pass the mobile phase through the column. Due to the small sample amount separated in analytical HPLC, typical column dimensions are 2.1-4.6 mm diameter, and 30-250 mm length. Also HPLC columns are made with smaller sorbent particles (2-50 micrometer in average particle size). This gives HPLC superior resolving power (the ability to distinguish between compounds) when separating mixtures, which makes it a popular chromatographic technique. The schematic of an HPLC instrument typically includes an auot- sampler, pumps, and a detector. The sampler brings the sample mixture into the mobile phase stream which carries it into the column. The pumps deliver the desired flow and composition of the mobile phase through the column. The detector generates a signal proportional to the amount of sample component emerging from the column, hence allowing for quantitative analysis of the sample components. A digital microprocessor and user software control the HPLC instrument and provide data analysis. Some models of mechanical pumps in a HPLC instrument can mix multiple solvents together in ratios changing in time, generating a composition gradient in the mobile phase. Various detectors are in common use, such as UV/Vis, photodiode array (PDA), Fluorescence, Refractive index detectors or those based on mass spectrometry. Most HPLC instruments also have a column oven that allows for adjusting the temperature the separation is performed.

Use of HPLC for B-complex vitamin analysis with detailed methods were explained theoretically and also demonstrated in the laboratory. Thiamine, riboflavin and niacin were estimated and hands-on experience was given to participants on B-complex vitamin analysis.

Pedagogy - Non-verbal Communication by Dr. I. Sreenivasa Rao, Professor and Head, Department of Extension Education, PJTSAU, Hyderabad; 15-09-2015.



Pedagogy training is mandatory for every 21-day training programme. The definition of communicating a message without using words was described as non-verbal communication or communication by implication. This was described as communication through appearance of objects. Silence was also mentioned as a method of communication. KINESICS or the study of some physical movements like facial expressions, gestures, postures, body movements and eye contact was explained. The main functions of non-verbal communication were described as repeating, contradicting, ignoring, pretending, marginal listening, selective listening, attentive listening, emphatic listening and active listening.

E- Resources for Agriculture and Allied Sciences by Dr. Veeranjaneyulu, University Librarian, PJTSAU, Hyderabad; 16-09-2015.



Different websites, login i.ds, and methods of accessing websites for journal articles, books and other web based learning material were enlarged upon. Advantages and disadvantages of the eresources were described. Types of journals like open access journals, subscribed journals, were enlarged upon. The speaker explained in detail about AGRICAT, KrishiKosh, Krishiprabha, CAB abstracts, CAB Journals, E-portals etc. He also stressed about open access journals of Foods & Nutrition like:

- ➤ J. Nutrition & Food Science
- Foods & Nutrition Sciences
- ➤ J. Food & Nutrition Research
- Food Science & Nutrition
- ➢ J. Human nutrition & food science

- Nutrition science & Food technology
- British Journal of Nutrition
- ➤ J. Nutrition & Metabolism
- ➤ J. food processing

User i.ds and gateways for accessing various websites were shared with participants.

Use of Gamma irradiation chamber for improving shelf life of foods and seed viability for germination: Theory & Practical. By Dr. M. Sreedhar, Principal Scientist, QC Lab, PJTSAU, Hyderabad. 16-09-2015.

The speaker explained about GC 5000, and its current dose rate 1.76KGY/hr and it's design. The gamma irradiation chamber was made by BRIT (Board of Radiation Isotopic Technology) He explained about different categories of irradiation. In category I dry source is used whereas the remaining categories used storage pool mainly water. The gamma irradiation chamber is compact shelf shielded Cobalt 60, category I gamma irradiator providing an irradiation volume of approximately 5000cc. The material for irradiation is placed in an irradiation chamber located in vertical drawer inside the Lead flask. This drawer can be moved up and down with the help of a system of motorized drive, which enables precise positioning of the irradiation chamber at the center of the irradiation field. Radiation field was provided by a set of stationary Cobalt-60 source placed in a cylindrical cage. The source was doubly encapsulated in corrosion resistant stainless steel pencils and was tested in accordance with international standards. The Lead shield provided around the source is adequate to keep the external radiation field well within permissible limits. An irradiator in which the sealed source is completely contained in a dry container constructed of solid materials, the sealed source is shielded at all times, and human access to the sealed source and the volume undergoing irradiation is not physically possible in its designed configuration.

The radiological safety officer should contain TLD badge, which is made up of 3 discs and the material used are Caso4, Dysprosium. Annual effective dose exposure of any person in a year is 30msv.

Later practical session was conducted and the irradiation units were demonstrated. The sample was placed in chamber and the time and temperature were set. The various dosages used for different food stuffs and grains were elaborated upon in the theory session. Mainly this technology is useful for improving the shelf life any food material or improves seed viability when it is stored for long periods.

Microbial Quality Analysis in Food grains and processed food testing by Dr. Sneha Gogte, Professor and HOD, St. Ann's College for Women, NAAC Reaccredited "A". Hyderabad; 18-09-2015.

The Scheme of Presentation of the lecture was as follows: Biometrics, Food from Microbiologist's point of view, Constant interaction of plants, animals and microbes, Food Categories as Microbiologist view, Some lessons from nature, Facts and figures of microbes-man-food- associations, Methods of testing including conventional and modern methods, ISO, GMP, GLP, HACCP, CCP, FDA, USDA, CDC, FSSAI.

She explained about the food sources which are constantly in touch with microbes and different conditions for growth of microbes. Microbes include bacteria, viruses, and fungi such as moulds and yeasts. Some microbes are very useful and are used to make foods such as yoghurt, bread, soy sauce. Some microbes can cause food poisoning Eg: E. coli, salmonella, campylobacter,

and Listeria, can make people sick. Nature is a great training institute for food safety; it is about protecting food from contamination. There are three types of contamination- Physical contamination, Chemical contamination, and Microbiological contamination.

Aims and objectives of all microbiological testing were elaborated, Standard microbiological testing methods like Conventional and **Plate count methods** which include Standard plate count (SPC) Aerobic plate count (APC),Total viable count (TVC), were explained in detail. Description was given about the two Testing Methods i.e. **Traditional methods** like -Plate counts, Membrane filtration, Most probable number, Direct microscopic count, Dye reduction tests, Indicators and **Rapid Methods** like- Micromanipulator, Direct epifluorescent filter technique (DEFT), Electrical impedance biosensors, Enzyme-linked immunosorbent assay(ELISA), PCR.



## Role of Laboratories in Food Safety Systems and VISIT to First Source Laboratories; by Mr. Y. Sudhakar, Vice President, First Source Laboratories, Hyderabad; 18-09-2015.

The visit to First Source Laboratories, Nacharam, Hyderabad was arranged along with a guest lecture delivered by the Vice President. The laboratory is accredited as per ISO/IEC: 17025:2005 and complies with the good laboratory practices to meet the current Global regulatory requirements (NABL, APEDA, FSSAI, and GAFTA). The speaker explained about the definition of Food Safety i.e. Concept that food will not cause any harm to the consumer when it is prepared or eaten according to its intended use (Codex Alimentarius Food Hygiene Basic Texts, FAO). He told about the food safety regulation in India, various testing requirements in food chain and three categories of laboratories (In-house laboratories, Contract laboratories, University laboratories).

Present Technology which is used for chemical and biology contaminants in the laboratory was also explained. He also shared his experiences in food safety monitoring systems in India.

The laboratory was of 11,000 sq feet area, the samples were stored in control sample room under -20 to -40°C. Preparation, extraction and turbo evaporator are located in standard preparation room. There was a separate inventory store room, instrumentation room and balance room in the lab. The technology used for the chemical contaminations in the laboratory are: LC IT TOF and GCMS for screening of targeted and non-targeted compounds, GC-MS-MS (Pesticide residue), LC-Q-TQF (Plant growth regulation), HPLC was used for Aflatoxins and NOTs, LCMS-MS(QQQ) was used for other mycotoxins, pesticides residues, Antibiotic residues, Industrial contamination), ICP-MS,ICP-OES, AAS(Toxic metal contamination), HR-GC HR-MS for Dioxin and PCP congeners.

The technology used for the biological contaminants estimation in the laboratory are: PCR and RT PCR (Pathogen, GMO detection and species authentication), Immunological (Pathogen detection determination of Mycotoxins, allergens, Antibiotic residue), API Methods (Bio typing of microorganisms), Automated enumeration instruments (Enumeration of microbiological quality indicators), Manual Methods ready media, films and chromogenic media] used for the pathogenic identification, Automated air samplers for samples for indoor air and compressed gasses (Microbiological environment monitoring of processing area).

## Practical on Microbial quality Analysis & Fatty acid by Dr. N. Prathima, Microbiologist (Research Associate) & Mr. Ch. Jagan, QC Lab, PJTSAU. Hyderabad. 19-09-2015.

Session 1: Standard operating procedure for determination of total bacterial count was explained on cereal flour sample. The Isolation of culture was explained as these methods.

- 1. Pour plate method
- 2. Spread plate method
- 3. Streak plate method
- 4. Serial dilution on algae plate method

The procedure for MRUP broth for determination of E. coli where the preparation of culture is not required was also explained. Then the entire participants were divided in to 3 groups for the practice. The pour plate method along with serial dilution of culture was explained in detail. The first group has done the experiment with plate count agar media for total bacterial count in sample. Second group has done the experiment with potato dextrose media for fungal contamination in the sample. Third group has done with rose Bengal agar media for fungus and environmental sample. The procedure followed in the determination of TBC and fungal contamination was as follows.

Media culture for plate count agar was developed by taking 4-7g in 200ml of distilled water, potato dextrose media by dissolving 7.8 g in distilled water and Rose Bengal agar of 6.3g200mi of distilled water. Saline solution of 8.5% (0.85 g NACL100ml) was prepared to enhance the culture growth. 10 broth tubes were taken and then 1 as blank and other tube as 1-9 and autoclaved at 15lb / ima for 30mins. After transferring the saline distilled water of 9ml in all the tubes and 10ml in blank.

1gm of food sample was weighed and transferred to first tube (1-10) mixed well in vortex. The 1ml & supernatant of the tube 1-10 was transferred into tube 2 that is 2-10 in the same manner 1ml of suspension was transferred to additional tasks containing 9ml to get serially diluted suspension of 3-10, 4-10,5-10,6-10,7-10,8-10, and 9-10.

1ml of liquid was taken in to Petri dishes (which was labeled as blank 1-10, 2-10,3-10,4-10,5-10,6-10,7-10,8-10 and 9-10)from each brath tubes and transferred in to similarly labeled Petri dishes. 24.3 ml prepared agar media was poured in to the Petri dish, kept for solidification and incubated at 25c for 24-48 hrs in inverted position in the incubators.

The plates were evaluated for the TBC. No mould was observed in the sample 1 colony count was observed in 9-10.

#### Session 2:

Analysis of Fatty acid by Gas Chromatography

The functioning of gas chromatography was explained for the detection of GC is F1D for fatty acids. Organic compounds on flame ionization detector were detected as fatty acid methyl esters. The organic compounds which are volatile will be estimated by GC the procedure includes.

- 1. Method validation
- 2. Sample contraction by converting the sample in to methonolic KOH
- 3. Blank is hexane
- 4. Column in GC is DBFFAD is nitroterephthalic-acid-modified polyethylene glycol

#### (PEG).

- 5. Mobile phase is nitrogen (carrier gas)
- 6. The FID hydrogen gas flow rate should be 30ml min.

The fatty acid content was measured based on area normalization.

NABL Accreditation-Process and requirements by Dr. Satish Yadav; Sr. Scientist, NIPHM, Hyderabad., 21-09-2015



The guest speaker explained about Accreditation Process, Preparation of Laboratory for Accreditation which includes Application to Accreditation, Maintenance of Accreditation like Conformance to applicable standards & NABL Requirements, NABL Terms and Conditions, Modification to Accreditation criteria, Adverse decision against the laboratories Surveillance & Renewal of Accreditation and Appeals & Complaints. He also covered about the Rights and Obligations of Laboratories, NABL Fees Structure and on General Requirement which includes Management Requirement Technical Requirement.

Application of Food Quality Testing in Crop quality Improvement-Rice-I; by Dr. C. Damodar Raju, Sr. Scientist, Rice Research Centre, PJTSAU, Hyderabad; 21-09-2015.

The speaker has described the research work on crop improvement being conducted at Rice Research Station, PJTSAU, where they were involved n plant breeding and modifying the rice grains by using different combinations of parents to make the grains grow faster and with better yield, etc. In spite of all efforts and research they were failing to promote the product as it was not meeting the quality of sona masoori rice. He gave many practical examples of hybrids, cross breeding and all other examples. His lecture was an eye opener to all food scientists that any varieties produced by scientist will not be accepted by people until they meet the food quality requirements. Very beautifully examples and diagrammatical representations were shown to indicate the importance of quality testing in rice breeding.

# Application of Food Quality Testing in Crop quality Improvement-Rice-II; by Dr. S. Vanisree, Pr. Scientist, Rice Research Centre, PJTSAU, Hyderabad; 21-09-2015.

The guest speaker explained about the appearance of rice grain, layers of rice grains, physical characteristic of rice grains and chemical properties of rice grain which are important to

determine before submitting release proposals for any newly developed varieties of food grains. Different properties were described, which need to be tested, including gelatinization, gel molecularization, gel consistency, chalkiness and translucency, water uptake ratio etc. She also explained about bio-fortification of rice giving example of "Golden rice".

She has focused on nutritional aspects of rice like its carbohydrate content, proteins content, fat content, etc, which need to be determined to assess the quality of rice grains before release. She gave a brief picture about how these nutrients are destroyed on dehulling/ dehusking, milling and processing. It gave us an overall picture about the food quality application for improvement of crops.

## From 11:00 -2:30: Documentation for NABL accreditation by Mr. C. V. Rao, Sr. Scientist NIPHM

In this session, requirements for accreditation laid down in the International Standard ISO/IEC17025:2005, documents to be maintained, terms and conditions which include selectivity, range, linearity, sensitivity, limit of detection, limit of quantification, ruggedness, accuracy, precision, reference material, certified reference material, sample, sample handling, sub-sample, sample preparation, test portion, scope were discussed. Technical requirements like personnel, environment and accommodation conditions, space, design, health and safety, validation, equipment, laboratory information management systems, sampling, sample preparation, sample retention and storage, reagents, physical standards, calibration and measurement of traceability were discussed in detail in this session.



Statistical methods and sampling methods by Dr. G. Nageswara Rao, Professor of Statistics, College of Home Science, PJTSAU, Hyderabad. 21-09-2015.

This session was conducted on the participants' request. The lecture on "Statistical Procedures in Research" was given by Dr. Nageswara Rao. This session covered basic statistical procedures for quantitative research methods. He discussed about various levels of measurement and different statistical methods that can be applied in the research projects, sampling and sample size, augmented design for single sample, etc.





INTERACTIVE M EET WITH DR. SHAILAJA NAIK, ICAR EXPERT REVIEWER



CAFT- HOME SCIENCE TRAINING - VALEDICTORY FUNCTION ON 23<sup>RD</sup> SEPTEMBER 2015.

#### PRE AND POST EVALUATION OF THE TRAINING

#### Pre and post evaluation report details with percentages

Pre evaluation and post evaluation were conducted to all the participants on day 1 and day 20, where they were given a questionnaire containing multiple choice questions in areas of food analysis, chemical analysis, biological analysis, laboratory accreditation procedures, irradiation, aflatoxins, climate change, vitamins, minerals, methods of estimation etc. The pre evaluation results indicated that 60% of participants had answered 10 to 13 questions out of 20 and remaining 40% of participants answered 14 - 17 questions out of 20 indicating knowledge levels. between 50% to 85%.



Fig.1: Knowledge levels of the participants before and after training programme

The post evaluation results indicated that 35% of participants had answered 14 to 17 questions out of 20 and remaining 65% of participants answered 18 -20 questions out of 20 indicating knowledge levels between 75% to 100%. There was an improvement in the knowledge levels of all the participants, especially in areas like laboratory accreditation procedures, irradiation, climate change, biological analysis etc.

S1.	No. of correct answers out of	Knowledge levels	Pre-evaluation	Post-evaluation
No	20 questions	(%)	(%)	(%)
1	1 – 9	0 - 49	0	0
2	10 - 13	50 - 65	60	0

3	14 – 17	66 - 85	40	35
4	18 & Above	86 & Above	0	65

 Table.1: Knowledge levels of the participants. Pre and post evaluation

#### **Report of Evaluation schedules**

**Pre evaluation**: The evaluation schedules had questions on reasons for attending the training programme for which the respondents cited reasons like – training relevant to their job, update the knowledge and skills in the training area, training related to subject area and few respondents attended the training as instructed by the organization.

The intended use of knowledge and skills acquired by the participants during training was for development of new research proposals, develop and refine methodologies for research activities in respective institutions, develop or revise course material based on experiences acquired, develop material for extension / outreach programmes and to set up a laboratory in their organization.

Based on participant's expectations, the top five areas of expectation was ranked in the following order:

- 1<sup>st</sup> Learn new knowledge and skills for improving my research competencies
- 2<sup>nd</sup> Know the current research trends to plan new research project
- 3<sup>rd</sup> Learn new knowledge and skills for improving my academic and teaching competencies
- 4<sup>th</sup> Create a new facility at my institute (lab, Food Testing facility, etc.)
- 5<sup>th</sup> Establish and strengthen professional network with other participants

**Post Evaluation**: Based on the post evaluation, the participants found all the topics "relevant" to the training and none were found to be in the "non-relevant" category. The topics which were found to be most satisfying were NABL accreditation procedure, theory and practical's on fatty acids, GC, gamma irradiation, microbial quality analysis, food safety and quality control, measurement of uncertainty, food compositional analysis. All the participants were satisfied with the skill training imparted, demonstrations and the field visits. The participants felt that the methodology adopted for delivering the technical content was apt and adequate. All the participants felt that their objective of attending the training programme was extremely satisfactory. The overall impression about the course such as theoretical back-up and content coverage, resource materials provided, extent of involvement of the guest faculty and level of seriousness maintained all through the course were rated as excellent. The training facilities like learning environment and capacity of institute's faculty

was rated as excellent. Other facilities like boarding and lodging arrangements were rated and good and very good respectively.

#### EVALUATION REPORT SUBMITTED TO CBP PORTAL

- 1. No. of participants (Applied): 27
- 2. No. of participants (Approved): 20
- 3. No. of participants (Attended): 20
- 4. No. of participants (Not attend): 0
- 5. Gender wise distribution of participant: Male 5 Female:15
- 6. ICAR / SAU wise distribution of participant: ICAR: 0 SAU (and Others):20
- 7. State wise distribution of participant:

Sl No.	State Name	No. of Participants
1	Andhra Pradesh	1
2	Assam	2
3	Gujarat	2
4	Karnataka	3
5	Telangana	10

8. Discipline wise distribution of participant:

Sl No.	Discipline Name	No. of Participants
1	Agricultural Chemistry	1
2	Bio-Chemistry (Plant Science)	2
3	Soil Science-Soil Chemistry/Fertility/Microbiology	1
4	Agricultural Structure & Process Engineering	1
5	Chemical Engineering	1
6	Food & Nutrition	11
7	Food Science & Technology	1
8	Agricultural Engineering	1
9	Microbiology	1

#### General information about training

#### Queries

#### No. of responses for each query

4 3

1.	How	did yo	u coi	me to k	know a	about	this	trainir	ng pr	ogram?	
	a)	CPB J	Porta	l / ICA	AR We	ebsite					
	• •	<b>A</b> 11									

b) Colleague in the same department / organization

	c)	Immediate superior (like HOD)	0
	d)	Head of the organization	11
	e)	Friend in other organization	1
	f)	Personally contacted by CAFT Director / Faculty	1
	g)	Any other	0
	h)	Not Specify	0
2.	What	t was your main motive to attend this training?	
	a)	Training theme was relevant to my job	6
	b)	Training was related to my subject area	5
	c)	To update my knowledge and skills	8
	d)	To fulfill CAS / promotion requirement	0
	e)	Desired by Head of the Organization	1
	f)	To seek change from daily routine	0
	g)	Any other	0
	h)	Not Specify	0

3. In your opinion what is your ranking with respect to knowledge / skills / attitude in the beginning and at the end of this training programme?

-			
a)	Rank 1	14	ŀ
b)	Rank 2	4	
c)	Rank 3	2	
d)	Rank 4	0	
e)	Rank 5	0	

### **Opinion towards training design and delivery:**

S. No.	Opinions	Excellent	Very Good	Good	Poor	Very Poor	Total
1.	Training program environment	16	3	1	0	0	20
2.	Laboratory facilities available for the training program	16	3	1	0	0	20
3.	Participation in decision making and planning of program in future	12	7	1	0	0	20
4.	Behavior of resource persons (faculty members)	16	2	2	0	0	20
5.	The course materials were designed as per the objectives of the training	16	4	0	0	0	20
6.	The tools / techniques used during the training	15	4	1	0	0	20
7.	Adequate resource	16	3	1	0	0	20

	persons involvement						
8.	Boarding facilities	10	5	5	0	0	20
9.	Lodging facilities	9	5	6	0	0	20
10.	Transport facilities	13	4	3	0	0	20
11.	Exposure visits / practical exposure / field orientation	17	3	0	0	0	20
12.	Providing platform for future networking / applications	17	2	1	0	0	20
	Total	173	45	22	0	0	

### Topics rating of training program:

Sl No.	Very Well	Fairly Well	Poor Covered	Topic Name	Most Useful	Useful	Not Useful
1	20	0	0	Introduction to ISO/IEC 17025- 2005 and Need for Accreditation	18	2	0
2	15	5	0	Pesticide Analysis	15	5	0
3	18	2	0	Food Safety for Food Security	19	1	0
4	17	3	0	Pesticide Residues Issues, SPS Agreement, Impact on Trade	19	0	1
5	19	1	0	Registration Inauguration and Keynote address	19	1	0
6	19	1	0	Climate change and crop quality- Applications of Food testing facilities for crop quality analysis.	17	3	0
7	19	1	0	Food Safety and Quality Assurance	18	2	0
8	20	0	0	Introduction to Food Quality Testing	19	1	0
9	20	0	0	Practical on Proximate analysis and Uncertainty measurement	18	2	0
10	18	2	0	Food Compositional Analysis-A long experience in analysis and documentation of Food Composition Tables	16	4	0

11	20	0	0	Proximate Analysis with Accuracy and Precision	17	3	0
12	19	1	0	Gas Chromatography	15	5	0
13	19	1	0	Practical on the use of GC for pesticide analysis	16	4	0
14	19	1	0	ICP-OES-Principles of Operation and practical for use of ICP-OES for mineral and heavy metal analysis	17	3	0
15	20	0	0	Importance of Food Quality and Nutrient Analysis for the Food Processing industry	19	1	0
16	20	0	0	PLC-Principles of operation and practical on use of HPLC for B- complex vitamin Analysis Practical on use of ICP-OES for mineral and heavy metal analysis	19	1	0
17	16	4	0	Project work on planning of setting up of a Food Testing Lab	17	3	0
18	20	0	0	Role of Laboratories in Food Safety Systems	19	1	0
19	20	0	0	Visit to First Source Laboratories	20	0	0
20	18	2	0	Application of Food Quality testing in Crop Quality Improvement-II	18	2	0
21	18	2	0	Aflatoxin Estimation Methods- Molecular Diagnostics versus LC- MS method	17	3	0
22	20	0	0	Documentation for NABL Accreditation	19	1	0
23	18	2	0	Post Evaluation	17	3	0
24	16	4	0	Hands-on experience on use of HPLC for B-complex vitamin analysis	18	2	0
25	19	1	0	E-Resources in Agriculture and Allied Sciences	19	1	0
26	16	4	0	Use of Gamma Irradiation chamber	16	4	0

				for improving shelf life of foods and seed viability for germination and demonstration of gamma irradiation chamber			
27	19	1	0	Application of Food Quality Testing in Crop Quality Improvement-I	17	3	0
28	18	2	0	Introduction to use of GC for Fatty Acid Analysis and practical on use of GC for fatty acid analysis.	16	4	0
29	20	0	0	Practical on Microbial Analysis	18	2	0
30	20	0	0	NABL Accreditation-Process and Requirements	19	1	0
31	18	2	0	Microbial quality Analysis in Food Grains and processed food testing	19	1	0
32	20	0	0	Interaction with ICAR expert	19	1	0
33	19	1	0	Implementation of ISO/IEC 17025- 2005 in Food Testing Laboratories	20	0	0
34	18	2	0	Visit to State Food Laboratories	15	5	0
35	20	0	0	Pedagogy-Communication skills	19	1	0
36	19	1	0	Valedictory Function	20	0	0
37	19	1	0	Statistical Methods for Sampling	19	1	0

### Overall opinion about training programme

2.

1.	How	was the daily program?	
	a)	Very tight	5
	b)	Tight	6
	c)	Comfortable	9
	d)	Light	0
	e)	Very light	0
	f)	Not Specify	0
2.	Have	your expectations from the training program fulfilled?	
	a)	To great extent	19
	b)	To some extent	1
	c)	Not at all	0

d) Not Specify

0

3. What should be the optimum duration of the training program? (Kindly suggest the optimum duration from 2 to 6 weeks)

a)	Two weeks	7
b)	Three weeks	10
c)	Four weeks	2
d)	Five weeks	0
e)	Six weeks	1

4. What would be your most preferred time to undergo training program? (Please name the month suit better for this training program)

a)	January	8
b)	February	1
c)	March	0
d)	April	0
e)	May	5
f)	June	0
g)	July	0
h)	August	2
i)	September	2
j)	October	1
k)	November	0
l)	December	1

5	What is y	your overall	opinion	about the	training	program?
5.	Willac 15	your overun	opinion	about the	uumng	program.

Outstanding	2
Excellent	16
Very good	2
Good	0
Average	0
Not Specify	0
	Outstanding Excellent Very good Good Average Not Specify

The overall perception, attitude of the participants towards the training was found to be excellent after the completion of the training programme.

# LECTURE NOTES AND PRESENTATIONS

### Guest Lecture and presentations.

Date	Topic of Lecture and Name of the speaker
04-09-2015	Climate change and crop quality-Applications of Food testing facilities for crop
	quality analysis by Dr. D. Raji Reddy, Director of Research, PJTSAU
04-09-2015	Food Compositional Analysis-A long Experience in analysis and documentation of
	Food Composition Tables by Dr. Bhaskara Chary, Scientist D, National Institute of
	Nutrition
05-09-2015	Food Safety and Quality Assurance by Dr. Anurag Chaturvedi, I/C Dean, Home
	Science, PJTSAU.
05-09-2015	Introduction to Food Quality Testing by Dr. K. Manorama Pr. Scientist, QC Lab,
	<u>PJTSAU</u> .
05-09-2015	Proximate Analysis with Accuracy and Precision by Dr. K. Aparna, Scientist, QC
	Lab, PJTSAU.
07-09-2015	Uncertainty of measurement by Dr. K. Manorama Pr. Scientist, QC Lab, PJTSAU.
08-09-2015	Implementation of ISO-17025 in Food Testing Labs by Dr. Abhay Ekbote, Pr.
	Scientist, NIPHM, Hyderabad.
09-09-2015	Gas Chromatography (GC), Principles of Operation and use of GC for pesticide
	analysis by Dr. V. Sashibhushan, Pr. Scientist and Head, PRL, Hyderabad.
11-09-2015	ICP-OES-Principles of operation by Dr. M. Sreedhar, Sr. Scientist, QCL, PJTSAU.
12-09-2015	Introduction to use of GC for Fatty Acid Analysis by Dr. K. Manorama, Pr. Scientist,
	QCL, PJTSAU.
14-09-2015	Importance of Food Quality and Nutrient Analysis for the Food Processing Industry
	by Mr. A. Poshadri, Assistant Professor, Food Science and Technology, College of
	Food Science and Technology, PJTSAU.
14-09-2015	HPLC-Principles of operation by Dr. K. Manorama, Pr. Scientist, QCL, PJTSAU.
15-09-2015	Pedagogy-Non-verbal Communication by Dr. I. Sreenivasa Rao, Professor and
	Head, Department of Extension Education, College of Agriculture, PJTSAU.
16-09-2015	E- Resources for Agriculture and Allied Sciences by Dr. K. Veeranjaneyulu,
	University Librarian, PJTSAU.
16-09-2015	Use of Gamma Irradiation chamber for improving shelf life of foods and seed
	viability for germination by Dr. M. Sreedhar, Sr. Scientist, QCL, PJTSAU.
18-09-2015	Microbial Quality Analysis in Food grains and processed food testing by Dr. Sneha
	Gogte, Head, Microbiology Department, St. Ann's College, Hyderabad.
21-09-2015	NABL Accreditation-Process and requirements by Dr. Satish Yadav, Sr. Scientist,
	NIPHM, Hyderabad.
21-09-2015	Application of Food Quality Testing in Crop quality Improvement-Rice-I by Dr. C.
	Damodar Raju, Sr. Scientist, Rice Research Centre, PJTSAU.
21-09-2015	Application of Food Quality Testing in Crop quality Improvement-Rice-II, Dr. S.
	Vanisree, Sr. Scientist, Institute of Biotechnology, PJTSAU
22-09-2015	Documentation for NABL Accreditation, Mr. C.V. Rao, Sr. Scientist, NIPHM,
	Hyderabad.
22-09-2015	Statistical Methods and Sampling by Dr. G. Nageswara Rao, Retd. Professor of
	Statistics, College of Home Science, PJTSAU.

#### CLIMATE CHANGE AND FOOD SAFETY

#### Dr. D. Raji Reddy

- Large amounts of gases such as carbon dioxide and methane were released into the atmosphere from the burning of fossil fuels, industrial processes and deforestation.
- Estimates for release of gases from food systems are between 19-29% (2008 data).
- These greenhouse gases in the atmosphere, traps energy and acts like a blanket around the Earth.

CO2 emissions in India: Great Natural Disasters of 1950 – 2005 and a Number of events

#### **Mycotoxins**

- Toxins such as mycotoxins are formed by some fungi as they grow on crops.
- They can be consumed through contaminated crops or indirectly through animal products (e.g. meat or milk from animals) that have eaten contaminated feed.
- The production of these toxins affected by temperature and moisture conditions.
- Mycotoxins can cause a wide range of toxic effects in both animal and humans.
- Some of the most common mycotoxins are carcinogenic, genotoxic, or may target specific organs in the body such as the kidney or liver.

#### Climate change impacts on animal health and veterinary public health: potential pathways

- $\checkmark$  Increase in the susceptibility of animals to disease.
- ✓ Increase in the range or abundance of vectors/animal reservoirs and prolonging the transmission cycles of vectors.
- ✓ Impact of climate change on farming/husbandry practices (including the use of veterinary drugs)
- ✓ Harmful algal blooms (HABs) and fishery product safety
- ✓ Impact of temperature change on persistence and patterns of occurrence of algal communities

	Diseases caused by HABs:	- Azaspiracid shellfish poisoning (AZP),
-	annesic siennish poisonnig (ASP),	– Paralytic shellfish poisoning (PSP),
-	diarrheic shellfish poisoning (DSP),	<ul> <li>Ciguatera fish poisoning</li> </ul>
-	Neurotoxic shellfish poisoning (NSP),	<ul> <li>Cyanobacteria poisoning</li> </ul>

Impact of sea level rise, increased precipitation and flash floods on harmful algal communities
Global distribution of HAB toxins and toxicities (reproduced from Dolah (2000) and detail of increase in PSP outbreaks (from Glibert et al. (2005).

- Environmental contaminants and chemical residues in the food chain
- Flooding and environmental contamination
- Contamination of waters
- Ocean warming
- Sea level rise
- Soil contamination
- Alternating periods of droughts and floods
- Environmental degradation and accelerated desertification
- Pesticide usage and residues in crops and the environment
- Veterinary drug use and residues in foods and the environment
- Illnesses that occur due to Climate Change
- Diarrheal syndromes
- Bacterial contaminants and food borne diseases:
- Salmonellosis
- Campylobacteriosis
- Vibriosis
- Other bacterial food borne diseases (FBD)
- Viral food borne diseases
- Parasitical agents and food borne diseases
- Protozoan parasites
- Food borne trematodes
- Vector-borne diseases and food borne transmission Alimentary tick-borne encephalitis
- Chagas' disease.
- American Trypanosomiasis

#### Addressing food safety implications of climate change Use of Predictive models

- Prevention of Mycotoxin contamination
- Maintenance of strategic food stocks

- Improved coordination among public health, veterinary health, environmental health and food safety services
- Agricultural policy and public information review
- Risk management guidance
- Good agricultural, animal husbandry, aquaculture and veterinary practices

#### FOOD SAFETY AND QUALITY ASSURANCE IN INDIA Dr. Anurag Chaturvedi

- The liberalization of the global trade, and the fact that the consumers in the industrialized countries are more and more demanding food to be not only economical, but also healthy, tasty, safe and sound and
- Changing the so far quantity-oriented food production, guaranteeing the nutrient supply for a nation, into an international quality-oriented food market, where commodities, production areas, production chains and brands complete each other.
- > The competitiveness of food production will soon be more dependent on the reliability of the safety and the quality of the food and acceptability of the production procedures than on quantity and price.
- In contrast to the quantity-oriented markets that are often subsidized and producers can always sell everything they produce, quality-oriented markets are market-driven.

Thus, apart from the steady increase of the national and international standards for food safety and public health, there is a growing influence of the consumer's demands on food production and its allied industries. All of this means that the agricultural supply of food production is facing remarkable changes in the years to come, which is both a challenge and opportunity for food producers.

- India became from a food importing economy to an export oriented country.
- To overcome the problems of food shortage priority was accorded to programmes aimed at augmenting food production but programmes for ensuring wholesomeness of food were relegated to secondary importance.
- Time has come to give sufficient importance to quality assurance of the food produced or processed.
- Health and nutrition areas much dependant on the wholesomeness of food and its freedom from microbial and chemical contamination, as on its adequacy with respect to quantity and nutritive value.
- **Intensification of agriculture and animal husbandry;** more efficient food handling, processing and distribution systems; introduction of newer technologies including appropriate application of biotechnology will all have to be exploited to increase food availability to meet the needs of growing populations. Some of these practices and technologies may pose potential problems of food safety and nutritional quality.

Relationship between lifestyle, food safety, and emerging food borne pathogens

- Food consumed is becoming more multinational in origin
- Increased availability of fast food

•

- More convenient for individuals and families to eat away from home
- Greater risk for developing food borne diseases

Globalization of food supply makes it difficult for traditional regulatory control Street Foods:

- In India, during recent years, there has been an increasing trend towards the sale and consumption of foods at the roadside. Most of these street sales centers have been mushrooming on the roadside in busy market areas. Such foods mostly satisfy the people, served specially to the taste of the consumer, with little attention bestowed on hygiene, food safety or nutritional aspects.
- Recent growth in this sector has been phenomenal with important economic and nutritional implications in the urban context. Street foods are readily accessible and affordable to urban populations, and they provide the energy and nutrient needs of large segments of workers and their families in the cities. Clean and nutritious street foods have a positive impact on food security; low quality and unsafe street foods can have a negative impact.
- The Corporation of Chennai has prescribed certain guidelines for urban street food vendors and collects an annual license fee from them. However, in most other Indian cities, no control is exercised over street food vendors by government authorities. Microbiological examination of various foods served by the street vendors in Pune indicated the presence of fecal coli form bacteria. In Hyderabad use of non-permitted coaltar food colors in sweetmeats and of Lathyrus sativus, in certain snack foods.

#### National food control systems suffer from serious inadequacies, including:

- They are not based on modern scientific and management concepts using compliance policies, risk assessment, HACCP, transparency, and broad-based involvement of industry, trade and consumers.
- Insufficient involvement of scientific expertise from the academia, industry, consumers to strengthen the scientific basis for food control decision making processes.
- > Lack of suitable facilities such as laboratories, trained inspectorate and laboratory staff, funding.
- > Lack of coherence and coordination among different governmental activities concerning agriculture, food, trade, industry and health to achieve optimal results.

**Customer expectations:** This is where quality programs begin. Marketing has defined the customer expectations. Product development has created a product that meets those expectations. Engineering has designed a process to make the desired product. Now, all QA must do is design a control system that verifies that everything is working as designed.

**What is Quality:** The ability to make the same thing the same way, over and over again Customer buys today is same as what they bought last week or will buy next week Product meets customer's expectations 100% of the time

**What is Safety:** Conflicts may exist between optimum quality and food safety Manufacturers must recognize that many processes that ensure food safety do not enhance product quality. Any time a process change occurs to improve quality, product safety requires re-verification

#### Responsibility may fall to QA

#### Supervision: Person with basic educational knowledge

Desire to do the job: "The job is relentless and does not go away over the weekend. The quality manager must address the issues as they arise. If one leaves an issue on Friday without making a decision, then on Monday, one is already two days behind. The consensus is that the good supervisors have a fire in their belly that keeps them on top of things and does not allow them to become complacent. One cannot ride along hoping that things will get better without some type of intervention." Dean Tjornehoj, director of quality assurance, Land O'Lakes, Inc.,

**The "pre-harvest" food safety and quality approach:** Industries with long experiences in growing competition, initially used quality control to cope with increasing quality standards. The need to produce and sell high quality products and increase the efficiency of the production process, however, has led to the development of quality assurance systems along production chains. The difference between quality control and quality assurance can be explained as follows:

**Quality control** is the evaluation of a final product prior to its marketing, i.e. it is based on quality checks at the end of a production chain aiming at assigning the final product to quality categories

such as "high quality", "regular quality", "low quality" and "non-marketable". Since, at the end of the production chain, there is no way to correct production failures or upgrade the quality of the final product, the low-quality products can only be sold at lower prices and the

non-marketable products have to be discarded.

Their production costs, however, had been as high as those of the high and regular quality products. Thus, quality control has only a limited potential to increase the quality and efficiency of a multi-step production procedure.

#### Food safety Standards: CODEX

1963 - The Codex Alimentarius Commission was created by FAO and WHO to develop food standards, guidelines and related texts.

1969 - The Codex Alimentarius Commission brought out the Recommended International Code of Practice-General Principles of Food, Hygiene GHP which has undergone four revisions. Ver 4. 2005- The ISO (International Organization for Standardization) stepped in and brought out ISO 22000: 2005. Harmonize on a global level, Food safety management systems - Requirements for any organization in the food chain.

- Primary objective food is safe and suitable for human consumption.
- Ensuring fair trade practices in the food trade.
- > Follows the food chain farm to fork.
- Takes into account the wide diversity of Food safety standards: CODEX activities and varying degrees of risk involved in food production.
- Lays a firm foundation for ensuring food hygiene with each specific code of hygiene practice applicable to each sector
- Codex recommends a HACCP based approach wherever possible to enhance food safety as desired.
- > HACCP (Hazard Analysis and Critical Control Point).
- Codex guidelines and HACCP approach forms the core of the entire food safety program.

#### Food safety Standards: ISO 22000

Since ISO 22000 is a generic food safety management standard. It can be used by any organization directly or indirectly involved in the food chain. It applies to all organizations in the food chain. It doesn't matter how complex the organization is or what size it is, ISO 22000 can help ensure the safety of its food products. It integrates the Codex Alimentarius Commission's 7 principles of

HACCP and dynamically combines with PRPs necessary to control and reduce any food safety hazards. PRPs (PRE-REQUISITE PROGRAM) are also referred to as good hygienic practices (GHP), good agricultural practices, good production practices, good manufacturing practices (GMP), good distribution practices, and good trading practices.

#### FSSAI-2006

The food safety and standards Act of 2006 came into force across the country on August 5, 2011, making it at par with the international standards. The act will ensure improved quality of food for the consumers and censure misleading claims and advertisement by those in food business.

FSSA, established under the overarching legislation, will lay down science based standards for food items and regulate their manufacture, storage, distribution, sale and import to ensure availability of safe and wholesome food for human consumption. As many as 22 states and Union Territories now have food commissioners in place as required under the Act, while seven were expected to do so by the time it is enforced.

#### INTRODUCTION TO FOOD ANALYSIS Dr. K. Manorama

#### Food analysis is the discipline dealing with

- > The development,
- Application and
- Study of analytical procedures
- > For characterizing the properties of foods and their constituents.
- These analytical procedures are used to provide information about a wide variety of different characteristics of foods, including their composition, structure, physicochemical properties and sensory attributes.
- This information is critical to our rational understanding of the factors that determine the properties of foods, as well as to our ability to economically produce foods that are consistently safe, nutritious and desirable and for consumers to make informed choices about their diet.

#### The following questions will be addressed in this introductory section:

- □ Who analyzes foods?
- □ Why do they analyze foods?
- □ What types of properties are measured?

#### **REASONS FOR ANALYZING FOODS**

- Foods are analyzed by scientists working in all of the major sectors of the food industry including:
- ✤ food manufacturers,

#### PURPOSE OF FOOD ANALYSIS

- How does one choose an appropriate analytical technique for a particular food?
- ➢ ingredient suppliers,
- analytical service laboratories,
- Sovernment laboratories, and
- > University research

Government Regulations and Recommendations

> Standards

> Authenticity

Nutritional Labeling

#### Food Inspection and Grading

#### STANDARDS Mandatory Standards:

- $\circ$   $\,$  Standards of Identity.
- These regulations specify the type and amounts of ingredients that certain foods must contain if they are to be called by a particular name on the food label.
- $\circ\,$  For some foods there is a maximum or minimum concentration of a certain component that they must contain,
- e.g., peanut butter must be contain less than 55% fat,
- ice-cream must have greater than 10% milk fat,
- Cheddar cheese must have greater than 50% milk fat and less than 39% moisture.
- Standards of Fill-of-Container.
- $\circ$  These standards state how full a container must be to avoid consumer deception, as well as specifying how the degree of fill is measured.

#### **Voluntary Standards**

#### **Standards of Grade:**

- A number of foods, including meat, dairy products and eggs, are graded according to their quality, e.g. from standard to excellent.
- > For example meats can be graded as prime, choice, select, standard etc according to their origin, tenderness, juiciness, flavor and appearance.
- There are clear definitions associated with these descriptors that products must conform to before they can be given the appropriate label.
- Specification of the grade of a food product on the label is voluntary, but many food manufacturers opt to do this because superior grade products can be sold for a higher price.
- The government has laboratories that food producers send their products too to be tested to receive the appropriate certification which is requested and paid for by the food producer.

#### NUTRITION LABELING

- > It mandatory for almost all food products to have standardized nutritional labels.
- One of the major reasons for introducing these regulations was so that consumers could make informed choices about their diet.
- Nutritional labels state the total calorific value of the food, as well as total fat, saturated fat, cholesterol, sodium, carbohydrate, dietary fiber, sugars, protein, vitamins, calcium and iron.

- The label may also contain information about nutrient content claims (such as low fat, low sodium, high fiber, fat free, etc), although government regulations stipulate the minimum or maximum amounts of specific food components that a food must contain if it is to be given one of these nutrient content descriptors.
- The label may also contain certain FDA approved health claims based on links between specific food components and certain diseases (e.g., calcium and osteoporosis, sodium and high blood pressure, soluble fiber and heart disease, and cholesterol and heart disease).
- The information provided on the label can be used by consumers to plan a nutritious and balanced diet, to avoid over consumption of food components linked with health problems, and to encourage greater consumption of foods that are beneficial to health.

#### Authenticity

- > The price of certain foods is dictated by the quality of the ingredients that they contain.
- For example, a packet of premium coffee may claim that the coffee beans are from Columbia, or the label of an expensive wine may claim that it was produced in a certain region, using a certain type of grapes in a particular year.
- How do we verify these claims? There are many instances in the past where manufacturers have made false claims about the authenticity of their products in order to get a higher price.
- It is therefore important to have analytical techniques that can be used to test the authenticity of certain food components, to ensure that consumers are not the victims of economic fraud and that competition among food manufacturers is fair.

#### **Food Inspection and Grading**

- The government has a Food Inspection and Grading Service that routinely analyses the properties of food products to ensure that they meet the appropriate laws and regulations.
- > Hence, both government agencies and food manufacturers need analytical techniques to provide the appropriate information about food properties.
- The most important criteria for this type of test are often the accuracy of the measurements and the use of an official method.
- Techniques need to be chosen which provide accurate and reliable results, but which are relatively simple and inexpensive to perform.

#### **Reasons for Analyzing Foods-Cont...**

- Food Safety
- > Quality control:
  - Characterization of raw materials.
- Monitoring of food properties during processing.
- Characterization of final product.

#### НАССР

Research and Development Food Safety

- ➢ One of the most important reasons for analyzing foods from both the consumers and the manufacturers' standpoint is to ensure that they are safe.
- It would be economically disastrous, as well as being rather unpleasant to consumers, if a food manufacturer sold a product that was harmful or toxic.
- A food may be considered to be unsafe because it contains harmful microorganisms (e.g. Listeria, Salmonella), toxic chemicals (e.g., pesticides, herbicides, heavy metals) or extraneous matter (e.g., glass, wood, metal, insect matter).
- ➢ It is therefore important that food manufacturers do everything they can to ensure that these harmful substances are not present, or that they are effectively eliminated before the food is consumed.

#### This can be achieved by

- □ following good manufacturing practices
- □ regulations specified by the government for specific food products and
- □ By having analytical techniques that are capable of detecting harmful substances.

## In many situations it is important to use analytical techniques that have a high sensitivity, i.e., that can reliably detect low levels of harmful material.

**Quality control:** One of the most important concerns of the food manufacturer is to produce a final product that consistently has the same overall properties, i.e. appearance, texture, flavor and shelf life.

#### This can be achieved by:

- > Characterization of raw materials
- Monitoring of food properties during processing
- Characterization of final product
- HACCP
- Characterization of raw materials
- Manufacturers measure the properties of incoming raw materials to ensure that they meet certain minimum standards of quality that have previously been.
- When a batch of raw materials has been accepted, variations in its properties might lead to changes in the properties of the final product.
- > By analyzing the raw materials it is often possible to predict their subsequent behavior during processing so that the processing conditions can be altered to produce a final product with the desired properties.
- > For example, the color of potato chips depends on the concentration of reducing sugars in the potatoes that they are manufactured from: the higher the concentration, the browner the potato chip.
- Thus it is necessary to have an analytical technique to measure the concentration of reducing sugars in the potatoes so that the frying conditions can be altered to produce the optimum colored potato chip.
- Monitoring during processing

- > Food manufacturers should be able to measure the properties of foods during processing.
- Traditionally, samples are removed from the process and tested in a quality assurance laboratory.
- This procedure is often fairly time-consuming and means that some of the product is usually wasted before a particular problem becomes apparent.

#### FOOD MONITORING

- ➢ For this reason, there is an increasing tendency in the food industry to use analytical techniques which are capable of rapidly measuring the properties of foods on-line, without having to remove a sample from the process.
- > These techniques allow problems to be determined much more quickly and therefore lead to improved product quality and less waste.
- > The ideal criteria for an on-line technique is that it be capable of rapid and precise measurements, it is non-intrusive, it is nondestructive and that it can be automated.

#### **Characterization of final product**

 $\therefore$  Once the product has been made it is important to analyze its properties to ensure that

It	meets	the	appropriate	legal	and	labeling	That it is safe, and
ree	quireme	ents,					That it is of high quality.

- > It is also important to ensure that it retains its desirable properties up to the time when it is consumed.
- Hazard Analysis and Critical Control Point (HACCP)
- > The aim of HACCP is to systematically identify the ingredients or processes that may
- Cause problems (hazard analysis),
- Assign locations (critical control points) within the manufacturing process where the properties of the food must be measured to ensure that safety and quality are maintained, and
- rightarrow To specify the appropriate action to take if a problem is identified.
- $\therefore$  The type of analytical technique required to carry out the analysis is often specified.
- In addition, the manufacturer must keep detailed documentation of the performance and results of these tests.
- > HACCP was initially developed for safety testing of foods, but it or similar systems are also now being used to test food quality.

#### **Research and Development**

- Change in consumer preferences towards healthier foods of high quality, low cost, etc
- Food manufacturers should change to meet demands and be competitive
- Need for research that will lead to new product development
- Improve existing food
- > Make them cost effective

		TYPES OF RESEARCH	
BASIC			
\$ UNIVERSITIES	>	GOVT. RESEARCH LABORATORIES	\$ LARGE FOOD COMPANIES
Properties of foods for analysi COMPOSITION	S		
Safety		Physio-chemical	Sensory properties
Nutritional		Quality attributes	Structure

#### The structure of a food can be examined at a number of different levels:

Molecular structure (~ 1 - 100 nm): Ultimately, the overall physicochemical properties of a food depend on the type of molecules present, their three-dimensional structure and their interactions with each other. It is therefore important for food scientists to have analytical techniques to examine the structure and interactions of individual food molecules.

Microscopic structure (~ 10 nm - 100 mm): The microscopic structure of a food can be observed by microscopy (but not by the unaided eye) and consists of regions in a material where the molecules associate to form discrete phases, e.g., emulsion droplets, fat crystals, protein aggregates and small air cells.

Macroscopic structure ( $\sim > 100$  mm): This is the structure that can be observed by the unaided human eye, e.g., sugar granules, large air cells, raisins, chocolate chips.

#### **PHYSICO-CHEMICAL PROPERTIES:**

- The optical properties of foods are determined by the way that they interact with electromagnetic radiation in the visible region of the spectrum, e.g., absorption, scattering, transmission and reflection of light.
- The rheological properties of foods are determined by the way that the shape of the food changes, or the way that the food flows, in response to some applied force. For example, margarine should be spreadable when it comes out of a refrigerator, but it must not be so soft that it collapses under its own weight when it is left on a table.
- The stability of a food is a measure of its ability to resist changes in its properties over time. These changes may be chemical, physical or biological in origin.
- Chemical stability refers to the change in the type of molecules present in a food with time due to chemical or biochemical reactions, e.g., fat rancidity or non-enzymatic browning.
- Physical stability refers to the change in the spatial distribution of the molecules present in a food with time due to movement of molecules from one location to another e.g., droplet creaming in milk. Biological stability refers to the change in the number of microorganisms present in a food with time, e.g., bacterial or fungal growth.
- The flavor of a food is determined by the way that certain molecules in the food interact with receptors in the mouth (taste) and nose (smell) of human beings.

- The perceived flavor of a food product depends on the type and concentration of flavor constituents within it, the nature of the food matrix, as well as how quickly the flavor molecules can move from the food to the sensors in the mouth and nose.
- Sensory Attributes
- > VISION
- SMELL
- > TASTE
- FEEL

- > HOW TO SELECT AN ANALYTICAL TECHNIQUE
- The analytical technique selected depends on
- $\triangleright$  The property to be measured,
- $\rightarrow$  The type of food to be analyzed, and

> HEARING

- $\Rightarrow$  The reason for carrying out the analysis.
- Information about the various analytical procedures available can be obtained from a number of different sources like:
- rightarrow That which is routinely used in the laboratory or company where you are working.
- rightarrow Available from an expert who could recommend a certain technique,
- Scientific and technical publications.

#### BOOKS

- > Food Analysis, 2<sup>nd</sup> Edition. S.S. Nielsen, Aspen Publishers
- Food Analysis: Theory and Practice. Y. Pomeranz & C.E. Meloan, Chapman and Hall
- Food Analysis: Principles and Techniques. D.W. Gruenwedel and J.R. Whitaker, Marcel Dekker
- Analytical Chemistry of Foods. C.S. James, Blackie Academic and Professional

#### TABULATED OFFICIAL METHODS OF ANALYSIS

- > AOAC > IS (BIS)
- > AOCS

- > VALIDATION
- Normally, a particular laboratory develops a new analytical procedure and proposes it as a new official method to one of the organizations.
- The method is then tested by a number of independent laboratories using the same analytical procedure and type of equipment stipulated in the original proposal.
- The results of these tests are collated and compared with expected values to ensure that the method gives reproducible and accurate results.
- After rigorous testing the procedure may be accepted, modified or rejected as an official method.
- Crganizations publish volumes that contain the officially recognized test methods for a variety of different food components and foodstuffs.
- > It is possible to consult one of these official publications and ascertain whether a suitable analytical procedure already exists or can be modified for your particular application.

#### **OTHER SOURCES**

> Journals

- > Internet
- Equipment manufacturers and reagent >> In-house development of a new technique suppliers
- > NEW TECHNIQUE DEVELOPMENT-PRINCIPLES
- Solution Identify the molecular and physio-chemical characteristics of the analyte that makes it different from other components in a food matrix
- > It is necessary to carry out preparatory steps to process the matrix in order to separate the other components / analytes
- > If one or more components have the same properties, they may be interferents
- > It is necessary to differentiate these from the component of interest and separate them

CRITERIA FOR SELECTING AN APPROPRIATE TECHNIQUE

- Precision: A measure of the ability to reproduce an answer between determinations performed by the same scientist (or group of scientists) using the same equipment and experimental approach.
- Reproducibility: A measure of the ability to reproduce an answer by scientists using the same experimental approach but in different laboratories using different equipment.
- Accuracy: A measure of how close one can actually measure the true value of the parameter being measured, e.g., fat content, or sodium concentration.
- Simplicity of operation: A measure of the ease with which relatively unskilled workers may carry out the analysis.
- Cost: The total cost of the analysis, including the reagents, instrumentation and salary of personnel required to carry it out.

#### CRITERIA FOR SELECTING AN APPROPRIATE TECHNIQUE

- Speed: The time needed to complete the analysis of a single sample or the number of samples that can be analyzed in a given time.
- Sensitivity: A measure of the lowest concentration of a component that can be detected by a given procedure.
- Specificity: A measure of the ability to detect and quantify specific components within a food material, even in the presence of other similar components, e.g., fructose in the presence of sucrose or glucose.
- Safety: Many reagents and procedures used in food analysis are potentially hazardous e.g. strong acids or bases, toxic chemicals or flammable materials.
- > Destructive/Nondestructive: In some analytical methods the sample is destroyed during the analysis, whereas in others it remains intact.
- > On-line/Off-line: Some analytical methods can be used to measure the properties of a food during processing, whereas others can only be used after the sample has been taken from the production line.

- Official Approval: Various international bodies have given official approval to methods that have been comprehensively studied by independent analysts and shown to be acceptable to the various organizations involved, e.g., ISO, AOAC and AOCS.
- > Nature of Food Matrix: The composition, structure and physical properties of the matrix material surrounding the analyte often influences the type of method that can be used to carry out an analysis, e.g., whether the matrix is solid or liquid, transparent or opaque, polar or non-polar.

#### METHODS OF FOOD ANALYSIS-PROTEINS

- Current status
- For many years, the protein content of foods has been determined on the basis of total nitrogen content, while the Kjeldahl (or similar) method has been almost universally applied to determine nitrogen content (AOAC, 2000).
- Nitrogen content is then multiplied by a factor to arrive at protein content.
- > This approach is based on two assumptions:
- That dietary carbohydrates and fats do not contain nitrogen, and
- That nearly all of the nitrogen in the diet is present as amino acids in proteins.
- On the basis of early determinations, the average nitrogen (N) content of proteins was found to be about 16 percent, which led to use of the calculation N x 6.25 (1/0.16 = 6.25) to convert nitrogen content into protein content.
- This use of a single factor, 6.25, is confounded by two considerations.
- First, not all nitrogen in foods is found in proteins: it is also contained in variable quantities of other compounds, such as free amino acids, nucleotides, creatine and choline, where it is referred to as non-protein nitrogen (NPN).
- > Only a small part of NPN is available for the synthesis of (non-essential) amino acids.
- Second, the nitrogen content of specific amino acids (as a percentage of weight) varies according to the molecular weight of the amino acid and the number of nitrogen atoms

> Kjeldahl Nitrogen:

it contains (from one to four, depending on the amino acid in question).

- Based on these facts, and the different amino acid compositions of various proteins, the nitrogen content of proteins actually varies from about 13 to 19 percent.
- This would equate to nitrogen conversion factors ranging from 5.26 (1/0.19) to 7.69 (1/0.13).
- In response to these considerations, Jones (1941) suggested that N x 6.25 be abandoned and replaced by N x a factor specific for the different foods, factors, now referred to as "Jones factors", have been widely adopted.
- Jones factors for the most commonly eaten foods range from 5.18 (nuts, seeds) to 6.38 (milk).
- Most foods with a high proportion of nitrogen as NPN contain relatively small amounts of total N
- As a result, the range of Jones factors for major sources of protein in the diet is narrower.
- ✤ Jones factors for animal proteins such as meat, milk and eggs are between 6.25 and 6.38;
- Those for the vegetable proteins that supply substantial quantities of protein in cereal-/legume-based diets are generally in the range of 5.7 to 6.25.
- Use of the high-end factor (6.38) relative to 6.25 increases apparent protein content by 2 percent.
- Use of a specific factor of 5.7 rather than the general factor of 6.25 decreases the apparent protein content by 9 percent for specific foods.
- In practical terms, the range of differences between the general factor of 6.25 and Jones factors is narrower than it at first appears (about 1 percent), especially for mixed diets.
- $\rightarrow$  Jones factors for a selection of foods.

#### SUGGESTIONS

- A specific Jones factor for nitrogen content of the food being analyzed should be used to convert nitrogen to protein when the specific factor is known.
- When the specific factor is not known, N x the general factor 6.25 should be used.
- Use of the general factor for individual foods that are major sources of protein in the diet introduces an error in protein content that is relative to the specific factors and ranges from -2 percent to +9 percent.
- Because protein contributes an average of about 15 percent of energy in most diets, the use of N x 6.25 should introduce errors of no more than about 1 percent in estimations of energy content from protein in most diets ([-2 to +9 percent] x 15).

#### **OTHER METHODS**

- Dumas method based on combustion of organic matter and release of gases
- Protein can be measured as the sum of individual amino acid residues (the molecular weight of each amino acid less the molecular weight of water) plus free amino acids, whenever possible.
- $\triangleright$  Lipids and fat estimation

1) For energy purposes, it is recommended that fats be analyzed as fatty acids and expressed as triglyceride equivalents, as this approach excludes waxes and the phosphate content of phospholipids, neither of which can be used for energy A gravimetric method, although less desirable, is acceptable for energy evaluation purposes. It includes conventional soxhlet extraction using organic solvents

#### CARBOHYDRATES

- Total carbohydrate content of foods has, for many years, been calculated by difference, rather than analyzed directly.
- Under this approach, the other constituents in the food (protein, fat, water, ash) are determined individually, summed and subtracted from the total weight of the food.
- This is referred to as total carbohydrate by difference and is calculated by the following formula:
- 100 (weight in grams [protein + fat + water + ash +] in 100 g of food)
- It should be clear that carbohydrate estimated in this fashion includes fiber, as well as some components that are not strictly speaking carbohydrate, e.g. organic acids
- > Available carbohydrate
- This represents that fraction of carbohydrate that can be digested by human enzymes, is absorbed and enters into intermediary metabolism.
- > (It does not include dietary fiber, which can be a source of energy only after fermentation)
- Available carbohydrate can be arrived at in two different ways:
- it can be estimated by difference, or analyzed directly
- To calculate available carbohydrate by difference, the amount of dietary fiber is analyzed and subtracted from total carbohydrate, thus:
- 100 (weight in grams [protein + fat + water + ash + dietary fiber] in 100 g of food)
- Total carbohydrate: By difference: 100 - (weight in grams [protein + fat + water + ash] in 100 g of food)

- By direct analysis: weight in grams (mono-+ disaccharides + oligosaccharides + polysaccharides, including fiber)
- Available carbohydrate:
   By difference: 100 (weight in grams [protein + fat + water + ash + fiber] in 100 g of food)
- By direct analysis: weight in grams (mono-+ disaccharides + oligosaccharides + polysaccharides, excluding fiber)\*

# **Dietary fiber**

> It is a physiological and nutritional concept relating to those carbohydrate components of

foods that are not digested in the small intestine.

- Dietary fiber passes undigested from the small intestine into the colon, where it may be fermented by bacteria (the microflora), The end result being variable quantities of shortchain fatty acids and several gases such as carbon dioxide, hydrogen and methane.
- Short-chain fatty acids are an important direct source of energy for the colonic mucosa; they are also absorbed and enter into intermediary metabolism.

# PROXIMATE ANALYSIS WITH ACCURACY & PRECISION Dr. K. Aparna

# Food analysis objectives

- Developing a theoretical and practical understanding of the methods used to analyze foods
- Chemical analysis and physical analysis to ensure high and consistent quality of foods in the food industry
- Estimation and determination of how much of the major food components, which are Moisture, CHO, Lipids, Proteins, Ash, Crude Fiber, exist in a given food.

The proximate analysis therefore comprises:

- 1. Moisture
- 2. Crude Fat
- 3. Crude Protein
- 4. CHO and Crude Fiber

Total carbohydrate = 100-[moisture + crude fat + crude protein + ash].

MOISTURE

**OBJECTIVE:** To determine moisture content in cereals and pulses.

# **REFERENCES:**

IS: 1155-1968, Indian Standard specification for wheat Atta (Second Revision) Reaffirmed 2010; Seventh Reprint, June 2006

IS: 4333(Part 2): 2002; ISO 712: 1998; Reaffirmed-2012.

# **PRINCIPLE:**

A test portion is dried at a temperature of  $130^{\circ}C \pm 3^{\circ}C$ , under conditions which enable a result to be obtained which is in agreement with that obtained by the basic reference method. The difference in weights between the sample and the dried material is the moisture lost on dehydration.

# **REQUIREMENTS:**

# **Equipments:**

Analytical Balance (Capable of weighing to an accuracy of  $\pm 0.001$ g)

**Constant Temperature Oven,** electrically heated, capable of being controlled in such a way that, during normal working, the temperature of the air and of the shelves carrying the test portions is 130  $^{\circ}C \pm 3 \,^{\circ}C$  in the vicinity of the test portions.

# **Apparatus:**

Glass Petri dishes with lids

Desiccator containing effective desiccant

# PROTEIN

**OBJECTIVE:** To determine protein content in cereals and pulses.

# REFERENCES

**AOAC 992 . 23**; 32.2.02. – Combustion method; (Leco FP-528 Nitrogen Analyzer)-Official Methods of Analysis of the Association of Analytical Chemists.2005. 18<sup>th</sup>edition. Association of official Analytical Chemists, International

# PRINCIPLE

Nitrogen freed by pyrolysis and subsequent combustion at high temperature in pure oxygen is quantified by thermal conductivity detection. Equivalent protein is calculated.

# **REQUIREMENTS:**

# **Equipments:**

Analytical Balance (Capable of weighing to an accuracy of  $\pm 0.0001$ g)

Leco protein analyzer FP-528 Protein/Nitrogen determinator with Digital Signal Processor (DSP); containing the following parts:

Furnace: U-shaped resistance maintaining minimum operating temperature of 950°C for pyrolysis of test portion in pure (99.9%) oxygen with a range of up to 975°C.

Isolation system: Isolating liberated nitrogen gas from other combustion products for subsequent measurement by thermal conductivity detector.

Thermal conductivity Detector: For interpreting detector response as percent nitrogen (w/w) with features such as calibration of standard material, blank determination. Calibration is based on theoretical percent nitrogen in pure standard organic material such as EDTA.

Grinder: To grind test samples to suitable fineness to pass No. 20 sieve.

# Apparatus:

Reticulated Crucibles porous

Quartz wool strips

Small tin foil cups

# Steel wool

Gas cylinder regulators: Helium: 0 to 125 psi, CGA 580, 15/16-14 Female R.H.; Oxygen: 0 to 125 psi, CGA 540, 7/8-14 Male R.H; Air: 0 to 125 psi, CGA 346, 13/16-14 Male R.H

# **Reagents:**

EDTA standards (Nitrogen 9.52-9.60)

Furnace reagent (Calcium oxide), Lecosorb (20/30 mesh), Alumina oxide pellets, Copper sticks, Anhydrone, Nitrogen catalyst, Magnesium oxide reagent, **Gases:** Carrier gas: 99.99% Helium; Combustion gas: 99.99% Oxygen; Compressed air: Oil and water free

FAT

• **OBJECTIVE:** To determine crude fat content in cereals and pulses.

# • **REFERENCES:**

- AOAC 922.06 and 2003.06; 4.5.06. –Crude fat in Feeds, Cereal grains and forages; Randall/Soxtec/Hexanes Extraction-Submersion method, Official Methods of Analysis of the Association of Analytical Chemists.2005. 18<sup>th</sup> edition. Association of official Analytical Chemists, International
- Gerhardt-Application SOXTHERM, Crude fat in oilseeds, peeled fruit and seeds, B.2.; 16-09-2010

# **PRINCIPLE:**

- The Randall modification of the standard Soxhlet extraction submerges the test portion in boiling solvent, reducing the time needed for extraction. The solvent dissolves the fats, oils, pigments and other soluble substances, collectively termed as "crude fat".

- A dried ground test portion is extracted by a 2-step process: In the first step, the thimble containing the test portion is immersed into the boiling solvent. The intermixing of the matrix with hot solvent ensures rapid solubilization of extractables.
- The thimble is then raised above the solvent and the test portion is further extracted by a continuous flow of condensed solvent. The solvent is evaporated and recovered by condensation. The resulting crude fat residue is determined gravimetrically after drying.
- The solubility characteristics of different solvents may result in slight differences in crude fat results, hence, the report should reflect the solvent used, eg., Crude fat, Hexanes extraction and so on.

# REQUIREMENTS

# Equipment

- Analytical Balance (Capable of weighing to an accuracy of  $\pm 0.001$ g)
- Solvent Extraction system-multiple position extraction unit conducting a 2-stage Randall extraction process with solvent recovery cycle, with Viton or Teflon seals compatible with ether or hexanes.
- Grinder: To grind test samples to suitable fineness to pass No. 20 seive.

# Apparatus

- Cellulose thimbles and stand to hold the thimble
- Extraction cups: Aluminum or glass (Extraction temperature settings based on manufacturer's instructions)

# Reagents

- Hexane/Petroleum Ether (60-80oC B.P.)
- Boiling stones

# ASH

**OBJECTIVE:** To determine ash content in cereals and pulses.

# **REFERENCES:**

IS: 1155-1968, Indian Standard specification for wheat Atta (Second Revision) Reaffirmed 2010;

Seventh Reprint, June 2006; (Incorporating Amendment No. 1 and including Amendment No. 2 & 3) **PRINCIPLE:** 

The sample is charred and ignited at 600°C to burn off all organic material. The inorganic material which does not volatilize at that temperature is called ash.

# • **REQUIREMENTS**

# – Equipments

- Analytical Balance (Capable of weighing to an accuracy of  $\pm 0.001$ g)
- Constant Temperature Oven, electrically heated, capable of being controlled in such a way that, during normal working, the temperature of the air and of the shelves carrying the test portions is  $130 \text{ oC} \pm 30 \text{ C}$  in the vicinity of the test portions.
- Muffle Furnace

# – Apparatus

- Silica crucibles with lids
- Desiccator containing effective desiccant
- Gas burner

# FIBER

- **OBJECTIVE:** To determine crude fibre content in cereals and pulses.
- **REFERENCES**
- AOAC 962.09–Crude fibre analysis in feeds by filter bag technique-Official Methods of Analysis of the Association of Analytical Chemists. 2005. 18<sup>th</sup> edition. Association of official Analytical Chemists, International, Reaffirmed 2010
- PRINCIPLÉ

Crude fibre is lost on ignition of dried residue remaining after digestion of sample with 1.25% (w/v) H2SO4 and 1.25% (w/v) NaOH solutions under specific conditions. Method is applicable to materials from which fat is extracted to obtain a workable residue, including grains, meals, flours, feeds, fibrous materials and pet foods.

# REQUIREMENTS

# Equipments

Analytical Balance (Capable of weighing to an accuracy of  $\pm 0.0001$ g)

Hot air Oven: capable of maintaining a temperature of  $105 \pm 2^{\circ}$ C.

Electric muffle furnace with rheostat control and pyrometer that will maintain a temperature of  $600 \pm 15^{\circ}$ C.

Desiccator with suitable desiccant.

Timer

Fume Cabinet

# Apparatus

Filter bags constructed from chemically inert and heat resistant filter media, capable of being heat sealed closed and able to retain 25 micron particles while permitting rapid solution penetration. Heat sealer sufficient for sealing the filter bags closed to ensure complete closure.

Crucibles for incineration.

#### Reagents

Sulphuric acid (1.25 %): 12.5 g in 1 liter water NaoH (1.25 %): 12.5 g in 1 liter water

#### UNCERTAINTY OF MEASUREMENT Dr. K. Manorama

# What is Uncertainty of Measurement?

"A parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measure and" (ISO Guide) **Related Terms and Phrases** 

- Accepted Reference Value- A value that serves as an agreed upon reference for comparison
- Accuracy of Measurement- The closeness of agreement between a test result and accepted reference value
- Arithmetic Mean- The sum of values divided by number of values
- Measurand- A quantity subject to measurement

**Repeatability:**Closeness of the agreement between the results of successive measurements of the same measure and carried out under the same conditions of measurement (Test results are obtained by same method on identical test items in the same lab by same operator by same equipment with in short interval of time)

**Reproducibility:** Closeness of the agreement between the results of measurements of the same measure and carried out under changed conditions of measurement (test results are obtained with the same method on identical test items in different labs by different operators using different equipments)

• Standard uncertainty- Uncertainty of the result of a measurement expressed as a standard deviation

- Type A Uncertainty- Uncertainty evaluated by the statistical analysis of series of observations
- Type B Uncertainty- Uncertainty evaluated by means other than statistical analysis of series of observations

#### **Identify sources of uncertainty**

• List all possible sources of uncertainty including sources from chemical assumptions. (Sources of uncertainty may arise from various sources, e.g.

Random

specific.)

reproducibility

approximation and assumptions,

variations,

of

equipment/instruments used, and operator

repeatability

method

and

used.

- Sampling,
- Matrix effect,
- Interferences,
- Environmental conditions,
- uncertainty due to mass and glassware, reference values being used,

**Quantify uncertainty components**: Measure uncertainty component associated with each potential identified source. Check whether available data accounts sufficiently for all sources of uncertainty.

**Calculate Standard uncertainty**: Each component of uncertainty that contributes to measurement of uncertainty is expressed by an estimated standard deviation termed as standard uncertainty  $U_1$  and is equal to the positive square root of variance  $U_1^2$ .

**Calculate Combined uncertainty**: Express the individual uncertainties calculated as standard deviation and combined them as per rule. U<sub>c</sub> is calculated square root of the sum of the squares based on the law of propagation of uncertainty.  $U_c = \sqrt{U_1^{2+} U_1^{2+} U_1^{2+} U_1^{2+} \dots U_n^{2}}$ 

**Calculate Expanded uncertainty**: Expanded uncertainty  $U_e$  is determined by multiplying  $U_c$  with coverage factor "k" such that the estimated true value of a measurement result Y may lie within "y -  $U_c$ " and "y +  $U_c$ " values, where "y" is the measured value of the parameter  $Y = y \pm U$ . The coverage factor k is generally taken as 2, which is equivalent to a confidence level of 95%.

**Types of Probability distributions associated with variables**: (B) Rectangular Distribution:- is used when the information is taken from a certificate or specifications, which gives associated uncertainty without specifying the level of confidence.

Example 1:- Concentration of the calibration standard is quoted in the certificate as =  $1000\pm 2$  mg/l. Example 2:- purity of cadmium is quoted as  $99.99\pm 0.1$  %.

These are cases of uniform or rectangular distribution. Here the distributions are such that individual units (purity) probability is more likely to be near extremes. Therefore, an estimate is made by applying rectangular distribution. Hence, assumed standard uncertainty = half width/ Sq. root of 3. (C) Triangular Distribution:- is used, when the distribution is such that occurrence of mean value from range is most probable. Therefore, an estimate is made by applying triangular distribution. Example: Manufacturer quoted volume of volumetric flask of 100ml

As  $100 \pm 0.1$  ml (Tolerance of 0.1ml) In this case a nominal value is most probable.

Therefore, an estimate is made by applying triangular distribution. Hence, assumed standard uncertainty = half width/ Sq. root of 6.

# **Combined Uncertainty:**

- The component uncertainties are combined to produce an overall uncertainty.
- Some of the uncertainties may cancel each other out.
- Some may be interdependent.
- Type A and Type B Uncertainty factors.
- When combining all factors need to be converted into similar unit of measurement eg. , %; gm; ml; °C; unit less.
- Sources of Uncertainty in Chemical testing
- Sampling: the sample measured may not represent the defined measure and

- Incomplete extraction and/or preconcentration of the measure and, contamination of the measurement sample, interferences and matrix effects
- Inadequate knowledge of the effects of environmental conditions on the measurement procedure or imperfect measurement of environmental conditions
- Cross-contamination or contamination of reagents or blanks
- Type B factors: Inadequate information

**Expanded Uncertainty:** The product of Combined Relative uncertainty, coverage factor (k) and the average value reported for the parameter gives the expanded uncertainty. The Expanded Uncertainty  $(U_E)$  calculated as

- $U_{E=} 2 X Uc X Avg.value$
- Where, 2 is the coverage factor at 95% confidence level.
- Final result = Mean Value  $\pm U_{E.}$

Method of Stating Test Results: When reporting the test results and its uncertainty, the use of excessive number of digits should be avoided. Unless otherwise specified, the test result should be reported together with the expanded uncertainty at 95% level of confidence in the following manner. Measured value: 100.1(Units); Uncertainty of measurement +/-0.1 (Units)

# IMPLEMENTATION OF IS/ISO/IEC 17025:2005 IN FOOD TESTING LABORATORIES Dr. A. Ekbote

- Introduction, all officers, supporting staff and Trainer
- Understanding the activities of the Laboratory
- Organogram of Laboratory
- Scope of work discussion. Different activities of Laboratory

Total number staff, Technical and supporting staff, Existing authorized signatory as per statutory requirement, Formation of core team, Appointment of Quality Manager, Technical manager and their deputies, ISO 17025: 2005

General Requirements for the Competence of Testing and Calibration Laboratories program awareness to all staff.

**ILAC:** Requirement of different standards (national, International), standard methods, documents statutory and technical.

ISO 9001 : 2000 Quality Management Systems – Requirements IS / ISO 9001 : 2000 Quality Management Systems – Requirements (Third revision)

# WHY USE ACCREDITED LABORATORY?

When selecting a laboratory to full fill you're testing, calibration or measurement needs, you need to be sure that they can supply you with accurate and reliable results. The technical competence of a laboratory depends on a number of factors including:

- ✓ The qualifications, training and experience of the staff
- ✓ The right equipment properly calibrated and maintained
- ✓ A quality assurance procedures
- ✓ Proper sampling practices
- ✓ Appropriate testing procedures

- $\checkmark$  Valid test methods
- ✓ Traceability of measurements to national standards
- ✓ Accurate recording and reporting procedures
- ✓ Suitable testing facilities
- ✓ All these factors contribute to a laboratory being technically competent to do your testing.

**Minimize risk:** Throughout the world today, customers seek reassurance that the products, materials or services they produce or purchase meet their expectations or conform to specific requirements. This often means that the product is sent to a laboratory to determine its characteristics against a standard or a specification. For the manufacturer or supplier, choosing a technically competent laboratory minimize the risk of producing or supplying a faulty product.

**Avoid expensive retesting**: Testing of products and materials can be expensive and time consuming, even when they are done correctly the first time. If not done correctly, then the cost and time involved in re-testing can be even higher if the product has failed to meet specifications or expectations. Not only do costs go up, but your reputation as a supplier or manufacturer can go down. You can also be held liable for any failure of your product, particularly if it involves public safety or financial loss to a client. Choosing a technically competent laboratory minimize the chance of retesting being required.

**Enhance your customers' confidence**: Confidence in your product is enhanced if clients know it has been thoroughly evaluated by an independent, competent testing facility. This is particularly so if you can demonstrate to them that the laboratory itself has been evaluated by a third party. Increasingly customers are relying on independent evidence, rather than simply accepting a supplier's word that the product is "fit for purpose".

**Reduce costs and improve acceptance of your goods overseas through a system of international agreements, technically competent, accredited laboratories receive a form of international recognition, which allows their data to be more readily accepted on overseas markets.** 

- This recognition helps to reduce costs for manufacturers and exporters that have their products or materials tested in accredited laboratories, by reducing or eliminating the need for retesting in the importing country.
- Laboratories can be audited and certified to an international management systems standard called ISO 9001.
- This standard is widely used in manufacturing and service organizations to evaluate their system for managing the quality of their product or service. Certification of an organization's quality management systems against ISO 9001 aims at confirming the compliance of the management system to this standard, but does not specifically evaluate the technical competence of a laboratory.

# What if the laboratory has ISO 9001 certification?

- Throughout the world, many countries rely on a process called laboratory accreditation as a means of determining technical competence.
- Laboratory accreditation uses criteria and procedures specifically developed to determine technical competence.
- Specialist technical assessors conduct a thorough evaluation of all factors in a laboratory that affect the production of test or calibration data.
- The criteria are based on the internationally accepted standards ISO/IEC 17025, or ISO 15189 for medical laboratories which are used for evaluating laboratories throughout the world.
- Laboratory accreditation bodies use this standard specifically to assess factors relevant to a laboratory's ability to produce precise, accurate test and calibration data, including the:
- $\checkmark$  Technical competence of staff
- ✓ Validity and appropriateness of test methods
- $\checkmark$  Traceability of measurements and calibrations to national standards
- ✓ Suitability, calibration and maintenance of test equipment
- Laboratory accreditation also covers the quality systems elements addressed in ISO 9001 certification. To ensure continued compliance, accredited laboratories are regularly re-examined to check that they are maintaining their standards of technical expertise.
- These laboratories may also be required to participate in regular proficiency testing programs as an on-going demonstration of their competence.
- Laboratory accreditation thus provides a means of evaluating the competence of laboratories to perform specific types of testing, measurement and calibration. It also allows a laboratory to determine whether it is performing its work correctly and to appropriate standards.

Manufacturing: organizations may also use laboratory accreditation to ensure the testing of their products by their own in-house laboratories is being done correctly. Very importantly, laboratory accreditation provides formal recognition to competent laboratories, thus providing a ready means for customers to find reliable testing and calibration services able to meet their needs.

#### How then can you be sure that a laboratory

Accredited laboratories usually issue test or calibration reports bearing some type of symbol or endorsement indicating their accreditation. You should also check with the laboratory as to what specific tests or measurements they are accredited for, and for what ranges or uncertainties.

This is normally specified in their Scope of Accreditation, which may be supplied by the laboratory upon request.

Accreditation bodies in many countries publish lists or directories of the laboratories they have accredited, together with laboratories' contact details and information on their testing capabilities.

If necessary, you can contact the accreditation body and find out whether there are any accredited laboratories who can perform the tests or calibrations you require.

To find out if your country has one or more laboratory accreditation bodies, try contacting your national standards body or your ministry for industry or technology. Alternatively, if you have access to the internet, you can visit the website of the International Laboratory Accreditation Cooperation

- ✓ Testing environment
- $\checkmark$  Sampling, handling and transportation of test items
- ✓ Quality assurance of test and calibration data

(ILAC) at **www.ilac.org** and use the directory of laboratory accreditation bodies available on this website. You will also find directories of accredited laboratories for certain countries on this website. Many countries around the world have one or more organizations responsible for the accreditation of their nation's laboratories. Most of these accreditation bodies have adopted ISO/IEC 17025 as the basis for accrediting their country's testing and calibration laboratories or ISO 15189 for accrediting medical laboratories. This has helped countries employ a uniform approach to determining laboratory competence. It has also encouraged laboratories to adopt internationally accepted testing and measurement practices, where possible.

This uniform approach allows countries to establish agreements among themselves, based on mutual evaluation and acceptance of each other's laboratory accreditation systems. Such international agreements, called mutual recognition arrangements (MRAs), are crucial in enabling test data to be accepted between these countries. In effect, each partner in such an MRA recognizes the other partner's accredited laboratories as if they themselves had undertaken the accreditation of the other partner's laboratories.

Over 40 laboratory accreditation bodies have signed a multi-lateral recognition agreement, called the ILAC arrangement, which greatly enhances the acceptance of data across the national borders of the signatory countries.

This system of international MRAs between accreditation bodies has enabled accredited laboratories to achieve a form of international recognition, and allowed data accompanying exported goods to be more readily accepted on overseas markets. This effectively reduces costs for both the manufacturer and the importers, as it reduces or eliminates the need for products to be retested in another country.

Countries without viable accreditation systems can seek to have their laboratories accredited by established accreditation systems, so that their test data and associated goods can be accepted on foreign markets. These countries can also endeavor to develop their own accreditation system based on the structure and experience of established systems in other countries.

International Laboratory Accreditation Cooperation is the peak international authority on laboratory accreditation, with a membership consisting of accreditation bodies and affiliated organizations throughout the world. It is involved with the development of laboratory accreditation practices and procedures; the promotion of laboratory accreditation as a trade facilitation tool and mechanism for ensuring decisions on public health and environment issues are based on reliable, reproducible, accurate data; providing assistance to developing accreditation systems; and the international recognition of competent test and calibration facilities around the globe ILAC actively cooperates with other relevant international bodies in pursuing these aims. ILAC also publishes a range of literature on topics covering accreditation, testing, trade facilitation and related subjects. Its Internet site at www.ilac.org provides a range of information on laboratory accreditation, as well as the location of its members world-wide. A brochure entitled "What is ILAC?" provides detailed information on ILAC and its activities, and is available from the website.

#### Asia Pacific Economic Cooperation

- APEC has drafted a Mutual Recognition Arrangement on Conformity Assessment of Foods and Food Products. This calls for consistency with SPS and TBT requirements as well as with Codex standards, including the recommendations of the Codex Committee on Food Import and Export Certification Systems.
- APEC and the Codex committee are supporting laboratory accreditation activities to ISO/ IEC :17025-2005 from a body that operates to now ISO/ IEC 17011.
- Accreditation Bodies that are signatories to the APLAC and ILAC MRAs have demonstrated that they meet ISO/ IEC 17011and that the laboratories they accredit meet ISO/ IEC 17025 for a specific scope of testing/ calibration.

#### **Food Analysis**

- Food analysis is inter-disciplinary in nature.
- Food testing is required to evaluate food products for their nutritive and safety values in terms of microbiology, Mycotoxin, pesticide and other chemical residues, toxic metals, additives and packaging materials, in addition to their proximate, biochemical, biophysical and engineering analysis.

#### Scope of Analytical work

- As per the food standard CODEX or any other organization
- Proximate analysis: Protein, Sugars, fatty acids, vitamins etc.
- Microbiological Analysis: Contamination ٠ pathogens

#### **Formulation of Core team**

- Laboratory requires to formulate core team •
- Quality Manager •
- Technical Manager ٠
- Deputy Quality Manager ٠
- ٠ **Deputy Technical Manager**
- Authorized signatory

- Sensory analysis
- Trace level analysis: Pesticide, antibiotic, heavy metals, aflatoxins.
- Infrastructure of laboratory

✓ For microbiological laboratories

 $\checkmark$  Food testing laboratory A laboratory that

performs tests on food products, ingredients,

in process samples, food packaging materials

chemical

microorganisms and associated environmental

analytes

and

- Building
- Water source
- Power supply
- Illumination
- Compatibility

 $\checkmark$  For sensory testing

additives,

#### ISO/ IEC 17025:"General requirements for the competence of testing and calibration laboratories".NABL-114: 2005 "NABL **GUIDELINES** for FOOD TESTING LABORATORIES" ✓ For chemical laboratories

- $\checkmark$  Scope of Testing
- $\checkmark$  The scope of Food Testing Laboratories is applicable mainly to the following disciplines/ areas of activity: Food Chemistry; Food Microbiology; Food Rheology and other Physical Testing; Food Toxicology; Functional Testing; Molecular **Biology** (including genetically modified organisms); Sensory Testing.

#### **MANAGEMENT REQUIREMENTS**

- Management System:  $\checkmark$
- All the Procedures, manuals, and records which monitor and control the laboratory organization
- **Document Control :**
- Documents of all procedure and activities Preparation,
- $\succ$  Checking,

 $\blacktriangleright$  Review,

for

aspects.

- $\triangleright$  Approval,
- ➢ Modification.

Issuance, and control

- Review of Requests, Tenders and Contracts: Self assessment of laboratory regarding resources, capabilities to carry out analytical assignment in relation to method, equipment, resources, method validation etc.
- Control of nonconforming testing work: Laboratory needs to develop mechanism to find out any non conforming activities are being happened. What is agreed? Not being carried out. Stopping of work, correct the situation, Re-assess the situation and then resume the work
- Corrective actions on non conforming, complaints, documentation and conversion of corrective action in to preventive action.
- Developing set of preventive action
   Control
   of
   Records

**Management Reviews:** Planned activity to find out weakness in the system. The management review should be conducted in a systematic manner using a formal agenda. This should include at least the following items:

- a. Matters arising from the previous management review;
- b. Reports on surveillance and re-assessment visits carried out by NABL or any other accreditation body and follow-up actions of the laboratory;
- c. Reports on audits by customers or other approval bodies and follow-up-actions;
- d. Results of internal audits carried out since the last management review and follow-up actions.
- e. trends analysis of results of in-house quality control checks;

f. Trends analysis of complaints received from customers;

g. Need for amendment of management system, including the quality manual;

h. Adequacy of current human and equipment resources;

i. Further plans and estimates for new work, additional staff, new equipment, changed methods

j. Training requirements for new staff and for updating of existing staff;

k. Review of quality policy and setting of objectives for the coming year

1. Plan a program for preventive action;

m. Trends analysis of results of the laboratory's participation in any proficiency testing or Inter-laboratory comparison programs; the corrective action taken where applicable and the need for such participation in other areas of calibration and/ or testing;

n. plan for implementation of decided changes to the management system, including a timetable.

o. Reports from managerial & supervisory personnel.

- p. plan a program for corrective actions
- q. Customer feedback
- r. Recommendations for improvement

# **TECHNICAL REQUIREMENTS**

•

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.

- A large multi disciplinary laboratory shall have trained, competent supervisory level, food scientists / chemists/ microbiologists on staff having at least a bachelor's Degree in Food science/ Nutrition/ Biochemistry/ Chemistry/ Microbiology/ Biotechnology with at least one year of relevant laboratory experience.
- The people filling these positions shall have successfully completed at least one training course from a recognized institution or worked in an accredited laboratory.
- Accommodation and environment
- Generic activities include wet chemistry, which requires extensive fixed benches with provision of water, power, sinks, cupboards, fume cupboards, reagent shelves, glassware cleaning and storage.
- Specialized rooms are required for clean-air-work or for work on substances, which need special care for reasons of safety (e.g., working with radioactive materials or storage or work on toxic substances)
- Dust, both from environmental sources or from other samples, must be avoided since dust contamination of test materials is sporadic and uneven and is likely to be missed b
- There shall be at least 300-lux light intensity at working surfaces other than those required for specified tests. y normal quality control checks.
- For certain chemical analysis, design of the laboratory needs to be specific to ensure segregation of trace analysis from highly concentrated formulations and from pure substances used in preparing analytical standards. The segregation must apply to all facilities for washing/ cleaning equipment, washing and storage of glassware, use of protective clothing.
- It is recommended that the media preparation and media/ glassware sterilization areas be separated from the testing areas.
- Laboratories located in facilities where products or ingredients are manufactured are not to test for infectious pathogens unless the laboratory is physically separated with limited access, equipped with bio-safety cabinets and supervised by a qualified microbiologist.
- Environmental contamination by microorganisms is to be controlled by appropriate air-filters and air-exchange systems.
- Eating, drinking and smoking should be prohibited in the laboratory. Separate area, physically separated from the laboratory, may be provided for such activities.
- Entry in laboratory areas shall be restricted as appropriate for reasons such as security, safety or sensitivity to contamination.
- a) cleaning of floors, vertical surfaces, horizontal surfaces, interiors of refrigerators, freezers, fume cupboards, controlled environment store;
- b) control of contents of refrigerators, freezers, fume cupboards, controlled environment stores; c) checking the performance of air-conditioning of dust extraction equipment and of fume cupboards;
- Test and calibration methods and method validation
- Selection of methods
   Method validation
- Estimation of uncertainty of measurement

- a) sampling and sub-sampling/ lack of sample homogeneity b) extraction/ digestion/ sample preparation c) inherent instability of reference standard and reference material d) calibration of equipment and instrument e) variation of environmental and supply condition f) operator variation g) non-repeatability of result
- Equipment

٠

Handling

Calibration Items

- General laboratory equipment such as incubators, refrigerators, freezers, ovens, water-baths, centrifuges, autoclaves, furnaces etc. shall be periodically cleaned, maintained and calibrated at predetermined intervals
- Specific equipment like balances, GLC, HPLC, spectrophotometers, AAS, GCMS/MS, LCMS/MS etc need also to be calibrated, maintained and intermediate checks
- Measurement of Traceability
- Identification Reference standards and number **Reporting Test Results** Reference Materials. should be given to sample to Name, address, phone and avoid loss of identity. Reference material: email of laboratory • Internationally accepted, Sample to be stored in such Name and address from NIST traceable. a fashion that its integrity whom sample received should be maintained. Stored properly ٠ Date of sample received Assuring the Quality of Test Checked inter - mittantly • Results • Date of sample analyzed Sampling • Use of Certified Reference Method and technique of **Materials** analysis used. ٠ Sampling plan and procedure is to be followed **Replicate Analysis** Unit of tests while drawing the sample. Retesting Name signature and . • Statistical design of designation of analysts sampling has to be followed. Inter Laboratory Comparisons
  - PESTICIDE RESIDUES, ISSUES, SPS AGREEMENT, IMPACT ON TRADE Dr. V. Sashibhushan

**Proficiency testing** 

It means any specified substances in food, agricultural commodities, or animal feed resulting from the use of a pesticide. The term includes any derivatives of a pesticide, such as conversion products, metabolites, reaction products, and impurities considered to be of toxicological significance. They are the very small amounts of pesticides that can remain in the crop after harvesting or storage and make up their way into the food chain. They also include any breakdown products of pesticides.

Pesticide residues can remain even when pesticides are applied in the right amount and at the right time. Sometimes they need to remain on the crop to do their job. For example, they may need to be on the surface of the fruit or the vegetable to protect it from pests during storage. Some pesticides are applied after harvest for this purpose.

# They were found everywhere

of Test

and

•	Found in all foods		• Found in all ecosyst	ems
•	Found in almost all	living creatures	• Found in all Huma	an beings belongs to all
•	Found in all enviro	nments	religions, races, color	Ir.
• • •	Pesticide in the sche Pesticide Registered Formulations registe Pesticide banned fo 28 Pesticide/formulatio allowed for export :	dule to the Act : 841 for use : 246 rred for use: 530 r manufacture/import use: n banned for use but 02	<ul> <li>Pesticide/formulation manufacture/import /</li> <li>Pesticides refused reg</li> <li>Pesticides Restricted</li> <li>Pesticides withdrawn</li> </ul>	banned for use: 04 gistration : 18 for use : 13 for use: 07
•	<b>FUNCTIONS OF</b> T To register pesticid formulae and	THE REGISTRATION CO es after scrutinizing their	DMMITTEE Poisoning etc to be Leaflets.	printed on Labels and
•	Verify claims regar to human beings and	ding Efficacy And Safety Animals	• Any other Function Matters.	on Incidental to these
·	Specify Dosage, M time and techniq <b>Potential Effects of</b> Acute-Short term: Irritation Coughing Nausea Vomiting <b>Chronic-Long t</b>	anner of Use Application ue, Precautions against <b>Pesticides on Health</b>	<ul> <li>▶ Ra</li> <li>▶ Br</li> <li>▶ Cor</li> </ul>	shes on skin eathlessness na
•	Asthma	Neurologic	al Effects • Ho	ormone Disruption
•	Birth Defects	• Cancer	• Im	munological problems
г		USA Refusals – Co	ountry wise - MAY 2014	
	Date	Product	Reason	Manufacturer / Exporter
	01/05/14	Capsicums	Salmonella	Jorhat, Assma
	02/05/14	Turmeric (Ground)	Salmonella	Mumbai
	05/05/14	Cumin	Salmonella	Rajkot, Gujarat
	05/05/14	Curry Powder	Salmonella	Pune
	05/05/14	Rice Basmati	Filthy	Delhi
	08/05/14	Capsicums	Pesticide	Cochin

Filthy

Pesticide

Delhi

Valsad, Gujarat

08/05/14

09/05/14

Dates

Leaf & Stem Vegetables

09/05/14	Tamarind Dried	Filthy	Calcutta
09/05/14	Tamarind, Dried	Filthy	Apollo, Hyderabad
12/05/14	Curry Powder	Salmonella	Desai Brothers, Pune
12/05/14	Herbals And Botanicals	Pesticide	Prahlada Res Farms, Bangalore
12/05/14	Okra	Pesticide	Vadilal,Gujarat
12/05/14	Spinach	Pesticide	Vadilal, Gujarat
13/05/14	Rice	Pesticide	Quality, Maharastra
13/05/14	Star Gooseberry	Pesticide	Papayil Exports, Kerala
13/05/14	Black Pepper	Salmonella	Pujan, Mumbai
29/05/14	Rice	Pesticide	Quality Spices, Maharastra
29/05/14	Pepper, Hot	Pesticide	Vadilal, Guj
29/05/14	Mango	Pesticide	Vadilal, Guj
29/05/14	Tamarind	Filthy	Roopak, New Delhi
29/05/14	Fenugreek	Salmonella	Gurgoan
29/05/14	Rice	Pesticide	Quality Spices, Mah
29/05/14	Turmeric, Ground	Pesticide	Laxmi Ext, Mumbai
29/05/14	Turmeric, Ground	Pesticide	Laxmi Ext, Mumbai
29/05/14	Turmeric, Ground	Pesticide	Laxmi Ext, Mumbai
29/05/14	Rice, Basmati	Filthy	Agronic Foods, Jodhpur
29/05/14	Rice, Basmati	Filthy	Agronic Foods, Jodhpur
29/05/14	Gourd	Pesticide	Grandmas, Kerala
29/05/14	Gooseberry	Pesticide	Grandmas, Kerala
29/05/14	Mango	Pesticide	Grandmas, Kerala
29/05/14	Gooseberry	Pesticide	Papayil, Kerala
29/05/14	Black Pepper	Filthy	Agronic Foods, Jodhpur
29/05/14	Rice, Basmati	Pesticide	Mr Overseas, Delhi
29/05/14	Tamarind	Filthy	Poojanjali, Mumbai

# SANITARY AND PHYTOSANITARY MEASURES

**Problem:** How do you ensure that your country's consumers are being supplied with food that is safe to eat — "safe" by the standards you consider appropriate? And at the same time, how can you

ensure that strict health and safety regulations are not being used as an excuse for protecting domestic producers?

An agreement on how governments can apply food safety and animal and plant health measures (sanitary and phytosanitary or SPS measures) sets out the basic rules in the WTO.

# **Regulatory and Analytical Challenges ahead**

Methods for detection of not only 246 pesticides (registered in India), but also for many more pesticides under umbrella for imported food safety regulations to be developed

- Fixing of MRLs for all 246 registered pesticides on all food commodities as per GAPs
- Revising GAPs as per ICAR / SAUs etc. and Label Expansions as per Insecticide Act, 1968
- Crop grouping

# WHAT DO WE DO?

- Promoting GAP (Good Agricultural Practices)
- Promoting Safe Use of Pesticides
- Promoting Safe Handling of Pesticides
- Recommending PHIs (Pre-Harvest Intervals)
- Recommending Decontamination Methods

- Harmonization of MRLs with International MRLs
- Development of MRMs for sensitivity at least 10 ppb
- Sensitizing food analysts and regulatory officials on sampling and reporting results
- Monitoring Agri Products for Pesticide Residues
- Validation of Methods
- > Training Programs
- Publications
- > Publicity

Decontamination Methods for Removal of Pesticide Residues were also discussed.

# GAS CHROMATOGRAPHY Dr. V. Sashibhushan

- In gas chromatography (GC), the sample is vaporized and injected onto the head of a chromatographic column.
- Gas liquid chromatography is based upon the partition of analyte between a gaseous mobile phase and a liquid phase immobilized on the surface of an inert solid.

**Instruments for GC:** The schematic of a gas chromatograph is shown below:

• <u>Carrier Gas Supply:</u> Carrier gases, which must be chemically inert, include helium, argon, nitrogen, etc. The choice of gases is often dictated by the detector used. Associated with the gas supply are pressure regulators, gauges, and flow meters.

# Desirable properties for the immobilized liquid phase in a gas liquid chromatographic column include...

• thermal stability

chemical inertness

# Sample Injector System...

The most common method of injection involves the use of a micro-syringe to inject a liquid or gaseous sample through a silicone rubber diaphragm or septum into a flash vaporizer port located at the head of the column.

# **Column Configurations and Column Ovens...**

Two general types of columns are encountered in gas chromatography:

- (1) Packed
- (2) Open tubular or capillary

Chromatographic columns vary in length from less than 2m to 50m or more. They are constructed of stainless steel, glass, fused silica, or Teflon. In order to fit into and oven for thermo stating, they are usually formed as coils having diameters of 10 to 30cm.

# Characteristics Of The Ideal Detector...

1.

•

- Adequate sensitivity (up to pg level)
- 2. Good stability and reproducibility.
- 4. A short response time that is independent
- of flow rate.
- 5. Nondestructive of sample.

3. A temperature range from room temperature to at least 400 deg C.

<u>Thermoionic Detector..(NPD)</u> The thermo ionic detector (TID) is selective toward organic compounds containing phosphorous and nitrogen. It is similar in structure to the flame detector.

**Electron Capture Detector (ECD) :** Electron-capture detector (ECD) operates in much the same way as a proportional counter for measurement of X-radiation. Here the effluent from the column passes over a beta-emitter, such as nickel-63 or tritium (adsorbed on platinum or titanium foil). An electron from the emitter causes potential difference.

**Flame Ionization Detector:** The flame ionization detector (FID) is one of the most widely used and generally applicable detectors for gas chromatography. The effluent from the column is mixed with hydrogen and air and then ignited electrically.

**Thermal Conductivity Detector:** A very early detector for gas chromatography is based upon changes in the thermal conductivity of the gas stream brought about by the presence of analyte molecules.

**Packed Columns:** Present day packed columns are fabricated from glass, metal (stainless steel, copper, aluminum), or Teflon tubes that typically have lengths of 2 to 3 m and inside diameters of 2 to 4mm. These tubes are densely packed with a uniform, finely divided packing material, or solid support, that is coated with a thin layer of stationary liquid phase. The tubes are formed as coils having diameters of roughly 15cm.

<u>Open Tubular Columns :</u> Open tubular, or capillary, columns are of two basic types, namely, wallcoated open tubular (WCOT) and support-coated open tubular(SCOT). WCOT columns are simply capillary tubes coated with a thin layer of the stationary phase. In SCOT columns the inner surface of the capillary is lined with a thin film of a support material, such as diatomaceous earth.

**The Retention time:** The retention time is a parameter for identifying solutes from chromatograms. The retention time for any given solute can be derived from a chromatogram of a mixture of that solute.

**Residues mg/kg=** (Area of the sample/Area of standard) X (ng of std. Injected / ul of sample injected) x (Final volume) / Weight of sample)

# INDUCTION COUPLED PLASMA OPTICAL EMISSION SPECTROSCOPY Dr. M. Sreedhar

# **Atomic Spectroscopy**

• Solar Spectrum and Dark Lines

• Emission and absorption lines

- When heated every element gives off light. When this light is decomposed using a prism it is found to be made up of a series of ``lines", that is, the output from the prism is not a smooth spectrum of, but only a few of them show up. This set of colors is unique to each element and provides a unique fingerprint.
- Similarly, when white light passes through a cold gas of a given element, the gas blocks some colour. when the ``filtered" light is decomposed using a prism the spectrum is not full but shows a series of black lines which correspond to emission lines of heated element. These are absorption lines.
- Atomic Transitions
   Applied Techniques
   The principle
- The plasma is produced by the interaction of an intense magnetic field (produced by radio frequency [rf] passing through a copper coil) on a tangential flow of gas (normally argon), at about 15 L/min flowing through a concentric quartz tube (torch).
- This ionizes the gas and, when seeded with a source of electrons from a high-voltage spark, forms a very high temperature plasma discharge (~10,000 K) at the open end of the tube.
- OES v/s MS
- In ICP-OES, the plasma, usually oriented vertically, is used to generate photons of light by the excitation of electrons of a ground-state atom to a higher energy level. When the electrons "fall" back to ground state, wavelength-specific photons are emitted that are characteristic of the element of interest.
- In ICP-MS, the plasma torch is positioned horizontally, and is used to generate positively charged ions rather than photons.
- About the Prodigy-XP
- a. Assay on I, O, P Qualified Inductively Coupled Plasma Optical Emission Spectrometry.
- b. True simultaneous, poly chromator system with analysis of 72 elements in 45 seconds up to ppb levels.

# c. The data generated is 21 CFR compliant and tamper proof with hierarchical access levels to the archives and retrieval system.

- d. Thermostatically purged optical system. 165 1100 nm wave length.
- e. CID based C PAD largest solid state detector with very strong optics and Echelle grating.
- f. Dedicated dual view (Axial- for high sensitivity, Radial- dynamic working range).
- g. SALSA software with intuitive navigation based on Internet.
- h. Method developed for high dissolved solid samples like food grains and products.
- ICP Parts
   ICP Spectrometers
   Sample Introduction
   Components

•	What is Plasma?	• Tł	ne Prodigy Camera	•	Metadata
•	How the Plasma Works	• Ca	amera Supplies	•	The Salsa
•	What Happens to a Sample	• Al	bout the Camera Chiller	•	Login
•	Torch Design	• Ca	amera Controller Features	•	Display
•	Concentric Design	• Sa	mple analysis	•	Getting Started
•	The Peristaltic Pump	• Pr	ior to Analysis	•	Open Method
•	Types of Detectors	• In	portant Considerations	•	Open Method
•	CCD Readout	• W	avelength Selection	•	Interlocks
•	CID Readout	• Se	election Procedure	•	Instrument Parameters
•	Camera Schematics	• Pl	asma Parameters	•	Sample digestion

# SAMPLE PREPARATION FOR MICROWAVE DIGESTION:

Weigh the 0.5 grams sample in digestion tube and add 7ml Nitric acid, 1ml hydrogen peroxide, close the digestion tubes with lids and put into microwave digestion system.

# **OPERATION:**

- Switch on the instrument.
- Insert the segments in to the microwave cavity and connect the temperature sensor.
- In the panel, enter administrator using the password 123456.
- Develop a method in the microwave program (digestion cook book) and save.
- Log out of the administrator and type password 123 to enter in to user.
- Select a method already created and press start.
- After completion of the method, remove temperature sensor, rotor segments and
- Switch off the instrument.

# **Starting Procedure**

- Switch ON the Mains Instrument and Computer System.
- Open the Argon and Nitrogen, check pressure at outlet gauge its should be 85 PSI, if it is not readjust to 85PSI.
- Open the "salsa" software. Camera purging warning will appears "Has camera is purged for recommended period" click on "Yes" if camera is purged with Nitrogen for at least 4-hrs or Click "No" wait for to complete the purging time and reopen the software and then click on "Yes" now the camera temperature will reduce from normal room temperature to -40 degree centigrade.

# **Initial Start up Optimization**

- Click on for Hg auto alignment, a pop up auto align window will appear click on "Auto Align" this will adjust the drifts automatically click on "Accept" & close this window.
- Make sure all tubing and torch are connected properly, before ignition switch on the "Water Circulator System" and "Exhaust System.
- Now open the method which contain element Mn & Fe, set Fe as radial view or open the method "Positioning \_ Plasma"(this is created method during installation) ignite the plasma by clicking "Auto Start" on Instrument c
- Control (before ignition check all parameters like power, coolant, aux, nebulizer and pump set according to instruction).
- Aspirate the "Mn & Fe" combined element standard of approx. 10 ppm through manual control of auto sampler and take a Echelle frame for Mn & Fe by clicking with different exposer time and align wavelength, now do the positioning plasma by clicking "Positioning Plasma" on diagnostic page select the "Mn" line from drop down list and click on "Peak Plasma" it will adjust the Max. Intensity for Axial view.
- Now close the window and once again click on "Positioning Plasma" select the "**Fe**" line from drop down list and click on "Peak Plasma" it will adjust the Max. Intensity **for Radial view**. And close the window (avoid step No 8 if radial is not required).

# Method Development

- Open the existing method or create a new method to create a method click on "method" and "**new**" enter the desired name in method name and chapter this creates new method click on instrument control and adjust all parameters like power, coolant, aux, nebulizer and pump set according to instruction.
- Click on "element Selection" select the required element from periodic table and select the best wavelength preferred \*\*lines are best line by default selected line is set to axial view, if you want to change it to radial, click on the desired element line and go to general tab and select radial.
- Aspirate high concentration standard from calibration standard and take the full frame Image by clicking with different exposure time (like 0.01, 0.05, 0.1, 0.5 and 1.0 sec for element which is below 195nm may require more exposure time or high concentration standard).
- click on each individual element in "Element selection" tab and click on "Align Wavelength " tab now click on Echelle images see the Echelle image in window if required check with different exposure time for better Echelle image and click on "Auto" and "Accept". If there is a more than one Echelle image identify correct image for that particular element and adjust manually by clicking up, down, left and right cursors and set to maximum intensity.
- Repeat step for all elements which is selected in "element selection" tab.

• Now take a scan for blank, by clicking repeat the scan for one sample and for low and high standard from calibration set, go to Analysis tab check the scans and select the element and adjust the background points and analytical region for all elements.

Select View Options (on Dual View Systems)

- Integration Times and Replicates
- Position Plasma

Generate an EchellogramScans: Zn 206.200 nm

• Positioning the Plasma

#### Automatic Calibration & Analysis with Auto Sampler

• To calibrate with auto sampler go to instrument control, click on "QC Automation" in "initial – before sample run" select all standard (click on check box with right mark) this makes the software to bring **all standard in running sequence**.)

Now click on "Sequence" click on "All Off" go to cup No 1 and enter the sample Identity, now go to cup No. 2 enter the sample Identity repeat the same for all remaining sample and make sure to put all samples in respective positions of sample racks and mark the checkbox with right for all entered samples click on "Update" selected rack sample positions colour will change to yellow.

- Ignite the plasma by clicking "auto Ignition" in instrument control click the check box "Extinguish after Rinse" drop down the list in delay select 2 min, go to "Sequence" and click on "Run Sequence.
- Auto sampler
  Run Calibration
  Sample analysis
  Sequences
  Calibration Curve –
  Sequence Navigation Panel
- Calibration Standards
- Accept Calibration

Example 1

- Calibration
- <u>Switching Off Procedure</u>
- After analysis extinguish plasma on instrument control page, aspirate the blank for cleaning of sample introduction system for 2-5 min.
- Switch Off the chiller and Exhaust system.
- close the salsa application software
- Switch off the both Argon and Nitrogen Regulator.
- Statistical procedures in research
- Levels of measurement
- Nominal

- Ordinal
- Interval
- Ratio
  - Nominal & ordinal
    - Interval & ratio level
- Interval level measurement
- measurement done between two limits
- ex: fahreign heat &centigrade thermometers

**Run Samples** 

Analysis Results

- Ratio level measurement
- measurement with zero point
- ex: height, weight, volume
- Data used in home science research
- All levels of measurements are used in home science research

- data based on qualitative characters are called qualitative data
- Ex: good, read colour, strongly agree etc.,
- Data based on quantitative characters are called quantitative data
- Ex: height, weight, etc.,
- Types of characters
- qualitative characters are measured
- at nominal & ordinal levels
- ex: yes/no type,very good,good, etc.,
- Quantitative characters are measured at interval &ratio levels
- ex: height, weight, temparature, etc.,
- One-sample tests (non-parametric)
- Home science research & statistical procedures:
- Qualitative data analysis requires nonparametric tests
- One sample tests: chi-square -test:ex: uniform distribution of infected food samples or not
- Kolmogorov-smirnov test:ex: grading of wheat,run-test ex:-births are taking place in random order not in hospital according to gender
- Two-sample tests (non-parametric)
- Two-sample tests : (related samples)
- Wilcoxon test ex: scores of two sisters one trained and another untrained in child rearing
- Sign –test
- Two-sample tests: (independent samples )
- Mann- whitney test ex: scores on experimental diet & control diet
- Median test
- Morethan two-samples
- P-related samples tests (p > 2)
- Friedman's test : ex: scores on aptitude,
- Physical & social aspects on children
- P-independent samples tests (p>2)
- Kruskal –wallis test :ex: scores on quality of chapathi prepared from three varieties of wheat
- Rank correlation
- Correlation between two variables
- Spearman's rank correlation coefficient
- two-sample t-test:
- Comparison between cotton and silk with respect to mean tensile strength with unknown population s.d.
- Analysis of variance (anova)

- Ex: ranks for two types of garments, two methods of cooking, two methods of treatments for maintaining flowers, etc.,
- Correlation among more than two variables
- Coefficient of concordance
- Ex: correlatio among ranks given for more than two types of garments,more than two methods of cooking,etc.,
- Measurement data
- Quantitative variables ( interval& ratio level of measurement)
- Data of type height, weight, volume, etc.,
- Graphic & diagrammitic representation
- Ex: bar diagrams, pie diagrams & frequency curves
- Discriptive statistics :
- Tools: ex: mean, median, s.d.,c.v. mean± s.d.
- Tests of hypotheses
- Null hypothesis, level of significance, degrees of freedom
- Assumption : normal distribution for population, sample is drawn at random
- One- sample tests ( one-tailed & two-tailed)
- Z-test (large sample, n > 30)
- Population s.d. is known & unknown
- T-test ( small sample ,n≤ 30 ),population s.d. is not known
- One-sample tests (parametric )
- One sample z- test:
- Ex: comparison between sample mean weight of children in a particular year and mean weight in all the years
- Ex: comparison between sample proportion & population proportion
- one sample t- test:
- Ex: comparison between sample mean protein content in a dal and standard protein content with unknown popn. S.d.
- Two-sample tests (parametric)
- Two-sample z-test: Ex: comparison between efficacy of two types of detergents after fixed number of washes Ex: comparison between two villages w.r.t. incidence of dengue fever
- Analysing the total variation in the experimental data according to different sources of variation

- Ex:pond with fishes
- Assumptions:
- 1.additive linear model
- 2.experimental errors are independent and follow standard normal distribution Anova
- Anova is used for comparison between more than two treatment means simultaneously.
- First it uses f-test and then t-test for pair wise comparison
- If f-test is found not-significant then there is no need to go for t-test
- If f-test is found significant then c.d. (l.s.d.) will be used for pair wise comparison of treatment means in the case when no. Of observations are equal for each treatment.
- Otherwise t-test will be used for comparison of each pair of treatment means.
- One-way anova
- anova involved with one factor
- Ex: comparison between recipe prepared with different food grains for controlling blood sugar level in diabotic patients

#### Anova table

•	Source	d.f.	s.s.	m.s.	f(cal)

- -----
- Between food grains
- Within food grains
- Total
- , \_\_\_\_\_

-----1717

Pair wise comparison

If f (cal.) Value is found significant then. Value is calculated and then compared with difference of means of treatments when the number of observations is equal for each treatment. Otherwise two-sample t-test is applied for each of treatments means.

Two-way anova : Anova involved with two factors is two-way anova Ex: comparison between age groups & between different diets w.r.t.gain in weight of children

Anova table Source d.f. s.s. m.s. f(cal.) Age groups Diets Error Total

Two-way Anova: When f-value is found significant for both age groups and diets then C.d. values will be calculated separately for both age groups and diets for pairwise comparison of means by arranging them in descending order.

Three-way Anova: If three factors such as body weight, age & diets are included in the experiment, then three-way Anova will be carried out.

ource	d.f.	<b>S.S.</b>	m.s.	f(cal.)
groups				
ly weights				
ets				
r				
ntal				

**Pairewise comparison:** if f(cal.) Is found significant for each of the factors such as body weights, age groups & diets then c.d. is calculated separately for each factor and compared with differece of means using pairwise comparison after arranging them in descending order.

- Factorial experiment: When each factor is experimented at different levels then there will be synergism or antoginism betweean the factors and this is denoted by interaction. Ex: protein is at different levels and iron is applied at different levels then all combinations are considered as treatments
- Factorial experiments: Then it is possible to find the effect of interaction of protein with iron and can be tested for its significance and this is called two factor interactions. If three factors are involved it is possible to test two & three factor interactions besides testing main factor effects Factorial experiments. Ex: if the experiment is conducted with 4 protein levels, 3 iron levels & 3 replications.
  - Anova table


Transformations: Sometimes experimental data may not follow normal distribution & which is the assumption for Anova then transformation has to be effected transformations

Square root
 Angular

- 3. Logarithmic
- 4. Reciprocal

# Transformations

- 1.square root: if the data follows poisson distribution
- Ex: no. Of children infected with rare disability, no. of bacterial colonies in a food samples the data is transformed to square root. If "y" is original data then  $\sqrt{y}$  is taken for analysis
  - Transformations
- Angular : if the data follows binomial distribution

• Ex: if the original data is in percentages such as no. Of percentage of children effected by malnutrition ,percentage seeds effected with rare disease etc., then original data "y" is transformed to angular values.

Transformations

- Logarithmic : if the original data follows exponential distribution
- Ex: annual income of individuals, human population, disease growth, etc., then the original is "y" is transformed to log "y"

# Transformations

- Reciprocal : when small values are to be given more importance
- Than big values
- Ex: if the data is in no. Of days in
- completing work ,
- Number of kilometers covered in an hour etc.,
- If the original is "y" then "1/y " is used for analysis
- Correlation
- If one variable changes in accordance with another variable then there exists correlation between them
- Ex: protein content in diet increases then body weight of individual also increases.
- "protein and "bodyweight" variables are correlated
- Correlation
- The extent of correlation is measured by correlation coefficient and is denoted by "r"

"r" always lies between -1 and 1. covariance (xy) r = ------

 $\sqrt{\text{variance}(x)\text{variance}(y)}$ 

"r" has no units whatever may be the units of "x" and "y"

r value should not be calculated for unrelated variables

Correlation

• Protein content in diet and bodyweight change in same direction so they have positive

Correlation

• Supply of commodity and its price will change in opposite direction so they have negative correlation

Regression

• G if "x" is independent variable and "y" is dependent variable then the regression equation of y on x is

 $\mathbf{Y} = \mathbf{a} + \mathbf{b} \mathbf{x}$ 

- b is regression coefficient of y on x and a is intercept
- b measures change in y for unit change in x
- a gives the value of y when x takes the value 0 Regression

a = y - b xRegression Ex: protein content in diet is independent variable (cause) and body weight is dependent variable (effect) b can take any value and is measured in the units of dependent variable If x and y are interdependent then regression equations of y on x and x on y can be fitted Ex: bodyweight & head weight of insects.

#### Regression

It is possible to predict the value of y given the value of x from y on x equation. Also the value of x given the value of y from the equation of x on y, r is the geometric mean of two regression coefficients b and b1, r, b and b1 will have same sign Multiple regression If more than one independent variable is involved in simple regression then it becomes multiple regression The multiple regression equation with two independent variables is

#### Y = b 0 + b 1x1 + b2x2

b1 and b 2 are partial regression coefficients and b0 is constant Multiple regression. From the fitted equation it is possible to predict the value of y given the values of x1 and x2. in order to include the independent variables in the multiple regression equation the variables may be screened through zero order correlation matrix for multi collinearity etc.,

Multiple regression

Regression sum of squares

Total sum of squares is called coefficient of determination, R is called coefficient of multiple correlation, Step down regression: variables will be dropped successively if their contribution to r2 is negligible Multiple regression

- The final equation will be arrived at based on r2 values at the start and at the end .the difference should be minimum.
- Step up regression:
- Independent variables will be added one by one depending upon their contribution to r2.if the contribution is negligible then that variable will not be added to the equation
- Multiple regression From multiple regression equation it is possible to estimate the influence of each independent variable on the dependent variable and also the influence of all the variables together on the dependent variable
- Path coefficient analysis: the correlation between independent variable and dependent variable is particular particular of independent variable on dependent variable and indirect effects of independent variable on dependent variable through other independent variables.
- Path coefficient analysis : the sets of important independent variables which effect directly and which effect indirectly on dependent variable can be identified. the direct effect estimates are called path coefficients.

Ex: effect of protein on BMI directly and indirectly through iron, calcium and micronutrients sampling

• Universe

- Sample
- Statistical population/ population
- SamplingPurposive sampling
- Sampling frame

# Probability sampling methods:

- ➤ Simple random sampling
- > Stratified random sampling
- Cluster sampling
- Two-stage stage sampling
   Multi-stage stage sampling
- Systematic sampling
- Sampling methods
- ➢ Sample size
- Estimate of population mean
- ➢ Estimate of standard error
- ➢ Interval estimate

Case studies

Sampling

Simple random sampling

All possible samples will have equal chance of being selected as sample This method of sampling is preferred when the units in the population are not very heterogeneous. Sample can be selected using random tables / chits/ computer packages from the sampling frame.

**Stratified sampling**: in this method the population is divided into different strata or group based on a particular character then sample is drawn from each group to represent the entire population. If the sample represents in the same proportion in which they exist in the population then it is called stratified sample with proportional allocation otherwise it is called non proportional allocation.

- A) Identify and define population
- B) Determine desired sample size
- C) Identify subgroups for which sample
- representation is needed

- D) Classify population members into different sub groups that have been identified
- E) Use random table and select sample needed under each sub group

**Cluster sampling** –here population is divided into small groups called clusters and sample of clusters is drawn at random and study all the units in each selected clusters in the sample. Here intact groups, not individuals are selected. It may be the only feasible method when the data on individual units are not available but data on clusters is available in the population.Examples of clusters - classrooms, schools, hospitals, offices, villages.

# Steps:

- A) Identify and define population
- B) Determine desired sample size
- C) Identify and define a logical cluster

**Systematic sampling** – is a process in which individuals are selected from a list taking every kth person. K is determined by the researcher based on the size of the list, say nk divided by the sample size say n required. For ex: if the population size is 30 and sample size is 6 then k=5 i.e every 5<sup>th</sup> person will be selected in the sample. Difference between this method and others is that all members in a population do not have equal chance to be part of the sample. Once k is decided, and the first sample member is selected, the rest of the sample is automatically decided.

#### Steps in sample selection

- A) Identify and define population
- B) Determine desired sample size
- C) Obtain a list of the population
- D) Determine k = size of population /sample size

E) Use random table and select the first member of the sample

F) If  $3^{rd}$  person is selected randomly then k+3, 2k+3, ...., (n-1)k+3 will be automatically selected for a sample of n.

Sample size

# **Determining sample size:**

In general, the minimum sample size depends on the type of research involved. Some quote a minimum sample of 30 for co-relational research, a minimum of 30 in each group for casual-comparative and true experimental research. For descriptive research it is common to sample 10% to 20% of the population.

#### General rules to determine sample size:

1. The larger the population size, the smaller the percentage of the population required to get a representative sample

2. For smaller populations, there is no point in sampling; the entire population should be selected

Choosing inappropriate samples, convenient groups, and willing individuals can lead to sampling bias.

#### Selecting non - random sample

It is not always possible for researchers to use random sampling techniques. Non random sampling is the process of selecting a sample using a technique which does not permit the researcher to specify the probability or chance that each member of a population has of being selected for the sample. When non random samples are used, it is difficult to generalize to the population from which sample is selected. However if the population is not heterogeneous then the sample can represent the population.

1.convenient sampling – referred to as accidental sampling or haphazard sampling, is a process of using as the sample who ever happens to be available at the time, using volunteers and existing groups. The volunteers are different from the non volunteers, in the sense that they are more motivated and interested.

2. Purposive sampling also known as judgment sampling – is a process of selecting a sample that is believed to be representative of a given population. The researcher selects a sample using his experience and knowledge of the group to be sampled. The researcher deliberately identifies criteria for selecting a sample. Clear criteria provide a basis for describing and defending purposive samples.

3. Quota sampling – is a process of selecting sample based on required exact numbers or quotas, of persons of varying characteristics. It is most often used in descriptive research when it is not possible to list all members of the population of interest. When quota sampling is used, investigators are given exact characteristics and quotas of persons to be interviewed. This sampling technique is widely used in large scale surveys. Data is obviously gathered from easily accessible individuals.

1. Intensity sampling – selecting participants who permit study of different levels of the research topic. Example: Experienced and fresh teachers.

2. Homogeneous sampling – selecting participants who are very similar in experience, perspective; this process produces a very narrow, homogeneous sample and makes data collection and analysis simple

a) The extent to which the selected participants represent the range of potential participants in the setting.

b) The redundancy of information gathered from the participants; when researcher gets the same information repeatedly, she will know that more sampling of the group from the setting is not required. This is commonly known as data saturation.

#### NON-VERBAL COMMUNICATION By Dr Veeranjaneyulu



#### NABL ACCREDITATION - PROCESS & REQUIREMENT By SATISH YADAV

#### **OBJECTIVE:**

By the end of this class, you will be able to learn:

- A. Accreditation process
- B. General Requirement (ISO 17025:2005)
- C. Specific Requirement (NABL Documents 102,103,114, 141,161,163)

#### A. <u>ACCREDITATION PROCESS</u>

Accreditation Process involves following steps;

- 1. Preparation of Laboratory for Accreditation
- 2. Application to Accreditation
- 3. Maintenance of Accreditation
- 4. Surveillance of Accreditation
- 5. Renewal of Accreditation

#### 1. PREPARATION OF LABORATORY FOR ACCREDITATION:

- a) Formation of core team
- b) Prepare scope of analysis for application
- c) Training of ISO 17025 and analysis
- d) Prepare Quality Manual as per ISO 17025
- e) Quality System procedure
- f) Preparation of Standard Operating Procedures & Working Instructions
- g) Preparation of different forms, formats of register, datasheet etc.
- h) Calibration of equipment

#### 2. APPLICATION TO ACCREDITATION:

The process of application to accreditation involves following steps;

(Instrument & glassware)

- i) Validation of methods and standards
- j) Start analysis as per norms of ISO 17025 and generate data
- k) Conduct Inter Laboratory Comparison Program
- 1) Participation in PT Program
- m) Conduct Internal audit
- n) Compliance & Follow up of Internal Audit
- o) Management Review
- p) Preparation of Application for NABL Accreditation



# 2.1 Application for Accreditation

The laboratory is required to apply in the prescribed application form (NABL 151 for testing laboratories), in three copies along with two copies of the quality manual of the laboratory that should describe the management system in accordance with ISO/ IEC 17025: 2005. The application is to be accompanied with the prescribed application fee as detailed in the application form. Laboratory has to take special care in filling

the scope of accreditation for which the laboratory wishes to apply. In case, the laboratory finds any clause (in part or full) not applicable to the laboratory, it is expected to furnish the reasons.

# 2.2 Acknowledgement and Registration of Application

NABL Secretariat on receipt of application forms, the quality manual and the fees issues an acknowledgement to the laboratory. After scrutiny of application for its completeness in all respects, a unique laboratory ID number is allocated to the laboratory, which is used for correspondence with the laboratory. NABL Secretariat may ask for additional information/ clarification(s) at this stage, if found necessary.

#### 2.3 Appointment of Lead Assessor

NABL secretariat appoints a Lead assessor from the list of empanelled assessors. The lead assessor evaluates the adequacy of the quality manual, conducts pre-assessment and final assessment of laboratory concerned on behalf of NABL and submits the report to NABL secretariat. Towards the task of on-site assessment, he will be assisted by a team of assessors commensurate with the scope of accreditation.

#### 2.4 Adequacy of Quality Manual

The preliminary review for the adequacy of the application and quality manual submitted by the laboratory is carried out by NABL Secretariat whereas the detailed review is carried out by Lead Assessor. The lead assessor informs NABL regarding the adequacy of the quality manual, indicating inadequacies (if any) in the quality manual. The laboratory amends the manual and also implements the management system accordingly.

#### 2.5 Pre-Assessment

In case there are no inadequacies in the quality manual or after satisfactory corrective action by the laboratory, a pre-assessment visit of the laboratory is organised by NABL. The laboratory must ensure their preparedness by carrying out an internal audit and a management review before the pre-assessment. The pre-assessment of the laboratory is conducted to:

a. evaluate non-conformities (if any) in the implementation of the quality system.

b. assess the degree of preparedness of the laboratory for the assessment

c. determine the number of assessors required in various fields based on the scope of accreditation, number of key location to be visited etc.

The lead assessor submits a pre-assessment report to NABL Secretariat with a copy to the laboratory. The laboratory takes corrective actions on the non-conformities raised on the documented management system and its implementation and submits a report to NABL Secretariat.

# 2.6 Assessment

After the laboratory has taken corrective actions, NABL proposes constitution of an assessment team. The team includes the lead assessor (already appointed), the technical assessor(s)/ expert(s) in order to cover various fields within the scope of accreditation sought. NABL may also nominate an observer. NABL seeks laboratory's acceptance for the proposed assessment team and the laboratory is free not to accept one or more members of the proposed assessment team by giving specific reason(s) for their non-acceptance. After the

constitution of assessment team is finalized, NABL fixes dates for on-site assessment of the laboratory in consultation with the laboratory, the lead assessor and technical assessor(s)/ expert(s). The assessment team reviews the laboratory's documented management system and verifies its compliance with the requirements of ISO/ IEC 17025: 2005 and relevant specific criteria and other NABL policies. The documented Management system, SOPs, work instructions, test methods etc. are assessed for their implementation and effectiveness. The laboratory's technical competence to perform specific tests/ calibrations is also evaluated.

The assessment report contains the evaluation of technical manpower, all relevant material examined, test witnessed including those of replicate testing/ measurement, compliance to ISO/ IEC 17025: 2005 and relevant NABL specific criteria. The non-conformities if identified are reported in the assessment report. It also provides a recommendation towards grant of accreditation or otherwise. The report prepared by the assessment team is sent to NABL Secretariat. However a copy of summary of assessment report and copies of non-conformities if any, are provided to the laboratory at the end of the assessment visit.

#### 2.7 Scrutiny of Assessment Report

The assessment report is examined by NABL Secretariat and follow up action as required is initiated. Laboratory has to take necessary corrective action on non-conformities/ concerns and submit a report to NABL Secretariat within 60 days. NABL monitors the progress of closing of non-conformities.

#### 2.8 Accreditation Committee

After satisfactory corrective action by the laboratory, the Accreditation Committee examines the assessment report, additional information received from the laboratory and the consequent verification, if any. In case the Accreditation Committee finds deficiencies in the assessment report, the NABL Secretariat obtains clarification from the Lead Assessor/ Assessor/ Laboratory concerned. In case everything is in order, the Accreditation Committee makes appropriate recommendations regarding accreditation of the laboratory to the Chairman, NABL. All decision taken by NABL regarding grant of accreditation are open to appeal by the laboratory. The appeal is to be addressed to the Director, NABL.

# 3. MAINTENANCE OF ACCREDITATION

Conformance to applicable standards & NABL Requirements

The accredited laboratories at all times shall conform to the requirements of ISO/ IEC 17025: 2005 and relevant specific criteria and NABL Policies.

# NABL Terms and Conditions

The accredited laboratories are required to comply at all times with the terms and conditions of NABL given in NABL 131 'Terms & Conditions for obtaining and maintaining NABL Accreditation'. The laboratories are required to submit a signed copy of NABL 131 indicating their willingness to abide by the terms and conditions given in NABL 131.

# Modification to Accreditation criteria

If the accreditation criteria are modified by ISO/ ILAC/ APLAC/ NABL, the laboratory is informed of this in writing giving a transition period of at least 6 months to align its operations in accordance with the modified criteria.

# Adverse decision against the laboratories

If the laboratory at any point of time does not conform to the applicable standards and NABL criteria; or does not maintain the NABL terms and conditions; or is not able to align itself to the modified criteria, NABL may take adverse decision against the laboratory like denial of accreditation, scope reduction, abeyance (a state of temporary disuse or suspension.), suspension or forced withdrawal. NABL 216 'Procedure for dealing with adverse decisions' gives the details.

# 4. SURVEILLANCE & REASSESSMENT OF ACCREDITATION

The NABL accreditation certificate is valid for a period of 2 years. NABL conducts annual Surveillance which is aimed at evaluating continued compliance with ISO/ IEC 17025: 2005 and relevant NABL specific criteria and Policies. The types of surveillance's are given below:

# 4.1 On-site surveillance

For the newly accredited laboratories, in the first cycle of Accreditation, NABL conducts an on-site surveillance within 12 months from the date of accreditation. The first surveillance is similar to initial assessment and covers entire extension to the scope, (if any).

# 4.2 Desktop Surveillance

The desktop surveillance consists of calling of records from the laboratory to ascertain that the laboratory continues to maintain the requirements of ISO/ IEC 17025: 2005 and relevant NABL specific criteria. From the second cycle onwards the laboratory is subjected to desktop surveillance within 12 months of each reaccreditation.

# 4.3. Renewal of Accreditation / Reassessment

The accredited laboratory is subjected to re-assessment every 2 years. The laboratory has to apply 6 months before the expiry of accreditation to allow NABL to organise assessment of the laboratory, so that the continuity of the accreditation status is maintained.

The renewal application is submitted in the prescribed form (NABL 151) in three copies along with two copies of Quality Manual of the laboratory which describes the latest management system in accordance with ISO/ IEC 17025: 2005.

The application is to be accompanied by the prescribed renewal fee, as detailed in the application form.

The laboratory may request extension to the scope of accreditation, which should explicitly be mentioned in the application form.

# 5. APPEALS & COMPLAINTS

# Appeals

NABL is open to appeals from the laboratories against its decisions. The decisions against which appeals are entertained relate to denial of accreditation, reduction of scope of accreditation or abeyance/ suspension/ forced withdrawal of accreditation. The details are provided in NABL 134

# Complaints

NABL is open to receiving complaints for any of the activities performed by its officials, assessors, accreditation committee members and the accredited laboratories. The details are provided in NABL 132 'Procedure for Dealing with Complaints'.

# 6. RIGHTS AND OBLIGATIONS OF LABORATORIES

6.1 Rights of Laboratories

Laboratories are entitled to receive information related to laboratory accreditation. They can access NABL's website www.nabl-india.org which gives information necessary for NABL accreditation.

NABL is obliged to make available information on laboratories' scope of accreditation, validity dates for its certificate(s) and contact details to users of the laboratories. This information is provided at NABL web-site The laboratories are free to approach any accredited laboratory for traceability of measurements provided they fulfill the conditions laid down in NABL 142 'NABL Policy on Calibration and Traceability of Measurements'.

Laboratory has the right to object to appointment of specific member(s) of assessment team by giving valid reasons. NABL accredited laboratory has the right to use 'NABL Symbol' on the test/ calibration reports issued by it, as long as the test / Calibration is included in its scope of Accreditation. Detailed requirements governing use of 'NABL Symbol' and claim of accreditation have been stated in NABL 133.
NABL is open to receiving complaints for any of the activities performed by its officials, assessors, accreditation committee members and the accredited laboratories.

NABL is open to appeals from the laboratories against its decisions. The cases may involve refusal of accreditation, scope reduction, abeyance, suspension or forced withdrawal.

6.2 Obligations of the Laboratories

An accredited laboratory is obliged to fulfill requirements of relevant standard and NABL Specific Criteria and NABL 131 'Terms and conditions for maintaining NABL accreditation', at all times.

The laboratory is obliged to disclose name of the consultant; if applicable, at the time of applying for accreditation.

The laboratory is expected to provide access to all premises where key activities of laboratory are performed and afford access to all relevant information, documents and records necessary to assess laboratory's compliance to the relevant criteria, standards and NABL 131.

The laboratory is expected to facilitate work of the assessment team by providing necessary amenities including arrangement of appropriate test samples / devices for calibration and staff to demonstrate tests / calibrations.

An accredited laboratory can claim accreditation only with respect to the scope for which it has been granted accreditation as detailed in NABL 133, and not use accreditation in a manner to bring disrepute to NABL.

The laboratory is required to notify NABL of any change that may affect the ability of the laboratory to fulfill requirements of accreditation, within 15 days. Noticeable changes include (but are not restricted to): change in legal status, change in ownership, changes in organization, change in top management, change in key personnel and authorized signatories, major change in policies, change in locations etc.

The laboratory is required to pay necessary fees as determined by NABL from time to time.

# **GENERAL REQUIREMENT**

The International Standard IS/ISO/IEC 17025 : 2005 "General requirements for the competence of testing laboratories" contained all of the requirements that testing laboratories have to meet if they wish to demonstrate that they operate a management system, are technically competent, and are able to generate technically valid results.

Clause 4 specifies the Management Requirements and Clause 5 specifies the requirements for technical competence for the type of tests the laboratory undertakes.

### **Clause No. 4 - Management Requirement**

- 4.1 Organization4.2 Management system4.3 Document control4.4 Review of requests, tenders and contracts
- 4.5 Subcontracting of tests and calibrations4.6 Purchasing services and supplies4.7 Service to the customer
- 4.8 Complaints

<ul><li>4.9 Control of nonconforming testing and/or calibration work</li><li>4.10 Improvement</li><li>4.11 Corrective action</li></ul>	<ul><li>4.12 Preventive action</li><li>4.13 Control of records</li><li>4.14 Internal audits</li><li>4.15 Management reviews</li></ul>
Clause No. 5 - Technical Requirement	
5.1 General	5.6 Measurement traceability
5.2 Personnel	5.7 Sampling
5.3 Accommodation and environmental conditions	5.8 Handling of test and calibration items
<ul><li>5.4 Test and calibration methods and</li><li>method validation</li><li>5.5 Equipment</li></ul>	<ul><li>5.9 Assuring the quality of test and calibration results</li><li>5.10 Reporting the results</li></ul>

# SPECIFIC REQUIREMENT

The requirements specified in ISO/ IEC 17025, General requirements for the competence of testing and calibration laboratories are stated in general terms and, while they are applicable to all test and calibration laboratories, explanations might be needed. Such explanations on applications are herein referred to as applications. Applications should not include additional general requirements not included in ISO/ IEC 17025. Applications can be thought of as an elaboration of the generally stated criteria (requirements) for specified fields of test, test technologies, products, materials or specific tests. (ISO/ IEC 17025 Appendix B).

# NABL - 102 (Specific Criteria for Biological Testing Laboratories)

This criteria document provides extra information and interpretation on classes of test, personnel, accommodation and environment, equipment, reference materials/cultures and other aspects of laboratory management practices which are considered to be minimum standards for biological testing laboratories being accredited against NABL Laboratory Accreditation Program.

### NABL - 103 (Specific Guidelines for Chemical Testing Laboratories)

This document has been produced by a TECHNICAL COMMITTEE constituted by NABL for the purpose. It supplements ISO/ IEC 17025: 2005 standard and provides specific guidance on the accreditation of chemical laboratories for both assessors and laboratories preparing for accreditation. It gives detailed guidance for those undertaking quantitative and qualitative examination of the composition, nature and properties of materials, products and substances.

# NABL - 114 (NABL Guidelines for Food Testing Laboratories)

This document is intended to provide guidance for laboratories implementing ISO/ IEC 17025 for food testing, especially in support of international food trade activities. This document does not re-state all the provisions of ISO/ IEC 17025 and laboratories are reminded of the need to comply with all of the relevant criteria detailed in ISO/ IEC 17025.

# NABL - 141 (Guidelines for Estimation and Expression of Uncertainty in Measurement)

The purpose of the document is to harmonize procedures for evaluating uncertainty in measurements. The document will also provide broad guidelines to all those who are concerned with measurements about uncertainty in measurement, estimation and apportionment of uncertainty and interpretation of uncertainty. In fact, the purpose is to provide guidelines to users about contemporary requirements for global acceptance of various kinds of measurements. Attempts have been made to make the provisions of this document easy to understand and ready for implementation.

# NABL - 161 (Guide for Internal Audit and Management Review for Laboratories)

This document has been prepared to give laboratories guidance on how to establish a program for internal audit and management review. The document consists of two sections: Section A – Internal Audit and Section B – Management Review.

# NABL - 163 (Policy for Participation in Proficiency Testing Activities)

This document stipulates the minimum requirements of PT participation during accreditation process of testing laboratories. This also gives information about the measures against poor performance of laboratories in defined in Proficiency Testing program.