



ISAS newsletter

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ISAS newsletter is a quarterly email publication of Indian Society of Analytical Scientists composed and edited by Dr. V. Babu, Secretary-ISAS Kerala Chapter. He works as Scientific Officer in the R&D Department of Hindustan Insecticides Limited, Udyogamandal, 683501, Kerala State. To join the ISAS newsletter mailing list, contact Dr V. Babu by email babuv40@yahoo.co.in

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Editor's Note:

Welcome to ISAS News Letter Volume 1, No 3. Members are requested to We plan to start ,”Letters to Editor Column”. Members of ISAS may kindly send articles for publication in the newsletter. Articles may please be sent to the editor by e-mail. I would like to thank all the contributors for the time and effort they have spent preparing articles for this edition which I hope you will find both informative and interesting.

Indian Analytical Science Congress -2009 (IASC-2009)

The Indian Analytical Science Congress-2009(IASC-2009) will be held at Lonavala, a hill station in Maharashtra State during November 12-13, 2009. The theme of the congress was ,”Analytical Science for Industrial Development and Technological Progress”. Around 200 delegates are expected to participate in the congress. Leading analytical scientists from all over India are participating and delivering plenary / invited talks in this congress. In addition to Scientists from India a few scientists from abroad are also likely to participate in the congress. Details are available in the ISAS website isasinternational.com. Those who are desirous of participating in the conference may contact Dr V.R. Nair (e-mail drvnrnair@yahoo.com) or Dr K.K.A Rashid(k.k.a.rashid@sud-chemie-india.com) .

Programme

All major areas in analytical sciences will be discussed in IASC III .The Scientific Programme comprises the following major areas:

- Bioanalytical Chemistry
- Electroanalytical Chemistry
- Thermal Analysis
- Atomic and Molecular Spectroscopy
- Mass Spectrometry
- Separation Science
- Sampling and Sensor Technology
- Miniaturization Technology
- Environmental Analysis
- Food and Drug Analysis
- Materials Analysis

In addition to these main categories, the scientific committee will accept contributions of papers that are relevant to the field of Instrumental Methods of Analysis in general, describing important new concepts, or providing insights of analytical processes and methods, as well as novel applications to the area of chemical analysis. Contributions from

commercial organizations are encouraged, including detailed descriptions of new instrumentation, specific applications, and assessment of future commercial trends and opportunities. Prizes will be given to students who present the best posters.

Along with the congress an exhibition of analytical equipments by leading analytical equipment suppliers in the country will also be organized. Those who are interested to participate in the exhibition may kindly contact Shri Devaraj Aiyer, chairman of the local organizing committee (e-mail sgeindia@vsnl.com)



Lonavala - Venue for Indian Analytical Science Congress 2009

Message from President-ISAS

This is an exciting time for the ISAS as the preparations for the third Indian Analytical Science Congress(IASC-2009) at Lonavala , Maharashtra state during November 12-13 are being put into place. The Local Organizing Committee, chaired by Shri Devaraj Aiyer, is doing an excellent job organizing this congress . This IASC-2009 will be somewhat unique from past ones. In perusing the congress brochure, it's clear that this will be an excellent meeting, packed with keynote talks, papers and posters covering a broad range of analytical science topics. This event provides a not-to-be-missed opportunity for analytical scientists from all over India to come together to learn new developments in the area of analytical science and

explore potential collaboration opportunities in India's fast growing chemical and biological industry. For the Analytical Instrument companies, the event will serve as a unique opportunity to showcase their capabilities in analytical chemistry, bioanalytical chemistry, clinical and pharmaceutical analysis , and will provide opportunities for potential collaborations with partners and investors to further spur growth in the Indian market.

The theme of this year's event is, "Analytical science for Industrial Development and Technological Progress". Through keynote addresses, plenary sessions, industry insights, trend analysis, case study presentations and panel discussions, the event will bring together experts from all over , India together, to share new technologies, new analytical methods, instrumental techniques and new strategies to solve problems in analytical science. If you need more information on this meeting go to our website and click on the widget.

Preparation continues in the ISAS –sponsored workshop, "Application of X-ray fluorescence spectrometry in Analytical Science and Technology", on September 21st,2009. This workshop is organized in association with the Physics department of The Cochin University of Science and Technology. I'd like to thank Professor (Dr) Anantharaman ,Head of the Department of Physics, Cochin University of Science and Technology for his efforts in driving this initiative.

Thank you

Dr V.R.Nair

President-ISAS

Workshop on application of X - ray fluorescence spectrometry in analytical science and technology

ISAS in collaboration with the Department of Physics, Cochin University of Science and Technology is organizing an one day workshop on application of X - ray fluorescence spectrometry in analytical science and technology at the Physics Department



Cochin University of Science and Technology

Auditorium, Cochin University of Science and Technology, Cochin 682 022 on September 21st,2009. Experts in the area of Xray fluorescence from Bhabha Atomic Research Centre, Indira Gandhi Centre for Atomic Research Kalpakkam, National Centre for compositional characterization of materials ,user industries,etc are delivering talks in the work shop For details please see ISAS website www.isasinternational.com. Make your plans and join analytical scientists all over India participating in the workshop. This workshop offers junior and senior analytical scientists an opportunity and forum to exchange ideas and experience.



Photograph Physics Department Cochin University of Science and Technology where the workshop is being held

QUALITY AND FOOD SAFETY-A SUSTAINABLE APPROACH

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Agriculture is emerging as a major industry in India and Asia providing not only ample opportunity to farmers, but also generating significant export revenues for the economy. Value addition in agriculture produce has attracted the food processors due to increase in demand brought about by rising incomes, changing life styles and significant shifts in food habits. The competitiveness of food production would in future be more dependent on the reliability of the Safety and Quality of the food and the acceptability of the production procedures than on Quantity and Price. Therefore, agricultural supply of food production shall face remarkable changes which will not only be a challenge but also an opportunity for food producers to sell their produce globally. In addition, consumers have widely open choice to select a food.

Food is one of the major areas India has an inherent strength to dominate the global markets. But the dramatic growth of world trade food has increased the risk of consumer exposure to food-borne illness. Food systems in India are not always well organized as compared to developed countries. The overall increase in population in particular poses great challenges to food systems. Well established quality assurance systems are the need to meet the requirements of food safety and quality.

People have the right to expect the food they eat to be safe and suitable for consumption. Food borne illness and food borne injury are at best unpleasant; at worst, they can be fatal. But there are also other consequences. Outbreaks of food borne illness can damage trade and tourism, and lead to loss of earnings, unemployment and litigation. Food spoilage is wasteful, costly and can adversely affect trade and consumer confidence.

International food trade and foreign travel are increasing, bringing important social and economic benefits. But this also makes the spread of illness around the world easier. Eating habits too, have undergone major changes in many countries over a decade and new food production, preparation and distribution techniques have developed to reflect this. Effective hygiene control, therefore, is vital to avoid the adverse human health and economic consequences of food borne illness, food borne injury, and food spoilage.

Due to liberalization of the global trade with increasing demands from the consumers for healthy, safe and wholesome food, the food producers are changing to the quality oriented food markets instead of quantity oriented food productions. More emphasis is being laid down on quality and safety, nutritional value, labeling and traceability, besides value additions in food.

Safety and Quality

The growing influence of the consumers' demands reflects the foreseeable changes in the Quality Assurance (Q.A.) System, which needs to take a wider view of satisfying consumers' needs. Apart from the steady increase of the National and International standards for Food

Safety, the Quality Assurance System has to focus on the prevention of problems rather than its cure. Curing the problems is more difficult and expensive. Quality Assurance Program therefore includes the whole production and distribution system right from the 'raw materials' to the 'finished products' till used by the customers.

Food safety is the assurance that food will not cause harm to the consumer when it is prepared and/or eaten or is used as an ingredient in food according to its intended use, whereas Food quality is the totality of features and characteristics of a product that bear on its ability to satisfy stated or implied needs. In other words, good quality exists when the product complies with the requirements specified by the client/legal authorities. This means quality is a term defined by the consumer, buyer, grader, or any other client based on a number of subjective and objective measurements of the food product. These shall include measures of purity, flavor, color, maturity, safety, wholesomeness, nutrition, or any other attribute or characteristic of the product. Thus, safety is a component of quality. In fact, experts argue that safety is the most important component of quality, since lack of safety can result in serious injury and even death for the consumer.

Safety differs from many other quality attributes. A product can appear to be of high quality, i.e. well colored, appetizing, flavorful, etc. and yet be unsafe because it is contaminated with undetected pathogenic organisms, toxic chemicals, or physical hazards. On the other hand, a product, that seems to lack many of the visible quality attributes though not injurious, can be safe.

Quality assurance systems enable the application and verification of control measures intended to assure the quality and safety of food. They are required at each step in the food production chain to ensure safe food and to show compliance with regulatory and customer requirements. The systems are a set of controls implemented and verified by the responsible persons at each step in the chain (e.g. producers, farmers, fishermen, food processors, retailers, distributors, storage and transport, personnel etc.)

Safety and quality assurance should be ongoing. Both safety and quality assurance should focus on the prevention of problems, not simply curing them. Once safety or quality is reduced, it is virtually impossible to go back and improve. It is possible, however, to assure that the same problem does not affect future products. The need to produce and sell high quality products and increase in the 'efficiency' of the production process, however, has led to the development of quality assurance systems along the entire food chains.

Quality assurance is a formal methodology to assess the quality. The reliable results can only be obtained by the systematic application of quality assurance measures which include appropriate and calibrated instruments, validated methods, trained personnel, proper documentation and adequate laboratory infrastructure. Quality assurance tools such as proficiency or inter laboratory studies, use of reference materials and application of statistical evaluation (repeatability, reproducibility and accuracy) should be appropriate and accurate. Systematic implementation of quality assurance programs & procedures, thus, allows the identification of problems and application of corrective actions; thereby giving reliable results

and satisfying the consumer needs. Quality assurance can be achieved only when the staff is technically competent, well trained and motivated.

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. The production costs, however, have 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.

Quality attributes are often classified external (appearance, feel, defects), internal (odour, taste, texture) or hidden (wholesome, nutritive value and safety). Grades provide an opportunity to the consumer to select a food of his choice and quality, though it may be safe for all grades. Grading is a voluntary program. Grade standards describe the quality requirements for each grade of the product, giving industry a common language for buying and selling. Food standards give precise criteria to ensure that products are fit for their stated and implied purposes. They provide common frames of references to define a food. The standards are bench mark for consumers to purchase a food, for food producers/industries to produce/manufacture the food according to specified needs/quality and for regulatory authorities to check the quality and safety of food. Standards include quality of packaging material/container, labeling requirements, method of analysis and sampling. The standards are often used to provide the consumers with information about the products, maintain uniformity in quality of a food, establish market value, prevent economic fraud and above all provide safe food to a consumer.

Inspection is usually a mandatory process done by government or other certifying agencies to ensure product's wholesomeness, safety and adherence to quality. Therefore Quality Assurance, in contrast to Quality Control, is the implementation of quality checks and procedures to immediately correct the failure and mistakes that is able to reduce the quality of the interim products at every step.

The needs to produce and sell high quality products and increase in the 'efficiency' of the production process, however, has led to the development of Quality Assurance Systems along the entire food chains.

A Sustainable approach – For achieving safety and quality in food towards sustainable approach, the following practices should be adopted.

1] Food chain approach

It includes from primary production to final consumption. Requirements for any organization in the food chain, can be applied to organizations ranging from feed producers, primary producers through food manufacturers, transport and storage operators and subcontractors to retail and food service outlets together with inter-related organizations such

as producers of equipments, packaging materials, cleaning agents, additives and ingredients.” As food safety hazards can be introduced at any stage of the food chain, adequate control and verification throughout the food chain is essential”. Thus food safety is a joint responsibility that is principally assured through the combined efforts of all the parties participating in the food chain. ISO 22000 specifies the requirements for a food safety management system in the food chain where an organization needs to demonstrate its ability to control food safety hazards in order to provide consistently safe end products that meet both the requirements agreed with the consumers and those of applicable food safety regulations.

2] Good Agricultural/Manufacturing Practices (GAP/GMP)/Good Distribution/Good Transport/Good Storage Practices (GDP/GTP/GSP)/ Good Hygienic Practices

Good Agricultural Practices represent the basic environmental and operational conditions necessary for production of safe and wholesome produce. The various steps necessary for good agricultural practices are:

Preparation of land for cultivation, Application of manures and fertilizers, Quality of seed and certification, Sowing/planting/transplantation, Irrigation-water quality, Weeding/mulching/inter cultivation, Application of booster dose of fertilizer, Application of pesticides, fungicides, Harvesting, Cleaning, Drying, Storing, Grading, Testing and certification, Packaging, Labeling, Transport and Marketing.

Sources of Potential Farm Contamination are soil, irrigation water, animal manure, inadequately composted manure, Wild and domestic animals, inadequate field workers’ hygiene, harvesting equipment, transport containers (field to packing facility), wash and rinse water, unsanitary handling during picking, sorting and packaging in packing, facilities, in wholesale or retail operations, and at home, equipments used to soak, pack, or cut produce, ice, cooling units (hydro coolers), Transport vehicles, improper storage conditions (temperature), improper packaging, cross contamination in storage, packing and transportation.

Good Manufacturing Practices & Good Distribution Practices

- Good manufacturing and Distribution practices are measures designed to ensure an effective over all approach to product quality control and risk management. They do so by setting appropriate standards and practices for product testing, manufacturing, storage, handling, transportation and distribution.

Key Elements for GAP, GMP & GDP.

These elements set out the requirements of good practices with the aim to ensure food safety in agricultural produce.

This incorporates consideration of the following items in the present context.

1. Site and Environment- To prevent contamination and enable production of safe produce (GAP/GMP/GDP)
2. Local Environment: Maintain to an appropriate standard as per requirement (GAP/GMP/GDP)
3. Facilities for raw materials handling, preparation, processing, packaging and storage - Should be suitable for intended purposes (GAP/GMP/GDP)
4. Equipment – should be suitable for internal purpose and to minimize safety and quality risk. (GAP/GMP/GDP)
5. Maintenance – A system of planned maintenance should be in place. (GAP/GMP/GDP)
6. Staff Facilities – should be designed and operated in context of the requirements (GAP/GMP/GDP)
7. Contamination Risk - Effective facilities and procedures should be in place for control of physical, chemical and biological contamination of primary produce (GAP/GMP/GDP)
8. Segregation of cross contamination – Procedures should be laid down and implemented to prevent cross contamination of raw material, packaging material, and finished produce (GAP/GMP/GDP)
9. Stock Management - Procedures should be in existence for ensuring that materials and primary products are used in correct order and within their shelf life (GAP/GMP/GDP)
10. House keeping, cleaning and hygiene – Proper standards be maintained at all times (GAP/GMP/GDP)
11. Water Quality Management – Quality of water should be in accordance with the requirements and should be regularly monitored (GAP/GMP)
12. Waste Management - System should be in place for collation, collection and disposal of waste management. (GAP/GMP/GDP)
13. Insects and Pest Control - A system should be in place for controlling or eliminating the risk of contamination and infestation (GAP/GMP/GDP)
14. Pesticides, Herbicides, and Fungicides Control and Veterinary medicine – Integrated management system should be in place for use of chemicals at all the required stages. The drugs where required, shall be appropriate and should not be banned in the destination country (GAP)
15. Transport - This shall be ensured that the quality of the product is not deteriorated and no contamination takes place during transportation (GDP)
16. Training–All the persons are adequately trained, instructed and supervised. (GAP/GMP/GDP)

Good Hygienic Practices

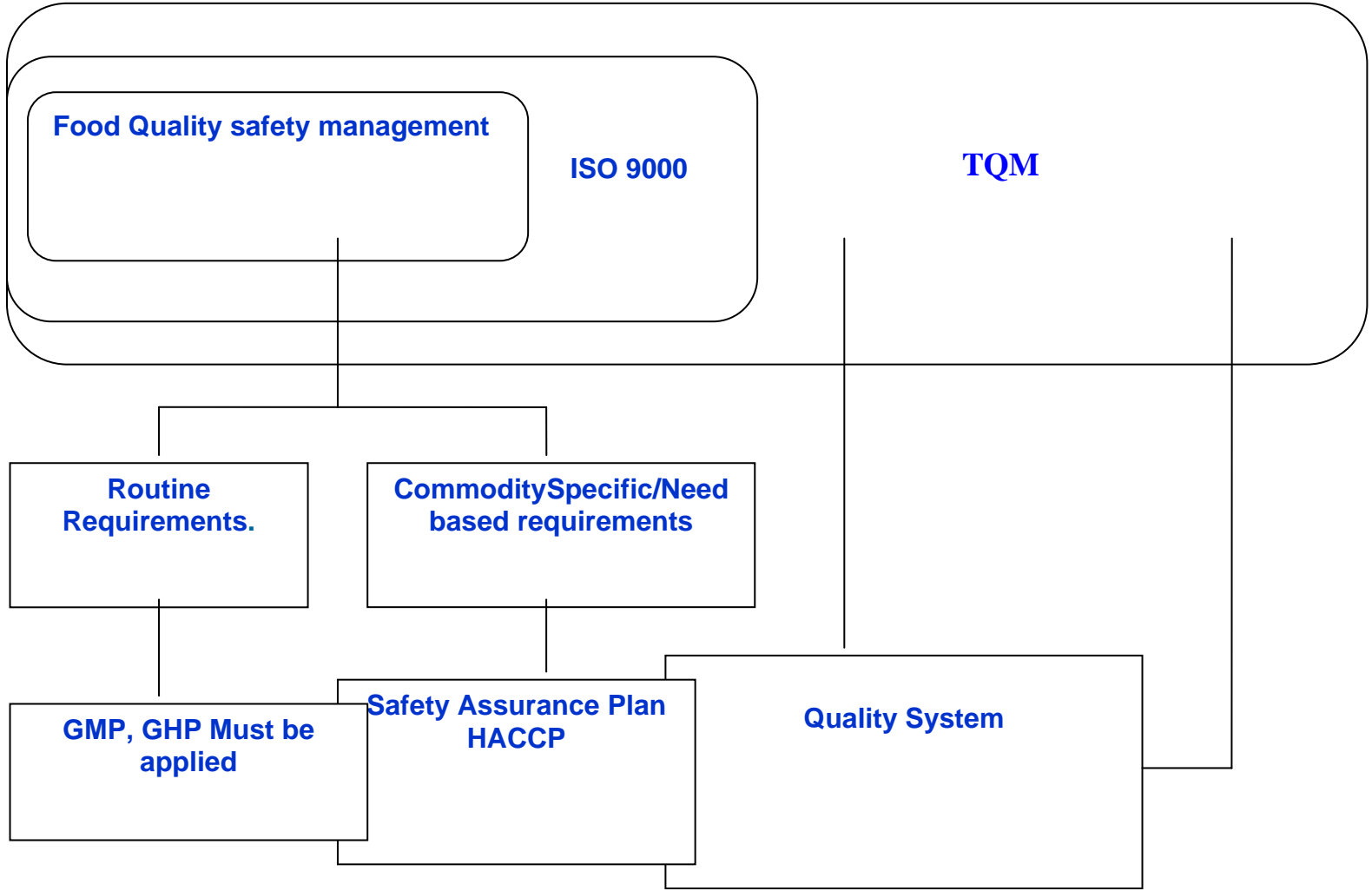
Effective hygiene control is vital to avoid contamination and adverse affects on human health and economic consequences of food borne illness, food borne injury and food spoilage. Important areas are given below:

1. Primary production :

Environment Hygiene, Hygienic production of food source ,Handling storage and transport, Cleaning, maintenance and personal hygiene at primary production.

2. Establishment, design and facilities :
Location, Premises and rooms, Equipments, Facilities like water supply, cleaning, personal hygiene facilities, and toilets, temperature control, air quality and ventilation, lighting, storage
3. Establishment, maintenance and sanitation:
Maintenance and ,Cleaning Program, Pest control system, Waste Management, Monitoring effectiveness
4. Establishment Personal Hygiene:
Health status, Illness and injuries, Personal cleanliness, Personal behavior, Visitor
5. Transportation:
General, Requirements, Use and maintenance
6. Product information and consumer awareness:
Lot identification, Product information, Labeling, Consumer education
7. Training:
Awareness and responsibility, Training program, Instruction and supervision, Refresher training.

Food Quality Safety Management



3] Quality and Safety Management System – key elements

1. General Requirements : The system should be documented, implemented, maintained and continually improved. Identify the process needed, criteria and methods required for operation etc.
2. Food Safety and Quality Policy : Food safety and Quality policy statement and objective should be specified to the extent of commitment to meet the quality and safety needs.
3. Food Safety and Quality Manual : It should contain scope appropriate to the activity with documented procedures or specific reference to those and describing the interaction of the related processes.
4. Management Responsibility : To establish a clear organizational chart that unambiguously states and document functions, responsibilities and reporting relationships, whose activities effect the safety and quality.
5. Management Commitment : The senior management/top management should demonstrate their commitments to develop and improve the quality and safety of the management system.
6. Management Review : Senior management well acquainted with the system and process should review the verification of management system at planned intervals to ensure its suitability, adequacy and effectiveness.
7. Resource Management : Senior management should determine and provide all the resources needed to implement the processes well in time and to meet the customer satisfaction.
8. General Documentation requirements : Documented procedures should be prepared and implemented. This could demonstrate compliance of the commitment made in the system and should ensure that all the records required to demonstrate the effective operational control of the processes and product safety are properly stored, effectively controlled and readily accessible.
9. Specifications : This shall be ensured that items and services purchased/provided do not adversely affect safety and quality requirement.
10. Procedures : Detail procedures and instructions for all the processes and operations having effect on the quality and safety shall be prepared and implemented.
11. Internal Audit : Internal audit system should be in place.
12. Control of non-conformity : Any non-conforming product in context of requirements is clearly identified and controlled to prevent intended use or delivery.
13. Corrective action : Procedure for determination and implementation of corrective action for significant non-conformance should be prepared and documented for implementation.
14. Product Release : Suitable procedures are laid down to ensure that produce is not released unless specific requirements are met.
15. Purchasing : All externally purchased items should conform to the requirements.
16. Supplier Performance Monitoring :
Procedures for approval and continuous monitoring of the supplies should be implemented.

17. Traceability: A proper system should be developed for purchase and supply of the product, ingredients or services.
18. Complaint : An effective system for management of complaint and their control and corrective evidence of the shortcoming should be in place.
19. Product Recall : An effective product recall procedure should be in place.
20. Control of measuring and monitoring device :Method to assure calibration and accuracy along with identification of measurement critical to quality and safety should be in place.
21. Product Analysis : A system should be prepared and implemented to ensure that final product, in-process ingredients conform to the required standards.

4] Hazard Analysis Critical Control Point (HACCP) / ISO 22000

- HACCP is a well-known technique used to analyze potential hazards in an operation; in identifying where these may occur and deciding which are critical to safety. HACCP can be applied throughout the food chain from the primary producer to final consumer and its implementation should be guided by scientific evidence of risks to human health.
- HACCP, therefore, is a tool to assess hazards and establish control systems that focus on prevention rather than relying mainly on end product testing. The hazards may be physical, chemical or microbiological and can occur during all stages from raw materials through to consumption by the consumer. Appropriate action can be taken to ensure that areas identified as **Critical Control Points (CCPs)** are kept under control and not allowed to endanger the items produced.
- The successful application of HACCP requires the full commitment and involvement of management and the workforce. It also requires a multidisciplinary approach. Application of HACCP systems can aid inspection by authorities and promote Quality.
- The HACCP system consists of the following seven principles
 1. Conduct a hazard analysis.
 2. Determine the Critical Control Points (CCPs).
 3. Establish critical limit(s) for each step.
 4. Establish a system to monitor control of the CCP.
 5. Establish the corrective action to be taken when monitoring indicates that a particular CCP is not under control.
 6. Establish procedures for verification to confirm that the HACCP system is working effectively.
 7. Establish documentation concerning all procedures and records appropriate to these principles and their application.
- Adoption of HACCP is becoming imperative to reach global standards and demonstrate compliance to regulations/requirements besides providing safer food. One of the main building blocks used for developing a Quality Assurance system is the “Hazard Analysis Critical Control Point” system (HACCP). HACCP is the

internationally recognized system to help assure safe food production. HACCP emphasizes prevention in the avoidance of food safety problems. This is based on Quality Control, Microbiological Analysis, Risk Management and is being adopted through out the world.

HACCP combines common sense with an evaluation of risks to identify the points along the Food Chain. The production of safe food requires all those involved from the primary production level to final consumption. Systematic implementation of Quality Assurance Program & procedures, thus, allows the identification of problems and application of corrective actions; thereby giving reliable results and satisfying the consumer needs. Quality Assurance can be achieved only when the staff is technically competent, well trained and motivated.

Conclusion

- Quality assurance and food safety is every one's responsibility throughout the food chain i.e. from farmer, market functionaries to consumers.
- Training and awareness on all aspects at different levels of their needs are the possible means for producing and providing a safe food to a consumer
- Follow good practices and hygienic practices to the extent of needs and requirements.
- Ensure compliance of mandatory requirements depending upon market/country.
- Control food safety at producer's level and contamination at every step.
- Apply HACCP and other management tools where safety and quality are involved.
- Final product testing in a competent laboratory.
- System benefits by way of reduction in rejections, creating confidence in quality product, economically viable, reduced costs for end product testing, enhanced reputation, consumer acceptance.

Compositional Characterization Of Carbon Electrode Material : A Study Using Simultaneous TG - DTA - FTIR

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Carbon materials have been widely used in both analytical and industrial electrochemistry. The diversity of carbon as an electrode material stems largely from its structural polymorphism, chemical stability, rich surface chemistry, and strong carbon-carbon bonds present both internally and often between the carbon material and a surface modifier[1]. The often-cited advantages of carbon electrodes include low cost, wide potential window, relatively inert electrochemistry, and electro catalytic activity for a variety of redox reactions.

It should be emphasized here that the physical and analytical electrochemistry using carbon electrode materials, depend on the electronic and chemical properties of carbon [2], which in turn, determine electrochemical performance of the electrode. Hence, it becomes necessary, to consider those aspects of the materials chemistry of carbon that dictate its electrochemical utility. All materials change their physical properties and their chemical characteristics under the influence of temperature. The properties most often studied are specific heat and enthalpy changes, weight loss or weight gain, thermal expansion and gas evolution[3]. Knowledge of these thermo-physical properties of solids and liquids has become very important for a sustainable energy industry and process economy. The temperature-contingent information on material properties determined by Thermal Analysis methods allows conclusions to be drawn regarding the identification of materials as well as their purity and composition, polymorphism and structural changes, thermal stability and temperature limits of application, aging behavior, thermo-mechanical behavior and viscoelastic properties, and processing conditions for shaping, casting, molding and extrusion. This comprehensiveness in material characterization encompasses all kinds of applications of

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Thermal Analysis for samples of organic, inorganic and biological origin [4].

Evolved gas analysis (EGA) [5] is a technique to determine the nature and amount of a volatile product/s formed during the thermal decomposition of materials. Continuous mode involves introducing the gaseous products directly into the detector system using an interface coupling, as in TG-FTIR, and TG-MS techniques. The main advantage of the continuous mode is the ability to conduct simultaneous and continuous real time analysis [6-7]. Several methods are available for the determination of carbon in different matrices. Essentially in all these methods, total carbon present in the sample is oxidized to give carbon dioxide and this liberated gas is detected by using various detectors [8-9]. The ability to measure the simultaneous and continuous mass loss of materials and monitor the gaseous products responsible for the mass loss allows TG-FTIR and TG-MS techniques to be widely employed in the material science field, including the study of coal pyrolysis and combustion, polymer degradation, evaluation of hazardous materials, thermal stability of materials, and formation of pharmaceuticals [10]. Considerable work [11-12] on characterizing materials, determining kinetic parameters, and quantitative analysis of degradation products has been performed with TG-FTIR.

In the absence of any systematic studies for the characterization of carbon with respect to its various forms in this sample matrix, current studies have been carried out. Authors have judiciously used available literature informations and took the advantage of inherent ability of TG-FTIR technique for the characterization of carbon electrode material with respect to its free and bound carbon content. All the experiments have been carried out using Netzsch Thermobalance (Model No.: STA 409PC Luxx) coupled to Bruker FTIR system (Model No.: Tensor 27) via a heated Teflon capillary (1m long , 2mm i.d.). Pt vs Pt 10%Rh was used as temperature sensor. Liquid nitrogen cooled MCT detector was used for the identification of IR absorbance/s in mid IR region (400 – 4000 cm^{-1}). Powdered carbon electrode samples (5 – 200mg) were heated to 450 – 1200⁰C in air / inert atmosphere with the heating rate of 5-10K/min.

Blank corrected thermo analytical curves for all the four samples have been depicted in Fig – 1. The corresponding total carbon content, the onset temperature for the combustion process and the temperature at which maximum absorbance was observed for the respective carbon dioxide evolution, has been listed in Table – 1. The observed

weight loss on TG curve is in the range of 95-99%. This suggests that the sample matrix should be highly crystalline graphite, based on the classification given elsewhere [13]. Integrity of the samples was checked by using suitable amounts of these samples and heating them in thermobalance to 1200⁰C at the heating rate of 10K/min in inert atmosphere. The observations have been depicted in Fig – 2. Identification of free carbon in these samples was carried out using the preconcentration mode of analysis. The typical recorded thermograms along with the simultaneous Gram Schmidt curves have been shown in Fig – 3 to Fig. - 6. The evolved carbon dioxide signal at lower temperature corresponds to amorphous carbon content in the sample matrix and the corresponding weight loss can be seen on DTG curve as shown in Fig. – 5. It can be seen from Fig. –6 that sample – 4 contains the least amount of amorphous carbon of these four different samples.

Current studies clearly indicate that simultaneous TG - DTA – FTIR is a sensitive and efficient tool for the identification and estimation of free carbon and bound carbon in any matrix. It should be emphasized here that this method of analysis can act as a preconcentration technique also, for the determination of free carbon, especially in the matrices like graphitic carbon i.e. the sample matrix used in this work where amorphous carbon is present in traces.

References:

1. K. Kinoshita, Carbon, Electrochemical and Physical Properties, John Wiley and Sons, New York, 1988.
2. R.L. McCreery, Chem. Rev., 108 (2008) 2646 – 2687.
3. T. Daniels, Thermal analysis, Kogan Page Ltd., London, 1973.
4. J. W. Hellgeth, Handbook of Vibrational Spectroscopy, (Eds J.M. Chalmers and P.R. Griffiths), John Wiley and Sons, New York, 2 (2002)1699.
5. W.W.Wendlandt, Thermal Analysis, IIIrd Ed., John Wiley and Sons, New York,1985.
6. J. Chiu, Anal. Chem., 40 (1968) 1516.
7. F. Zitomer, Anal. Chem., 40 (1968)1091.
8. ASTM, 12.01 (2003) C 26.05.
9. M.K. Ahmed, R. Geetha, N.K. Pandey, S. Murugesan, S.B. Koganti, B. Saha, P. Sahoo and M.K. Sundararajan, Talanta, 52 (2000) 885.

10. W. Xie and W.P. Pan, J. Therm. Anal. Cal. 65 (2001) 669-685.
11. C.L. Putzing, M.A. Leugers, M.L. Mckelvy, G.E. Mitchell, R.A. Nyquist, R.R. Papenfuss and L. Yurga, Anal. Chem., 66/12 (1994) 51R.
12. W.M. Groenewoud and W. de Jong, Thermochim Acta, 286 (1996) 341-354.
13. Michel Dumont, Graphite, Canadian Minerals Year Book, 2004, p-25.1.

Table – 1

Simultaneous TG – DTA – EGA data observed for different carbon electrode samples:

Sample No.	Sample wt. (mg)	Wt. loss (%)	TG _{onset} (°C)	TG _{offset} (°C)	DTA _{peak} (°C)	EGA _{peak} (°C)
1.	4.9	98.04	652	747	692	695
2.	5.0	96.77	593	686	641	642
3.	5.4	97.28	587	680	639	641
4.	5.3	98.56	613	705	662	664

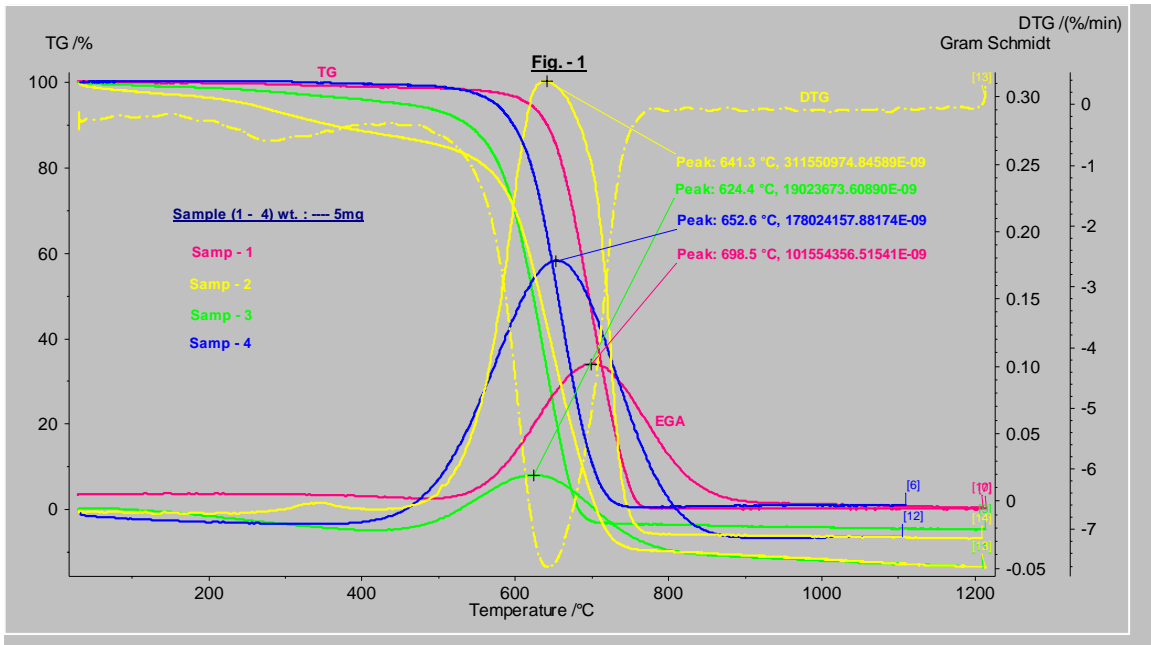
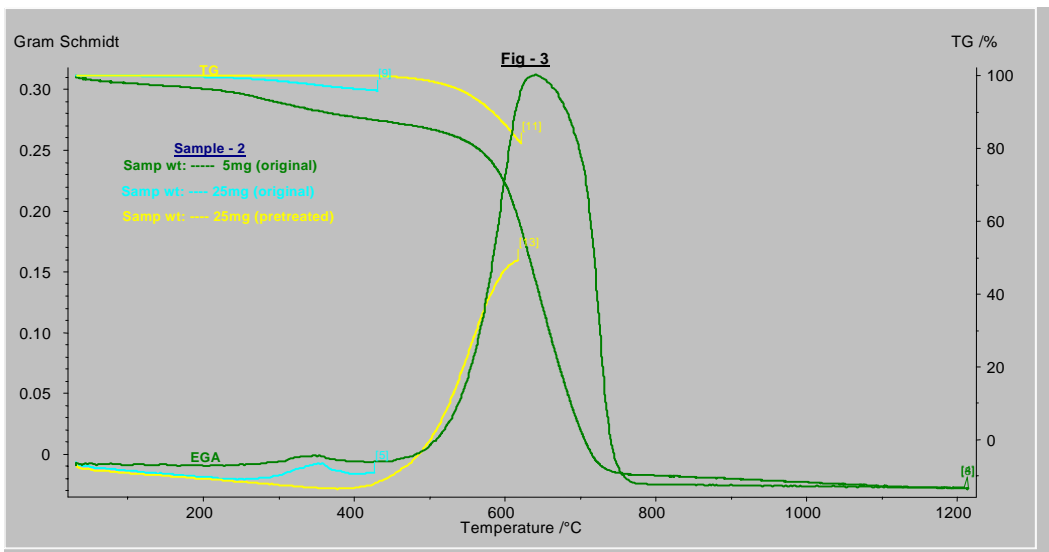
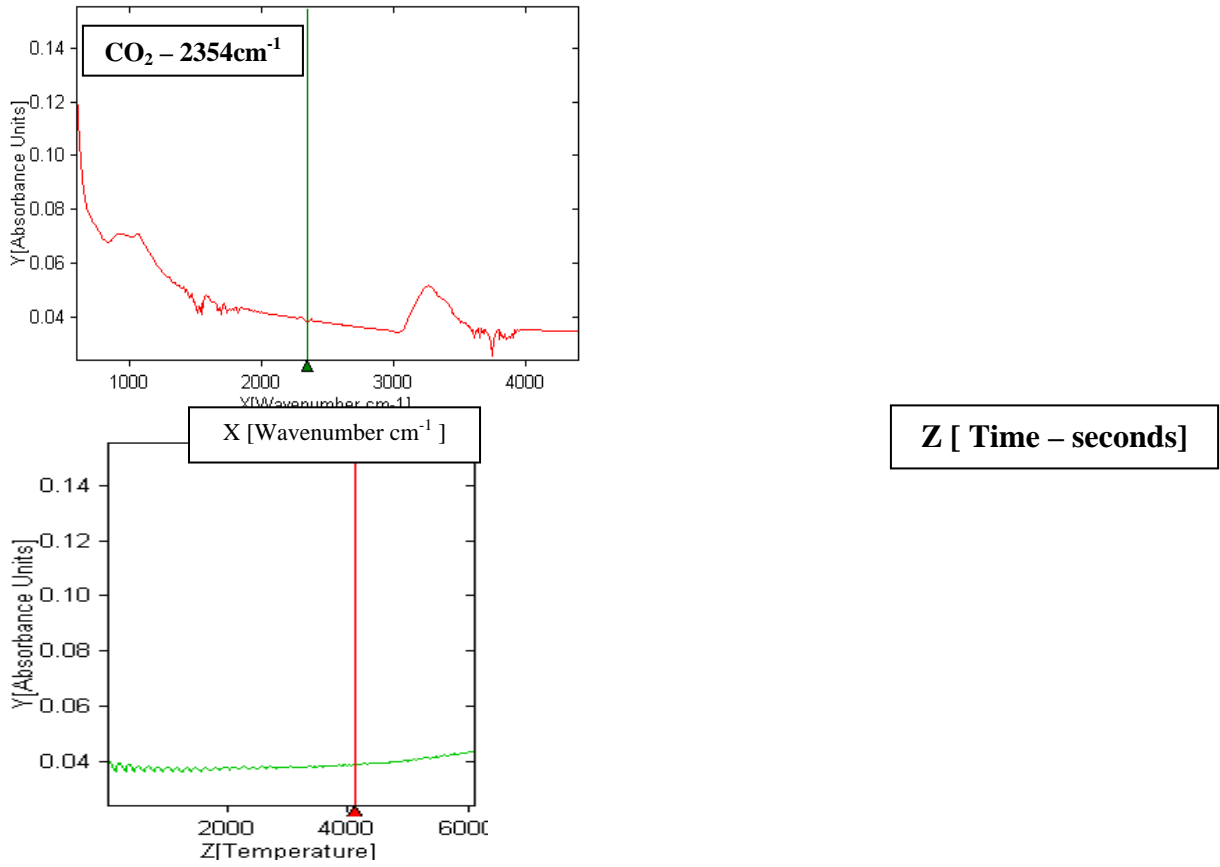


Fig. – 2

Gram Schmidt & CO₂ evolution pattern of carbon electrode sample in inert atmosphere:



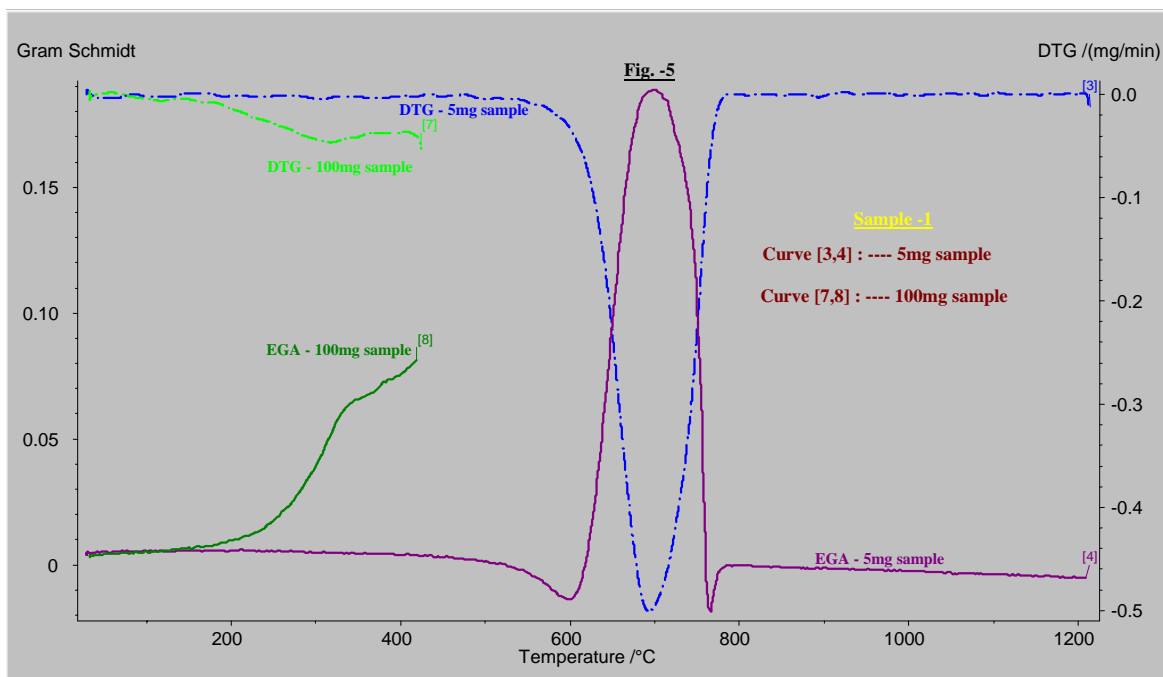
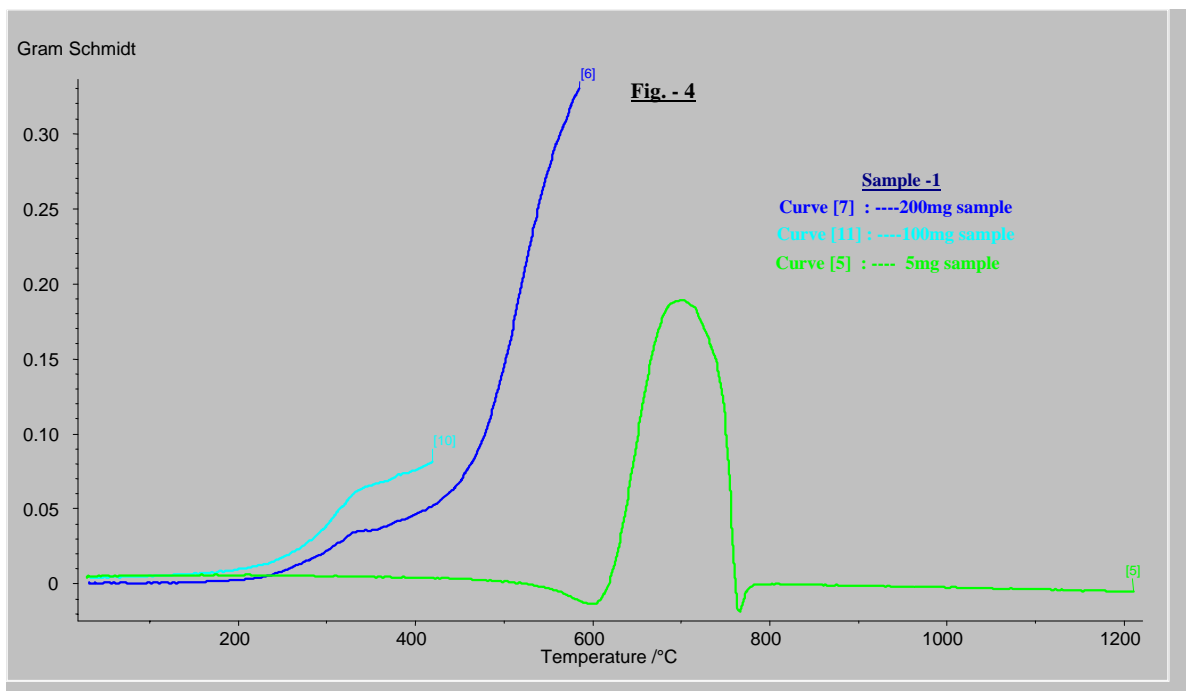
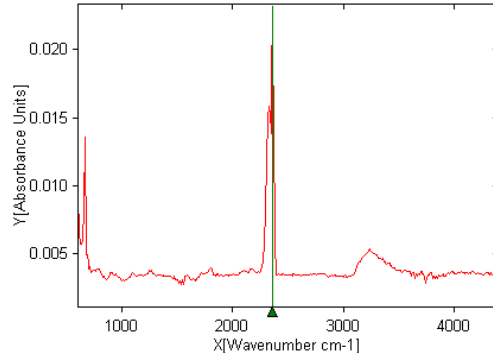
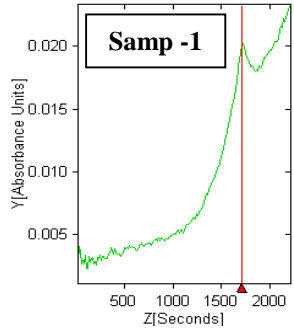
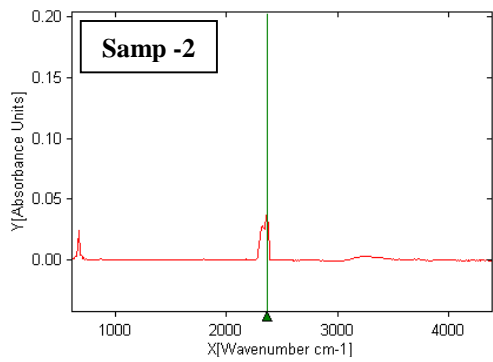
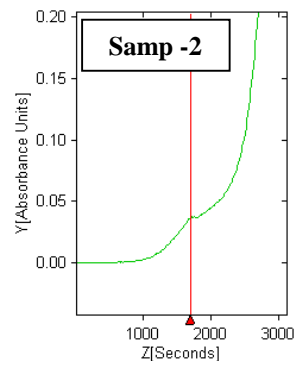


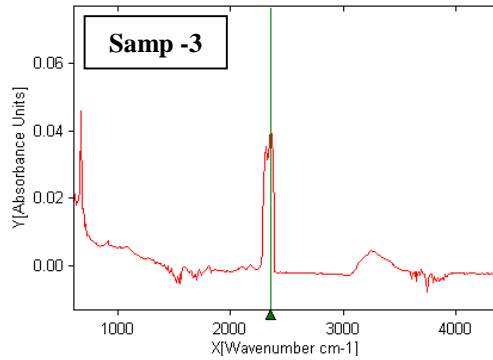
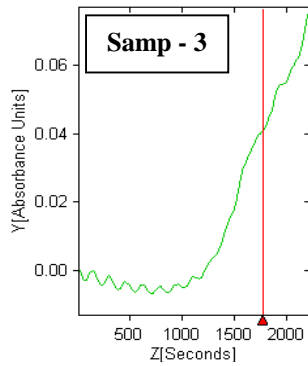
Fig. – 6
Effect of preconcentration (sample wt – 200mg):



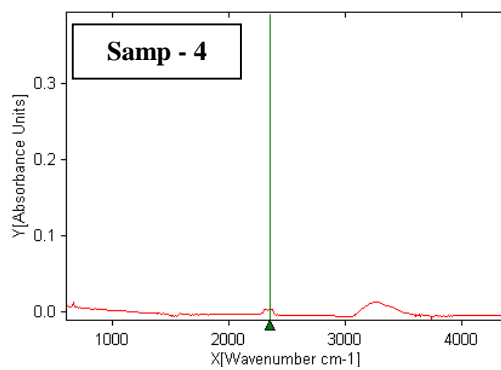
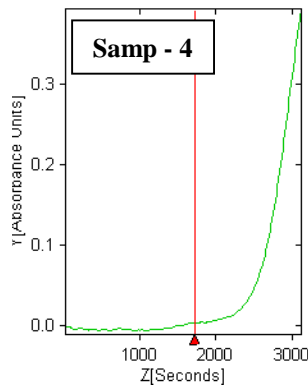
CO₂ – 2354cm⁻¹



CO₂ – 2354cm⁻¹



CO₂ – 2354cm⁻¹



CO₂ – 2354cm⁻¹