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For Efficient Dairy Plant Operation

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ADVANCED INSTRUMENTAL TECHNIQUES FOR MILK AND MILK PRODUCTS ANALYSIS

This bulletin includes technical information based on latest developments on products, systems, techniques etc. reported in journals, companies' leaflets and books and based on studies and experience. The technical information in different issues is on different areas of plant operation. It is hoped that the information contained herein will be useful to readers.

The theme of information in this issue is **Advanced Instrumental Techniques for Milk and Milk Products Analysis.** It may be understood that the information given here is by no means complete.

In this issue:

1. INTRODUCTION

Analysis (chemical / microbiological / physical) of dairy products is an important tool in assessing and controlling the quality of products (raw, intermediate and final), determining and standardizing product composition, and effectively controlling production and manufacturing processes on a real time basis. The analytical chemical methodology used in the dairy industry ranges from traditional wet chemistry to the modern instrumental techniques.

Traditional wet chemistry methods being time consuming and generally suitable for analyzing major components of milk and dairy products can seldom be used for real-time monitoring for effective product and process control. To overcome these difficulties, various advanced instrumental techniques have been developed that are capable of analyzing major and minor components, contaminants and other processing induced chemical changes in dairy products, take drastically less time in product analysis and give accurate analytical results.

This issue of *Technews* provides a brief review of advanced instrumental techniques that have provided newer approaches to effectively deal with the product and process requirements in the emerging food quality and safety scenario the world over.

Whereas the focus in this issue is on chemical analysis, certain techniques are also useful in the microbiological analysis and are, hence, mentioned appropriately at various places.

2. ADVANCED INSTRUMENTAL TECHNIQUES

2.1 Spectroscopy techniques

Spectroscopy is the study of physical systems by the electromagnetic radiation or ultrasonic waves with which they interact or that they produce. Spectrometry is the measurement of such electromagnetic radiations/ultrasonic waves as a means of obtaining information about the systems and their components. In certain types of optical spectroscopy, the radiation originates from an external source and is modified by the system, whereas in other types, the radiation originates within the system itself.

Spectroscopic techniques used in dairy analysis are:
- UV-VIS Spectroscopy
- Microwave spectroscopy
- Low resolution nuclear magnetic resonance spectroscopy
- Infrared spectroscopy
- Atomic spectroscopy
- Ultrasound spectroscopy

2.1.1 Infrared Spectroscopy[1]: Infrared Spectroscopy is the most important and widely used rapid quantitative analytical tool in the dairy industry. The technique is based on the absorption of radiation by the sample and is characterized by the wavelength of the light used, commonly mid infrared (MIR) and near infrared (NIR). The advantages of the technique include the following:
- The method is inexpensive, fast, reliable, accurate and environment friendly.
- Provides for simultaneous determination of various major milk constituents.

Applications in dairy industry: Nearly all major constituents in all dairy products can be analysed by infrared spectroscopy. Some important parameters measured by infrared spectroscopy are listed in the table below:

Products	**Constituents**
Raw milk	Fat, protein, casein, whey protein, lactose
Skim milk	Dry matter, protein, casein
Market milk	Fat, protein, dry matter
Cream	Dry matter, fat
Evaporated milk	Dry matter, fat
Skim milk powder	Water, fat

Products	Constituents
Milk powder	Water, fat, protein, lactose
Creamer	Water, fat
Coffee creamer	Water, fat, protein
Ice cream mix	Dry matter, fat
Feeding stuffs	Various constituents

Besides, infrared spectroscopy being accurate and fast is also used in the process line for better process control. It fulfills the analytical task nearly simultaneously with the production line (on-line). In dairy industry the following major applications of infrared spectroscopy are used for process control:

- The water content of milk powder could be determined directly after the drying chamber with a near infrared spectrometer. This allows on the spot regulation of feed to the chamber for moisture control.
- To standardize fat and protein in milk, a mid infrared spectrometer could be used to measure these components on-line.

Good laboratory practice (GLP) for infrared spectroscopy calibrations: Infrared spectroscopy is an indirect method and hence requires a calibration step with a reference chemical method and subsequent regular check on the performance of the method. To ensure proper performance of instruments, it is necessary to establish GLP guidelines such as provided below:

- Take the infrared spectra of an inert standard (a certified material of known concentration) periodically. If the difference between the standard spectrum and the spectrum obtained with the instrument is unacceptable, the instrument must be recalibrated.
- In cases where a reference material does not exist, the performance of the equipment must be monitored by analyzing corresponding samples by a reference method. Any difference between the results obtained by reference method and infrared spectrometer beyond acceptable limits warrants new calibration of the infrared equipment.

International standards: The following international standards have been specified for application of infrared spectrometric techniques in the analysis of dairy products:

- *ISO 21543 / IDF 201:2006 – Milk products - Guidelines for the application of near infrared spectrometry*
- *IDF 141C:2000 – Whole milk - Determination of milkfat, protein & lactose content – Guide for the operation of mid-infra-red instruments*
- *AOAC 972.16 – Fat, lactose, protein and solids in milk (Mid-Infrared Spectroscopic Method) (Also AOAC 975.18 – Protein in Milk and AOAC 975.19 Lactose in Milk)*

2.1.2 Atomic Spectroscopy[(2)]: Atomic spectrometric techniques are used for qualitative and quantitative determination of approximately 70 elements.

In atomic spectroscopy, the sample is placed in an environment that is hot enough to break molecular bonds and produce atoms. The atoms are then identified and their concentration measured by emission or absorption of characteristic radiation. In atomic absorption spectrometry (AAS), a light source emitting radiation characteristic of a specific element is passed through the atomised sample and the transmitted radiation is measured. In the atomic emission spectrometry (AES), the sample is heated to sufficiently high temperatures that the electrons of the atoms are excited. When the electrons return to their normal position, they emit radiation characteristic of the element present. The radiation transmitted through the atomizer is detected by either photon detectors or mass spectrometers. Mass spectrometers provide a very high sensitivity.

Applications in dairy industry: Atomic spectrometry has been used successfully for the analysis of trace and major elements in milk and milk products. These include elements of nutritional importance (such as Ca, P, I, Fe, Mg, Cu, Zn etc.) and toxic contaminants (Pb, Hg, As etc.).

The most commonly used instruments for the analysis of trace elements in milk and dairy products are:

- Flame atomic emission spectrometry (FAES) - A low cost technique useful for determination of the relatively high concentrations of sodium and potassium that are present in dairy products.
- Flame atomic absorption spectrometry (FAAS) - Useful for analysing liquid samples (milk etc.) directly, eliminating the need for sample preparation. The method is useful for the detection of a number of elements in dairy products, including Ca, Cu, Fe, Mg, Mn and Zn.
- Electrothermal atomic absorption spectrometry (ETAAS) – Capable of *in situ* sample preparation and, therefore, solid samples can be analysed directly for several elements. However, use of slurries or suspension of powdered samples with conventional liquid sample handling system facilitates better analysis of solid samples.
- Inductively coupled plasma atomic emission spectrometry (ICPAES) – Capable of fast-sequential and simultaneous multi-element analysis with lower limits of detection. Also provide wide linear dynamic range of calibration which is desirable for milk and milk products.
- Inductively coupled plasma mass spectrometry (ICPMS) – Provides enhanced sensitivity, multi-element and isotopic analysis, and detection of non-metals (such as phosphorous and iodine) that are of interest in milk and dairy products

A summary of the analytical characteristics of the above instruments are given in the table below:

Technique	Sample volume (ml)	Limit of detection (ppb)	Multi-element
FAES	5-10	1-100	Yes
FAAS	5-10	1-1000	Possible
ETAAS	0.01-0.1	0.01-0.1	Possible
ICPAES	1-10	0.05-10	Yes
ICPMS	1-10	0.001-0.01	Yes

Method validation: Atomic spectrometry based analytical methods are validated through the following approaches:

- Analysis of standard reference materials
- Comparison of results obtained using a second analytical method (reference method)
- Comparison of results obtained from another analytical laboratory (reference method)

AOAC has specified the following standard for estimation of various elements in cheese by atomic absorption spectroscopy:

AOAC 991.25 Calcium, Magnesium and Phosphorus in Cheese

2.1.3 Ultrasound Spectroscopy[3]: Ultrasonic techniques are finding increasing use in the food industry for the analysis of foods. Low-intensity ultrasound is a non-destructive technique that is based on the measurement of parameters of low energy ultrasonic waves propagating through the analysed samples. The technique can provide information about physicochemical properties of food products, such as composition, physical state and flow rate.

2.2 Chromatography techniques[4]

Chromatography is a physical method of separation in which the components to be separated are distributed between two phases, one of which is stationary (stationary phase) while the other (the mobile phase) moves in a definite direction.

Chromatographic methods are very useful for analysis of complex foods such as milk and dairy products.

In general chromatography comprises separation techniques in which the analytes partition between different phases that move with different velocities relative to each other. Two important highly automated chromatographic techniques used for analysis of dairy products are:

- High performance liquid chromatography (HPLC): In principle anything that can be dissolved can be analysed by HPLC.

- Gas chromatography (GC): Components that are volatile, or could be made volatile by heating or derivatization, can be analysed by gas chromatography.

International standards: Some important international standards that have been specified for chromatographic analysis of specific components of milk and milk products are given in the tables below:

HPLC –based methods

Component	Product	Standard
Vitamin A	Dried skimmed milk	IDF 142:1990
Vitamin D	Dried skimmed milk	IDF 177:2002
Aflatoxin M^1	Milk and milk powder	ISO 14501/IDF 171:2007 and ISO 14674/IDF 190:2005
Benzoic and sorbic acid	Milk, dried milk, yoghurt and other fermented milks	IDF 139:1987
Natamycin	Cheese, cheese rind and processed cheese	ISO 9233-2 / IDF 140-2:2007
Lactose	Milk and milk products	ISO 22662/IDF 198:2007
Lactulose	Heat treated milk	ISO 11868/IDF 147:2007
Heat treatment intensity	Dried milk	ISO 11814/IDF 162:2002
Acid soluble β-lactoglobulin	Liquid milk	ISO 13875/IDF 178:2005

GC/GLC-based methods

Component	Product	Standard
Vegetable fat	Milk fat	IDF 054:1970
Sterol composition	Anhydrous milk fat	ISO 12078/IDF 159:2006 and ISO 18252/IDF 200:2006
Fatty acid composition	Milk fat	ISO 15885/IDF 184:2002
Poly chlorinated biphenyls	Milk and milk products	IDF 130A:1991
Pesticides (organochlorine compounds)	Milk and milk products	IDF 144:1990 and 75C:1991

2.3 Immunochemical (immunoassay) techniques[5]

Immunoassays can be defined as quantitative binding reactions between antibodies and target antigens. The technique uses a specific antigen or antibody, capable of binding to the analyte, to identify and quantify substances. The antibody can be linked to a radioisotope (radioimmunoassay), or to an enzyme which catalyses an easily monitored reaction (enzyme-linked immunosorbent assay), or to a highly fluorescent compound by which the location of an antigen can be visualized (immunofluorescene).

Immunoanalytical techniques, listed below, have been increasingly applied in the analysis of food components, chemical contaminants in food and food borne pathogens:
- Enzyme immunoassay (EIA)
- Radio immunoassay (RIA)
- Enzyme-linked immunosorbent assay (ELISA)
- Enzyme-linked coagulation assay (ELCA)
- Immunosensor with multiple amperometric detection (ISA)
- Immunosensor based on antibodies attached to quartz crystal (ISC)

Applications in dairy industry: Immunochemical techniques are successfully used in detecting bacterial and fungal cells and toxins, veterinary drugs, pesticides etc.

- Antibiotics, toxins and other undesirable compounds: Various immunochemical techniques are capable of detecting residues of these contaminants in milk with high degree of sensitivity and accuracy.
- Detection of bacteria and moulds: ELISA combined with an enrichment step substantially reduces the time in detecting bacteria and moulds in foods as compared with conventional microbiological methods. ELISA can detect moulds before their growth can be seen on dairy products.
- Milk proteins, enzymes and enzyme inhibitors: ELISA with antibodies specific for native ά-lactalbumin and β-lactoglobulin may be used to differentiate between raw, pasteurized and ultra high

temperature (UHT) milk. Test methods based on ELISA can indirectly identify recombinant chymosin from three different organism (*E. coli, Kluyveromycis lactis* and *Aspergillus* niger) which are used for manufacturing cheeses.

Test methods, based on these techniques, have been developed for milk and are listed in the table below:

Method	Analyte	Detection limit
EIA or RIA	Staphylococcal enterotoxins A, B, C, D, E	0.1-1 ppb
	Pesticides and herbicides	0.2-1 ppb
EIA	Aflatoxin M_1	0.25 ppb
	Ochratoxin A	-
	β-Lactam antibiotics	0.05-1 ppb
	Chloramphenicol	0.5-2 ppb
	Suphamethazine	2-10 ppb
	Fragment of κ-casein macropeptide to detect proteolysis	100 ppb
ELISA	*Clostridium perfringens* enterotoxins A	5 ppb
	Chymosin	0.25 ppb
	Heat-stable proteinase from *Pseudomonas*	0.08 ppb
	Staphylococcus spp.	10^4-10^5 cells / ml
ELCA	*Clostridium botulinum* toxins A, B, E	0.01 ppb
ISA	*Listeria monocytogenes*	$2x10^2$ cells / ml
ISC	*Listeria monocytogenes*	$2.5x10^5$ cells / ml

AOAC has specified several test methods based on immunoassays for screening of Salmonella, Listeria, Staphylococcal toxins etc. in foods.

2.4 Biosensor techniques[6 and 7]

Biosensors are sensors that use biological material to make measurements. They use specific biochemical reactions mediated by isolated enzymes, immunosystems, tissues, organelles or whole cells to detect chemical compounds usually by electrical, thermal or optical signals. Biosensors consist of a recognition element (biological receptor), a signal conversion unit (transducer) and an output interface (the electronic component for interacting with the instrument). The biological receptors could be non-catalytic (such as cell receptors or antibodies) or catalytic (such as enzymes, microorganisms, plant or mammalian tissue etc.). Biosensors that use antibodies are called immunobiosensors.

Biosensors are a potentially powerful tool for quality control testing of food products, product composition, the control of production and processing and the management of dairy animals. They could also be used for analysis of products for the presence of toxins, pathogens, antibiotics, pesticides and chemicals. Biosensors have the following potential advantages over other methods of analysis:

- Fast and real-time detection enable producers to take corrective action before a product is further processed or released for consumption
- Many biosensors allow analysis of larger volumes of liquids in a continuous flow.
- Biosensors can be miniaturized and can be integrated into dairy equipment such as milking systems, dairy tanks, centrifuges etc.
- Biosensors can be integrated with on-line process monitoring schemes to provide real-time information about multiple parameters at each production step or at multiple time points during a process, enabling better control and automation of milk and dairy facilities
- Biosensors can also be integrated into HACCP programme, enabling critical examination of the entire food manufacturing process.

Dairy industry applications: Various applications of biosensor based analytical methods in dairy industry include the following:

- Antibiotic analysis: Various biosensors have been developed to detect antibiotics in milk.
- Bacterial analysis: A list of biosensors developed for the analysis of microbial contaminants in milk and dairy products is given in the table below:

Biosensor technique	Organism	Detection range (cells /ml)
Electrochemical	• *Listeria monocytogenes* • *Salmonella typhimurium* • *Staphylococcus aureus* • *E. coli* O157:H7	10^3 - 10^6
Resonant mirror	• *Staphylococcus aureus* • *Salmonella* spp. • *E. coli*	10^5 – 10^7
Light addressable potentiometric sensor	• *Salmonella typhimurium* • *E. coli* O157:H7	10^2 – 10^4
Fluorescent fibre optic	• *E. coli* O157:H7 • *Salmonella typhimurium*	10^2 – 10^5
Piezoelectric	• *Salmonella* spp. • *E. coli* • *Vibrio cholerae*	10^5 – 10^8

- Mastitis detection: An electric conductivity biosensor has been developed as a mastitis detection tool.
- Microbial toxin analysis: Optical biosensors have been used for direct real-time analysis of *Staphylococcal* enterotoxins in milk. There are many biosensors that have been designed to analyse milk for aflatoxin M_1. Detection limits and time for some biosensors developed for detection of various microbial toxins in milk are provided in the table below:

Toxin	Detection limit	Detection time
Staphylococcal eneterotoxin A	~10 ppb	< 5 min.
Staphylococcal eneterotoxin B	5 ppb	< 2 min.
Aflatoxin M_1	0.05 ppb	18 min.
Aflatoxin M_1	0.1 ppb	< 2 min

- Effectiveness of milk heat treatment: Lactulose which is produced during UHT process and generally present in UHT milk at a level of around 500 ppm can be monitored by the biosensors techniques to assess the intensity of thermal process.
- Biosensors for quality control: Biosensors can be used to monitor

levels of L- and D- amino acids in milk, the content of which in milk may decline depending upon the type of processing and the duration and conditions of storage. Such analysis would give information about effects of milk ageing.

- Biosensors have been developed for on-line measurement of major components of milk solids such as fat, protein, lactose and calcium.
- Integration with HACCP programme: There are several possible uses for biosensors in HACCP monitoring, including screening of antibiotics residues, assaying for pathogenic organisms and assessing the effectiveness of milk heat treatments in dairy plants.

3. SPECTROSCOPY BASED EQUIPMENT AVAILABLE FOR RAPID ANALYSIS OF MILK AND DAIRY PRODUCT

Some spectroscopy based equipment suitable for dairy plants for rapid analysis of milk and dairy products developed and currently available in the market include the following:

A. Visible spectroscopy based[8]

- **Chemspec 150:** An automatic, fast and compact visible spectroscopy based equipment specifically designed for the determination of urea (or MUN) in milk (up to 150 samples per hour).

B. Infrared Spectroscopy based [9 and 10]:

- **MilkoScan™:**

  MilkoScan™ Minor: Infrared based equipment for analysis of 6 parameters (fat, protein, lactose, total solids, solids-not-fat, and freezing point depression) of milk samples.

  MilkoScan™ FT: Fourier Transform Infrared Technology (FTIR) based equipment for high speed automatic multicomponent analysis (including saturated and unsaturated fatty acids, freezing point depression, urea, casein and free fatty acids) of raw milk.

MilkoScan™ FT 120: FTIR based versatile equipment for analysis of raw milk, intermediates and final products. It has a wide range of ready to use calibrations. An improved version of the equipment is MilkoScan™ FT 2.

- **Food Scan™:** Dairy Analyzer: Infrared based equipment for rapid analysis of moisture, fat, protein etc. in solid and semi-solid dairy products like cheese, butter etc.
- **InfraXact™:** Near Infra Red (NIR) based equipment for compositional analysis of food and feed. It can be placed in the laboratory or in a production environment.
- **Process Analytics™ Dairy:** NIR based equipment designed for real-time, in-line control of quality of butter, powder, processed cheese, cream cheese and other dairy products.
- **ProcessScan™ FT:** FTIR based equipment suitable for real-time, on-line standardization of milk and other liquid dairy products.
- **LactoScope™:** Infrared based spectrophotometer capable of analysing the composition (fat, protein, lactose and total solids) of milk and milk products. Various models include C3, C4 and FTIR (both manual and automatic)

C. Ultrasound Spectroscopy based[11]:

- **Milk-Lab *COMPACT*:** Ultrasound spectroscopy based small and automatic equipment suitable for analyzing various parameters (fat, SNF, protein, total solids and temperature) in raw milk. Designed for use in dairy farms, milk collection centres, milk collection trucks, small dairies etc.
- **Milk-Lab *PRO*:** Ultrasound spectroscopy based equipment suitable for analyzing various parameters (fat, SNF, protein,

lactose, total solids, temperature, freezing point, added water, conductivity and pH) in milk. Also available in different product types suitable for cream, ice-cream mix, milk shake, flavoured milk, reconstitute milk, evaporated milk and whey.

4. OVERVIEW OF ANALYTICAL TECHNIQUES IN DAIRY PRODUCTS ANALYSIS(12 and 1-6)

A. General analytical techniques for milk and milk products

Test methods	Suitable for
Fat (gravimetric)	
Rose-Gottlieb method	Liquid and dried milks (all types), cream, dried whey and ice cream
Schmid-Bondzynski-Ratzlaff method	Many types of cheeses, and caseins and caseinates
Weibull-berntrop method	Dairy products containing relatively high levels of thickeners, stabilizers, emulsifiers, vegetables, fruits etc.
Evers method	Butter, and a wide range of spreadable fats (margarine, low-fat spreads, blends etc.)
Protein	
Kjeldahl method	Milk and milk products
Dumas method	Milk and milk products
Amido black method	Milk
Lactose	
Phenol-sulphuric acid method	Caseins and caseinates
Chloramine-T method	Milk
Water (or total solids)	
Oven drying gravimetric method	Most dairy products
Karl-Fisher titration	Dairy products having fairly low water content, such as milk powders and anhydrous fat (ghee etc.)

Test methods	Suitable for
Ash	
Muffle furnace method	Milk and milk products
Calcium and magnesium	
Oxalate method	Milk
EDTA method	Wide range of dairy products, including liquid and dried milks, buttermilk, buttermilk powders, cheese and protein powders
Phosphorous	
Dry ashing method	Cheese, liquid and dried milks, and wheys.
Wet digestion method	Above and many other dairy products
Chloride	
Mohr method	Butter
Volhard method	Wide range of dairy products including cheese, cheese powders, butter, buttermilk powders, milk powders and certain protein products

B. Advanced analytical techniques for milk and milk products

Analytical technique	Suitable for
Spectrometric techniques	
UV-VIS spectroscopy	Enzymatic analysis and enzyme-linked immunosorbent essay (ELISA) plates
Microwave spectroscopy	Water content of powders (Laboratory analysis as well as on-line monitoring)
Low-resolution nuclear magnetic resonance spectroscopy	Fat and water content of low-moisture products
Infrared spectroscopy	Various major constituents in milk and dairy products (Laboratory analysis as well as on-line monitoring)
Ultrasonic spectroscopy	Major constituents, structure and physical state of milk and milk products
Atomic spectrometric techniques	
Flame emission spectrometry	Elemental (sodium, potassium, calcium) analysis of milk and dairy products
Flame atomic absorption spectrometry	Analysis of liquid dairy products for elements such as Ca, Cu, Fe, Mg, Mn and Zn

Analytical technique	**Suitable for**
Electrothermal atomic absorption spectrometry	Elemental analysis of dairy products; low detection limits of nano-gram (ng) per ml / parts per billion (ppb) using microlitre-sized sample volumes
Inductively coupled plasma atomic emission spectrometry	Fast and simultaneous elemental analysis of milk and dairy products
Inductively coupled plasma Mass spectrometry	Multi-element analysis of milk and dairy products with detection limits at sub-ng per ml level and providing isotopic data. Also capable of detection of non-metals (phosphorous and iodine) that are of importance in milk and dairy products.
Chromatographic techniques	
High performance liquid chromatography	Vitamins, antibiotic residues, protein components in milk and various dairy products
Gas chromatography	Cholesterol, pesticides (organophosphorous and organochlorines compounds) residues, flavouring components in milk and various dairy products
Immunochemical techniques	
Enzyme-linked immunosorbent assay	• Antibiotic residues in milk • Toxins of *Clostridium botulinum, C. perfringens, Staphylococcal toxins* in milk • Mycotoxins in milk • Bacteria and moulds in milk and dairy products • Proteolysis in milk and cheese
Electrophoresis techniques	
Electrophoresis	Protein analysis (caseins, whey proteins) of cheeses
Biosensor techniques	
Biosensor analysis	• Antibiotic analysis of milk • Pathogen analysis of pasteurized milk • Mastitis detection • Microbial toxins analysis in milk • Freshness of milk • Effectiveness of milk heat treatment (process control) • On-line measurement of milk composition

5. DEFINITIONS OF SOME COMMONLY USED ANALYTICAL TERMINOLOGY[13]

International Union of Pure and Applied Chemistry (IUPAC) definitions of some commonly used terminology in laboratory analysis are given under:

- **Reference method:** A method having small, estimated inaccuracies relative to the end use requirement. The accuracy of a reference method must be demonstrated through direct comparison with a definitive method or with a primary reference material.
- **Reference material:** A substance or mixture of substances, the composition of which is known within specified limits, and one or more of the properties of which is sufficiently well established to be used for the calibration of an apparatus, the assessment of a measuring method or for assigning values to materials.
- **Repeatability:** The closeness of agreement between independent results obtained with the same method on identical test material, under the same conditions (same operator, same apparatus, same laboratory and after short intervals of time). The measure of repeatability is the standard deviation qualified with the term: 'repeatability' as *repeatability standard deviation.*
- **Reproducibility:** The closeness of agreement between independent results obtained with the same method on identical test material but under different conditions (different operators, different apparatus, different laboratories and/or after different intervals of time). The measure of reproducibility is the standard deviation qualified with the term 'reproducibility' as reproducibility standard deviation.

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9. **FOSS Instruments (Website: www.foss.dk/Solutions/BusinessAreas/ MilkAndDairy.aspx)**

10. **Delta Instruments (Website: www.aitests.com/DeltaCD/diproduct.htm)**

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12. **Evers, J.M.. Aand Hughes, C.G. (2002), *Chemical Analysis,* Encyclopedia of Dairy Sciences (Volume 1), pp 31-40, Elsevier Science Limited**

13. **IUPAC Gold Book (Website: http://goldbook.iupac.org/I02979.html)**

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NEWS SECTION

Indian Food Laws

- **Notification S.O. 1246 (E) of 28 May 2008 of the Ministry of Health and Family Welfare:** The notification appoints 28 May 2008 as the date on which the provisions under Section 3 and Section 30 of the *Food Safety and Standards Act, 2006* shall come into force. The Section 3 provides definitions of terms relevant to the Act and the Section 30 describes functions of the Commissioner of Food Safety of the State.

- **Notification GSR 383 (E) of 16 May 2008 of the Ministry of Health and Family Welfare:** It is a corrigendum to the GSR 491 (E) of 21 August 2006 of the Ministry of Health and family Welfare that amended the PFA Rules by defining several new terms, revising Rules 32 (labeling requirements), 37A (proprietary foods), 37B (infant milk substitute / infant foods) and 42 (form of labels). The GSR 491(E) was scheduled to become applicable from 20 August 2007. Subsequently, the date of applicability of GSR 491 (E) was postponed to 20 February 2008 through the GSR 518 (E) of 31 July 2007 and then once again to 20 May 2008 through the GSR 70 (E) of 5 February 2008. The Corrigendum now extends the date of its applicability further, and accordingly the provisions of GSR 491 (E) would now be applicable from 20 October 2008.

- **Draft Notification GSR 380 (E) of 15 May 2008 of the Ministry of Health and Family Welfare:** The draft notification proposes to further amend the PFA rules, as amended by the GSR 491 dated 21 August 2006 (applicable from 20 October 2008 as indicated above). The revisions are proposed in Rules 32 (labelling requirements), 37 A (Manufacture of proprietary foods) and 42 (Form of labels – Declaration of allergenic ingredients).

The important revision proposed in Rule 32 is the requirement to specify the quantity of sugar when declaring amount of carbohydrate. Additionally, exemptions to some label declaration in certain situations are proposed:

Rule 37 A is proposed to be revised to remove the requirement to obtain a separate license for manufacture of proprietary products.

Rule 42 is proposed to be revised to remove the requirement to declare the presence of allergenic ingredients in foods.

Codex Alimentarius Commission (CAC)

40th Session of the Codex Committee on Food Additives was held during 21-25 April 2008 in Beijing, China (refer *Technews* issue 72, January – February 2008). It endorsed the following food additive provisions proposed by the 8th Session of the Codex Committee on Milk and Milk Products (February 2008, New Zealand):

- the maximum levels for annatto extracts and beta-carotene (vegetable) for use in butter, fat spreads and cheeses;
- the list of food additives for use in fermented milks, including food additives provisions for the drinks based on fermented milks; and
- the use of diacetyltartaric and fatty acid esters of glycerol at the level of 5000 mg/kg in creams and prepared creams.

These provisions will now be considered for adoption by the 31st Session of the Codex Alimentarius Commission, June-July 2008, Geneva.

International Dairy Federation (IDF)

IDF has published the following Bulletins recently:

- IDF Bulletin No.428/2008: Determination of Omega-3 and Omega-6 fatty acid content by gas-liquid chromatography in milk fat from Enriched products - Interlaboratory collaborative studies;

- IDF Bulletin No.429/2008: Physiological and Functional Properties of Probiotics (This Bulletin is available for free downloading at http://www.fil-idf.org/WebsiteDocuments/429-2008.pdf); and
- IDF Bulletin No. 430/2008: Standards, Hygiene and Food Safety of Dairy products (Risk Management; Practical Food Safety Management; Predictive Modelling; Emerging Issues).

For purchasing the IDF publications, the following may be contacted:

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ADVANCED INSTRUMENTAL TECHNIQUES FOR MILK AND MILK PRODUCTS ANALYSIS

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