



Technews

**National Dairy Development Board
For Efficient Dairy Plant Operation**

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GOOD LABORATORY PRACTICES FOR DAIRY PLANTS

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 **Good Laboratory Practices for Dairy Plants**. It may be understood that the information given here is by no means complete.

In this issue:

- ***Introduction***
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1. INTRODUCTION

Assuring quality and safety of products has now become an essential requirement for manufacturing units to meet domestic consumer expectations as well as to tap the international market.

Not only should the quality and safety of food products be ensured, one must be able to demonstrate that it has been ensured. Appropriate food quality and safety assurance systems have been developed to meet these objectives. All these systems rely significantly on the analyses carried out by the dairy laboratories. The dairy laboratories are involved in all the major aspects of assuring quality like control of raw material, process control, hygiene control and control of finished products. In this respect, the accuracy and reliability of the laboratory analyses is very important. As a result, application of Good Laboratory Practices and Analytical Quality Assurance in dairy laboratories has been constantly growing in importance. These practices improve the confidence that laboratory personnel have in the analytical results they obtain and help ensure the acceptability of the results to others.

The Good Laboratory Practices (GLPs) is a set of requirements to be met and practices to be followed with respect to different aspects like the laboratory personnel, basic resources, sampling and analysis that are considered as prerequisites for obtaining accurate and reliable results⁽¹⁾. Analytical Quality Assurance Programme is the sum total of a laboratory's activities aimed at ensuring that the information generated by the laboratory is correct⁽²⁾.

This issue of Technews provides information on the Good Laboratory Practices and Analytical Quality Assurance for dairy laboratories.

2. GOOD LABORATORY PRACTICES^(1, 2)

i) Laboratory personnel

- The laboratory personnel should have understanding of the principles of analysis.
- The laboratory manager must have an appropriate professional qualification and possess understanding of the requirements of analytical quality assurance. He should be experienced and competent in required analysis, interpretation of results and decision-making.
- The laboratory staff must be fully trained and experienced in the correct use of apparatus and in appropriate laboratory skills. They must understand the importance of following the methods exactly as described and noting any unavoidable deviations. They must also be trained in interpretation of analytical results and taking appropriate decisions based on these.
- The new laboratory personnel should be trained in relevant analyses. Each analyst using an analytical method for the first time should train himself by repeatedly analyzing the samples already analyzed by other experienced analysts and obtaining comparable results.
- Records of training and experience must be maintained.

ii) Basic resources

a) Laboratory building

Location and layout

- The dairy laboratory should be located to permit easy access to all the major activities of the dairy.
- It should be so located as to avoid vibrations of all kinds and should preferably be away from extreme environments like boiler houses.
- The microbiological laboratories should be located away from the production areas to avoid any potential microbiological contamination of products from the laboratory. Its location should also permit limited access.
- The layout must permit a free and logical movement as per different activities to be undertaken in the laboratory.
- The washing and sterilization facilities should be separated, but not far from, the main working area.

Design and construction

- Laboratories should be constructed of, and utilize, materials resistant to chemicals likely to be used within them.
- The main working area should be designed and equipped for storage and handling of an appropriate range of chemicals and other related material.
- There should be sufficient number of appropriately located electrical points.
- There should be appropriate number of water points. For most of the dairy laboratories, 1 water point for distillation apparatus, 1-2 for washing facilities and around 2 for main working area would suffice.
- There should be exhaust / fume hoods to allow exit of gases / vapours / toxic fumes.

- Where necessary, it should have air-conditioning facilities for specific areas. E.g. areas designated for microbiological testing or for housing sophisticated instruments.
- Microbiological laboratory should have adequate provisions to prevent contamination. E.g. air curtain at main door.

b) Equipment

- All the equipment must be fit for purpose.
- They should be maintained in working conditions and serviced regularly.
- All the measuring equipment, including automated pipettes / dispensers, should be calibrated as per manufacturer's instructions or standard procedures using certified standards where necessary. Regular calibration and re-calibration of measuring equipment must be done where the possible change in nominal value may significantly compromise accuracy of measurements.
- Records of servicing and calibration must be maintained for each equipment.
- Operating temperatures of refrigerators and freezers, and of ovens and incubators, should be continually monitored or be checked at specified intervals and recorded.

The IDF standard IDF 128A:1999 titled 'Definition and Evaluation of Overall Accuracy of Indirect Methods of Milk Analysis Application to Calibration Procedure and Quality Control in the Dairy Laboratory', in its Part 2, provides practical details and recommendations for calibration of measuring equipment.

c) Chemicals and supplies

- The laboratory should have adequate supplies of chemicals, reagents, glassware, etc of suitable quality.
- It should have adequate and reliable supply of water and electricity.
- All stock solutions and reagents should be properly labelled including preparation date, analyst's identification and storage conditions. Those compounds whose integrity could be influenced by degradative processes must be clearly labelled with an expiry date and stored under appropriate conditions, such as under refrigeration, away from direct sunlight and heat etc.
- All the reagents prepared must be standardized if utilized for quantitative analyses. Ready-made reagents purchased from chemists should also be standardized unless the manufacturer provides a certificate of its accurate strength. Reagents prepared in bulk and stored should be standardized before use, that is, the aliquot drawn for use must be standardized. Long stored reagents must always be standardized before use to ascertain that they have not been degraded by the effect of light or heat or become concentrated due to evaporation of solvents. Records of standardization of reagents should be maintained.
- All laboratory glassware used for measurement purpose such as pipette, burette, measuring cylinders, thermometer, lactometer, butyrometers etc., should be calibrated using standard procedures or against glassware certified by the National Physical Laboratory or equivalent reputed institute. Glassware supplied with certificate of accuracy may not need calibration. Records of calibration of glassware should be maintained.
- It is advisable to calibrate and store a few pieces of each

type of measuring glassware to meet eventualities during analysis.

Indian Standard IS: 2981-1964 of the Bureau of Indian Standards provides detailed guidance on laboratories for different size of dairies.

iii) Sampling

a) Standard sampling procedures

Standard sampling procedures should be established based on national legislation and reputed sources like BIS, CODEX, AOAC, ISO, IDF etc., documented and followed.

b) Obtaining samples

- Goal of sampling is to obtain a representative sample from a batch or lot. Correct sampling requires most careful attention. Sampling is at least as important as, if not more than, the analysis.
- Samples should be drawn as per the sampling protocol.
- The standard sampling procedures should be followed to the extent possible. However, judgement, skill and experience should be used where necessary.
- Trained and experienced laboratory personnel having necessary skills for sampling and understanding of the sampling requirements should be authorized to draw samples. A person experienced in the sampling technique for microbiological purposes shall be authorized to undertake sampling for microbiological examination.

- Samples meant for microbiological analysis should always be drawn first. The person drawing sample should be free from any infectious disease.
- The container and the sampling equipment used for sampling should be clean and dry (also sterilized in case of sampling microbiological analyses).
- Any information that may be useful in correct interpretation of results of analysis should be recorded at the time of obtaining samples.
- The quantity of the sample drawn must be adequate to perform the intended analysis (refer Table in subsection d).

c) Identification/coding

The sample should be appropriately identified by suitable markings / coding. This can include, but is not limited to, the date, name of the product, temperature at sampling, purpose, source, name and initials of the person undertaking sampling, etc. as per the specific requirements.

d) Storage of samples

- Samples need to be stored under appropriate storage conditions to ensure that the original sample characteristics at the time of sampling are not adversely affected till it is analyzed.
- The storage conditions generally relate to the temperature and light conditions. Dried products like milk powders also need protection against exposure to moisture. Other specific storage requirement, if any, must be adhered to.

- It may be necessary to use preservatives to some samples if prolonged storage is necessary. Only legally permitted preservatives should be used to preserve the samples. Samples for microbiological analysis or those intended to be subjected to microbiology-based analysis should never be added with preservatives.
- The Table below provides important guidance on appropriate temperature of storage for, and use of preservatives in, samples of milk and milk products based on the International Dairy Federation (IDF) standard on sampling⁽³⁾.

Table: Sample preservation, storage and minimum sample size

Sl. No.	Product	Preservatives permitted for samples for chemical and physical analysis	Temperature ¹ of storage (°C)	Minimum sample size ²
1	Non-sterilized milk and liquid milk products	Yes	0-4	100 ml or g
2	Sterilized milk, UHT milk and sterilized liquid milk products in unopened containers	No	Ambient, max. 30	100 ml or g
3	Sterilized milk, UHT milk and sterilized liquid milk products after sampling from the production line or from one or more original pack (s)	Yes	0-4	100 ml or g

Sl. No.	Product	Preservatives permitted for samples for chemical and physical analysis	Temperature ¹ of storage (°C)	Minimum sample size ²
4	Evaporated milk, sweetened condensed milk and milk concentrates	No	Ambient, max. 30	100 g
5	Semi-solid and solid milk products except butter and cheese	No	0-4	100 g
6	Edible ices and semi-finished ice products	No	-18 or lower	100 g
7	Dried milk and dried milk products	No	Ambient, max. 30	100 g
8	Butter and butter products	No	0-4 (in the dark)	50 g
9	Butterfat (butteroil) and similar products	No	0-4 (in the dark)	50 g
10	Fresh cheese	No	0-8	100 g
11	Processed cheese	No	Ambient, max. 30	100 g
12	Other cheeses	No	4-8	100 g

¹ The temperatures mentioned in the Table are meant as general guidelines. For specific analysis purposes other temperatures can be more appropriate. It may be, under certain practical conditions, not always easy or even impossible to maintain the temperatures specified in this Table or required for specific analysis purposes. It is, therefore, recommended to use suitable container in all cases where it is necessary and to monitor and record temperature in the suitable way.

² A larger sample size may be necessary according to the tests required and type of product.

- Product samples for future reference (e.g. for comparing with samples obtained from market or for use as reference during handling of market complains) should be invariably stored as finished product packages and under the storage conditions declared on the product label. These samples need to be discarded once their declared shelf life is over.

The following national and international standards can form useful guidance documents for establishing sampling procedures and related requirements:

- *Milk and Milk Products – Guidance on Sampling (IS: 11546-1999)*
- *Milk and Milk Products – Guidance on Sampling (IDF: 50C-1995)*
- *Codex General Guidelines on Sampling (CAC/GL 50-2004)*

iv) Analysis

a) Standard analytical procedures

- Appropriate analytical procedures should be identified from national legislation and reputed sources like BIS, Codex, AOAC, ISO, IDF etc. and documented in detail.
- The identified methods should be clearly distinguished as the routine and reference methods. Only those routine methods that can be checked against a reference method should be used in the laboratory.
- Only the identified methods should be used for analysis without any deviations as far as possible.
- All the analysis should be carried out in duplicate.

- When analysis involves use of measuring equipment/instrument, the equipment/instrument manufacturer's instructions must be strictly adhered to.
- Should any deviation from standard analytical procedure or equipment/instrument manufacturer's instructions take place, it should be recorded in detail.

b) Standardization of methods

- The reference methods should be standardized by analyzing samples containing known concentration of the analyte. This helps in knowing whether all the steps of the method have been understood and can be followed appropriately in the laboratory and that whether the set of chemicals used by the laboratory is yielding the results expected from the method.
- Most of the laboratories utilize routine methods for day-to-day analysis for practical reasons. These routine methods used should have been standardized against appropriate reference method. For example, the Gerber test for milk is a routine method and should be standardized against a reference method like Rose-Gottlieb method.
- When the analytical procedures identified for a particular product are adopted for other similar products, these should be standardized for use on the new product matrix appropriately before use.
- Records of such standardization must be maintained.

c) Recording and interpretation of results

- The results should be recorded in suitable formats developed for each test. The format should allow recording of all necessary details from the log-

books/rough record books used during analysis.

- All records must be initialed.
- No pages, including those containing incorrect or erroneous results, should be removed from the records. Incorrect or erroneous data may be crossed out and initialed.
- The results of any analysis should be interpreted as per the guidance provided in the method of analysis and must take into consideration any other relevant information recorded during sampling and/or analysis.

d) Communicating results

The results of analysis should be communicated to the concerned officials in other sections of the dairy without delay to enable them take timely decisions.

The article titled Critical Control Points in Dairy Chemical Analysis (International Dairy Federation Special Issue No. 9302. International Dairy Federation, Brussels, pp 278-288) provides important information on specific points where care should be taken during chemical analysis in general and also for some specific analyses like preparation of cheese samples for chemical analyses and drying oven methods.

The following ISO/IDF standards are useful with respect to microbiological analyses of milk and milk products in dairy laboratories:

- *Milk and Milk Products: Quality Control in Microbiological Laboratories. Part 1: Analyst Performance Assessment for Colony Counts (ISO 14461-1 / IDF 169 –1).*

- *Milk and Milk Products: Quality Control in Microbiological Laboratories. Part 2: Determination of the Reliability of Colony Counts of Parallel Plates and Subsequent Dilution Steps (ISO 14461-2 / IDF 169 –2).*

It may be noted that the dairy laboratories engaged in specific analyses may implement specific GLPs for their requirement such as Codex Guidelines on Good Laboratory Practices in Residue Analysis (CAC/GL 40 – 1993, Rev.1-2003).

3. ANALYTICAL QUALITY ASSURANCE⁽⁴⁾

i) Criteria for choice of methods

- Through the establishment of choice of methods, the operating laboratory must define the precision with which it wishes to analyze.
- The choice of method should also include what the result is to be used for. If this is to be used for current process control of a production, it may be appropriate to choose routine methods with less precision than those required by the official authorities. But if the result is to be used for documentation for forwarding to the authorities, it may be appropriate to use the official reference methods as the basis for examination. In other words, what the result is to be used for is of vital importance to the choice of method.

ii) Description of applied analytical methods

The Quality Assurance Programme should include a

description of the analytical methods used and its application. This makes the methods easier for the operating laboratory staff to understand and also enables them to easily explain the background for the choice of analytical methods to other employees at the dairy, whenever required.

iii) Choice of reference methods

The Quality Assurance Programme should include a description of the officially approved reference methods to be used for verifying the accuracy of routine methods used in the laboratory. The following points must be considered while choosing a reference method:

- Routine methods used by the laboratory that require standardization.
- Facilities available with laboratory to carry out analysis as per the reference method.

iv) Scheme for control procedure and control frequency of the analysis

- The control procedures and frequencies of analysis should be established. This should take into account the sampling requirements of the HACCP system followed in the dairy.
- A sampling protocol should be formulated which shall indicate:
 - * Which samples are to be taken
 - * Stage in processing at which the samples are to be taken
 - * Frequency of sampling
 - * Parameters to be tested

- * Precision limits required

- Control procedures and frequencies should be evaluated to judge their efficacy in achieving desired levels of control. This should include consideration of the risk of obtaining false positive or false negative results.

v) Scheme for administrative routines

Administrative routines should be established for the laboratory. These should include:

- Sampling responsibility
- Analysis responsibility
- Details on handling of results, including statistical processing
- Protocols for reporting the result

vi) Scheme for instruction and training of laboratory staff

- Policies for deciding the degree of instruction and training of laboratory staff should be established. Well-educated and trained laboratory staff is an important requirement to ensure that results obtained are correct and can be used in running the organization.

vii) Determination of organizational position of the laboratory

- It is important that the laboratory and its leader have the authority and competence to establish that the results and samples have been obtained in accordance with the established protocol.
- The laboratory leader should refer directly to the person responsible for managing the quality assurance

functions and not to the production manager. This is useful in achieving the independence and impartiality necessary for the laboratory to carry out its work.

viii) Determination of responsibilities and competence

The responsibilities and competence of the laboratory should be determined based on its organizational position, and documented. This is necessary to ensure effective participation of the laboratory in the general quality assurance programme of the dairy.

REFERENCES

1. IDF (2001). Quality control in the laboratory, **Bulletin of the International Dairy Federation, Special Issue No.365**. International Dairy Federation, Brussels, pp 31-33.
2. Codex Alimentarius Commission (2003). **Codex Guidelines on Good Laboratory Practices in Residue Analysis, CAC/GL 40-1993, Rev.1-2003**. Codex Alimentarius Commission, Geneva/Rome.
3. IDF (1995). Milk and Milk Products – Guidance on Sampling, **International Dairy Federation Standard 50C-1995**. International Dairy Federation, Brussels.
4. IDF (1993). Requirements of analytical quality assurance, **International Dairy Federation, Special Issue No.9302**. International Dairy Federation, Brussels, pp 80-84.

Information on critical points in important analyses commonly performed in dairy laboratories will be included in a future issue of Technews.

NEWS SECTION

Indian Food Laws

- **Draft Notification GSR 278 (E) of 9 May 2006 of the Ministry of Health & Family Welfare:** The Draft Notification proposes some amendments to cream standard under PFA Rules. The amendments pertain to revised definitions, minimum fat content requirements, additives permitted and microbiological standards for different types of creams which are largely based on the Codex Standard for Creams and Prepared Creams (CODEX STAN A/9-1976, Rev.1-2003) except the fat content requirements and microbiological specifications.
- **Draft Notification GSR 298 (E) of 18 May 2006 of the Ministry of Health & Family Welfare:** The Draft Notification proposes some amendment to PFA Rules and includes proposal of a maximum level for aflatoxin M₁ in milk.
- **Draft Notification GSR 299 (E) of 18 May 2006 of the Ministry of Health & Family Welfare:** The Draft Notification proposes use of standard methods for analyses to judge compliance to PFA Rules. It refers to the Manual of methods of analysis developed by the Ministry of Health and Family welfare and also provides a list of renowned books/manuals that may be referred for alternative methods of analysis. The manual of methods of analysis for milk and milk products is available in electronic form at the website: <http://www.mohfw.nic.in/manmethod.htm>).

- **Draft Notification GSR 325 (E) of 29 May 2006 of the Ministry of Health & Family Welfare:** The Draft Notification includes a proposal, among others, of an amendment to PFA Rules, reducing the permitted level of the coal tar dyes erythrosine / sunset yellow FCF to 50 ppm from currently permitted level of 100 ppm in sweets. It also proposes to allow use of mono - and diglycerides in infant formula up to a maximum level of 0.4 mg/100 ml of ready to serve formula.
- **Draft Notification GSR 355 (E) of 6 June 2006 of the Ministry of Health & Family Welfare:** The Draft Notification proposes application of a 'Food Recall Procedure' under the PFA Rules. The amendment proposes the requirements of, and the conditions for application of a 'Food Recall Procedure'. It proposes that a food business shall voluntarily recall any article of food or any ingredient or any substance that is adulterated, misbranded or injurious to health failing which the Government shall order to recall such product from the market, which shall be complied with.

Codex Alimentarius Commission (CAC)

- **Codex Committee on Pesticide Residues (CCPR):** The 38th Session of the CCPR was held during 3-8 April 2006 in Fortaleza, Brazil. The Committee forwarded maximum residue limits for 13 pesticides (Paraquat, Carbendazim, Oxydemeton methyl, Chlorpropham, Pyraclostrobin, Fludioxonil, Trifloxystrobin, Phorate, Methoprene, Glyphosate, Terbufos, Dimethenamid-P and Novaluron) in milk and 1 pesticide (Novaluron) for milk fat to the Commission for final adoption.

- **Codex Committee on Food Additives and Contamination (CCFAC):** The 38th Session of CCFAC was held during 24-28 April 2006 in The Hague, The Netherlands. The Committee forwarded several food additive provisions for milk powder, cream powder (plain), cream, whey cheese, whey protein cheese, dried whey and whey products, butter and concentrated butter, and butteroil (anhydrous milk fat, ghee) to the Commission for final adoption. The Committee also forwarded the 'Code of Practice for the Prevention and Reduction of Dioxin and Dioxin-like PCB Contamination in Foods and Feeds' for final adoption. The maximum levels for tin in canned foods (other than beverages) and in canned beverages were forwarded by the Committee to the Commission for preliminary adoption.
- **Codex Committee on Food Labelling (CCFL):** The 34th Session of CCFL was held during 1-5 May 2006 in Ottawa, Canada. The Committee forwarded a definition of Trans Fatty Acids for final adoption by the Commission.
- **Codex Committee on Residue of Veterinary Drugs in Foods (CCRVDF):** The 16th Session of the CCRVDF was held during 8-12 May 2006 in Cancun, Mexico. The Committee forwarded maximum residue limits (MRLs) for 4 veterinary drugs (Trichlorfon (Metrifonate), Pirlimycin, Cypermethrin/Alpha-Cypermethrin and Doramectin) and the 'Compendium of Methods of Analysis Identified as Suitable for Support to Codex MRLs' to the Commission for final adoption. The Committee also forwarded the 'Guidelines for the Design and Implementation of National Regulatory Food Safety Assurance Programmes Associated with the Use of

Veterinary Drugs in Food Producing Animals’ for preliminary adoption.

- **Codex Committee on Methods of Analysis and Sampling (CCMAS):** The 27th Session of the CCMAS was held during 15-19 May 2006 in Budapest, Hungary. The Committee forwarded methods of analysis and sampling for different parameters in several milk products for final adoption and the ‘Guidelines for Settling Disputes Over Analytical (Test) Results’ for preliminary adoption, by the Commission.

The reports of the above Committees’ Sessions can be downloaded from the Codex website: <http://www.codexalimentarius.net/web/archives.jsp?lang=en>.

International Dairy Federation (IDF)

The IDF has published the following Bulletins / Standards recently:

- IDF Bulletin 406/2006: *"New Applications of Mid-infra-red Spectrometry for the Analysis of Milk and Milk Products"*.
- IDF Bulletin 407/2006: *"Determination of Water Content of Butter Oil By Karl Fischer Titration"*.
- IDF Special Issue: *"International Dairy Journal Vol.16/6 (June 2006) 499-716 – Special Issue: First IDF Symposium on Indigenous Enzymes in milk"*.
- IDF Special Issue: *"Proceedings of the IDF Symposium on Cheese: Ripening, Characterization & Technology – Prague, 2004"*.
- IDF-ISO Standard: *"Cheese and Processed Cheese Products – Determination of Citric Acid Content – Enzymatic Method (IDF 34 / ISO 2693)"*.

- IDF-ISO Standard: *"Milk and Milk-based Products – Detection of Thermonuclease Produced by Coagulase-positive Staphylococci (IDF 83 / ISO 8870)"*.
- IDF-ISO Standard: *"Milk and Milk Products – Detection of Alkaline Phosphatase Activity – Part 1: Fluorimetric Method for Milk and Milk-based Products (IDF 155-1 / ISO 11816-1)"*.
- IDF-ISO Standard: *"Milk Products – Enumeration of Presumptive Lactobacillus Acidophilus on a Selective Medium – Colony Count Technique at 37 °C (IDF 192 / ISO 20128)"*.

For purchasing the IDF publications, Mr. Oscar Chavez, Office Manager, International Dairy Federation, Brussels, Belgium (Email: OChavez@fil-idf.org) may be contacted.



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