INTERNATIONAL STANDARD

ISO 23291

IDF 248

First edition
2020-01

Milk and milk products — Guidelines for the application of in-line and on-line infrared spectrometry

Lait et produits laitiers — Lignes directrices pour l'application de la spectrométrie infrarouge en ligne

Reference numbers
ISO 23291:2020(E)
IDF 248:2020(E)

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

IDF (the International Dairy Federation) is a non-profit private sector organization representing the interests of various stakeholders in dairying at the global level. IDF members are organized in National Committees, which are national associations composed of representatives of dairy-related national interest groups including dairy farmers, dairy processing industry, dairy suppliers, academics and governments/food control authorities.

ISO and IDF collaborate closely on all matters of standardization relating to methods of analysis and sampling for milk and milk products. Since 2001, ISO and IDF jointly publish their International Standards using the logos and reference numbers of both organizations.

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The work was carried out by the IDF/ISO Action Team (S12) of the *Standing Committee on Statistics and Automation* under the aegis of its project leaders, Dr S. Holroyd (NZ) and Dr A. Larsen (DK).

Milk and milk products — Guidelines for the application of in-line and on-line infrared spectrometry

1 Scope

This document gives guidelines for using infrared spectrometry in in-line and on-line applications for dairy processing. These applications are distinct to those covered in ISO 21543 | IDF 201.

It is applicable, but not limited, to:

— the determination of protein, fat and total solids in liquid milk and milk products using mid and near infrared spectrometry;

— the determination of protein, fat and moisture in solid or semi-solid products, such as milk powder, and butter and liquid dairy streams using near infrared spectrometry.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
in-line analysis
analysis of a product line where the sensor probe interfaces directly with the product stream being measured, or a reflectance measurement through an optical window into the product stream

3.2
on-line analysis
analysis of a product line where the sensor probe interfaces indirectly with the product stream being measured by way of a bleed loop, automated grab sampler or other means of subsampling

3.3
at-line analysis
analysis of a product where the instrument is physically remote to the product stream being measured and the sample is manually introduced to the instrument

Note 1 to entry: While not covered in this document, this definition is added here in order to distinguish this type of spectometric analysis from in- and on-line apparatus.

3.4
near infrared instrument
NIR instrument
proprietary apparatus utilizing wavelengths within the range 400 nm to 2 500 nm or 25 000 cm^{-1} to 4 000 cm^{-1} (both visible and NIR range) or 12 820 cm^{-1} to 4 000 cm^{-1} (NIR range only) which, when used under certain conditions, estimates mass fractions or other parameters of use

3.5
mid infrared instrument
MIR instrument
proprietary apparatus utilizing wavelengths in the range 4 000 cm^{-1} to 400 cm^{-1}, which, when used under the conditions specified in this document, estimates the mass fractions or other parameters of use specified in Clause 1

4 Principle

A laboratory in-line or on-line instrument is installed according to the manufacturer's guidelines for the type of process under measurement. Absorbance within the wavelength regions mentioned above is measured by transmission, reflectance and a combination of both, or by attenuated total reflectance (ATR). The resulting spectral information is transformed to constituent concentrations or constituent values with other units by calibration models developed by representative samples from the population to be tested.

5 Apparatus

5.1 Infrared instrument, based on diffuse reflectance or transmittance measurement in the near (400 nm to 2 500 nm or 25 000 cm^{-1} to 4 000 cm^{-1} or 12 820 cm^{-1} to 4 000 cm^{-1}) or mid (4 000 cm^{-1} to 400 cm^{-1}) wavelength region or segments of this or at discrete wavelengths. The optical operation principle may be dispersive (e.g. grating monochromators), interferometric or diode-array based. The instrument should be provided with an appropriate diagnostic test system for testing photometric instrument noise, wavelength accuracy and wavelength precision (for scanning spectrophotometers). The instrument shall be able to optically view into the product stream with an appropriate interface. There are many commercial such devices with a wide variety of technology depending on specific applications.

6 Installation and sampling considerations

6.1 General

Any installation shall cover both the integrity of the infrared instrument as well as interface with the process stream. Key aspects for the integrity of the instrument include:

— protection from cleaning regimes;

— isolation from vibration, dust and other environmental contaminants;

— an appropriate temperature regime for the specific instrument;

— many regions have strict protocols for materials in contact with human food products, such as Regulation (EC) No 1935/2004[3] or US equivalent 3A, and this is an important consideration for probes or cell construction.

A reliable, stable and consistent sampling interface is the key to successful use of in-line and on-line spectrometry. The following aspects are important.

— The ability to sample a representative flow of product. This can be ascertained by experimentation as well as an understanding of the fluid dynamics of the process flow.

— The ability to be consistently cleaned to the same level as the rest of the installation for good grade hygiene. For liquid product streams, this may mean that the probe is cleaned by the regular cleaning in place protocols. For powder, an air jet or similar can be necessary to remove sample prior to each measurement. Experimentation is often required to determine the most effective cleaning protocol for a specific environment.

— Stability over time. The interface shall not be altered by changes in plant or process as these can impact spectral quality and thus predictive performance of the instrument.

— Pipe, flow direction, sampling valve position and other technical considerations.

A critical component of any in-line or on-line system is how it samples from the process stream. The following key aspects shall be considered:

— the relationship between sample and spectra;

— the probe: optical interface, location and type;

— sampling and sample handling and timing, especially afterwards until the sample is analysed by the reference method;

— combinations and composition ranges of major and minor sample components: analytes (total solids, fat and protein) and non-analytes (component that can interfere with the results);

— seasonal, geographic and genetic effects on milk composition;

— different processing conditions, the design of production line and the speed of the process flow;

— the temperature, pressure and homogeneity;

— the turbulent/laminar flow, air entrapment or foaming of the process stream.

The infrared measurements and reference analyses shall be performed on the same test sample in order to minimize effects related to sampling uncertainty. It is suggested that sampling should take place in a steady-state situation, meaning there is very little fluctuation in sample composition in the piping at the moment of sampling. With in-line analysis, this means that a spectrum shall be taken preferably with a manual signalling option actually on the instrument housing that will flag the moment a manual sample is taken. This serves to accurately record the time of the sample collection and match it with the corresponding spectra.

An understanding of the process time constants is also important to assess to ensure alignment between the reference sample and spectroscopic measurement. The infrared measurements and the initiation of reference analyses should also be performed with a minimum time lag (preferably less than one day) and should reflect the stability of the matrix. The handling and treatment of samples from the time they are taken until their analysis by the reference method is also important: it shall be consistent and maintain the integrity of the sample. For example, milk powder may be cooled for a specific period prior to reference analysis. It is important this is similar for all samples used to build and validate the reference set and in routine use.

Sampling procedures should be respected uniformly during the calibration, validation and monitoring steps and not changed over time. The ability to obtain a representative sample from the process and link it closely to an infrared spectrum is important. Effective sampling will vary depending on the nature of the process stream. In addition, as it is not normally a single sample that gets laboratory tested for fat, protein and solids but sub-samples, the ability to sub-sample effectively should also be considered.

The sampling point should be defined properly. Samples should be collected in such a way that they represent the same or similar portion of sample that was measured by the infrared instrument. Considerations such as homogeneity of the process stream are important and it can be challenging to ensure a consistent representative sample is obtained in all process conditions. It is important to accurately record the time of the sample collection and match it with the corresponding spectra.

6.2 In-line analysis systems

An in-line system shall be set up so that a truly representative flow of material passes across the optical interface, and that there is no build-up of either material or contamination on the optical surface of the