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Milk fat products — Determination of water content — Karl Fischer method

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 5536|IDF 23 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.

This second edition of ISO 5536|IDF 23 cancels and replaces the first edition (ISO 5536|IDF 23:2002), which has been technically revised.

Foreword

IDF (the International Dairy Federation) is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

The main task of Standing Committees is to prepare International Standards. Draft International Standards adopted by the Standing Committees are circulated to the National Committees for endorsement prior to publication as an International Standard. Publication as an International Standard requires approval by at least 50 % of IDF National Committees casting a vote.

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All work was carried out by the Joint ISO-IDF Action Team on *Water* of the Standing Committee on *Main components in milk* under the aegis of its project leader, Prof. Dr. H.-D. Isengard (DE).

This edition of ISO 5536|IDF 23 cancels and replaces ISO 5536|IDF 23:2002, which has been technically revised.

Milk fat products — Determination of water content — Karl Fischer method

1 Scope

This International Standard specifies a method for the determination of the water content of milk fat products by the Karl Fischer (KF) method.

The method is applicable to butteroil (anhydrous butteroil, anhydrous butterfat, anhydrous milk fat) with a water content not exceeding 1,0 % mass fraction.

2 Terms and definitions

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

2.1
water content of milk fat products
w_{H_2O}
mass fraction of water determined by the procedure specified in this International Standard

NOTE The water content is expressed as a percentage mass fraction.

3 Principle

The test sample is directly titrated with a commercially available two-component Karl Fischer reagent at a temperature of approximately 40 °C. The water content is calculated from the amount of reagent used.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

Avoid absorption of moisture from the environment.

4.1 Karl Fischer (KF) reagent, a commercially available pyridine-free two-component reagent consisting of a titration component (4.2) and a solvent component.

Alternatively, a pyridine-free one-component KF reagent consisting solely of a titration component (4.2) can also be used, in which case, methanol (4.6) is used as the solvent component.

4.2 Titration component.

The titration component of the KF reagent (4.1) shall have a water equivalent of approximately 2 mg/ml.

4.3 1-Octanol ($C_8H_{17}OH$)

4.4 Working medium.

Prepare the working medium by mixing equal volumes of the solvent component of the KF reagent (4.1) and 1-octanol (4.3) (1+1) directly in the titration vessel of the KF apparatus (5.1).

4.5 Water standard, w_{H_2O} = 10 mg/g.

For the determination of the water equivalent of the titration component, use a commercially available liquid water standard.

4.6 Methanol (CH_3OH), with a water content of not more than 0,05 % mass fraction.

5 Apparatus

Usual laboratory apparatus and in particular the following.

Ensure that all equipment is completely dry before use.

5.1 Karl Fischer (KF) apparatus. Use an automatic volumetric KF apparatus, with a burette of capacity 5 ml (preferably) or 10 ml and a double-jacketed titration vessel with a water inlet and outlet to carry out the titration at approximately 40 °C.

5.2 Water bath, capable of maintaining a temperature of 40 °C ± 5 °C; equipped with a thermostat and a pump for heating the double-jacketed titration vessel of the KF apparatus (5.1).

5.3 Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.

5.4 Disposable syringes, of capacity 5 ml or 10 ml, for dosing the water standard (4.5) and the test portion (7.5).

6 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707|IDF 50[1].

7 Procedure

7.1 KF apparatus drift

7.1.1 Determine the drift of the KF apparatus (5.1) every day and/or when the working conditions change as follows.

Mix approximately 30 ml of working medium (4.4) in the titration vessel of the KF apparatus (5.1). Heat the vessel and its contents to 40 °C using the water bath (5.2). Pre-titrate the working medium. Then carry out a titration over 5 min (without addition of a test sample).

IMPORTANT — During each titration the amount of working medium used shall completely cover the electrodes of the KF apparatus.

7.1.2 Calculate the KF apparatus drift, q_V, expressed in millilitres per minute, by using Equation (1):

$$q_V = \frac{V_\mathrm{d}}{t} \tag{1}$$

where

V_d is the volume, in millilitres, of the amount of titration component (4.2) used in the titration (7.1.1);

t is the time, in minutes.

Or calculate the KF apparatus drift, q_m, expressed in milligrams per minute, by using Equation (2):

$$q_m = \frac{V_\mathrm{d}\,\rho_\mathrm{e}}{t} \tag{2}$$

where ρ_e is the water equivalent (7.2) of the titration component (4.2), in milligrams per millilitre, used in the titration (7.1.1).

7.2 Water equivalent of the titration component of the KF reagent

7.2.1 Titration

To calculate for each determination the water equivalent of a titration component accurately, carry out the titration in this subclause and the calculation step (7.2.2) five times.

Mix approximately 30 ml of working medium (4.4) in the titration vessel of the KF apparatus (5.1) (see also 7.1.1, “important” notice). Heat the vessel and its contents using the water bath (5.2) to 40 °C. Pre-titrate the working medium.

Take up between 0,6 g and 0,9 g of water standard (4.5) (corresponding to about 6 mg to 9 mg of water) in a disposable syringe (5.4). Weigh the syringe and its contents, and record its mass to the nearest 0,1 mg.

It is also possible to take up more than 0,6 g to 0,9 g of water standard and to use the same filling of the syringe for several titrations (one immediately after the other). In that case, approximately 0,6 g to 0,9 g of water standard should also be used for each titration, the exact amount being calculated each time by weighing the syringe and its remaining contents after each injection.

Add the water standard to the titration vessel. Weigh the empty syringe again and record its mass to the nearest 0,1 mg. Subtract the mass of the empty syringe from that of the syringe with water standard before addition of the water standard to the vessel to obtain the net mass of the water standard added to the titration vessel, $m_{w,\,i}$ (for i = 1 … 5). Record the net mass of the added water standard to the nearest 0,1 mg.

Carry out a titration while using as shut-off criterion the 10 s stop delay time mentioned in 7.3. Record, for i = 1 … 5, the volume, $V_{\rho_{\mathrm{e},\,i}}$, in millilitres, of titration component.

Do not perform more than three titrations on one portion of working medium.

After three titrations on one portion, carry out further titrations by again mixing 30 ml working medium (4.4) in the titration vessel of the apparatus.

7.2.2 Calculation

Calculate the water equivalent of the titration component, $\rho_{e,\,i}$, expressed in milligrams of water per millilitre, as the mean value of the five (for i = 1 … 5) single titrations (7.2.1), by using Equation (3).

$$\rho_{e,i} = \frac{m_{w,i} \quad w_{H_2O,s}}{V_{\rho_{e,i}}} \tag{3}$$

where

$m_{w,i}$ is the mass, in grams, of water standard used for titration i (7.2.1);

$w_{H_2O,s}$ is the water mass fraction, in milligrams per gram, of the water standard;

$V_{\rho_{e,i}}$ is the volume, in millilitres, of titration component (4.2) in titration i (7.2.1).

7.2.3 Mean value

Calculate the water equivalent of the titration component, ρ_e, expressed in milligrams of water per millilitre, as the mean value of the five (i = 1 … 5) single titrations (7.2.1) by using Equation (4):

$$\rho_e = \frac{\sum_{i=1}^{5} \rho_{e,i}}{5} \tag{4}$$

7.2.4 Expression of water equivalent

Express the (mean) value of water equivalent to three decimal places.

7.3 Shut-off criterion

According to the features of the apparatus used, the stop criterion shall either be a stop delay time of 10 s or a stop drift slightly above the measured drift (7.1).

7.4 Preparation of the test sample

Mix the test sample by blending thoroughly at 35 °C to 40 °C to completely disperse the water.

7.5 Determination

Mix approximately 30 ml of working medium (4.4) in the titration vessel of the KF apparatus (5.1) (see also 7.1.1, “important” notice). Heat the vessel and its contents using the water bath (5.2) at 40 °C. Pre-titrate the working medium.

Take up approximately 5 g of test sample (7.4) in a disposable syringe (5.4). Weigh the syringe with the test portion and record the total mass to the nearest 0,1 mg.

Add the test portion to the titration vessel. Weigh the empty syringe again and record its mass to the nearest 0,1 mg. Subtract the mass of the syringe from that of the test portion and the syringe to obtain the net test portion mass, m. Record the net mass to the nearest 0,1 mg.

Carry out a titration and record the volume, V, in millilitres, of titration component. Do not perform more than three titrations on one portion of the working medium.

If the titrator does not automatically calculate the results (see 8.1), record the titration time, t.

It is recommended to carry out several replicates for each sample to obtain more accurate results. In that case, record for each replicate, i, the individual test portion mass, m_i, the amount of titration component, V_i, and the titration time, t_i.

8 Calculation and expression of results

8.1 Calculation

Appropriate automatic calculation by the titrator may also be used, if available.

8.1.1 Calculate the water content of the test sample, $w_{\mathrm{H_2O}}$, expressed as a percentage mass fraction, by using either Equation (5) or (6):

$$w_{\mathrm{H_2O}} = \frac{(V - q_V t)\rho_{\mathrm{e}}}{m} \times 100 \tag{5}$$

or

$$w_{\mathrm{H_2O}} = \frac{V\rho_{\mathrm{e}} - q_m t}{m} \times 100 \tag{6}$$

where

V is the volume, in millilitres, of titration reagent (7.5);

q_V is the numerical value of the drift (7.1.2), in millilitres per minute;

q_m is the numerical value of the drift, in milligrams per minute (7.1.2);

t is the titration time, in minutes (7.5);

ρ_{e} is the water equivalent of the titration component, in milligrams per millilitre (7.2.3);

m is the mass, in milligrams, of the test portion (7.5).

8.1.2 If n replicates for a sample have been carried out, calculate for each of the replicates, i, the individual water content of the test sample, $w_{\mathrm{H_2O},i}$, expressed as percentage mass fraction, by using either Equation (7) or (8):

$$w_{\mathrm{H_2O},i} = \frac{(V_i - q_V t_i)\rho_{\mathrm{e}}}{m_i} \times 100 \tag{7}$$

or

$$w_{\mathrm{H_2O},i} = \frac{V_i\,\rho_{\mathrm{e}} - q_m\,t_i}{m_i} \times 100 \tag{8}$$

where

V_i is the volume, in millilitres, of titration reagent for replicate i (7.5);

t_i is the titration time, in minutes, for replicate i (7.5);

m_i is the mass, in milligrams, of the test portion for replicate i (7.5);

i is the number of replicates for i = 1 to n.

8.1.3 Calculate, for the n replicates, the mean value of the water content of the test sample, $\bar{w}_{H_2O}$, expressed as a percentage mass fraction, by using Equation (9):

$$\bar{w}_{H_2O} = \frac{\sum_{i=1}^{n} w_{H_2O,i}}{n} \tag{9}$$

8.2 Expression of results

Express the test results to two decimal places.

9 Precision

9.1 Interlaboratory test

The values for repeatability and reproducibility derived from this interlaboratory test were determined in accordance with ISO 5725-1[2] and ISO 5725-2[3].

Details of an interlaboratory test on the precision of the method are given in Annex A. The values obtained may not be applicable to concentration ranges and matrices other than those given.

9.2 Repeatability

The absolute difference between two individual single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, shall in not more than 5 % of cases be greater than 0,01 % mass fraction.

9.3 Reproducibility

The absolute difference between two individual single test results, obtained with the same method on identical test material in different laboratories with different operators using different equipment, shall in not more than 5 % of cases be greater than 0,05 % mass fraction.

10 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained; and if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Interlaboratory trial

An international collaborative test (see Reference [4]) involving 12 laboratories was carried out on six samples of butteroil obtained from the Netherlands. The levels of water content varied from 0,03 % mass fraction to 0,08 % mass fraction.

NOTE The water content of butteroil on the market is far below 1 % mass fraction and usually lies between 0,02 % and 0,1 %. Therefore, samples with similarly low water contents were used in the collaborative trial.

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1[2] and ISO 5725-2[3] to give the precision data shown in Table A.1.

Table A.1 — Results of the interlaboratory test

Parameter	**Sample**						**Mean**
	1	**2**	**3**	**4**	**5**	**6**	
Participating laboratories after eliminating outliers, n_r	11	12	12	12	12	12	
Mean value, $\bar{w}_{H_2O}$, %	0,036	0,080	0,057	0,082	0,035	0,064	
Repeatability standard deviation, s_r, %	0,005	0,004	0,004	0,005	0,005	0,003	0,004
Repeatability limit $r = 2{,}8\ s_r$, %	0,013	0,010	0,011	0,014	0,013	0,008	0,012
Coefficient of variation of repeatability, $C_{V,\,r}$, %	13,28	4,40	7,20	6,12	13,19	4,50	
Reproducibility standard deviation, s_R, %	0,010	0,027	0,017	0,026	0,010	0,021	0,019
Reproducibility limit, $R = 2{,}8\ s_R$, %	0,027	0,074	0,048	0,074	0,027	0,058	0,051
Coefficient of variation of reproducibility, $C_{V,\,R}$, %	27,21	33,00	30,38	32,30	27,40	32,17	