

AGENDA PWG

1. ADDRESS METHODS FROM CX/MAS 18/39/3

CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU39)

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CX 72-1981)

The Committee agreed to submit the methods for biotin, vitamin D, and chloride to CCMAS for typing, endorsement and inclusion in the *Recommended Methods of Analysis and Sampling* (CX 234-1999) and request CCMAS to re-type the related existing methods for biotin, vitamin D and chloride in CX 234-1999.

The Committee **is invited to endorse** the methods of analysis and consequential retyping of existing methods in PWG Table 1.

PWG TABLE 1: Methods of analysis for infant formula

| Commodity | Provision | Method | Principle | Proposed Type |
|------------------|------------------|---|----------------------|----------------------|
| Infant Formula | Biotin | EN 15607 | HPLC | # III |
| | | AOAC 2016.02 | HPLC | II |
| | Vitamin D | AOAC 992.26 | HPLC | III |
| | | EN 12821 | HPLC | # III |
| | | AOAC 995.05 | HPLC | III |
| | | AOAC 2016.05 ISO DIS 20636 | LC-MS | II |
| | Chloride | AOAC 986.26 | Potentiometry | III |
| | | AOAC 2016.03 ISO DIS 21422 IDF 242 | Potentiometry | II |

COMMITTEE ON MILK AND MILK PRODUCTS (CCMMP)
Methods of analysis for dairy permeate powders

NOTE: The Commission adopted the Standard for Dairy Permeate Powders at Step 8 subject to the endorsement of the provisions on food labelling and methods of analysis by CCFL44 and CCMAS39, respectively.

The Committee **is invited to endorse** the methods of analysis in PWG TABLE 2.

PWG TABLE 2: Methods of analysis for dairy permeate powders

| Provisions | Method | Principle | Type |
|--------------------|--|--|-------------|
| Lactose, anhydrous | ISO 22662 IDF 198:2007 - Milk and milk products - Determination of lactose* | HPLC (high-performance liquid chromatography) | II |
| Milkfat | ISO 1736 IDF 009:2008 - Dried milk and dried milk products - Determination of fat content | Gravimetry (Röse-Gottlieb) | I |
| Nitrogen | ISO 8968-1 IDF 020-1:2014 - Milk and milk products - Determination of nitrogen content - Part 1 | Titrimetry, Kjeldahl principle | I |
| Moisture** | ISO 5537 IDF 026:2004 - Dried milk Determination of moisture content | Gravimetry (drying at 87°C) | I |
| Ash | NMKL 173:2005 Ash, gravimetric determination in foods AOAC 930.30-1930 - Ash of Dried Milk | Gravimetry (ashing at 550 °C) | IV |

2. ADDRESS METHODS/SAMPLING PLANS FROM CX/MAS 18/39/3 Add 1

CODEX COMMITTEE ON CEREALS, PULSES AND LEGUMES (CCCPL)

Methods of analysis for quinoa (CL 2018/25-CPL Annex II)

Note: The draft standard for quinoa is forwarded to the Commission for adoption at Step 8. The Committee **is invited to endorse** the methods in PWG Table 3.

PWG Table 3: Methods of analysis for Quinoa

| Provision | Method | Principle | Type |
|--|----------|----------------------|------|
| Moisture content | ISO 712 | Gravimetric | 1 |
| Protein Content (N x 6.25) Dry weight basis | ISO 1871 | Titrimetry, Kjeldahl | 1 |

*Methods of analysis for quinoa*¹

CCCPL identified saponin as a quality requirement; however, the EWG on the development of the draft standard for quinoa concluded that there was no method validated internationally for the determination of saponin in quinoa.

The Committee **is invited to advise** on suitable testing methods for saponin in quinoa.

*Sampling plan for MLs for methylmercury in fish (CXS 193-1995)*²

The Committee **is invited to endorse** the sampling plan below.

¹ CL 2018/25-CPL paras. 12 and 16

² REP18/CF, para 91, /Appendix IV, Part B

COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)**PROPOSED DRAFT SAMPLING PLAN FOR METHYLMERCURY CONTAMINATION IN FISH****DEFINITIONS**

The following definitions should apply:

| | |
|---------------------------|---|
| Lot | An identifiable quantity of a food commodity delivered at one time and determined by the official to have common characteristics, such as origin, variety, type of packing, packer, consignor, or markings. |
| Sublot | Designated part of a larger lot in order to apply the sampling method on that designated part. Each sublot must be physically separate and identifiable. |
| Incremental sample | The quantity of material taken from a single random place in the lot or sublot. |
| Aggregate sample | The combined total of all the incremental samples that is taken from the lot or sublot. The aggregate sample has to be at least as large as the laboratory sample or samples combined. |
| Laboratory sample | A sample intended for a laboratory. |

SAMPLING METHODS**GENERAL PROVISIONS****Personnel**

Sampling should be performed by an authorised person as designated by the national authority.

Material to be sampled

Each lot or sublot which is to be examined should be sampled separately.

Precautions to be taken

In the course of sampling, precautions should be taken to avoid any changes which would affect the levels of contaminants, adversely affect the analytical determination or make the aggregate samples unrepresentative.

Incremental samples

As far as possible, incremental samples should be taken at various places distributed throughout the lot or sublot.

Preparation of the aggregate sample

The aggregate sample should be made up by combining the incremental samples.

Samples for enforcement, defence and referee purposes

The samples for enforcement, defence and referee purposes should be taken from the homogenised aggregate sample unless this conflicts with the rules of the national authority as regards the rights of the food business operator.

Packaging and transmission of samples

Each sample should be placed in a clean, inert container offering adequate protection from contamination, from loss of analytes by adsorption to the internal wall of the container and against damage in transit. All necessary precautions should be taken to avoid any change in composition of the sample which might arise during transportation or storage.

Sealing and labelling of samples

Each sample taken for official use should be sealed at the place of sampling and identified following the locally applicable rules.

A record should be kept of each sampling, permitting each lot or subplot to be identified unambiguously (reference to the lot number should be given) and giving the date and place of sampling together with any additional information likely to be of assistance to the analyst.

SAMPLING PLAN

Division of lots into sublots

Large lots should be divided into sublots on condition that the subplot may be separated physically. For products traded in bulk consignments Table 1 should apply. For other products Table 2 should apply. Taking into account that the weight of the lot is not always an exact multiple of the weight of the sublots, the weight of the subplot may exceed the mentioned weight by a maximum of 20%.

Number of incremental samples

The aggregate sample should be at least 1 kg except where it is not possible, e.g. when the sample consists of 1 package or unit.

The minimum number of incremental samples to be taken from the lot or subplot should be as given in Table 3.

The incremental samples should be of similar weight/volume. The weight/ volume of an incremental sample should be at least 100 grams, resulting in an aggregate sample of at least about 1 kg. Departure from this method should be recorded.

Table 1 Subdivision of lots into sublots for products traded in bulk consignments

| Lot weight (ton) | Weight or number of sublots |
|---------------------------|-----------------------------|
| $\geq 1\ 500$ | 500 tonnes |
| > 300 and $< 1\ 500$ | 3 sublots |
| ≥ 100 and ≤ 300 | 100 tonnes |
| < 100 | — |

Table 2 Subdivision of lots into sublots for other products

| Lot weight (ton) | Weight or number of sublots |
|------------------|-----------------------------|
| ≥ 15 | 15-30 tonnes |
| < 15 | — |

Table 3 Minimum number of incremental samples to be taken from the lot or subplot

| Weight or volume of lot/sublot (in kg) | Minimum number of incremental samples to be taken |
|--|---|
| < 50 | 3 |
| ≥ 50 and ≤ 500 | 5 |
| > 500 | 10 |

If the lot or subplot consists of individual packages or units, then the number of packages or units which should be taken to form the aggregate sample is given in Table 4.

Table 4 Number of packages or units (incremental samples) which should be taken to form the aggregate sample if the lot or subplot consists of individual packages or units

| Number of packages or units in the lot/ subplot | Number of packages or units to be taken |
|---|---|
| ≤ 25 | at least 1 package or unit |
| 26-100 | about 5%, at least 2 packages or units |
| > 100 | about 5%, at maximum 10 packages or units |

Specific provisions for the sampling of large fish arriving in large lots

In case the lot or subplot to be sampled contains large fish (individual fish weighing more than about 1 kg) and the lot or subplot weighs more than 500 kg, the incremental sample should consist of the middle part of the fish. Each incremental sample should weigh at least 100 g.

SAMPLING AT RETAIL STAGE

Sampling of foodstuffs at retail stage should be done where possible in accordance with the sampling provisions set out in this sampling plan.

Where it is not possible to carry out the method of sampling set out above because of the unacceptable commercial consequences (e.g. because of packaging forms, damage to the lot, etc.) or where it is practically impossible to apply the abovementioned method of sampling, an alternative method of sampling may be applied provided that it is sufficiently representative for the sampled lot or subplot and is fully documented.

SAMPLE PREPARATION AND ANALYSIS

LABORATORY QUALITY STANDARDS

Laboratories should be able to demonstrate that they have internal quality control procedures in place. Examples of these are the 'ISO/ AOAC/IUPAC Guidelines on Internal Quality Control in Analytical Chemistry Laboratories'³.

Wherever possible the trueness of analysis should be estimated by including suitable certified reference materials in the analysis.

Precautions and general considerations

The basic requirement is to obtain a representative and homogeneous laboratory sample without introducing secondary contamination.

All of the sample material received by the laboratory should be used for the preparation of the laboratory sample.

Compliance with maximum levels laid down in the General Standard for Contaminants and toxins in Food and Feed should be established on the basis of the levels determined in the laboratory samples.

Specific sample preparation procedures

The analyst should ensure that samples do not become contaminated during sample preparation. Wherever possible, apparatus and equipment coming into contact with the sample should not contain mercury and be made of inert materials, e.g. plastics such as polypropylene, polytetrafluoroethylene (PTFE) etc. These should be acid cleaned to minimise the risk of contamination. High quality stainless steel may be used for cutting edges.

There are many satisfactory specific sample preparation procedures which may be used for the products under consideration. For those aspects not specifically covered by this sampling plan, the CEN Standard 'Foodstuffs. Determination of elements and their chemical species. General considerations and specific requirements'⁴ has been found to be satisfactory but other sample preparation methods may be equally valid.

³ Edited by M. Thompson and R. Wood, Pure Appl. Chem., 1995, 67, 649-666.

⁴ Standard EN 13804:2013, 'Foodstuffs. Determination of elements and their chemical species. General considerations and specific requirements', CEN, Rue de Stassart 36, B-1050 Brussels.

Treatment of the sample as received in the laboratory

The complete aggregate sample should be finely ground (where relevant) and thoroughly mixed using a process that has been demonstrated to achieve complete homogenisation.

Samples for enforcement, defence and referee purposes

The samples for enforcement, defence and referee purposes should be taken from the homogenised material unless this conflicts with the applicable rules at the national level on sampling as regards the rights of the food business operator.

METHODS OF ANALYSIS

Definitions

| | |
|-----------------------------|--|
| r | Repeatability the value below which the absolute difference between single test results obtained under repeatability conditions (i.e., same sample, same operator, same apparatus, same laboratory, and short interval of time) may be expected to lie within a specific probability (typically 95%) and hence $r = 2,8 \times s r$. |
| s r | Standard deviation calculated from results generated under repeatability conditions. |
| RSD r | Relative standard deviation calculated from results generated under repeatability conditions $[(s r /) \times 100]$. |
| R | Reproducibility the value below which the absolute difference between single test results obtained under reproducibility conditions (i.e., on identical material obtained by operators in different laboratories, using the standardised test method), may be expected to lie within a certain probability (typically 95%); $R = 2,8 \times s R$. |
| s R | Standard deviation, calculated from results under reproducibility conditions. 'RSD R' = Relative standard deviation calculated from results generated under reproducibility conditions $[(s R /) \times 100]$. |
| LOD | Limit of detection, smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty. The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations ($n > 20$). |
| LOQ | Limit of quantification, lowest content of the analyte which can be measured with reasonable statistical certainty. If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to 10 times the standard deviation of the mean of blank matrix determinations ($n \geq 20$). |
| HORRAT⁵ r | The observed RSD r divided by the RSD r value estimated from the (modified) Horwitz equation (2) (cf. point C.3.3.1 ('Notes to the performance criteria')) using the assumption $r = 0,66 R$. |
| HORRAT⁶ R | The observed RSD R divided by the RSD R value estimated from the (modified) Horwitz equation ⁷ (cf. point 'Notes to the performance criteria'). |
| u | Combined standard measurement uncertainty obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model ⁸ |
| U | The expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95% ($U = 2u$). |
| Uf | Maximum standard measurement uncertainty. |

General requirements

Methods for analysis for total mercury are appropriate for screening purpose for control on methylmercury levels. If the total mercury concentration is below or equal to the maximum level for methylmercury, no further

⁵ Horwitz W. and Albert, R., 2006, The Horwitz Ratio (HorRat): A useful Index of Method Performance with respect to Precision, Journal of AOAC International, Vol. 89, 1095-1109. (2) M. Thompson, Analyst, 2000, p. 125 and 385-386.

⁶ Horwitz W. and Albert, R., 2006, The Horwitz Ratio (HorRat): A useful Index of Method Performance with respect to Precision, Journal of AOAC International, Vol. 89, 1095-1109.

⁸ International vocabulary of metrology – Basic and general concepts and associated terms (VIM), JCGM 200:2008.

testing is required and the sample is considered to be compliant with the maximum level for methylmercury. If the total mercury concentration is at or above the maximum level for methylmercury, follow-up testing should be conducted to determine if the methylmercury concentration is above the maximum level for methylmercury.

Specific requirements

Performance criteria

Where no specific methods for the determination of contaminants in foodstuffs are prescribed at the Codex level, laboratories may select any validated method of analysis for the respective matrix provided that the selected method meets the specific performance criteria set out in Table 5.

It is recommended that fully validated methods (i.e. methods validated by collaborative trial for the respective matrix) are used where appropriate and available. Other suitable validated methods (e.g. in-house validated methods for the respective matrix) may also be used provided that they fulfil the performance criteria set out in Tables 5.

Where possible, the validation of in-house validated methods should include a certified reference material.

Table 5 Performance criteria for methods of analysis of mercury and methylmercury

| Parameter | Criterion | | |
|-------------------------------------|--|------------------------|-----------------------|
| Applicability | Fish specified in the General Standard for Contaminants and Toxins in Food and Feed (GSCTFF, CXS 193-1995) | | |
| Specificity | Free from matrix or spectral interferences | | |
| Repeatability (RSD _r) | HORRAT _r less than 2 | | |
| Reproducibility (RSD _R) | HORRAT _R less than 2 | | |
| Recovery | The provisions of 'Recovery calculations' apply | | |
| LOD | = three tenths of LOQ | | |
| LOQ | Methylmercury | ML is < 0,100mg/kg | ML is ≥ 0,100 mg/kg |
| | | ≤ two fifths of the ML | ≤ one fifth of the ML |

Notes to the performance criteria:

The Horwitz equation⁹ (for concentrations $1,2 \times 10^{-7} \leq C \leq 0,138$) and the modified Horwitz equation¹⁰ (for concentrations $C < 1,2 \times 10^{-7}$) are generalised precision equations which are independent of analyte and matrix but solely dependent on concentration for most routine methods of analysis.

Modified Horwitz equation for concentrations $C < 1,2 \times 10^{-7}$:

$$RSD R = 22\%$$

where:

- RSD R is the relative standard deviation calculated from results generated under reproducibility conditions $[(s R /) \times 100]$
- C is the concentration ratio (i.e. 1 = 100 g/100 g, 0,001 = 1 000 mg/kg). The modified Horwitz equation applies to concentrations $C < 1,2 \times 10^{-7}$.

Horwitz equation for concentrations $1,2 \times 10^{-7} \leq C \leq 0,138$:

$$RSD R = 2C^{(-0,15)}$$

⁹ W. Horwitz, L.R. Kamps, K.W. Boyer, J.Assoc.Off.Analy.Chem., 1980, 63, 1344.

¹⁰ M. Thompson, Analyst, 2000, p. 125 and 385-386.

where:

- RSD R is the relative standard deviation calculated from results generated under reproducibility conditions $[(s R /) \times 100]$
- C is the concentration ratio (i.e. 1 = 100 g/100 g, 0,001 = 1 000 mg/kg). The Horwitz equation applies to concentrations $1,2 \times 10^{-7} \leq C \leq 0,138$.

Fitness-for-purpose' approach

For in-house validated methods, as an alternative a 'fitness-for-purpose' approach¹¹ may be used to assess their suitability for official control. Methods suitable for official control must produce results with a combined standard measurement uncertainty (u) less than the maximum standard measurement uncertainty calculated using the formula below:

$$Uf = \sqrt{(LOD/2)^2 + (\alpha C)^2}$$

where:

- Uf is the maximum standard measurement uncertainty ($\mu\text{g}/\text{kg}$).
- LOD is the limit of detection of the method ($\mu\text{g}/\text{kg}$). The LOD must meet the performance criteria set in point C.3.3.1 for the concentration of interest.
- C is the concentration of interest ($\mu\text{g}/\text{kg}$);
- α is a numeric factor to be used depending on the value of C. The values to be used are given in Table 6.

Table 6 Numeric values to be used for α as constant in formula set out in this point, depending on the concentration of interest

| C ($\mu\text{g}/\text{kg}$) | α |
|-------------------------------|----------|
| ≤ 50 | 0,2 |
| 51-500 | 0,18 |
| 501-1 000 | 0,15 |
| 1 001-10 000 | 0,12 |
| $> 10 000$ | 0,1 |

Table 7: Calculated performance criteria for $ML \geq 0.1 \text{ mg}/\text{kg}$

| | ML mg/kg | LOD mg/kg | LOQ mg/kg | Min. applicable range | | Precision RSDR (%) |
|------------|-------------|--------------|--------------|-----------------------|-------------|-----------------------|
| | | | | From mg/kg | To mg/kg | |
| All Tuna | 1.2 | 0.12 | 0.24 | 0.64 | 1.76 | 31.1 |
| Alfonsino | 1.5 | 0.15 | 0.3 | 0.823 | 2.177 | 30.1 |
| All Marlin | 1.7 | 0.17 | 0.34 | 0.947 | 2.453 | 29.5 |
| Shark | 1.6 | 0.16 | 0.32 | 0.885 | 2.315 | 29.8 |

¹¹ M. Thompson and R. Wood, Accred. Qual. Assur., 2006, p. 10 and 471-478.

REPORTING AND INTERPRETATION OF RESULTS

Expression of results

The results should be expressed in the same units and with the same number of significant figures as the maximum levels laid down in the *General Standard for Contaminants and Toxins in Food and Feed* (GSCTFF) (CXS 193-1995).

Recovery calculations

If an extraction step is applied in the analytical method, the analytical result should be corrected for recovery. In this case the level of recovery must be reported.

In case no extraction step is applied in the analytical method, the result may be reported uncorrected for recovery if evidence is provided by ideally making use of suitable certified reference material that the certified concentration allowing for the measurement uncertainty is achieved (i.e. high accuracy of the measurement), and thus that the method is not biased. In case the result is reported uncorrected for recovery this should be mentioned.

Measurement uncertainty

The analytical result should be reported as $x \pm U$ whereby x is the analytical result and U is the expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95% ($U = 2u$).

INTERPRETATION OF RESULTS

Acceptance of a lot/sublot

The lot or subplot is accepted if the analytical result of the laboratory sample does not exceed the respective maximum level as laid down in the *General Standard for Contaminants and Toxins in Food and Feed* (GSCTFF, CXS 193-1995), taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.

Rejection of a lot/sublot

The lot or subplot is rejected if the analytical result of the laboratory sample exceeds beyond reasonable doubt the respective maximum level as laid down in the *General Standard for Contaminants and Toxins in Food and Feed* (GSCTFF, CXS 193-1995), taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.

Applicability

The present interpretation rules should apply for the analytical result obtained on the sample for enforcement. In case of analysis for defence or reference purposes, the locally applicable rules should apply.

3. ADDRESS METHODS FROM CX/MAS 18/39/2 Add. 1

COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)

Sampling plan for MLs for methylmercury in fish (CXS 193-1995)¹²

CCCF12 agreed to request CCMAS advice on the following:

- The necessary performance criteria for the MLs;
- Whether there is evidence that methyl mercury can vary widely between individual fish sampled at the same time. How this would apply to large fish sold as individual units and whether the sampling plan provides enough basis to deal with this; and
- Whether the whole fish should be analyzed or only specific fractions of edible portions. Currently only mention is made that the mid-section should be sampled for some large fish. (see CX/MAS 18/39/3 Add. 1)

The Committee **is invited to provide advice** as requested by CCCF.

Validated method of analysis for Sterigmatocystin (STC)

CCCF agreed to inform the Standards development organizations of the need for an internationally validated method of analysis for STC through CCMAS.

The Committee **is invited to take note** of the request.

¹² REP18/CF, para 87, /Appendix IV, Part B

4. ADDRESS PROPOSED NEW METHODS FROM CX/MAS 18/39/4 Add. 1

PWG Table 4: Proposed new methods from CX/MAS 18/39/4 Add. 1

| Commodity | Provision | Method | Principle | Type |
|---|-----------------------|-------------------------------------|------------------------------------|-----------|
| <u>Cheese</u> | <u>Propionic acid</u> | <u>ISO/TS 19046-1I IDF/RM 233-1</u> | <u>Gas chromatography</u> | <u>IV</u> |
| <u>Cheese</u> | <u>Propionic acid</u> | <u>ISO/TS 19046-2I IDF/RM 233-2</u> | <u>Ion exchange chromatography</u> | <u>IV</u> |
| <i>Comment: CXS 283 General Standard for Cheese has a maximum level of 3000 mg/kg for propionic acid.</i> | | | | |

4a. ADDRESS OTHER DAIRY METHODS NOT CAPTURED IN CX/MAS 18/39/4 Add. 1

In review of the PWG Agenda, it was found that the Codex standard for Emmental (STAN 269) has a min level of 150 mg/100g of cheese ready for sale. Can this be covered by the lines above or require additional lines?

| Commodity | Provision | Method | Principle | Type |
|-----------------|-----------------------|-------------------------------------|------------------------------------|-----------|
| <u>Emmental</u> | <u>Propionic acid</u> | <u>ISO/TS 19046-1I IDF/RM 233-1</u> | <u>Gas chromatography</u> | <u>IV</u> |
| <u>Emmental</u> | <u>Propionic acid</u> | <u>ISO/TS 19046-2I IDF/RM 233-2</u> | <u>Ion exchange chromatography</u> | <u>IV</u> |

5. ADDRESS QUESTIONS RAISED BY IDF/ISO/AOAC in CX/MAS 18/39/4 Add. 1

Clarify rules for determining when a defining method should be Type I or Type IV method.

For example:

- Is it necessary to have precision figures for a Type I method?
- If a defining method has been subjected to an international collaborative study involving dairy commodities A, B and C, and the method is generally known to work on commodity D, but this commodity was not included in the study, should the method then be listed as Type I or Type IV in STAN 234 for commodity D?

- Clarify for the situation where there are two defining methods (from different organisations) and the degree of validation differs (i.e. one method has been subjected to an international collaborative study, whereas the other method has not), whether one method be Type I and the other method Type IV, or only one (the best validated) method should be accepted and be listed as Type I.
- Clarify for those cases where a provision is not specifically listed in the Commodity Standard, what decision process is to be followed to determine whether or not to include such provision in CXS234 (e.g., see provisions for iron in milk products, lead in edible casein products, and MSNF in cream in the table below).
- Apply a consistent approach in listing provisions that require a calculation based on two or more analyses. In some cases, all concerned methods are listed; in other cases only a single method (see example of inconsistency below).

6 ADDRESS Table in CX/MAS 18/39/4 Add 1 and WORKABLE PACKAGE “DAIRY GROUP”

Note: PWG Table 5 is a combination of table listed in CX/MAS 18/39/4 Add. 1 and from Dairy Group Workable Package (DG WP) provided as link in CX/MAS 18/39/4 Appendix II. The red **bold red underline text** is from the REMARKS column of the DG WP and refers to row just above. Additionally, IDF had provided responses, so they have been included for information as *red italics* text.

PWG Table 5: 18/39/4 Add. 1 and Dairy Group Workable Package Remarks (red bold underline) and IDF Response (*red italics*)

| Milk and Milk Products | | | | |
|--|-----------|--|---|-----|
| Milk products | Iron | NMKL139 AOAC999.11 (Codex general method) | Atomic absorption spectrophotometry | II |
| Comment: <i>There is no standard for milk products and no provision for iron in nutrition labelling nor GSTCFF. There are provisions for iron in butter, milk fat products, whey powders and edible casein products. AOAC 999.11 as written has only been validated in milk powder, does not contain precision data or specify applicability to milk products.</i> <i>A separator is needed between NMKL 139 AOAC 999.1, forward slash or vertical line as appropriate.</i> | | | | |
| Milk products | Iron | NMKL161/ AOAC999.10 | Atomic absorption spectrophotometry | III |
| Comment: <i>There is no standard for milk products and no provision for iron in nutrition labelling nor GSTCFF. There are provisions for iron in butter, milk fat products, whey powders and edible casein products. AOAC 999.10 as written is not applicable to foods ≥40%fat, specifically states not applicable to milk powder, does not contain precision data or specify applicability to milk products.</i> | | | | |
| Milk products | Iron | AOAC984.27 | Inductively Coupled Plasma optical emission spectrophotometry | III |
| Comment: <i>There is no standard for milk products and no provision for iron in nutrition labelling nor GSTCFF. There are provisions for iron in butter, milk fat products, whey powders and edible casein products. AOAC 984.27 as written has only been validated in infant formula and does not include precision data or specify applicability to milk products.</i> | | | | |
| Milk products | Iron | ISO6732 IDF103 | Photometry (bathophenanthroline) | IV |
| Comment: <i>There is no standard for milk products and no provision for iron in nutrition labelling nor GSTCFF. There are provisions for iron in butter, milk fat products, whey powders and edible casein products.</i> | | | | |
| Milk and Milk Products | Melamine | ISO/TS15495 IDF/RM230 | LC-MS/MS | IV |
| Comment: <i>There is no standard for milk products.</i> | | | | |
| Milk products (products not completely soluble in ammonia) | Milk fat | ISO8262-3 IDF124-3 | Gravimetry (Weibull-Berntrop) | I |
| Comment: <i>There is no standard for milk products.</i> | | | | |
| Blend of evaporated skimmed milk and vegetable fat | Total fat | ISO1737 IDF13 | Gravimetry (Röse-Gottlieb) | I |

Milk and Milk Products

| | | | | |
|--|--|--------------------------------|---|----|
| Blend of evaporated skimmed milk and vegetable fat | Milk solids-not-fat ¹³ (MSNF) | ISO6731 IDF21andISO1737 ID F13 | Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb) | I |
| Blend of evaporated skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | IV |
| Comment from DG WP: This provision has no type II method | | | | |
| <i>Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed Ref: CRD 1 CCMAS 2010</i> | | | | |
| Blend of evaporated skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | AOAC991.20 | Titrimetry (Kjeldahl) | IV |
| Comment from DG WP: This provision has no type II method. The Codex Stan 234 mentions incorrectly the AOAC method . The correct one is AOAC 991.20 | | | | |
| <i>Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010</i> | | | | |
| Reduced fat blend of evaporated skimmed milk and vegetable fat | Total fat | ISO1737 IDF13 | Gravimetry (Röse-Gottlieb) | I |
| Reduced fat blend of evaporated skimmed milk and vegetable fat | Milk solids-not-fat ¹⁵ (MSNF) | ISO6731 IDF21 andISO1737 IDF13 | Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb) | I |
| Comment: Note 15 is needed | | | | |
| Reduced fat blend of evaporated skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | ISO8968-1 IDF20-1/ | Titrimetry (Kjeldahl) | IV |
| Comment from DG WP: This provision has no type II method | | | | |
| <i>Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010</i> | | | | |
| Reduced fat blend of evaporated skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | AOAC991.20 | Titrimetry (Kjeldahl) | IV |
| Comment from DG WP: This provision has no type II method | | | | |
| <i>Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010</i> | | | | |
| Blend of skimmed milk and vegetable fat in powdered form | Total fat | ISO1736 IDF9 | Gravimetry (Röse-Gottlieb) | I |
| Blend of skimmed milk and vegetable fat in powdered form | Water ¹⁴ | ISO5537 IDF26 | Gravimetry, dryingat87°C | I |
| Blend of skimmed milk and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | ISO8968-1 IDF20-1/ | Titrimetry (Kjeldahl) | IV |

¹³ Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

¹⁴ Water content excluding the crystallized water bound to lactose (generally known as "moisture content")

Milk and Milk Products

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|--|------------------------------------|-------------------|------------------------------|-----------|
| Blend of skimmed milk and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | AOAC991.20 | Titrimetry (Kjeldahl) | IV |
|--|------------------------------------|-------------------|------------------------------|-----------|

Comment: The content of the line was missing

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|---|-----------|--------------|----------------------------|---|
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Total fat | ISO1736 IDF9 | Gravimetry (Röse-Gottlieb) | I |
|---|-----------|--------------|----------------------------|---|

| | | | | |
|---|---------------------|---------------|---------------------------|---|
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Water ¹⁶ | ISO5537 IDF26 | Gravimetry, drying at87°C | I |
|---|---------------------|---------------|---------------------------|---|

| | | | | |
|---|------------------------------------|-------------------|-----------------------|----|
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | IV |
|---|------------------------------------|-------------------|-----------------------|----|

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|---|------------------------------------|------------|-----------------------|----|
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | AOAC991.20 | Titrimetry (Kjeldahl) | IV |
|---|------------------------------------|------------|-----------------------|----|

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|---|-----------|---------------|----------------------------|---|
| Blend of sweetened condensed skimmed milk and vegetable fat | Total fat | ISO1737 IDF13 | Gravimetry (Röse-Gottlieb) | I |
|---|-----------|---------------|----------------------------|---|

| | | | | |
|---|---------|---------------|-------------|----|
| Blend of sweetened condensed skimmed milk and vegetable fat | Sucrose | ISO2911 IDF35 | Polarimetry | IV |
|---|---------|---------------|-------------|----|

| | | | | |
|---|---|---------------|--|----|
| Blend of sweetened condensed skimmed milk and vegetable fat | Milksolids-not-fat ¹⁵ (MSNF) | ISO6734 IDF15 | Calculation from total solids content, fat content and sugar content | IV |
|---|---|---------------|--|----|

Comment: note 15 needed.

Milk and Milk Products

| | | | | |
|--|------------------------------------|-------------------|-----------------------|----|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | IV |
|--|------------------------------------|-------------------|-----------------------|----|

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|--|------------------------------------|------------|-----------------------|----|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | AOAC991.20 | Titrimetry (Kjeldahl) | IV |
|--|------------------------------------|------------|-----------------------|----|

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|---|-----------|---------------|----------------------------|---|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Total fat | ISO1737 IDF13 | Gravimetry (Röse-Gottlieb) | I |
|---|-----------|---------------|----------------------------|---|

| | | | | |
|---|---|---------------|---|----|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Milksolids-not-fat ¹⁵ (MSNF) | ISO6734 IDF15 | Calculation from total solids content and sugar content | IV |
|---|---|---------------|---|----|

Comment: note 15 needed.

| | | | | |
|---|------------------------------------|-------------------|-----------------------|----|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Milk protein in MSNF ¹⁵ | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | IV |
|---|------------------------------------|-------------------|-----------------------|----|

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|---|---|------------|-----------------------|----|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Milk protein <u>in</u> MSNF ¹⁵ | AOAC991.20 | Titrimetry (Kjeldahl) | IV |
|---|---|------------|-----------------------|----|

Comment from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|--------|--------|--------------------------|------------------------------------|----|
| Butter | Copper | ISO5738 IDF76 AOAC960.40 | Photometry, diethyldithiocarbamate | II |
|--------|--------|--------------------------|------------------------------------|----|

Comment: The IDF/ISO method and AOAC method are different and should be written in different lines. AOAC 960.40 as written does not contain precision data or specify applicability to butter.

Milk and Milk Products

| | | | | |
|--------|------|----------------------------------|-------------------------------------|----|
| Butter | Lead | AOAC972.25(Codex general method) | Atomic absorption spectrophotometry | II |
|--------|------|----------------------------------|-------------------------------------|----|

Comment: AOAC 972.25 as written does not contain precision data or specify applicability to butter.

| | | | | |
|--------|---|-------------------|------------|---|
| Butter | Milksolids-not-fat ¹⁵ (MSNF) | ISO3727-2 IDF80-2 | Gravimetry | I |
|--------|---|-------------------|------------|---|

Comment: note 15 needed.

| | | | | |
|--------|---------|-----------------|--|---|
| Butter | Milkfat | ISO17189 IDF194 | Gravimetry Direct determination of fat using solvent extraction | I |
|--------|---------|-----------------|--|---|

| | | | | |
|--------|----------------|-----------------|---|---|
| Butter | Milkfat purity | ISO17678 IDF202 | Calculation from determination of triglycerides by gas chromatography | I |
|--------|----------------|-----------------|---|---|

| | | | | |
|--------|------|--------------------------|---|-----|
| Butter | Salt | ISO1738 IDF12/AOAC960.29 | Titrimetry(Mohr: determination of chloride, expressed as sodium chloride) | III |
|--------|------|--------------------------|---|-----|

| | | | | |
|--------|------|-----------------|---|----|
| Butter | Salt | ISO15648 IDF179 | Potentiometry (determination of chloride, expressed as sodium chloride) | II |
|--------|------|-----------------|---|----|

| | | | | |
|--------|-------------------------|-----------------|--------------------|----|
| Butter | Vegetable fat (sterols) | ISO12078 IDF159 | Gas chromatography | II |
|--------|-------------------------|-----------------|--------------------|----|

| | | | | |
|--------|-------------------------|-----------------|--------------------|-----|
| Butter | Vegetable fat (sterols) | ISO18252 IDF200 | Gas chromatography | III |
|--------|-------------------------|-----------------|--------------------|-----|

| | | | | |
|--------|---------------------|--------------------|------------|---|
| Butter | Water ¹⁶ | ISO3727-1 IDF80-1 | Gravimetry | I |
|--------|---------------------|--------------------|------------|---|

Comment from DG WP: The correct method is ISO 3727-1|IDF 80-1; The ISO method mentions moisture, non fat solids and fat content in butter

Response from IDF: This method has indeed three parts. Only part 1 determines moisture (so entry in 234 should be corrected to ISO 3727-1|IDF 80-1). Part 2 determines non-fat solids, and part 3 the calculation for fat. The equivalence between moisture and water is explained in the provision column, and is important to keep.

Comment: Correct references are ISO 3727-1|IDF 80-1

| | | | | |
|--------|-------------|---------------------|------------------|----|
| Cheese | Citric acid | ISO/TS2963 IDF/RM34 | Enzymatic method | IV |
|--------|-------------|---------------------|------------------|----|

| | | | | |
|--------|-------------|------------|------------|----|
| Cheese | Citric acid | AOAC976.15 | Photometry | II |
|--------|-------------|------------|------------|----|

Comment: AOAC 976.15 as written does not include precision data.

| | | | | |
|--------|---------|--------------|---|---|
| Cheese | Milkfat | ISO1735 IDF5 | Gravimetry (Schmid-Bondzynski-Ratslaff) | I |
|--------|---------|--------------|---|---|

| | | | | |
|--------|----------|--------------|------------------------------|---|
| Cheese | Moisture | ISO5534 IDF4 | Gravimetry, drying at 102 °C | I |
|--------|----------|--------------|------------------------------|---|

Milk and Milk Products

| | | | | |
|--------------------------|-----------|--------------------|--|-----|
| Cheese (and cheese rind) | Natamycin | ISO9233-1 IDF140-1 | Molecular absorption spectrophotometry | III |
| | | ISO9233-2 IDF140-2 | HPLC | II |

Comment: Shall the two lines above be fully separated as the two methods have different types?

| | | | | |
|---------------------|---|---------------|---|----|
| Cheese | Sodium chloride | ISO5943 IDF88 | Potentiometry (determination of chloride, expressed as sodium chloride) | II |
| Cheeses, individual | Dry matter (Total solids) | ISO5534 IDF4 | Gravimetry, drying at 102°C | I |
| Cheeses, individual | Milkfat in dry matter | ISO1735 IDF5 | Gravimetry (Schmid-Bondzynski-Ratzlaff) | I |
| Cheeses, individual | Dry matter (Total solids) ¹⁵ | ISO5534 IDF4 | Gravimetry, drying at 102°C | I |

Comment: The two lines above may need to be combined, as both ISO 1735|IDF5 and ISO 5534|IDF 4 are needed to determine milkfat in dry matter (see fat-free dry matter for cottage cheese for instance). CCMAS to clarify a consistent format when combination of several methods.

| | | | | |
|------------------|-----------------------------|-------------------------------|---|---|
| Cheeses in brine | Milkfat in dry matter (FDM) | ISO1735 IDF5 | Gravimetry (Schmid-Bondzynski-Ratzlaff) | I |
| Cottage cheese | Fat-free dry matter | ISO5534 IDF4 and ISO1735 IDF5 | Calculation from dry matter content and fat content Gravimetry, drying at 102°C Gravimetry (Schmid-Bondzynski-Ratzlaff) | I |
| Cottage cheese | Milkfat | ISO1735 IDF5 | Gravimetry (Schmid-Bondzynski-Ratzlaff) (for samples containing lactose up to 5%) | I |
| | | ISO8262-3 IDF124-3 | Gravimetry (Weibull-Berntrop) (for samples containing lactose over 5%) | I |
| Cottage cheese | Milkfat in dry matter | ISO8262-3 IDF124-3 | Gravimetry (Weibull-Berntrop) | I |

Comment: ISO5534|IDF4 must be added for dry matter determination. Also, ISO 1735 | IDF 5 is preferable to ISO 8262-3 | IDF 124-3. ISO 1735 |IDF 5 is fully applicable to cottage cheese unless the cheese contains non-dairy ingredients like sugar, jam, muesli.... For these "added" products ISO 8262-3 | IDF 124-3 is more appropriate.

| | | | | |
|--|----------------------|-------------------|------------------------------|---|
| Cheese, Unripened Including Fresh Cheese | Milk Protein | ISO8968-1 IDF20-1 | Titrimetry, Kjeldahl | I |
| Cream and Prepared Creams | Milk protein | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | I |
| Cream | Milkfat | ISO2450 IDF16 | Gravimetry (Röse-Gottlieb) | I |
| Cream | Solids ¹⁵ | ISO6731 IDF21 | Gravimetry (drying at 102°C) | I |

Milk and Milk Products

Comment: note 15 needed.

| | | | | |
|---|---|----------------------------------|---------------------------|---|
| Creams Lowered in Milkfat Content | Milkfat | ISO2450 IDF16/AOAC995.19 | Gravimetry(Röse-Gottlieb) | I |
| Creams, Whipped Creams and Fermented Creams | Milksolids-not-fat (MSNF) ¹⁵ | ISO3727-2 IDF80-2 AOAC920.116 | Gravimetry | I |

Comment: There appears to be no requirement for MSNF in CXS 288 for creams and prepared creams, therefore CCMAS to confirm the need for this provision in CXS 234.

**AOAC 920.116 is not equivalent to the ISO|IDF method, therefore the method should be listed in separate lines
Neither the ISO|IDF method nor the AOAC method have been validated for this commodity.**

| | | | | |
|------------------------|----------------------------|-----------------|---|-----|
| Cream cheese | Dry matter | ISO5534 IDF4 | Gravimetry drying at102°C (forced air oven) | I |
| Cream cheese | Moisture on fat free basis | ISO5534 IDF4 | Calculation from fat content and moisture content | I |
| | | ISO1735 IDF5 | Gravimetry drying at102°C(forced air oven) | |
| | | ISO17678 IDF202 | Gravimetry (Schmid-Bondzynski-Ratzlaff) | |
| Dairy fat spreads | Milkfat purity | ISO17678 IDF202 | Calculation from determination of triglycerides by gas chromatography | I |
| Dairy fat spreads | Total fat | ISO17189 IDF194 | Gravimetry | I |
| | | | Direct determination of fat using solvent extraction | |
| Dairy fat spreads | Vegetable fat (sterols) | ISO12078 IDF159 | Gas chromatography | II |
| Dairy fat spreads | Vegetable fat (sterols) | ISO18252 IDF200 | Gas chromatography | III |
| Edible casein products | Acids, free | ISO5547 IDF91 | Titrimetry (aqueous extract) | IV |

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|------------------------|--|--|--------------------------------------|-----|
| Edible casein products | Ash(includingP ₂ O ₅) | ISO5545 IDF90 or ¹⁵ ISO5544 IDF89 | Gravimetry (ashingat825°C) | I |
| Edible casein products | Copper | AOAC985.35 | Atomic absorption spectrophotometry | II |
| Edible casein products | Copper | ISO5738 IDF76 | Colorimetry(diethyldiethiocarbamate) | III |

¹⁵ Refer to scope of the methods

Milk and Milk Products

| | | | | |
|------------------------|---------|----------------|---|----|
| Edible casein products | Lactose | ISO5548 IDF106 | Photometry (phenol and H ₂ SO ₄) | IV |
|------------------------|---------|----------------|---|----|

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|------------------------|------|---|-------------------------------------|----|
| Edible casein products | Lead | NMKL139 (Codex general method) AOAC999.11 | Atomic absorption spectrophotometry | II |
|------------------------|------|---|-------------------------------------|----|

Comment: There is no provision for lead in CXS 290 for edible casein products. AOAC 999.11 as written has only been validated in milk powder and does not contain precision data or specify applicability to edible casein products.

| | | | | |
|------------------------|------|--------------------|-------------------------------------|-----|
| Edible casein products | Lead | NMKL161/AOAC999.10 | Atomic absorption spectrophotometry | III |
|------------------------|------|--------------------|-------------------------------------|-----|

Comment: There is no provision for lead in CXS 290 for edible casein products. AOAC 999.10 as written does not contain precision data or specify applicability to edible casein products.

| | | | | |
|------------------------|------|----------------------------------|-------------------------------------|-----|
| Edible casein products | Lead | AOAC972.25(Codex general method) | Atomic absorption spectrophotometry | III |
|------------------------|------|----------------------------------|-------------------------------------|-----|

Comment: There is no provision for lead in CXS 290 for edible casein products. AOAC 972.25 as written does not contain precision data or specify applicability to edible casein products.

| | | | | |
|------------------------|------|----------------------------------|------------------------------|-----|
| Edible casein products | Lead | AOAC982.23(Codex general method) | Anodic stripping voltammetry | III |
|------------------------|------|----------------------------------|------------------------------|-----|

Comment: There is no provision for lead in CXS 290 for edible casein products. AOAC 982.23 as written does not contain precision data or specify applicability to edible casein products.

| | | | | |
|------------------------|------|----------------------|--|----|
| Edible casein products | Lead | ISO/TS6733 IDF/RM133 | Spectrophotometry(1,5-diphenylthiocarbazone) | IV |
|------------------------|------|----------------------|--|----|

Comment: There is no provision for lead in CXS 290 for edible casein products.

| | | | | |
|------------------------|---------|----------------|---|---|
| Edible casein products | Milkfat | ISO5543 IDF127 | Gravimetry (Schmid-Bondzynski-Ratslaff) | I |
|------------------------|---------|----------------|---|---|

| | | | | |
|------------------------|----|----------------|--------------|----|
| Edible casein products | pH | ISO5546 IDF115 | Electrometry | IV |
|------------------------|----|----------------|--------------|----|

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|------------------------|---|-------------------|----------------------|---|
| Edible casein products | Milk Protein (total Nx6.38 in dry matter) | ISO8968-1 IDF20-1 | Titrimetry, Kjeldahl | I |
|------------------------|---|-------------------|----------------------|---|

| | | | | |
|------------------------|-------------------------------|----------------|---|----|
| Edible casein products | Sediment (scorched particles) | ISO5739 IDF107 | Visual comparison with standard disks, after filtration | IV |
|------------------------|-------------------------------|----------------|---|----|

Milk and Milk Products

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|------------------------|-------------------------|----------------|------------------------------|----|
| Edible casein products | Water ¹⁶ | ISO5550 IDF78 | Gravimetry (drying at 102°C) | I |
| Emmental | Calcium >=800mg/100g | ISO8070 IDF119 | Flame atomic absorption | IV |

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|------------------|--|-------------------|------------------------------|----|
| Evaporated milks | Milkfat | ISO1737 IDF13 | Gravimetry (Röse-Gottlieb) | I |
| Evaporated milks | Milk Protein in MSNF ¹⁵ | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | I |
| Evaporated milks | Solids, total ¹⁵ | ISO6731 IDF21 | Gravimetry (drying at 102°C) | I |
| Fermented milks | Colony-forming units of yeasts and/or moulds | ISO6611 IDF94 | Colony-count at 25°C | IV |
| Fermented milks | Dry matter (total solids) ¹⁵ | ISO13580 IDF151 | Gravimetry (drying at 102°C) | I |

Comment: note 15 needed.

| | | | | |
|--|--|---------------------------|---|----|
| Fermented milks | Total acidity expressed as percentage of lactic acid | ISO/TS11869 IDF/RM150 | Potentiometry, titration to pH 8.30 | I |
| Fermented milks | <i>Lactobacillus acidophilus</i> | ISO20128 IDF192 | Colony count at 37°C | I |
| Fermented milks - Yoghurt and yoghurt products | <i>Lactobacillus delbrueckii</i> subsp <i>bulgaricus</i> & <i>Streptococcus thermophilus</i> | ISO7889 IDF117 | Colony count at 37°C | I |
| Fermented milks - Yoghurt and yoghurt products | <i>Lactobacillus delbrueckii</i> subsp <i>bulgaricus</i> & <i>Streptococcus thermophilus</i> | ISO9232 IDF146 | Test for strain identification | I |
| Fermented milks | Microorganisms constituting the starter culture | ISO27205 IDF149 (Annex A) | Colony count at 25°C, 30°C, 37°C and 45°C according to the starter organism in question | IV |
| Fermented milks | Milkfat | ISO1211 IDF1/AOAC989.05 | Gravimetry (Röse-Gottlieb) | I |

Milk and Milk Products

Comment: The IDF/ISO and AOAC methods are different and neither have been specifically validated for fermented milks.

| | | | | |
|--------------------------------|---------------------|-------------------|---|----|
| Fermented milks | Milk Protein | ISO8968-1 IDF20-1 | Titrimetry(Kjeldahl) | I |
| Milk powders and cream powders | Acidity, titratable | ISO6091 IDF86 | Titrimetry,titrationtopH8.4 | I |
| Milk powders and cream powders | Milkfat | ISO1736 IDF9 | Gravimetry (Röse-Gottlieb) | I |
| Milk powders and cream powders | Milk Protein | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | I |
| Milk powders and cream powders | Scorched particles | ISO5739 IDF107 | Visual comparison with standard disks, after filtration | IV |

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|--------------------------------|---------------------|-----------------------------|-----------------------------|---|
| | Solubility Index | ISO8156 IDF129 | Centrifugation | I |
| Milk powders and cream powders | Water ¹⁶ | ISO5537 IDF26 18 | Gravimetry (drying at 87°C) | I |

Comment from DG WP: The ISO method mentions moisture

Response from IDF: The notes explains that water and moisture are equivalent: ("Water content excluding the crystallized water bound to lactose (generally known as "moisture content")

| | | | | |
|------------------|--------|-----------------------------|------------------------------------|----|
| Milkfat products | Copper | ISO5738 IDF76 AOAC960.40 | Photometry, diethyldithiocarbamate | II |
|------------------|--------|-----------------------------|------------------------------------|----|

**Comment: AOAC 960.40 as written does not contain precision data or specify applicability to milk fat products.
The IDF/ISO method and AOAC method are different and should be written in different lines.**

| | | | | |
|---|--|-----------------|---|---|
| Milkfat products | Fatty acids, free (expressed as oleic acid) | ISO1740 IDF6 | Titrimetry | I |
| Milkfat products | Milkfat purity | ISO17678 IDF202 | Calculation from determination of triglycerides by gas chromatography | I |
| Milkfat products (anhydrous milkfat) | Peroxide value (expressed as meq. of oxygen/kg fat) | ISO3976 IDF74 | Photometry | I |

Comment: Clarification to match provision in CXS 280 for Milkfat Products.

| | | | | |
|--------------------------------------|----------------|------------|------------|---|
| Milkfat products (anhydrous milkfat) | Peroxide value | AOAC965.33 | Titrimetry | I |
|--------------------------------------|----------------|------------|------------|---|

Comment: AOAC 965.33 as written does not contain precision data.

Milk and Milk Products

| | | | | |
|------------------|------------------------|-----------------|--------------------|-----|
| Milkfat products | Vegetable fat(sterols) | ISO12078 IDF159 | Gas chromatography | II |
| | | ISO18252 IDF200 | Gas chromatography | III |

Comment: Shall the two lines above be fully separated as the two methods have different types?

| | | | | |
|---|---------------------------|--------------------------|--------------------------|--------------|
| Milk fat products | Water | ISO5536 IDF23 | Titrimetry (KarlFischer) | II |
| Milk fat products (anhydrous milk fat) | Peroxide value | ISO3976 IDF74 | Photometry | I |
| Milk fat products (anhydrous milkfat) | Peroxide value | AOAC965.33 | Titrimetry | I |

Comment: Duplicates of lines above.

| | | | | |
|---------------------------|---|-------------------|-------------------------------------|---|
| Mozzarella | Milk fat in dry matter with high moisture | ISO1735 IDF5 | Gravimetry after solvent extraction | I |
| Mozzarella | Milk fat in dry matter–with low moisture | ISO1735 IDF5 | Gravimetry after solvent extraction | I |
| Sweetened Condensed Milks | Milkfat | ISO1737 IDF13 | Gravimetry (Röse-Gottlieb) | I |
| Sweetened Condensed Milks | MilkProteininMNSF ¹⁵ | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | I |
| Sweetened Condensed Milks | Solids ¹⁵ | ISO6734 IDF15 | Gravimetry, dryingat102°C | I |

Comment: note 15 needed

| | | | | |
|-------------------------------|------------------------|-------------------------------|--|----|
| Whey cheeses by coagulation | Milkfat | ISO1735 IDF5 | Gravimetry (Schmid-Bondzynski-Ratzlaff) | I |
| Whey cheeses by coagulation | Milk fat in dry matter | ISO1735 IDF5 andISO5534 IDF4 | Calculation from fat content and dry matter content Gravimetry (Schmid-Bondzynski-RatzlaffGravimetry, dryingat102°C | I |
| Whey cheeses by concentration | Milkfat | ISO1854 IDF59 | Gravimetry (Röse-Gottlieb) | I |
| Whey cheeses by concentration | Milk fa in dry matter | ISO1854 IDF59andISO2920 IDF58 | Calculation from fat content and dry matter content Gravimetry (Röse-Gottlieb) Gravimetry, drying at 88°C | I |
| Whey powders | Ash | ISO5545 IDF90 | Gravimetry (ashingat825°C) | IV |

Milk and Milk Products

Comments from DG WP: This provision has no type II method

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010

| | | | | |
|--------------|--------|------------|-------------------------------------|----|
| Whey powders | Copper | AOAC985.35 | Atomic absorption spectrophotometry | II |
|--------------|--------|------------|-------------------------------------|----|

Comment from DG WP: Methods aplicated for Baby Foods/Infant Formula, Pet Foods, Baby Foods/Enteral Products. The Codex Stan 289 does not mention this provision.

| | | | | |
|--------------|--------|---------------|------------------------------------|-----|
| Whey powders | Copper | ISO5738 IDF76 | Photometry(diethyldithiocarbamate) | III |
|--------------|--------|---------------|------------------------------------|-----|

| | | | | |
|--------------|---------|-----------------------|---|----|
| Whey powders | Lactose | ISO5765-1/2 IDF79-1/2 | Enzymatic method:Part1-GlucosemoietyorPart2-Galactosemoiety | II |
|--------------|---------|-----------------------|---|----|

| | | | | |
|--------------|------|-----------------------------------|-------------------------------------|----|
| Whey powders | Lead | AOAC972.25 (Codex general method) | Atomic absorption spectrophotometry | II |
|--------------|------|-----------------------------------|-------------------------------------|----|

Comment: AOAC 972.25 as written does not contain precision data or specify applicability to whey powders.

| | | | | |
|--------------|---------|---------------|----------------------------|---|
| Whey powders | Milkfat | ISO1736 IDF 9 | Gravimetry (Röse-Gottlieb) | I |
|--------------|---------|---------------|----------------------------|---|

| | | | | |
|--------------|---------------------------|-------------------|-----------------------|---|
| Whey powders | Milk protein(totalNx6.38) | ISO8968-1 IDF20-1 | Titrimetry (Kjeldahl) | I |
|--------------|---------------------------|-------------------|-----------------------|---|

| | | | | |
|--------------|------------------|---------------|----------------------------------|----|
| Whey powders | Moisture, "Free" | ISO2920 IDF58 | Gravimetry (drying at 88°C ±2°C) | IV |
|--------------|------------------|---------------|----------------------------------|----|

Comment from DG WP: This provision has no type II method. The Stan 289 doesn't mention "Moisture free", just "Water". The ISO method mentions dry matter in whey cheese.

Response from IDF: The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010. The current version of stan 234 correctly lists water with a note explaining the equivalence between moisture and water.

| | | | | |
|--------------|---------------------|---------------|-----------------------------|---|
| Whey powders | Water ¹⁶ | ISO5537 IDF26 | Gravimetry (drying at 87°C) | I |
|--------------|---------------------|---------------|-----------------------------|---|

7. ADDRESS STRUCTURE OF CODEX STAN 234 AS PRESENTED IN CX/MAS 18/39/4 (if time permits)

Note: This discussion will not replace the discussion in plenary, but if the PWG reaches consensus on any aspects of Draft Version of CODEX STAN 234, those conclusions will be presented during the plenary discussion. We will move through the document in a paragraph-by-paragraph nature. We will use CA/MAS 18/39/4 as a guide for this discussion.

Appendix 1

Annex 1

Annex 2