

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda Item 6

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

Thirty-eighth Session
Budapest, Hungary, 8 – 12 May 2017

REVIEW AND UPDATE OF METHODS IN CODEX STAN 234-1999

(Comment submitted by the International Dairy Federation (IDF))

IDF wishes to thank the delegation of Brazil and Uruguay for putting together this extensive discussion paper. This paper addresses a large range of diverse and complex issues to be addressed by CCMAS. Each part presented in the paper requires attention from all stakeholders to ensure accuracy.

It may be an option to divide the work in several workings groups.

Proposed changes are indicated in bold/underline and bold/strikethrough text. Additional comments are provided in bold underline text where relevant.

Annex 1: LIST OF COMMODITIES CATEGORIES AND NAMES

IDF is not sure to understand how this list would be used within the endorsement process (endorsement by categories versus validation on specific matrices for example) as explained in the document: "The list of commodities is hierarchical, meaning that when a method of analyses is recognized for use in a general category, it is recognized for use in all its sub-categories, unless otherwise stated".

This list was not part of the document circulated within the electronic working group so further clarification would be welcome.

This list could also be used for information purpose at first without the quoted line above.

If maintained, some commodities are repeated in different categories (for example butter under 7.1.2.1 and 8.5.1). For the dairy categories, IDF would suggest the following changes, understanding it would likely require further discussion:

- 8. Milk and Milk Products
 - 8.1. Fluid milk, Cream and Milk Powders
 - 8.1.1. ~~Fermented~~ **Fluid** Milk
 - 8.1.2. Whole Milk Powder
 - 8.1.3. Partly Skimmed Powder
 - 8.1.4. Skimmed Milk Powder
 - 8.1.5. Cream Powder
 - 8.1.6. Half Cream Powder
 - 8.1.7. High Fat Cream Powder
 - 8.2. Cream and Prepared Creams
 - 8.2.1. Cream
 - 8.2.2. Reconstituted cream
 - 8.2.3. Recombined cream
 - 8.2.4. Prepared creams
 - 8.2.5. Prepackaged liquid cream
 - 8.2.6. Whipping cream
 - 8.2.7. Cream packed under pressure

8.2.8. Whipped cream

8.2.9. Fermented cream

8.2.10. Acidified cream

8.3. Cheese

8.3.1. Unripened Cheese

8.3.1.1. Mozzarella

8.3.1.2. Cottage

8.3.1.3. Cream Cheese

~~8.3.1.4. Extra Hard Grating Cheese~~

8.3.2. Ripened Cheese

8.3.2.1. Cheddar

8.3.2.2. Danbo

8.3.2.3. Edam

8.3.2.4. Gouda

8.3.2.5. Havarti

8.3.2.6. Samsø

8.3.2.7. Emmentaler

8.3.2.8. Tilsiter

8.3.2.9. Saint Paulin

8.3.2.10. Provolone

8.3.2.11. Coulommiers

8.3.2.12. Camembert

8.3.2.13. Brie

8.3.2.14 Extra Hard Grating Cheese

8.3.3. Whey Cheese

8.4. Milk Fractions and Dairy Products

~~8.4.1.1. Edible acid casein~~

~~8.4.1.2. Edible rennet casein~~

~~8.4.1.3. Edible caseinate~~

~~8.4.1.4. Whey~~

~~8.4.1.5. Acid whey~~

~~8.4.2. Evaporated Skimmed Milk and Vegetable Fat~~

~~8.4.3. Skimmed Milk and Vegetable Fat in Powdered Form~~

~~8.4.4. Skimmed Milk and Vegetable Fat~~

~~8.4.5. Dairy Fat spread~~

8.5. Milkfat Products

8.5.1. Butter

8.5.2. Anhydrous Milkfat

8.5.3. Milkfat

8.5.4. Anhydrous butteroil

8.5.5. Butteroil

8.5.6. Ghee

8.5.6 Dairy Fat spread

8.6 Fermented milks

8.7 Blends of milk and other ingredients

8.7.1. Evaporated Skimmed Milk and Vegetable Fat

8.7.2. Skimmed Milk and Vegetable Fat in Powdered Form

8.7.3. Skimmed Milk and Vegetable Fat

Annex 2: LIST OF PROVISIONS

IDF comments: While a lot of IDF suggestions have been taken into consideration by the eWG, we would like to provide additional changes.

As a general comment, we think it is important to stick to the provision mentioned in the commodity standard and any of its specificity as it is most likely to affect the method chosen.

Attention is needed on how some provisions are written for instance: Fat-free dry matter is suggested to be replaced with 'Fat, Fat-free dry matter', but 'fat' and 'Fat-free dry matter' are different provisions. This way of writing is confusing.

Provision in CODEX STAN 234	Provision suggested	IDF comments
Acid Insoluble Ash	Ash, insoluble	<u>These provisions are not equivalent</u>
Acid Soluble Ash	Ash, soluble	<u>These provisions are not equivalent</u>
Acids, free	Acidity, free	<u>Free acids and free acidity are not synonymous.</u>
Amino acid nitrogen	Nitrogen, ammoniacal <u>amino acid</u>	<u>Replace with Nitrogen, amino acid</u> <u>Ammoniacal nitrogen is something else. Ammoniacal nitrogen (NH3-N), is a measure for the amount of ammonia, a toxic pollutant often found in landfill leachate and in waste products.</u>
Ash (including P2O5)	Ash	<u>Some products need to be analyzed by the fixed ash methods, other products not. This needs to be carefully checked to ensure there is no ambiguity.</u>
Dry extract – soluble solids	Dry matter <u>Soluble solid</u>	<u>Dry matter is not equivalent to soluble solids. Not all solids are necessarily soluble.</u>
Fat acidity	<u>Acidity</u> , Fat acidity	<u>Acidity pertains to the whole matrix. Fat acidity depicts the acidity of the fat phase.</u>
Fat-free cocoa solids	Fat , Fat-free cocoa solids	
Fat-free dry matter	Fat , Fat-free dry matter	
Fatty acids, free (expressed as oleic acid)	Fatty acid, free (<u>expressed as oleic acid</u>)	
Free fatty acids	Fatty acids s , free fatty acids s	
Halogenated solvents, traces	Halogenated solventes	
Invert sugar	Sugar, Invert ed sugar	
Iron and copper	Iron	
	Co o pper	
Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus	Lactobacillus delbrueckii subsp bulgaricus	
	Streptococcus thermophi l eus	
Milk fat in dry matter	Fat, Milk fat <u>in dry matter</u>	<u>Also requires method for total solids</u>

Milk fat in dry matter (FDM)	Fat, Milk fat <u>in dry matter</u>	<u>Also requires method for total solids</u>
Milk fat in dry matter with high moisture	Fat, Milk fat <u>in dry matter</u>	<u>Also requires method for total solids</u>
Milk fat in dry matter with low moisture	Fat, Milk fat <u>in dry matter</u>	<u>Also requires method for total solids</u>
Milk fat purity	Fat, triglycerides <u>Milk fat purity</u>	
Milk protein	Protein, <u>milk protein</u>	<u>Milk protein should remain as a different provision than protein. Justification: in the standards that contain the provisions milk protein, it is a requirement that the protein is only milk protein.</u>
Milk protein (total N x 6.38 in dry matter)	Protein <u>(total N x 6.38) (also requires method for total solids)</u>	
Milk protein (total N x 6.38)	Protein <u>(total N x 6.38)</u>	
Milk protein in MSNF	Protein, <u>protein in MSNF (also requires methods for total solids and fat)</u>	
Milk solids-not-fat (MSNF)	Dry matter, Fat-free	<u>Also requires method for fat</u>
Milkfat	Fat, <u>milk fat</u>	<u>For consistency with lines above</u>
Mineral impurities	<u>Ash, insoluble Mineral impurities</u>	<u>These provisions are not</u>
Mineral Impurities (Sand)	<u>Ash, insoluble Mineral impurities</u>	<u>These provisions are not</u>
Moisture on fat free basis	<u>Dry-matter, fat-free moisture Moisture on fat free basis</u>	<u>Fat free moisture is not equivalent to moisture on fat free basis. Fat free moisture is a plain content in g/100 g product, Moisture on a fat free basis is a content in g/100 g fat free product.</u>
Moisture, "Free"	<u>Dry-matter, fat-free moisture Moisture</u>	<u>Dry matter and moisture are not synonymous, they are in fact complementary. Free moisture is not equivalent to fat free</u>
MSNF	Dry-matter, fat-free <u>moisture</u>	
Peroxide value	Peroxide <u>value</u>	
Peroxide value (expressed as meq. of	Peroxide <u>value</u>	
Peroxide value (PV)	Peroxide <u>value</u>	
Protein (N x 5.7)	Protein <u>(N x 5.7)</u>	
Protein (Nx6.21)	Protein <u>(Nx6.21)</u>	
Protein (Nx6.25)	Protein <u>(Nx6.25)</u>	
Protein Efficiency Ratio (PER)	Protein <u>Efficiency Ratio (PER)</u>	
Salt	Chloride expressed as NaCl	<u>Check with commodity standard</u>
Salt content	Chloride expressed as NaCl	<u>Check with commodity standard</u>
Salt in brine	Chloride expressed as NaCl	<u>Check with commodity standard</u>
Solids (soluble)	Solids, soluble solids <u>Dry matter, soluble</u>	
Solids, alcohol insoluble	Solids, insoluble solids in alcohol <u>dry matter, alcohol insoluble</u>	

Soluble solids	Solids, soluble solids Dry matter, <u>soluble</u>	
Soluble solids, total	Solids, soluble solids Dry matter, <u>soluble</u>	
Stable carbon isotope ratio in the pulp of fruit	<u>Stable</u> carbon stable isotope ratio	
Stable carbon isotope ratio of sugars from fruit	<u>Stable</u> carbon stable isotope ratio	
Stable hydrogen isotope ratio of water from fruit juices (Sections)	<u>Stable</u> hydrogen stable isotope ratio	
Stable oxygen isotope ratio in fruit juice water	<u>Stable</u> oxygen stable isotope ratio	
Sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}\text{O}$	<u>Stable</u> carbon stable isotope ratio	
Total acidity (as lactic acid)	Acidity, <u>total expressed as percentage of lactic acid</u>	
Total fat for milk-based infant formula (products not completely soluble in ammonia)	Fat <u>Total fat for milk-based infant formula (products not completely soluble in ammonia)</u>	<u>This clarification impacts the recommended method</u>
Total nitrogen	Nitrogen, total	
Total phospholipids	Phospholipids, total	
Water	Water activity –	<u>Check in commodity standard whether this is moisture, water content or water activity. Karl Fischer method measure the chemical water, which is different from drying methods which measure volatiles at a certain time-temperature combination, expressed as moisture.</u>

Annex 3: LIST OF THE PRINCIPLES OF METHODS

Principle in CODEX STAN 234	Principle suggested	IDF comments
Analysis of triglycerides of HPLC and Calculation	High Performance Liquid Gravimetry <u>Calculation</u>	<u>Spelling to be checked</u> <u>This suggest it is an actual analysis, whereas it is a computation (although from two other analyses based on</u>
Chemical & HPAEC-PAD	High Performance <u>Liquid Anion Exchange</u> Chromatography	
Enzymatic & HPAEC-PAD	High Performance <u>Liquid Anion Exchange</u> Chromatography	
Gravimetry (Schmid-Bondzynski-Ratzlaff) (for samples containing lactose up to 5%) Gravimetry (Weibull-Berntrop) (for samples containing lactose over 5%)	Gravimetry <u>(for samples containing lactose up to 5%) Gravimetry (Weibull-Berntrop) (for samples containing lactose over 5%)</u>	<u>This is important to be included otherwise the analyst may select the wrong method.</u>
Kjeldahl digestion	Titrimetry <u>Sample preparation</u>	
Stretching	Rheological measurement	
Titrimetry (Mohr: determination of chloride, expressed as sodium	Titrimetry <u>Potentiometry</u>	
Titrimetry , Kjeldahl digestion	Titrimetry <u>Sample Preparation</u>	
Titrimetry C h	Titrimetry <u>Potentiometry</u>	
Titrimetry, Kjeldahl digestion	Titrimetry <u>Sample Preparation</u>	
Titrimetry, Kjeldahl digestion; after extraction of milk protein	Titrimetry Sample Preparation	

IDF agrees with the following statements:

- Some participants proposed not to use the term “Codex general methods” considering, for example:
 - Once methods are presented in the (new) format of this Standard, users of the Standard would have sufficient information for the selection of a method and to which commodity the method can be applied so that the information whether a method is “Codex general method” or not would no longer be necessary.
- For “all food methods”:

Most of the eWG participants proposed that all methods need to be validated in each specific matrix, or group of matrices if a robustness test were done to demonstrate its applicability. As with all analytical methods, they need to be validated to ensure that the method performance is fit for purpose for any new matrices for a given method.

Appendix III - METHODS OF ANALYSES WORKABLE PACKAGES**General comments****WORKABLE PACKAGE 2- Multiple Type I Methods**

Several cases are as described on page 4 of document. There are several methods listed because they are complementary or identical.

WORKABLE PACKAGE 5- Provisions without a Type II method

For all methods in this package, it is our understanding that it is appropriate to list a type IV when no other type I or II methods are available, as decided by CCMAS when this method was recommended. Therefore IDF recommends that no change is needed.

WORKABLE PACKAGE 2- Multiple Type II Methods

ACTIONS SUGGESTED: Identify which one is the Type I method and delete the others. Amend the Codex standards accordingly.

IDF comments: See our comments in the table below.

Commodities	CODEX STAN	Provision	Method/ version	Principle	Type	Committee	Remarks
Blend of evaporated skimmed milk and vegetable fat	CODEX STAN 250	Milk solids-not-fat (MSNF) ("Milk total solids and Milk solids- not-fat (MSNF) content include water of crystallization of lactose")	ISO 6731 IDF 21 and ISO 1737 IDF 13	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I	CCMMP	Multiple type I method IDF: No change necessary. Both methods are complementary (as indicated on page 4 of CX/MAS 17/38/6).
Edible casein products	CODEX STAN 290	Ash (including P2O5)	ISO 5545 IDF 90 or ISO 5544 IDF 89	Gravimetry (ashing at 825 °C)	I	CCMMP	Multiple type I method IDF: No change necessary. The scopes of both methods are complementary.
Fermented milks	CODEX STAN 243	Milk fat	ISO 1211 IDF 1 AOAC 989.05	Gravimetry (Röse-Gottlieb)	I	CCMMP	Multiple Type I method. IDF: Both methods are identical.
Infant formula	CODEX STAN 72	Total fat	AOAC 989.05 ISO 8381 IDF 123	Gravimetry (Röse-Gottlieb)	I	CCNFSDU	Multiple type I method. IDF: Method AOAC 989.05 is for Milk. Method ISO 8381 IDF 123 is for milk-based infant foods with less than 5% dry matter from another origin.
Reduced fat blend of evaporated skimmed milk and vegetable fat	CODEX STAN 250	Milk solids-not-fat (MSNF)	ISO 6731 IDF 21 and ISO 1737 IDF 13	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I	CCMMP	Multiple type I method IDF: No change necessary. Both methods are complementary (as indicated on page 4 of CX/MAS 17/38/6).

WORKABLE PACKAGE 4- Methods with Incorrections**ACTIONS SUGGESTED: Check the methods information, identify other methods when necessary and amend the Codex documents accordingly.**

Commodities	CODEX STAN	Provision	Method/ version	Principle	Type	Committee	Remarks
Butter	CODEX STAN 279	Water ("Water content excluding the crystallized water bound to lactose (generally known as "moisture content")	ISO 3727 IDF 80	Gravimetry	I	CCMMP	The correct method is ISO 3727-1 IDF 80-1 The ISO method mentions moisture, non fat solids and fat content in butter <u>IDF: This is correct. The provision water is equivalent to moisture in this standard. No change needed.</u>
Milk powders and cream powders	CODEX STAN 207	Water ("Water content excluding the crystallized water bound to lactose (generally known as "moisture content")	ISO 5537 IDF 26	Gravimetry (drying at 87°C)	I	CCMMP	The ISO method mentions moisture. <u>IDF: This is correct. The provision water is equivalent to moisture in this standard. No change needed.</u>
Whey powders	CODEX STAN 289	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II	CCMMP	Methods applicable for Baby Foods/Infant Formula, Pet Foods, Baby Foods/Enteral Products The CODEX STAN 289 does not mention this provision
Whey powders	CODEX STAN 289	Moisture, "Free"	ISO 2920 IDF 58	Gravimetry (drying at 88 °C ±2°C)	IV	CCMMP	CODEX STAN 289 doesn't mention "Moisture free", just "Water". The ISO method mentions dry matter in whey cheese. This provision has no type II method <u>IDF: The provision water is equivalent to moisture in this standard. The moisture is obtained by calculation from dry matter. No change needed.</u>