CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 2 **NFSDU/41 CRD 29**

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Forty-first Session

Dusseldorf, Germany
24 – 29 November 2019

MATTERS REFERRED BY THE CODEX ALIMENTARIUS COMMISSION AND/OR OTHER SUBSIDIARY BODIES

Comments of ISDI

B. Matters Arising from Subsidiary Bodies as Related to the Work of CCNFSDU

Matters for Action

The 40th Session of the Codex Committee on Methods of Analysis and Sampling (CCMAS40)

Endorsement of methods of analysis and sampling plans for provisions in Codex standards

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

Endorsement of AOAC 2011.14 / ISO 15151 | IDF 229

ISDI supports CCNSFDU endorsement of AOAC 2011.14 / ISO 15151 | IDF 229 as a Type III method for calcium, copper, iron, magnesium, manganese, phosphorous, potassium, sodium and zinc. This method is another recently validated, collaboratively studied and published method for mineral analysis.[1,2,3] The method uses microwave digestion followed by determination of elemental concentrations by ICP-AES detection, and is a "sister" technique to AOAC 2015.06 / ISO 21424 | IDF 243, which was endorsed as Type II by the Codex Committee on Methods of Analysis and Sampling in May 2019 and adopted as Type II by the Codex Alimentarius Commission in July 2019. Table 1 below outlines how this method would appear in CXS 234-1999.

Possible Establishment of Method Performance Criteria

ISDI does not support establishing numerical method performance criteria for calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium and zinc. The Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN), which is administered by AOAC INTERNATIONAL (AOAC) was formed to develop updated/modernized analytical methods for the determination of priority nutrients in infant formula. The International Dairy Federation (IDF) and the International Standardization Organization (ISO) have cooperated and contributed to the work of SPIFAN. A primary goal of SPIFAN is for Codex to adopt one Type II method for each priority nutrient for the purpose of dispute resolution.

During the 37th and 38th Sessions of the Codex Committee on Methods of Analysis and Sampling (CCMAS), the Committee considered a method for the determination of chromium, selenium and molybdenum (AOAC 2011.19 / ISO 20649 | IDF 235). CCNFSDU agreed during its 37th Session to refer this method to CCMAS for technical review and typing. In considering this method, CCMAS37 noted the method provision in CXS 72-1981 indicated that none of the existing methods for these analytes, including current Codex methods (at the time) and AOAC 2011.19 / ISO 20649 | IDF 235, would meet the criteria, specifically the minimum limit (ML), and questioned whether a criteria approach may be preferred. CCMAS therefore asked CCNFSDU for further guidance related to the criteria to help determine whether the method should remain Type III or become Type II. More specifically, CCMAS asked CCNFSDU to review the numeric values for the method criteria and advise CCMAS on the correct values and how to proceed.

In response to this feedback, the SPIFAN community gathered additional validation data on the quantitation limit of the method as well as reproducibility data on placebos, which was then published in *JAOAC.* These

[1] In publication
[2] ISO 15151 | IDF 229: 2018 https://www.iso.org/standard/70900.html
[3] ISO 15151 | IDF 229: 2018 https://store.fil-idf.org/product/iso-15151-i-idf-229/

additional data demonstrated that the method, without modifications, clearly gives results at or above the quantitation limit and demonstrated acceptable reproducibility at the minimum levels set through CXS 72-1981. In addition to providing these data to CCNFSDU for review during its 38th Session, the SPIFAN community confirmed the need for Type II methods for the purpose of dispute resolution. Below, for reference is background information on method types, as taken from the Codex Procedural Manual.

(a) Defining Methods (Type I)

Definition: A method which determines a value that can only be arrived at in terms of the method per se and serves by definition as the only method for establishing the accepted value of the item measured.

(b) Reference Methods (Type II)

Definition: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). ***It should be recommended for use in cases of dispute*** and or for calibration purposes.

(c) Alternative Approved Methods (Type III)

Definition: A Type III Method is one which meets the criteria required by the Committee on Method of Analysis and Sampling for methods that may be used for control, inspection or regulatory purposes.

(d) Tentative Method (Type IV)

Definition: A Type IV Method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Committee on Methods of Analysis and Sampling have not yet been determined.

The SPIFAN community recommended that CCNFSDU confirm to CCMAS that a dispute resolution method is needed for these analytes and, as AOAC 2011.19 / ISO 20649 | IDF 235 is the only method that meets the method criteria, it should be endorsed as a Type II method. It was also noted this recommendation is in line with that made by CCNFSDU37.

After considering this information, CCNFSDU38 agreed to:

a) Inform CCMAS that it did not support a criteria approach because none of the current methods in CODEX STAN 234-1999, nor the newer methods AOAC 2011.19 / ISO 20649 / IDF 235 meet the criteria (REP16/MAS, para. 31).

b) Request that CCMAS reconsider the method for chromium, selenium and molybdenum, AOAC 2011.19 / ISO 20649 | IDF 235 as Type II in light of published validation data measuring the minimum level for chromium, selenium and molybdenum in CODEX STAN 72-1981.

c) Inform CCMAS that the other methods for chromium, selenium and molybdenum other than the AOAC method were still fit for purpose and to reconsider their classification, if necessary.

CCMAS considered this feedback during its 38th Session and agreed to endorse 2011.19 / ISO 20649 | IDF 235 as Type II. Recognizing previous discussions by CCNFSDU and CCMAS regarding a possible criteria approach for the determination of nutrients in infant formula, and acknowledging previous agreement that Type II methods are needed for the purpose of dispute resolution, ISDI urges CCNFSDU to follow these past decisions and support Type II methods for the determination of infant formula nutrients.

ISDI recognizes the value of a criteria approach with regard to the endorsement of analytical methods by Codex. However, we believe the applicability of the criteria approach is for situations where there is no need for Type II methods.

Replacement of Type II Methods for Vitamin K in Follow-Up Formula

ISDI supports replacing the methods for Vitamin K in follow-up formula currently in CXS 234 (AOAC 999.15 / EN 14148 with AOAC 2015.09 / ISO 21446, which has now been adopted by the Codex Alimentarius Commission as Type II in infant formula. AOAC 2015.09 / ISO 21446 underwent Single Laboratory Validation and Multi-Laboratory Validation testing using 17 and 19 sample matrices, respectively. Included in these sample matrices are products representative of follow-up formula. The data from this testing have been published by AOAC[4] and by ISO[5].

Additionally, AOAC 2015.09 / ISO 21446 demonstrates improved extraction efficiency of vitamin K_1 using a less labor intensive sample preparation procedure, allows for separation of the biologically inactive cis isomer from the biologically active trans isomer, and allows for the determination of total (cis + trans) vitamin

[4] *J. AOAC Int*. 2018 Jul 25. DOI: https://doi.org/10.5740/jaoacint.18-0155
[5] ISO 21446 https://www.iso.org/standard/70938.html

K_1, if required. For this method, vitamin K_1 is extracted from products with iso-octane after proteins are precipitated and lipids are released with methanol. Prepared samples are injected onto a silica HPLC column where cis and trans vitamin K_1 are separated with an iso-octane-isopropanol mobile phase. Column eluant is mixed with a dilute ethanolic solution of zinc chloride, sodium acetate, and acetic acid before it passes through a zinc reactor column where cis and trans vitamin are chemically reduced. The resulting hydroquinones are then detected by fluorescence at an excitation wavelength of 245 nm and an emission wavelength of 440 nm.

For these reasons, ISDI supports replacing AOAC 999.15 / ISO 14148 with AOAC 2015.09 / ISO 21446 as the Type II method for determining vitamin K in follow-up formula in CXS 234-1999 and making AOAC 999.15 / ISO 14148 Type III.

TABLE 1. AOAC Official Methods validated for Infant Formula

Commodity	Provision	Method	Principle	Proposed Type
Infant formula	Calcium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
		AOAC 985.35	Flame atomic absorption spectrometry	III
	Copper	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		AOAC 985.35	Flame atomic absorption spectrophotometry	III
	Iron	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		AOAC 985.35	Flame atomic absorption spectrometry	III
		AOAC 999.11 | NMKL 139	AAS after dry ashing	III
	Magnesium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
		AOAC 985.35	Flame atomic absorption spectrometry	III
	Manganese	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		AOAC 985.35	Flame atomic absorption spectrometry	III
	Phosphorus	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF	**ICP emission spectroscopy**	**III**

		229		
		AOAC 986.24	Spectrophotometry (molybdovandate)	III
	Potassium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
	Sodium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
	Zinc	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	II
		AOAC 2011.14 / ISO 15151 | IDF 229	**ICP emission spectroscopy**	**III**
		AOAC 985.35	Flame atomic absorption spectrometry	III