CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 3 **CX/MAS 24/43/3 Add.3**

April 2024

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Forty-third Session
Budapest, Hungary
13 – 18 May 2024

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS IN CODEX STANDARDS

Comments in reply to CL 2024/08-MAS

Comments by Brazil, Colombia, Costa Rica, Egypt, Guatemala, Guyana, Paraguay, Peru, Philippines, Saudi Arabia, Uruguay and IOC, NMKL

Background

1. This document compiles comments received through the Codex Online Commenting System (OCS) in response to CL 2024/08-MAS issued in January 2024. Under the OCS, comments are compiled in the following order: general comments are listed first, followed by comments on specific sections.

Explanatory notes on the Annex

2. The comments submitted through the OCS are hereby attached as **Annex I** are presented in table format.

ANNEX I

GENERAL COMMENTS

COMMENT	MEMBER / OBSERVER
Egypt appreciates the work done with the document	**Egypt**
Paraguay no tiene comentarios al documento.	**Paraguay**
El Perú agradece al Comité del Codex sobre Métodos de Análisis y Toma de Muestras (CCMAS) y su 42.a reunión, al debatirse la ratificación de métodos de varios comités, y donde se tomaron siguientes decisiones, lo que nos da la oportunidad para presentar comentarios.	**Peru**

SPECIFIC COMMENTS

Review of methods of analysis for contaminants: performance criteria for lead and cadmium in foods	
Comments and information on i) the suitability of anlytical methods for lead in Appendix II as example methods that can meet the performance criteria for lead listed in Appendix I; and ii) other methods that can meet the performance criteria for lead and cadmium in foods list in Appendix I.	
We could verify that the following methods listed in Appendix II can meet the performance criteria listed in Appendix I: • AOAC 994.02 - Lead in edible oils and fats. Direct graphite furnace atomic absorption spectrophotometric method. • AOCS Official Method Ca 18c-91 - Lead by Graphite Furnace Atomic Absorption Spectrophotometry. • AOAC 999.11 - Determination of Lead, Cadmium, Copper, Iron, and Zinc in Foods. • EUsalt/AS 015-2015 Determination of Elements Emission Spectrometric Method (IPC-OES). We note that there is no performance data for the following methods: • NMKL 139 - Metals. Determination by atomic absorption spectrophotometry in foodstuffs. • AOAC 972.25 - Lead in Food. Therefore, we would appreciate to have access to these data to evaluate the suitability of these methods. In relation to EUsalt/AS 013 - 2005 Determination of Total Lead Flame Atomic Absorption Spectrometric Method – it is applicable to products of lead content (Pb) equal to or greater than 0.5 mg per kilogram of salt. However, it is not a suitable method because the LOQ deviation is very high. In the table below, we provide example of applicable methods that meet the criteria including those listed in Appendix II. Natural mineral waters: Example of applicable methods that meet the criteria: EPA 200.8 (Principle: ICP-MS) Edible fats and oils Example of applicable methods that meet the criteria: AOAC 994.02 (Principle: GF-AAS) AOCS Ca 18c-91 (Principle: GF-AAS)	**Brazil**

Fish Example of applicable methods that meet the criteria: AOAC 999.11 (Principle: GF-AAS) Salt, food grade Example of applicable methods that meet the criteria: EUsalt/AS 015 (Principle: ICP-OES) Natural mineral waters Example of applicable methods that meet the criteria: EPA 200.8 (Principle: ICP-MS) Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice Example of applicable methods that meet the criteria: ISO 23637:2021 (Principle: GF-AAS) Wheat (common wheat, durum wheat, spelt and emmer) Example of applicable methods that meet the criteria: ISO 23637:2021 (Principle: GF-AAS) Rice, polished Example of applicable methods that meet the criteria: ISO 23637:2021 (Principle: GF-AAS) Salt, food grade Example of applicable methods that meet the criteria: EUsalt/AS 015 (Principle: ICP-OES)	
La Carta circular propone elaborar criterios numéricos de rendimiento para el plomo y el cadmio en los alimentos, con la posterior revocación de los Métodos de análisis generales para los contaminantes (CXS 228-2001) y de los métodos de la norma CXS 234 para la detección de plomo y cadmio en los productos a los que se aplican los criterios de rendimiento Para lo cual se solicita seguir examinando los métodos (véase el Apéndice II de esta carta circular) y otros métodos a fin de encontrar ejemplos de métodos disponibles que cumplan los criterios de rendimiento. Los ejemplos de las metodologías analíticas, segun examen del citado Apendice II, se fundamentan en que el Laboratorio Nacional de Referencia de Colombia actualmente las emplea. **Ver cuadro anexo**: Se envió la consulta a los miembros del Comité Nacional del Codex, recibiendo los siguientes comentarios refereridos como ejemplos de métodos de análisis acreditados desarrollados por el Laboratorio Nacional de referencia: **Para el caso de determiancion de Aflatoxinas B1,B2, G1 Y G2:** El método empleado por el Laboratorio, es un método interno (acreditado ISO 17025) que emplea para la extracción de los analitos cartuchos de fase sólida para matrices sencillas como el maíz y el arroz, en el caso de matrices complejas como mezcla de cereales y maní se utilizan las columnas de inmunoafiniodad y para la derivatización de las aflatoxinas B1 Y G1 se emplea un fotoreactor con luz ultravioleta, el análisis se realiza por cromatografía líquida de alta eficiencia con detector de fluorescencia. El desempeño del método en cuanto a los parámetros solicitados es el siguiente:	**Columbia**

El límite de detección (LOD) y límite de cuantificación (LOQ) es de:

Aflatoxina	LOD (ng/g)	LOQ (ng/g)
B1	0,29	1,08
G1	0,35	1,08
B2	0,10	0,36
G2	0,15	0,36

La recuperación del método se encuentra en el rango esperado de 60 - 115% para concentraciones en razón de 10^{-8} (10 µg/kg) y 40 - 120% para concentraciones en razón de 10^{-9} (1 µg/kg).

Para el caso de metales pesados (Cd y Pb): Metodo Validado ICP-OES MEDIANTE DIGESTIÓN ASISTIDA POR MICROONDAS. Este procedimiento describe la técnica de análisis para determinar el contenido de trazas de Cadmio (Cd), Plomo (Pb) y demás metales en alimentos, bebidas y materiales en contacto con alimentos mediante espectrometría de emisión óptica con plasma de acoplamiento inductivo (ICP-OES).

Commodity	**Provision**	**ML (mg/kg)**	**Minimum applicable range (mg/kg)**	**Limit of Detection (LOD) (mg/kg)**	**Limit of Quantification (LOQ) (mg/kg)**	**Precision (RSDR) (%) No more than**	**Recovery (%)**	**COMENTARIOS COLOMBIA**
Polished Rice; Cereal-based food for infants and young Afla B1, B2, G1 y G2	**Afla**	**5**	**≤1**	**<44**	**≤2**	**2.8 - 7.2**	**40-120**	**Método incluido en la circular CL 2024/08/MAS- Apéndice IV- (Method criteria for total aflatoxins in cereals, considering AFB1: AFB2:AFG1:AFG2 of 1:1:1:1.)**
Corn: Afla B1, B2, G1 y G2 Rice: Afla B1, B2, G1 y G2	Afla	0.1		0,29 0,35 0,10 0,15			60 - 115% para concentraciones en razón de 10^{-8} (10 µg/kg) y 40 - 120% para concentraciones en razón de 10^{-9} (1 µg/kg).	Se emplea método fotoreactor con luz ultravioleta, análisis por cromatografía líquida de alta eficiencia con detector de fluorescencia (HPLC-FL)

Rice, polished	**cadmium**	**0.4**	**0.18 - 0.62**	**0.04**	**0.08**	**37**	**80-110%**	**Método incluido en la circular CL 2024/08/MAS- Appendix I :Performance Criteria for Lead and Cadmium in Foods**
Rice	Lead		0.04 a 0.5				80-110	Se emplea wel método ICP-OES MEDIANTE DIGESTIÓN ASISTIDA POR MICROONDAS
	Cadmiun			0.01	0.04			

CUADRO DE METODOLOGIAS POR GRUPO DE MATRICES

Commodity	*Provision*	*Method*	*Principle*
Natural mineral waters	*Lead & Cadmium*	*ISO 17294-2*	*ICP-MS*
Infant Formulae			
Infant Formulae	*Lead & Cadmium*	*FDA Method 4.7 Ver. 1.2* *AOAC 2013:06* *EN 15763:2010*	*ICP-MS*
Milk and Secondary Products			
All milk and milk products	*Lead & Cadmium*	*FDA Method 4.7 Ver. 1.2* *AOAC 2013:06* *EN 15763:2010*	*ICP-MS*
Fruits and Vegetables, and Related Products			
Fruits and Vegetables (fresh or processed and related products)	*Lead & Cadmium*	*FDA Method 4.7 Ver. 1.2* *AOAC 2013:06* *EN 15763:2010*	*ICP-MS*
Meats and Secondary Products			
All meats and meats products	*Lead & Cadmium*	*FDA Method 4.7 Ver. 1.2* *AOAC 2013:06* *EN 15763:2010*	*ICP-MS*
Miscellaneous Products			
Food grade salt	*Lead & Cadmium*	*EuSalt/AS 015*	*ICP-OES*

Performance Criteria for Lead and Cadmium in Foods

Appendix I

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%	*ISO 17294-2*	*ICP-MS*
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN 15763:2010*	*ICP-MS*
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	*FDA Method 4.7 Ver. 1.2*	*ICP-MS*
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 - 0.043	0.006	0.012	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Edible fats and oils	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%		
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN 15763:2010*	*ICP-MS*

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06*	*ICP-MS*
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*

Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN 15763:2010*	*ICP-MS*
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*

Wine (wine and fortified / liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Fish	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Fresh farmed mushrooms (common mushrooms (*Agaricus bisporous*), shiitake mushrooms (*Lentinula edodes*), and oyster mushrooms (*Pleurotus ostreatus*))	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*

Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN 15763:2010*	*ICP-MS*
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%	*EuSalt/AS 015*	*ICP-OES*
Natural mineral waters	cadmium	0.003	0.0017 - 0.0043	0.0006	0.0012	44	40-120%	*ISO 17294-2*	*ICP-MS*
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06*	*ICP-MS*

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[f]	Principle
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*

Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac –	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06*	*ICP-MS*
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	*FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN*	*ICP-MS*
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 -0.32	0.02	0.04	41	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%	*EuSalt/AS 015*	*ICP-OES*
Chocolate containing or declaring >30% to <50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%		
Chocolate containing or declaring ≥50% to <70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table chocolate	cadmium	0.8	0.40 - 1.20	0.08	0.16	33	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[1]	Principle
Chocolate containing or declaring ≥70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table	cadmium	0.9	0.46 - 1.34	0.09	0.18	33	80-110%		
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		

Costa Rica considera que el porcentaje de recuperación indicado para los métodos cuyo principio es Atomic absorption spectrophotometry (direct graphite furnace), es restrictivo para las siguientes provisiones: Para los productos: carne de bovino, porcino y oveja; disposición plomo, se recomienda un rango de 80 a 115%. Para los productos: cefalópodos y moluscos bivalvos marinos; disposición cadmio, se recomienda un rango de 80 a 115%.	**Costa Rica**
Egypt agrees with the methods listed in Appendix II with the recommendation to add the following methods for lead determination : AOAC 999.10 - AOAC 999.11 due to the fact that these methods include a larger range in the analysis and are used to estimate cadmium - zinc - iron - copper, in addition to lead estimation, also these methods are approved by Egyptian laboratories.	**Egypt**
ICP-OES: Espectrómetro de Emisión de Plasma Acoplado Inductivamente ICP Inductively Coupled Plasma Optical Emission Spectrocopy (ICP-OES)	**Guatemala**

Sugerencia: que se incluya el uso de otra metodología alternativa más accesible con excelente resultado de sensibilidad y especificidad como es el método ICP-OES, para no limitar el análisis de metales pesados en alimentos. ICP-OES: Espectrómetro de Emisión de Plasma Acoplado Inductivamente ICP Inductively Coupled Plasma Optical Emission Spectrocopy (ICP-OES) El método de Absorción Atómica como método para detección de plomo en alimentos varios, este es un método de excelente sensibilidad pero de poca accesibilidad en muchos países, como Guatemala. ICP-OES es un método de alta confiabilidad que es más accesible en muchos países.	
Indonesia agrees to transfer the method in Appendix II to Appendix I as "example of applicable methods that meet the criteria" without any proposed revision.	**Indonesia**
i) la idoneidad de los métodos del Apéndice II como ejemplos de métodos que pueden cumplir los criterios de rendimiento enumerados en el Apéndice I, Perú ha analizado los métodos y ha determinado los criterios de rendimiento de dichos métodos: • AOAC 994.02 • ISO 12193 • AOCS Ca 18c-91 • AOAC 972.25 • NMKL 139 • AOAC 999.11 • NMKL 161 • AOAC 999.10 • ISO/TS 6733 | IDF/RM 133 • EuSalt/AS 015 • EuSalt/AS 013	**Peru**

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria[7]	Principle
Fats and Oils (all) AOAC 994.02 /	lead	0.08	> 0.02	-	0.02	29.2	-	Soybean oil with mean of 0.02 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Fats and Oils (all) ISO 12193	lead	0.08	> 0.001	0.001	0.01	15.4	110	Soybean oil with mean of 0.022 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Fats and Oils (all) AOCS Ca 18c-91	lead	0.08	0.02	-	0.02	28.3	-	Cocao butter with mean of 0.0265 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Named Vegetable Oils AOAC 994.02	lead	0.08	> 0.02	-	0.02	29.2	-	Soybean oil with mean of 0.02 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Named Vegetable Oils ISO 12193	lead	0.08	> 0.001	0.001	0.01	27.7	130	Cocoa butter with mean of 0.022 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Named Vegetable Oils AOCS Ca 18c- 91	lead	0.08	0.02	-	0.02	29.2	-	Soybean oil with mean of 0.02 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Olive Oils and Olive Pomace Oils AOAC 994.02	lead	0.1	> 0.02	-	0.02	29.2		Soybean oil with mean of 0.02 mg/kg	Atomic absorption spectrophotometry (directgraphite furnace)
Olive Oils and Olive Pomace Oils ISO 12193	Lead	0.1	> 0.001	0.001	0.01	15.4	110	Soybean oil with mean of 0.022 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)

Olive Oils and Olive Pomace Oils AOCS Ca 18c-91	lead	0.1	0.02	-	0.02	29.2		Soybean oil with mean of 0.02 mg/kg	Atomic absorption spectrophotometry (direct graphite furnace)
Butter AOAC 972.25 (Codex method)	lead	0.1	-	-	-	-		-	We recommend deleting the method of the standard
Edible casein products NMKL 139 (Codex general method)	lead	0.08	-	-		-	-	-	We do not have the method for evaluation
Edible casein products AOAC 999.11	lead	0.08	> 0.04	-	0.04	46	-	Milk powder with mean of 0.040	Atomic absorption spectrophotometry
Edible casein products NMKL 161	lead	0.08	-	-	-	-	-	-	We do not have the method for evaluation
Edible casein products AOAC 999.10	lead	0.08	-	-	-	-	-	-	We recommend deleting the method of the standard
Edible casein products ISO/TS 6733	lead	0.08		0,01		75.5		Casein	Graphite furnace atomic absorption spectrometric We recommend deleting the method of the standard, the Results of interlaboratory test do not meet the precision levels for casein
Edible casein products IDF/RM 133	lead	0.08	-	-	-		-	-	This method is withdraw
Table olives AOAC 999.11 | NMKL 139 (Codex general method)	lead	0.4	> 0.04	-	0.04	38	-	Apple sauce with mean of 0.27	AAS (Flame absorption)
Food grade salt EuSalt/AS 015	lead	1		0.06	0.018			Salt	ICP-OES
Food grade salt EuSalt/AS 013	lead	1	≥ 0.5	-	0.5	51.1	-	Salt with mean of 0.808	Atomic absorption spectrophotometry

ii) otros métodos que pueden cumplir los criterios enumerados en el Apéndice I.

Perú no tiene comentarios sobre este punto.

AOAC 994.02 method using AAS-GF for the determination of Lead is suitable for the analysis of Lead in Fats and Oils (All), Named vegetable oil, Olive oils and Olive Pomace Oils. AOAC 999.11 method using AAS-Flame is suitable for the determination of Lead in Table Olives. AOAC 999.10 method using AAS-Flame or AAS-GF is suitable for the determination of Lead in Edible Casein products.	**Philippines**
Firstly, The Kingdom of Saudi Arabia would like to thank the committee of CCMAS for their efforts. The Kingdom of Saudi Arabia respectfully submits the following comments on the Request for information relating to methods of analysis / examples of methods of analysis: Response to i and ii) The followed Points illustrates the method that can meet the Performance Criteria for Lead and Cadmium in Foods: Commodity: Foodstuffs Analyte: cadmium and lead Method Technique: The test solution, obtained by pressure digestion, is nebulized and the aerosol transferred to a high frequency inductively coupled argon plasma. The high temperature of the plasma is used to dry the aerosol and to atomize and ionize the elements. The ions are extracted from the plasma by a set of sampler and skimmer cones and transferred to a mass spectrometer where the ions are separated by their mass/charge ratio and determined by a pulse-count and/or analogue detector. Analytical Instrument: ICP-MS Reference: EN 15763:2009 Moreover, we recommend to add Horwitz ratio (HorRat) in the Performance Criteria for Lead and Cadmium in Foods which used to assess the acceptability of analytical techniques in terms of interlaboratory precision, or reproducibility. It is defined as the ratio of the observed relative standard deviation among laboratories, RSDR (%), to the corresponding predicted relative standard deviation, PRSDR which is derived from the Horwitz equation. The limits for performance acceptability are 0.5–2; however, 0.5 < HORRAT ≤ 1.5—Method reproducibility is normally expected while HORRAT > 1.5—method reproducibility is higher than normally expected for some analyte in a specific matrix, and further investigation needed.	**Saudi Arabia**
About part ii) Other methods that can meet the performance criteria list in Appendix I, for the provision "Lead and Cadmium", Uruguay suggests the following methods for the commoditties detailed: Commoditty: Natural mineral waters Method: ISO 17294-2	**Uruguay**

Principle: ICP-MS Commoditty: Infant Formulae Methods: FDA Method 4.7 Ver. 1.2, AOAC 2013:06, EN 15763:2010 Prinicple: ICP-MS Commoditty:Milk and Secondary Products All milk and milk products Methods: FDA Method 4.7 Ver. 1.2, AOAC 2013:06, EN 15763:2010 Principle: ICP-MS Commoditty: Fruits and Vegetables, and Related Products Fruits and Vegetables (fresh or processed and related products) Methods: FDA Method 4.7 Ver. 1.2, AOAC 2013:06, EN 15763:2010 Principle: ICP-MS Commoditty: Meats and Secondary Products All meats and meats products Methods: FDA Method 4.7 Ver. 1.2, AOAC 2013:06, EN 15763:2010 Principle: ICP-MS Miscellaneous Products Food grade salt Method: EuSalt/AS 015 Principle: ICP-OES Se presentan ejemplos para las disposiciones y comodities solicitados en dos cuadros complementarios: • cuadro con metodologías para grupos de matrices • cuadro disponible en la CL 2024/08-MAS Please consider the validation data presented in document: CX/MAS 23/42/3 Add.1 APPENDIX II TABLE 2 where it indicates: The Group responsible for preparing this document (Uruguay, Argentina, and Brazil) supported by other CCLAC countries recommends to CCMAS to endorse as Type I for the determination of moisture in the following commodities: Blend of skimmed milk and vegetable fat in powdered form, Reduced fat blend of skimmed milk powder and vegetable fat in powdered form, Dairy permeate powders, Milk powders and cream powders, Whey powders.	

Performance criteria for lead in butter, edible casein and whey powders (developed by CCMAS41, adopted by CAC44 and included in CXS234)

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum applicable range Minimum	Maximum	Examples of applicable methods that meet the criteria	Principle
Butter,edible casein products and whey powders, (secondary milk products)	Lead	0.02	0.004	0.008	≤ 44	60-115%	0.011	0.029	- *FDA Method 4.7 Ver. 1.2 AOAC 2013:06 EN 15763:2010*	- ICP-MS

The methods proposed for the determination of lead in olive oil and olive pomace oil in Appendix II of CL 2024/08-MAS are suitable for the intended purpose and are examples of methods that meet the performance criteria for determination, as provided for in Appendix I of the same document.	**IOC**
Review of methods of analysis for irradiated foods in the General Methods for the Detection of Irradiated Foods (CXS 231-2001) and their incorporation into CXS 234	
Information on methods in CXS 231 (See Appendix III) to determine whether they are still in use and "fit for purpose" to allow endorsement and incorporation into CXS 234 and subsequent revocation of CXS 231.	
Egypt agrees with the General Methods for the Detection of Irradiated Foods (for review and possible inclusion in CXS 234) as theses still in use and "fit for purpose"	**Egypt**
Indonesia agrees to delete the information of the publication year of the method, the method require the latest version of method revision. Indonesia also proposes some editorial revision for maintaining the consistency in the principles, as follows: - Gas chromatographic - Gas chromatographic/ mass spectrometric - Electron Spin Resonance (ESR) or Electron Paramagnetic resonance (EPR) spectrometric	**Indonesia**

Commodity	Provision	Method	Principle	Type	Todavía se usan	Adecuados al fin previsto
Food containing fat **(e.g. raw meat and chicken, cheese, fruits)**	Detection of irradiated food **- Detection of radiation-induced hydrocarbons**	EN 1784 ~~: 1996~~	Gas chromatographic analysis of hydrocarbons **Análisis de hidrocarburos por cromatografía de gases inducido mediante Irradiación**	Type II	Norma vigente / 2004-02-13 □.	Si son adecuados: Productos alimenticios. Detección de alimentos irradiados que contienen grasa
Food containing fat **(e.g. raw meat and chicken, liquid whole egg)**	Detection of irradiated food **- Detection of radiation-induced 2-alkylcyclobutanones**	EN 1785[1] ~~: 1996~~	Gas chromatographic/ mass spectrometric analysis of 2-alkylcyclobutanones **Análisis de las 2-alquilciclobutanonas por cromatografía de gases/espectrometría de masas radioinducidas**	Type III	Norma vigente / 2004-02-13. Productos alimenticios.	Si son adecuados: Detección de alimentos irradiados que contienen grasa
Food containing bone	Detection of irradiated food - **Radiation induced Electron Spin Resonance (ESR) signal attributed to hydroxyapatite (principal component of bones)**	EN 1786~~: 1996~~	ESR spectroscopy	Type II		
Food containing cellulose **(e.g. nuts and spices)**	Detection of irradiated food **- Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline cellulose**	EN 1787~~: 2000~~	ESR spectroscopy **Detección de alimentos irradiados que contienen celulosa mediante espectroscopía RSE** **princiio: análisis del espectro de resonancia del spin del electrón,**	Type II	UNE-EN 1787:2022 Norma vigente / 2022-10-11	Si son adecuados: Productos alimenticios que contienen celulosa y han sido sometidos a radiación ionizante.

Peru

			conocido como espectro de resonancia electrónica paramagnética EPR			
Food containing silicate minerals **(e.g. herbs, spices, their mixtures and shrimps)**	Detection of irradiated food - **Thermoluminescence glow ratio used to indicate the irradiation treatment of the food**	EN 1788~~: 2001~~	Thermoluminescence **Principio: análisis de termoluniscencia de los silicatos minerales contaminantes**	Type II	UNE-EN 1788:2002	Si son adecuados: Producto: productos y/o ingredientes alimenticios, productos alimenticios a partir de los cuales es posible aislar una cantidad suficiente de silicatos minerales, hierbas, especias y mezclas de ambas, y otros productos alimenticios
Food containing silicate minerals **(e.g. shellfish, herbs, spices, seasonings)**	Detection of irradiated food **- Measurement of photostimulated luminescence intensity**	EN 13751[2]: ~~2002~~	Photostimulated luminescence **Principio: medición inicial de intensidad de LFE que puede usarse con fines de cribado y un método de calibración para determinar la sensiblidad de LFE que permita la clasificación**	Type III	Norma vigente 2010-01-05 / UNE-EN 13751:2010	Si son adecuados: Productos: crustáceos y hierbas, especias y condimentos, sin embargo, se aplica a otra gran variedad de alimentos
Food containing crystalline sugar **(e.g. dried fruits and raisins)**	Detection of irradiated food **- Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline sugar**	EN 13708~~: 2001~~	ESR spectroscopy **análisis del espectro de resonancia paramagnética electrónica (RPE)**	Type II	UNE-EN 13708:2022 Norma vigente: 2022-11-23	Si son adecuados: Productos alimenticios. Detección de alimentos irradiados que contienen azúcar cristalizado mediante espectroscopia RPE azúcares cristalizados tratados con radiación ionizantes
Herbs **and** spices ~~and raw minced meat~~ [3]	Detection of irradiated food ~~**- Difference between total microorganism count and viable microorganism count**~~	EN 13783: ~~2001~~ NMKL 231 ~~(2002)~~	Direct Epifluorescent Filter Technique/Aerobic Plate Count (DEFT/APC) (screening method)	Type III	UNE-EN 13783:2002 Norma vigente: 2002-06-28	Si son adecuados: Productos alimenticios. Detección de alimentos irradiados usando la técnica de epifluorescencia después de filtrado y recuento en placa de la flora aeróbica (DEFT/APC). Método de cribado.
Food containing DNA **(e.g. food products, both of animal and plant origin such as various meats, seeds, dried fruits and spices)**	Detection of irradiated food **– Detection of DNA fragmentation presumptive to irradiation treatment**	EN 13784:~~2001~~	DNA comet assay (screening method)	Type III	UNE-EN 13784:2002 Norma vigente: 2023-06-05	Si son adecuados: Productos alimenticios. Ensayo cometa de ADN para la detección de alimentos irradiados. Método de cribado.

The Philippines support the decision of CCMAS that the General Methods for the Detection of Irradiated Foods not be endorsed into CXS 234 at this time due to insufficient information available on the methods and the application and as a consequence to maintain the methods in CXS 231	**Philippines**
Eystein Oveland: NMKL 231 does not exist (delete).	**NMKL**
Performance criteria – sum of components: for methods to determine MLs for aflatoxins in certain cereals and cereal-based products including foods for infants and young children	
Information on example methods that meet the performance criteria for methods for determination of MLs for aflatoxins in certain cereals and cereal-based products including foods for infants and young children.	
In Egypt , we are using the Flurometer as (in house method) for determination of MLS for aflatoxins which met the provided performance criteria Principle : Aflatoxin, a toxin from a naturally occurring mold, is a Group 1 carcinogen proven to cause cancer in humans. Aflatoxin can also cause economic losses in livestock due to disease or reduced efficiency of production. Afla Test is a fast, simple, safe and highly accurate method for quantitatively measuring aflatoxin in many commodities. Samples are prepared by mixing with an extraction solution, blending and filtering. The extract is then applied to the Afla Test column bound with specific antibodies to aflatoxin. At this stage, the aflatoxin binds to the antibody on the column. The column is then washed with water to rid the immunoaffinity column of impurities. By passing methanol through the column, the aflatoxin is removed from the antibody. This methanol solution can then be measured in a flurometer.	**Egypt**
Perú no tiene comentarios sobre este punto.	**Peru**
The Enzyme-Linked Immunoassay method (ELISA/AOAC) was used and validated for the analysis of Total Aflatoxins (AFB1 + B2 + G + G2) in maize, maize flour and corn chips. The limit of detection using the ELISA is ≤ 2 and the range of quantitation is 5.0 –50 ug/kg. HPLC-FLD was used to determine the amount of AFB1, AFB2, AFG1, AFG2 in Maize, maize flour and corn chips. The limit of detection using the HPLC-FLD is ≤ 0.2 ug/kg and range of quantitation is 1.0 to 50 ug/kg	**Philippines**
Methods of analysis for determination of moisture content in dried milk	
Information / data on the applicability of the method to determine moisture content in dried milk for dairy permeate and whey powders.	
Egypt agrees with the proposed method for estimating the moisture of dried milk and suggests the addition of the international reference number AOAC 927.05 .	**Egypt**
The outlined method in Appendix V for the determination of moisture content in dried milk is outdated. The process is time consuming and prone to errors, because it exposes the sample to moisture in the air while the test is being conducted by the analyst. Therefore, the use of a moisture analyzer is proposed. A moisture analyzer is a one-step analysis that measures the loss of mass on drying at the required temperature, calculates the loss in the required units and can be completed in less than twenty minutes. Use of a moisture analyzer is faster with increased accuracy.	**Guyana**

Indonesia would like to propose changes to the text as follows: 4.2. Drying oven, with good ventilation, as far as possible with forced ventilation, capable of being thermostatically maintained at (105 ± 2) °C throughout the workspace, with a temperature controller. Rationale: Based on AOAC 925.45 and NFTA 2.2.5 method, recommended temperature for oven drying covers 105 °C, or in the range of 100 °C - 105 °C.	**Indonesia**
Perú no tiene información ni datos sobre la aplicabilidad del método para determinar el contenido de humedad en la leche en polvo (Apéndice V) a los permeados lácteos y los sueros de leche en polvo.	**Peru**
The Philippines request consideration of the applicability of the method to determine moisture content in dried milk (CL 2024/08 -MAS Appendix V) for dairy permeate and whey powders as the method is simple and the equipment used for the method is widely available. It is also being used in the Philippines. Inclusion of the method as Type IV would allow countries to use the method if the ISO | IDF method is not available.	**Philippines**
See comments in Annex II	**Uruguay**
Any other methods for provisions in Codex commodity standards developed by committees adjourned sine die for consideration / endorsement by the PWG on endorsement and CCMAS43[1].	
Egypt proposes endorsement of analytical method for detecting / determining soybean proteins in processed meat products (such as sausage , Luncheon , ... etc.) in order to reduce the food fraud for a such products by soy proteins instead of meat proteins, in this concern Egypt issued in the Egyptian Standard No. 8452/2021 which is adopted by three international methods as follows : 1- Identification of Marker Proteins for the Adulteration of Meat Products with Soybean Proteins by Multidimensional Liquid Chromatography−Tandem Mass Spectrometry Reference: ACS Publication 2- TaqMan Real-time PCR assay Quantitative detection of soybean in meat products by a TaqMan real-time PCR assay Reference: Sónia Soares, Joana S. Amaral, M. Beatriz P.P. Oliveira, Isabel Mafra, Meat Science 98 (2014) 41–46 3- Sprint Protein Analyzer Protein in Raw and Processed Meats (Automated Dye-Binding Method)By using Sprint Protein Analyzer Reference: AOAC Official Method 2011.04	**Egypt**
Perú no tiene comentarios sobre este punto.	**Peru**