CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Items 3 and 10 **CRD05**

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

43rd Session
Budapest, Hungary

13 – 18 May 2024

(Comments of European Union)

Agenda Item 3: Endorsement of methods of analysis provisions and sampling plans in Codex standards

Mixed Competence
Member States Vote

The following is the response of the European Union to CL 2024/08-MAS.

Review of methods of analysis for contaminants: performance criteria for lead and cadmium in foods

Comments and information on i) the suitability of methods in Appendix II as example methods that can meet the performance criteria listed in Appendix I; and ii) other methods that can meet the performance criteria list in Appendix I.

The EUMS proposes to include the following CEN standards as examples of available methods that meet the performance criteria listed in Appendix I:

- EN 17851:2023 Foodstuffs - Determination of elements and their chemical species - Determination of Ag, As, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Tl, U and Zn in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion,
- EN 15763:2010 Foodstuffs - Determination of trace elements - Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion, and
- EN 14083:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion
- OIV-MA-AS323-07 Multielemental analysis using ICP-MS

as methods of analysis that meet the performance criteria listed in Appendix I of CL 2024/08-MAS

EN 17851:2023 was validated by a collaborative study involving 18 laboratories from different European countries, with 10 different samples (banana, cocoa powder, wheat noodles, black currant nectar, milk powder, oyster, celery, dogfish liver, liver, kale) covering a broad range of food matrices and element concentrations (lead and cadmium included). The samples banana, black currant nectar and liver were produced from retail market samples partly by spiking the elements of interest. Three certified reference material were included: ERM BD150 Skimmed milk powder, NIST SRM 1566b Oyster tissue and NRC DOLT-5 Dogfish liver. Celery and kale were reference materials from previous PT rounds. The cocoa and the wheat noodles were freshly prepared for the trial by spiking and homogenization.

Recent proficiency test data confirm that the method performance characteristics are fulfilled by official control laboratories especially at very low contents.

EN 17851:2023 performance characteristics for lead and cadmium in comparison with the Codex criteria are summarized in Table 1.

EN 15763:2010 was tested on a total of 7 foodstuffs: carrot puree, fish muscle, mushroom (CRM from the National Food Administration, Sweden), wheat flour, simulated diet (CRM from the National Food Administration, Sweden), scampi, mussel powder, and lobster hepatopancreas (Tort-2, CRM from NRC Canada). The elemental concentrations in mg/kg dry matter (dm) ranged from 0.03 to 28.3 for Cd and 0.01 to

2.4 for Pb. The reproducibility relative standard deviations (RSDR) for Cd from 2.8 to 18% and for Pb from 8.0 to 11 %, except for wheat flour (RSDR 50%). The HorRat values were less than 1.5 for all test samples, except for the determination of Pb in wheat flour at a level close to the limit of quantitation (0.01 mg/kg dm).
EN 14083:2003 is state of the art in determination of especially lead and cadmium by the widely used graphite furnace measurement technique and an appropriate sample preparation. The method is widely used in the EU. It is a well-established method for backup measurement of critical lead and cadmium contents exceeding MLs first measured by ICP-MS. The method was horizontal validated by different validation studies with more than 10 participating laboratories. A broad range of food matrices (bovine liver, wholemeal wheat flour, bovine muscle, green paprika, tomato powder and spinach powder),were tested, including 3 CRMs.

EN 14083:2003 performance characteristics for lead and cadmium in comparison with the Codex criteria are summarized in Table 2.

OIV-MA-AS323-07 was validated in two collaborative studies using samples of red, white, fortified and sparkling wine (in total 21 samples). This method can be applied to the analysis of lead in wines in the range of 0.010 and 0.20 mg/l with HorRat values less than 0.5. OIV-MA-AS323-07 performance characteristics for lead in comparison with the Codex criteria are summarized in Table 3.

Table 1: Codex performance criteria for lead and cadmium in foods in comparison to the performance characteristics of EN 17851

Commodity	**Provision**	**Codex Requirements (CL 2024/08-MAS App.I)**						**EN 17851**				
		ML (mg/kg)	Minimum applicable range (mg/kg)	(LOD) (mg/kg)	(LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Minimum applicable range (mg/kg)[1]	(LOD) (mg/kg)[1]	(LOQ) (mg/kg)[1]	Precision (RSDR) (%)[1] No more than	Recovery (%)[1]
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%	0.004-0.58	0.002	0.004	17	96-104
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%	0.004-0.58	0.002	0.004	30	96-104
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	0,004-0,58	0.002	0.005	30	96-104
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	0,004-0,58	0.002	0.005	30	96-104
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%	0,004-0,58	0.006	0.012	30	96-104
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	0,004-0,58	0.008	0.016	30	96-104
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	0,004-0,58	0.008	0.016	25	96-104
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	0,004-0,58	0.010	0.020	25	96-104
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%	0,004-0,58	0.010	0.020	25	96-104
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	0,004-0,58	0.010	0.020	25	96-104
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	0.02-0.58	0.010	0.020	25	96-104
Edible fats and oils	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%	0,004-0,58	0.016	0.032	30	96-104
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	30	96-104
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104

Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0,004-0,58	0.01	0.02	20	96-104
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%					
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%	0,004-0,58	0.015	0.03	37	96-104
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0,004-0,58	0.02	0.04	37	96-104
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0,004-0,58	0.02	0.04	20	96-104
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0,004-0,58	0.02	0.04	20	96-104
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0,004-0,58	0.02	0.04	20	96-104
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0,004-0,58	0.02	0.04	20	96-104
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%	0.004-0.58	0.03	0.06	20	96-104
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%	0.004-0.58	0.03	0.06	20	96-104
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	0,004-0,58	0.03	0.06	20	96-104
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0,004-0,58	0.04	0.08	20	96-104
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.004-0.58	0.04	0.08	20	96-104
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.10-0.58	0.04	0.08	20	96-104
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%	0.02-0.58	0.1	0.2	30	96-104
Natural mineral waters	cadmium	0.003	0.0017 - 0.0043	0.0006	0.0012	44	40-120%	0.006-15.2	0.0006	0.0012	35	101-107
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	0.006-15.2	0.01	0.02	25	101-107
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	0.006-15.2	0.01	0.02	25	101-107
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	0.006-15.2	0.01	0.02	25	101-107
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.006-15.2	0.01	0.02	20	101-107
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.006-15.2	0.01	0.02	20	101-107
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.006-15.2	0.01	0.02	20	101-107
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.006-15.2	0.01	0.02	20	101-107
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.006-15.2	0.01	0.02	20	101-107
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.006-15.2	0.01	0.02	20	101-107
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.006-15.2	0.01	0.02	20	101-107
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%	0.006-15.2	0.01	0.02	20	101-107
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.006-15.2	0.04	0.08	20	101-107
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%	0.006-15.2	0.05	0.10	30	101-107
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%	0.006-15.2	0.05	0.10	20	101-107

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%	0.006-15.2	0.05	0.10	20	101-107
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%	0.006-15.2	0.05	0.10	20	101-107
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	0.006-15.2	0.05	0.10	15	101-107
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	0.006-15.2	0.05	0.10	15	101-107
Butter,edible casein products and whey powders, (secondary milk products)	Lead	0.02		0.004	0.008	≤ 44	60-115%	0.004-0.58	0.002	0.005	30	96-104

Table 2: Codex performance criteria for lead and cadmium in foods in comparison to the performance characteristics of EN 14083

Commodity	**Provision**	**Codex Requirements (CL 2024/08-MAS App.I)**						**EN 14083**				
		ML (mg/kg)	Minimum applicable range (mg/kg)	(LOD) (mg/kg)	(LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Minimum applicable range (mg/kg)[2]	(LOD) (mg/kg)[2]	(LOQ) (mg/kg)[2]	Precision (RSDR) (%)[2] No more than	Recovery (%)[2]
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%	0.001 – 0.044	0.0004	0.001	18	91-102
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%					
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	0.01 – 0.44	0.004	0.008	44	91-102
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	0.01 – 0.44	0.004	0.008	44	91-102
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%	0.01 – 0.44	0.004	0.008	44	91-102
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%					
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	0.01 – 0.44	0.004	0.008	25	91-102
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	0.02 – 0.88	0.008	0.02	25	91-102
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%	0.01 – 0.44	0.004	0.008	25	91-102
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	0.01 – 0.44	0.004	0.008	25	91-102
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	0.01 – 0.4458	0.004	0.008	25	91-102
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%	0.01 – 0.44	0.004	0.008	18	91-102
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.02 – 0.88	0.008	0.02	25	91-102
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.02 – 0.88	0.008	0.02	25	91-102
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.02 – 0.88	0.008	0.02	25	91-102
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.01	0.02	18	91-102
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01 – 0.44	0.008	0.02	18	91-102

Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%					
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%	0.02 – 0.88	0.008	0.02	25	91-102
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.02 – 0.88	0.008	0.02	25	91-102
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.1 – 4.4	0.02	0.04	25	91-102
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.01 – 0.44	0.004	0.008	18	91-102
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%	0.02 – 0.88	0.008	0.02	18	91-102
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%	0.01 – 4,4	0.004	0.008	18	91-102
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	0.01 – 4,4	0.004	0.008	18	91-102
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.02 – 0.88	0.008	0.02	18	91-102
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.02 – 0.88	0.008	0.02	18	91-102
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.02 – 0.88	0.008	0.02	18	91-102
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%	0.1 – 4.4	0.04	0.1	18	91-102
Natural mineral waters	cadmium	0.003	0.0017 - 0.0043	0.0006	0.0012	44	40-120%	0.0014 – 0.20	0.0006	0.0012	22	97-108
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	0.014 – 2.0	0.04	0.01	22	97-108
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	0.014 – 2.0	0.04	0.01	22	97-108
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	0.014 – 2.0	0.04	0.01	22	97-108
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
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%	0.014 – 2.0	0.04	0.01	22	97-108
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Chocolate containing or declaring ≥30% to	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%	0.014 – 2.0	0.04	0.01	22	97-108

<50% total cocoa solids on a dry matter basis												
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%	0.014 – 2.0	0.04	0.01	22	97-108
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%	0.014 – 2.0	0.04	0.01	22	97-108
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	0.014 – 2.0	0.04	0.01	22	97-108
Butter,edible casein products and whey powders, (secondary milk products)	Lead	0.02		0.004	0.008	≤ 44	60-115%	0.02-0.58	0.008	0.02	44	91-102

Table 3: Codex performance criteria for lead in wine in comparison to the performance characteristics of OIV-MA-AS323-07

Commodity	**Provision**	**Codex Requirements (CL 2024/08-MAS App.I)**						**OIV-MA-AS323-07**				
		ML (mg/kg)	Minimum applicable range (mg/kg)	(LOD) (mg/kg)	(LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Minimum applicable range (mg/kg)	(LOD) (mg/kg)	(LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	0.01-0.20			7	
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%	0.01-0.20			7	
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%	0.01-0.20			7	

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.

The methods mentioned in Appendix III are still in use in the EU and fit for purpose, except EN 13783 (Difference between total microorganism count and viable microorganism count) and EN 13784 (Detection of DNA fragmentation presumptive to irradiation treatment), which should be withdrawn because other factors than the irradiation treatment have an influence on the analytical results like freezing followed by thawing. Therefore, the EUMS support the endorsement and incorporation into CXS 234 of EN 1784, EN 1785, EN 1786, EN 1787, EN 1788, EN 137512 and EN 13708 together with the proposed typing of the methods.

The footnote to EN 1785 should be removed as it was recently proven that 2-alkylcyclobutanones do not occur naturally in cashew nuts and nutmeg (https://DOI.org/10.1016/j.foodchem.2016.01.032, https://doi.org/10.1021/jf403500j).

The footnote to EN 13751 should also be removed as confirmation with EN 1788 (Thermoluminescence) is only necessary for intermediate results.

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. The performance criteria are provided in Appendix IV.

The EUMS proposes to include the following CEN standards as examples of available methods that meet the performance criteria listed in Appendix IV:

- EN 17641:2022 Multimethod for the determination of aflatoxins, deoxynivalenol, fumonisins, ochratoxin A, T-2 toxin, HT-2 toxin and zearalenone by LC-MS/MS, and
- EN 15851:2010 Determination of aflatoxin B1 in cereal based foods for infants and young children - HPLC method with immunoaffinity column cleanup and fluorescence detection

EN-17641:2022 is currently the most comprehensive internationally recognized method for the determination of multi-mycotoxins in food using a confirmatory technique. It includes aflatoxin B1 (AFB1), aflatoxin B2 (AFB2), aflatoxin G1 (AFG1) and aflatoxin G2 (AFG2). Analyte separation and detection is performed by liquid chromatography (LC) coupled with tandem mass spectrometry (MS/MS). Each mycotoxin is quantified by isotope dilution using its 13C-labelled isotopomer as internal standard (ISTD).

The method was collaboratively validated; 23 laboratories, either from authorities or private sectors participated. Test items were paprika, black pepper, almonds, hazelnuts, dried raisins, dried figs, wheat, maize (n = 2), milk powder (n = 2), and maize-, rice- and wheat-based infant cereal samples. All items but one were former proficiency test materials from years 2017-2019, still commercially available as Quality Control materials.

EN-17641:2022 performance characteristics for aflatoxins in cereals and cereal-based foods for infants and young children in comparison to the Codex requirements are summarized in Table 4. The group "Cereals" includes all type of grains or flours such as maize, wheat, sorghum, rice, barley, oat, rye, etc. Detailed results of the collaborative study are given in https://doi.org/10.3390/toxins11110658.

EN-15851:2010 uses HPLC with post column derivatization involving bromination followed by fluorescence detection for the determination of AFB1. This method has been validated in an interlaboratory study in 1999 (14 laboratories) via the analysis of both naturally contaminated and spiked samples at concentration levels ranging from 0,07 µg/kg to 0,18 µg/kg. EN-15851:2010 performance characteristics for AFB1 in baby food in comparison to the Codex requirements are summarized in Table 5. AFB2, AFG1 and AFG2 alone are not included in the scope of EN-15851:2010.

Table 4. Codex requirements vs. EN 17641:2022 method performance characteristics. Only data for aflatoxins are shown. AFLAs refers to the sum of AFB1, AFB2, AFG1 and AFG2.

		Codex requirements (CL 2024/08 -MAS)				EN-17641:2022 method performance				
Commodity	**Analyte**	**LOQ (µg/kg)**	**Range (µg/kg)**	**Precision (%)**	**Rec (%)**	**LOQ (µg/kg)**	**Range (µg/kg)**	**RSD_r (%)**	**RSD_R (%)**	**Rec (%)**
Maize grain	AFLAs	≤6	8.4 - 21.6	<44	60-115	4	0.1 -128	6 - 7	10 - 15	97 - 107
	AFB1	≤1.5	2.1 – 5.4	<44	40-120	1	0.025 - 32	6 - 19	12 - 23	100 - 116
	AFB2	≤1.5	2.1 – 5.4	<44	40-120	1	0.025 - 32	7 - 12	11 - 12	105 - 109
	AFG1	≤1.5	2.1 – 5.4	<44	40-120	1	0.025 - 32	8 - 10	13 - 14	101 - 108
	AFG2	≤1.5	2.1 – 5.4	<44	40-120	1	0.025 - 32	9 - 15	17 - 21	84 - 106
Maize flour, meal, semolina and flakes derived from maize; Sorghum grain; cereal-based foods for infants and young children	AFLAs	≤4	5.6 - 14.4	<44	60-115	0.1	0.1 - 128	3 - 11	5 - 13	94 - 96
	AFB1	≤1.0	1.4 - 3.6	<44	40-120	0.025	0.025 - 32	5 - 12	7 - 18	91 - 99
	AFB2	≤1.0	1.4 - 3.6	<44	40-120	0.025	0.025 - 32	5 - 10	6 - 16	97 - 108
	AFG1	≤1.0	1.4 - 3.6	<44	40-120	0.025	0.025 - 32	4 - 13	7 - 14	98 - 101
	AFG2	≤1.0	1.4 - 3.6	<44	40-120	0.025	0.025 - 32	9 - 15	9 - 16	94 - 100
Husked Rice	AFLAs	≤8	11.2 - 28.8	<44	60-115	4	0.1 -128	6 - 7	10 - 15	97 - 107
	AFB1	≤2.0	2.8 – 7.2	<44	40-120	1	0.025 - 32	6 - 19	12 - 23	100 - 116
	AFB2	≤2.0	2.8 – 7.2	<44	40-120	1	0.025 - 32	7 - 12	11 - 12	105 - 109
	AFG1	≤2.0	2.8 – 7.2	<44	40-120	1	0.025 - 32	8 - 10	13 - 14	101 - 108
	AFG2	≤2.0	2.8 – 7.2	<44	40-120	1	0.025 - 32	9 - 15	17 - 21	84 - 106
Polished Rice; Cereal-based food for infants and young children	AFLAs	≤2	2.8 - 7.2	<44	40-120	0.1	0.1 - 128	3 - 11	5 - 13	94 - 96
	AFB1	≤0.5	0.7 – 1.8	<44	40-120	0.025	0.025 - 32	5 - 12	7 - 18	91 - 99
	AFB2	≤0.5	0.7 – 1.8	<44	40-120	0.025	0.025 - 32	5 - 10	6 - 16	97 - 108
	AFG1	≤0.5	0.7 – 1.8	<44	40-120	0.025	0.025 - 32	4 - 13	7 - 14	98 - 101
	AFG2	≤0.5	0.7 – 1.8	<44	40-120	0.025	0.025 - 32	9 - 15	9 - 16	94 - 100

Table 5. Codex requirements vs. EN 15851:2010 method performance characterisitics. AFLAs refers to the sum of AFB1, AFB2, AFG1 and AFG2.

		Codex requirements (CL 2024/08 -MAS)				EN-15851:2010 method performance				
Commodity	**Analyte**	**LOQ (µg/kg)**	**Range (µg/kg)**	**Precision (%)**	**Rec (%)**	**LOQ (µg/kg)**	**Range (µg/kg)**	**RSD_r (%)**	**RSD_R (%)**	**Rec (%)**
Maize grain	AFLAs	≤6	8.4 - 21.6	<44	60-115	-	-	-	-	-
	AFB1	≤1.5	2.1 – 5.4	<44	40-120	-	-	-	-	-
	AFB2	≤1.5	2.1 – 5.4	<44	40-120	-	-	-	-	-
	AFG1	≤1.5	2.1 – 5.4	<44	40-120	-	-	-	-	-
	AFG2	≤1.5	2.1 – 5.4	<44	40-120	-	-	-	-	-
Maize flour, meal, semolina and flakes derived from maize; Sorghum grain; cereal-based foods for infants and young children	AFLAs	≤4	5.6 - 14.4	<44	60-115	-	-	-	-	-
	AFB1	≤1.0	1.4 - 3.6	<44	40-120	0.05	0.025 – 0.35	3 - 14	12 -23	92 -101
	AFB2	≤1.0	1.4 - 3.6	<44	40-120	-	-	-	-	-
	AFG1	≤1.0	1.4 - 3.6	<44	40-120	-	-	-	-	-
	AFG2	≤1.0	1.4 - 3.6	<44	40-120	-	-	-	-	-
Husked Rice	AFLAs	≤8	11.2 - 28.8	<44	60-115	-	-	-	-	-
	AFB1	≤2.0	2.8 – 7.2	<44	40-120	-	-	-	-	-
	AFB2	≤2.0	2.8 – 7.2	<44	40-120	-	-	-	-	-
	AFG1	≤2.0	2.8 – 7.2	<44	40-120	-	-	-	-	-
	AFG2	≤2.0	2.8 – 7.2	<44	40-120	-	-	-	-	-
Polished Rice; Cereal-based food for infants and young children	AFLAs	≤2	2.8 - 7.2	<44	40-120	-	-	-	-	-
	AFB1	≤0.5	0.7 – 1.8	<44	40-120	0.05	0.025 – 0.35	3 - 14	12 -23	92 -101
	AFB2	≤0.5	0.7 – 1.8	<44	40-120	-	-	-	-	-
	AFG1	≤0.5	0.7 – 1.8	<44	40-120	-	-	-	-	-
	AFG2	≤0.5	0.7 – 1.8	<44	40-120	-	-	-	-	-

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 (Appendix V) for dairy permeate and whey powders.

The EUMS take note of the decision of CCMAS42 to endorse the method described in Appendix V as Type IV for certain dairy matrices and the decision of CAC46 to adopt the method. However, the EUMS do not favour to have for the same provision a Type IV next to a Type I method as this can lead to confusion and conflicting test results, in particular for internationally traded commodities. The method described in Appendix V is in essence the one described in IDF 26A:1993. It has satisfactory repeatability but reproducibility is insufficient, which led to the withdrawal of IDF 26A:1993 and replacement by ISO 5537|IDF 26:2004.

The EUMS looks forward to the discussion of this topic under Item 11 of CCMAS43 (CX/MAS 24/43/12).

Agenda Item 10: Listing of Type IV methods in CXS 234 when a Type I method is listed for the same commodity and provision

Member States Competence
Member States Vote

The Member States of the European Union (MSEU) welcome and appreciate the work of Uruguay as chair and Brazil as co-chair of the Electronic Working Group to prepare the discussion paper CX/MAS 24/43/12 on Listing of Type IV methods in CXS 234 when a Type I method is listed for the same commodity and provision.

The Procedural Manual defines a Type I method as *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*. Having other methods of whatever type next to a Type I method is, therefore, counter-intuitive. However, certain circumstances such as non-availability of certain reagents, instruments, etc., in a geographical region, or legal requirements restricting the use of certain chemicals, etc., can justify that, as an exception, a Type IV method co-exists with a Type I method.

The MSEU support to accept Type I and Type IV method for the same provision only on a case-by-case basis provided that well-motivated and justified reasons exist as described in para 34 of CX/MAS 24/43/12.

The MSEU are not in favour of developing co-existence or equivalence criteria for Type I and Type IV methods.