

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda Item 16

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON CONTAMINANTS IN FOOD

15th Session

Virtual

9 – 13 May and 24 May 2022

REVIEW OF METHODS OF ANALYSIS FOR CONTAMINANTS

(Prepared by Brazil, the United States of America and Japan)

Codex members and observers wishing to submit comments on this document should do so as instructed in [CL 2022/22-CF](#) available on the Codex webpage¹

BACKGROUND

1. The 14th Session of the Codex Committee on Contaminants in Foods (CCCF14, 2021) agreed to review the methods in the *Standard for General Methods of Analysis for Contaminants* (CXS 228-2001) with the view to transfer them to the *General Standard for Recommended Methods of Analysis and Sampling* (CXS 234-1999) if applicable, and subsequent revocation of the CXS 228².
2. CCCF14 also agreed that Brazil, with the assistance of the United States of America (USA) and Japan, would review the methods with the aim of assessing the appropriateness of the methods or replacement by other more appropriate methods and possible conversion to performance criteria for consideration by CCCF15 (2022). The work focused only on those methods related to compounds in CXS 228 that fall within the definition of contaminant.
3. Considering the definition of contaminant in the *General Standard for Contaminants in Food and Feed* (CXS 193-1995), the methods for copper, iron and zinc, which are regarded as quality factors in CXS 228, were not considered to be within the scope of this document. Therefore, the following methods were identified as falling within the definition of methods for contaminants for the purpose of this work:

Table 1: Methods of analysis for contaminants in CXS 228 that fall within the definition of contaminant in CXS 193

FOOD	PROVISION	METHOD	PRINCIPLE	TYPE
All foods (except fats and oils)	Lead and cadmium	NMKL 139 (1991) AOAC 999.11	AAS after dry ashing	II
All foods (except fats and oils)	Lead and cadmium	NMKL 161 (1998) AOAC 991.10	AAS after microwave digestion	III
All foods	Cadmium	AOAC 986.15	Anodic stripping voltametry	III
All foods	Lead	AOAC 972.25	AAS	III
All foods (except fats and oils)	Lead	AOAC 982.23	Anodic stripping voltametry	III
All foods	Lead	AOAC 986.15	Anodic stripping voltametry	III

¹ Codex webpage/Circular Letters: <http://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/>.

Codex webpage/CCCF/Circular Letters:

<http://www.fao.org/fao-who-codexalimentarius/committees/committee/related-circular-letters/en/?committee=CCCF>

² REP21/CF14, para. 9

4. The analytical methods in Table 1 were reviewed and the applicable range for lead and cadmium of these methods were summarised in Table 2. While these analytical methods may be applicable when checking compliance with some of the MLs for lead and cadmium in the current CXS 193, these methods might not be appropriate for use in checking compliance with the recently revised lower MLs for lead. In addition, although these methods are listed in CXS 228 as applicable to all foods, there is no evidence that they are applicable to all foods for which MLs are currently established in the Codex.
5. In view of the above, it would be desirable for the CCCF to consider developing and including method criteria in CXS 234, rather than including the reference analytical methods for these contaminants in foods. This will also be in line with the decision of CCMAS41 (2021) to develop numeric performance criteria for methods to determine lead in butter, edible casein products and whey powders (secondary milk products) (and as adopted by CAC44) with the intent to revoke the published methods for lead in these products in CXS 234. CCMAS will continue to review the methods for lead in these products at its next session to determine if they should be included as example methods that meet the numeric criteria³.

Table 2: Applicable range of the analytical methods listed in the CXS 228-2001

Reference methods	Applicable range for Pb (mg/kg)	Applicable range for Cd (mg/kg)
NMKL 139 (1991) AOAC 999.11	≥0.04	≥0.05
NMKL 161 (1998) AOAC 999.10	≥0.1	≥0.01
AOAC 986.15	NA	NA
AOAC 972.25	≥0.3	NA
AOAC 982.23	≥0.010	≥0.005

NA: information not available from method standard

6. The Procedural Manual of the Codex Alimentarius Commission establishes that any Codex Committee may develop a set of criteria to which a method used for the determination must comply. The guidelines for establishing numeric values for the criteria are detailed on Table 1 of Section II: Elaboration of Codex texts, reproduced below in Table 3. These criteria are applicable to fully validated methods except for methods such as PCR and ELISA, which require another set of criteria.

*Table 3: Guidelines for establishing numeric values for the criteria
(Procedural Manual of the Codex Alimentarius Commission)*

Applicability:	The method has to be applicable for the specified provision, specified commodity and the specified level(s) (maximum and/or minimum) (ML). The minimum applicable range of the method depends on the specified level (ML) to be assessed, and can either be expressed in terms of the reproducibility standard deviation (sR) or in terms of LOD and LOQ
Minimum applicable range:	For ML ≥ 0.1 mg/kg, [ML - 3 sR , ML + 3 sR] For ML < 0.1 mg/kg, [ML - 2 sR , ML + 2 sR] sR ⁴ = standard deviation of reproducibility
Limit of Detection (LOD):	For ML ≥ 0.1 mg/kg, LOD ≤ ML · 1/10 For ML < 0.1 mg/kg, LOD ≤ ML · 1/5
Limit of Quantification (LOQ):	For ML ≥ 0.1 mg/kg, LOQ ≤ ML · 1/5 For ML < 0.1 mg/kg, LOQ ≤ ML · 2/5

³ REP21/MAS41, paras 28 - 33

⁴ The sR should be calculated from the Horwitz/Thompson equation. When the Horwitz/Thompson equation is not applicable (for an analytical purpose or according to a regulation) or when "converting" methods into criteria then it should be based on the sR from an appropriate method performance study.

Precision:	For ML ≥ 0.1 mg/kg, HorRat value ≤ 2 For ML < 0.1 mg/kg, the $RSD_{TR} < 22\%$. $RSDR^5$ = relative standard deviation of reproducibility. $RSDR \leq 2$. PRSDR			
Recovery (R):	Concentration	Ratio	Unit	Recovery (%)
	100	1	100% (100g/100g)	98 – 102
	≥ 10	10^{-1}	$\geq 10\%$ (10g/100g)	98 – 102
	≥ 1	10^{-2}	$\geq 1\%$ (1g/100g)	97 – 103
	≥ 0.1	10^{-3}	$\geq 0.1\%$ (1mg/g)	95 – 105
	0.01	10^{-4}	100 mg/kg	90 – 107
	0.001	10^{-5}	10 mg/kg	80 – 110
	0.0001	10^{-6}	1 mg/kg	80 – 110
	0.00001	10^{-7}	100 μ g/kg	80 – 110
	0.000001	10^{-8}	10 μ g/kg	60 – 115
	0.0000001	10^{-9}	1 μ g/kg	40 – 120

6. Using the guidelines of Table 3 above, the performance criteria listed in Appendix I for lead and cadmium were derived considering the maximum levels established for each commodity in CXS 193.
7. Several recommended analytical methods for lead in foods are already listed in CXS 234 as Type II (reference methods), Type III (alternative approved methods) or Type IV (tentative methods). These analytical methods are identified in Appendix II. CCCF may wish to consider whether these analytical methods should be replaced by performance criteria. transferred to the column of “example of applicable methods that meet the criteria” in Appendix I if they met the performance criteria established.
8. Although performance criteria for lead and cadmium in natural mineral waters are already listed in CXS 234, it was observed that the values are not in accordance with the guidelines of the Procedural Manual as listed in Table 3, especially related to precision. Therefore, Appendix I includes performance criteria for lead and cadmium in natural mineral waters.

RECOMMENDATIONS

9. CCCF15 is invited to consider the following:
 - a. To consider the performance criteria listed on Appendix I for lead and cadmium and to decide if the table should be submitted to CCMAS for consideration of inclusion in the *General Standard for Recommended Methods of Analysis and Sampling* (CXS 234-1999).
 - b. To consider recommending to CCMAS the revocation of the *Standard for General Methods of Analysis for Contaminants* (CXS 228-2001), including the methods for copper, iron and zinc, because analytical methods for these metals in foods are already listed in CXS 234.
 - c. To consider whether to request CCMAS to:
 1. remove analytical methods listed in Appendix II for lead from CXS 234; and
 2. transfer these methods to the column of “example of applicable methods that meet the criteria” in Appendix I, if they met the performance criteria established.
 - d. To request CCMAS to identify and suggest examples of applicable analytical methods meeting performance criteria in Appendix I.
 - e. To request CCMAS to evaluate the appropriateness of replacing the existing performance criteria in CXS 234 for lead and cadmium in natural mineral waters according to Appendix I.

⁵ The RSDR should be calculated from the Horwitz/Thompson equation. When the Horwitz/ Thompson equation is not applicable (for an analytical purpose or according to a regulation) or when “converting” methods into criteria then it should be based on the RSDr from an appropriate method performance study.

APPENDIX I
(For comments)

Numeric performance criteria for lead and cadmium in foods

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 (RSD _R) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Berries and other small fruits, except cranberry, currant and elderberry	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Cranberry	lead	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Currants	lead	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Elderberry	lead	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Fruits, except cranberry, currants and elderberry	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Bulb vegetables	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Leafy vegetables, except spinach	lead	0.3	0.127 a 0.473	0.03	0.06	38	80-110%		
Legume vegetables	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Fresh farmed mushrooms (common mushrooms (<i>Agaricus bisporus</i>), shiitake mushrooms (<i>Lentinula edodes</i>), and oyster mushrooms (<i>Pleurotus ostreatus</i>))	lead	0.3	0.127 a 0.473	0.03	0.06	38	80-110%		
Pulses	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Root and tuber vegetables	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Canned fruits	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Jams, jellies and marmalades	lead	0.4	0.180 a 0.620	0.04	0.08	37	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 (RSD _R) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Mango chutney	lead	0.4	0.180 a 0.620	0.04	0.08	37	80-110%		
Canned vegetables	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Preserved tomatoes	lead	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Table olives	lead	0.4	0.180 a 0.620	0.04	0.08	37	80-110%		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 a 0.043	0.006	0.012	44	60-115%		
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Grape juice	lead	0.04	0.022 a 0.058	0.008	0.016	44	60-115%		
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 a 0.014	0.002	0.004	44	60-115%		
Fish	lead	0.3	0.127 a 0.473	0.03	0.06	38	80-110%		
Meat of cattle, pigs and sheep	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Meat and fat of poultry	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Cattle, edible offal of	lead	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Pig, edible offal of	lead	0.15	0.054 a 0.246	0.015	0.03	43	80-110%		
Poultry, edible offal of	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Edible fats and oils	lead	0.08	0.045 a 0.115	0.016	0.032	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 (RSD _R) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Fat spreads and blended spreads	lead	0.04	0.022 a 0.058	0.008	0.016	44	60-115%		
Milk	lead	0.02	0.011 a 0.029	0.004	0.008	44	60-115%		
Secondary milk products ⁶	lead	0.02	0.011 a 0.029	0.004	0.008	44	60-115%		
Natural mineral waters	lead	0.01	0.006 a 0.014	0.002	0.004	44	60-115%		
Salt, food grade	lead	1	0.52 a 1.48	0.1	0.2	32	80-110%		
Wine (wine and fortified / liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.078 a 0.322	0.02	0.0400	41	80-110%		
Wine from grapes harvested after July 2019	lead	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.054 a 0.246	0.015	0.03	43	80-110%		
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Bulb vegetables	cadmium	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.028 a 0.072	0.01	0.02	44	60-115%		
Leafy vegetables	cadmium	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Legume vegetables	cadmium	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Pulses, except soya bean (dry)	cadmium	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac	cadmium	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Stalk and stem vegetables	cadmium	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		

⁶ Note that CXS 234 has been updated following CAC44 (2021) and contains numeric performance criteria for lead in butter, edible casein products and whey powders. This proposal will allow these numeric performance criteria to apply to methods for determining lead in all secondary milk products (including butter, edible casein products and whey powders)

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 (RSD _R) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.032 a 0.168	0.01	0.02	44	80-110%		
Rice, polished	cadmium	0.4	0.180 a 0.620	0.04	0.08	37	80-110%		
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.078 a 0.322	0.02	0.04	41	80-110%		
Marine bivalve mollusks (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.135 a 2,865	0.2	0.4	29	80-110%		
Cephalopods	cadmium	2	1.135 a 2,865	0.2	0.4	29	80-110%		
Natural mineral waters	cadmium	0.003	0.002 a 0.004	0.0006	0.0012	44	40-120%		
Salt, food grade	cadmium	0.5	0.234 a 0.766	0.05	0.1	36	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	0.403 a 1.197	0.08	0.16	33	80-110%		
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	0.461 a 1.339	0.09	0.18	33	80-110%		
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.127 a 0.473	0.03	0.06	38	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 (RSD _R) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Chocolate containing or declaring \geq 30% to <50% total cocoa solids on a dry matter basis	cadmium	0.7	0.346 a 1.054	0.07	0.14	34	80-110%		

APPENDIX II
(For information)

For CCCF review: Type II, III and Type IV methods of analysis for lead in foods listed in CXS 234-1999.

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>
Fats and Oils and Related Products				
Fats and Oils (all)	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Named Vegetable Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	AAS	II
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	ISO/TS 6733 IDF/RM 133	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Processed Fruits and Vegetables				
Table olives	Lead	AOAC 999.11 NMKL 139 (Codex general method)	AAS (Flame absorption)	II
Miscellaneous Products				
Food grade salt	Lead	EuSalt/AS 015	ICP-OES	III
Food grade salt	Lead	EuSalt/AS 013	Atomic absorption spectrophotometry	IV

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