

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 16

CRD05

April 2022

ORIGINAL LANGUAGE ONLY

PRIORITY LIST OF CONTAMINANTS FOR EVALUATION BY JECFA For consideration by the virtual meeting of the Working Group on the Review of Methods of Analysis for Contaminants

3, 5 May 2022

13.00 – 16.00 CET

(Prepared by Brazil

Chair of the Working Group)

BACKGROUND

1. CCCF14 (2021) agreed that Brazil, with the assistance of the United States of America (USA) and Japan, would 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 228¹.
2. CCCF14 also agreed that 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.

CIRCULAR LETTER REQUEST (CL 2022/22-CF)

3. The circular letter issued in advance of CCCF 15² requested comments on the performance criteria listed in Appendix I of CX/CF 22/15/15 and on the additional recommendations in paragraph 9 of the aforesaid document which are uploaded to the Codex Online Commenting System (OCS):
<https://ocs.codexalimentarius.org/>.

CIRCULAR LETTER COMMENTS (CL 2022/22-CF)

4. Eight countries and one observer provided comments in response to CL 2022/22-CF: Canada, Chile, Ecuador, Iraq, Peru, Saudi Arabia, Singapore, USA and FoodDrinkEurope.
5. In general, comments received on the CL 2022/22-CF supports the recommendations in CX/CF 22/15/15. It was observed that the performance criteria were calculated using the guidelines for establishing numeric values for the criteria provided in 'Section II: Elaboration of Codex texts, Principles for the Establishment of Codex Methods of Analysis', of the 27th edition of the Codex Alimentarius Commission Procedural Manual. The observer that provided comments also stated that they are fully aligned with the discussions within CCMAS.
6. Appendix I of CX/CF 22/15/15 was amended considering editorial changes as shown in Annex I (**bold underline**).
7. There was general support to send to CCMAS the table in Appendix I of CX/CF 22/15/15 (amended according to Annex I) to consider its inclusion in CXS 234-1999. One country highlights that the way that performance criteria are expressed in the table would facilitate the interpretation of the *Codex Alimentarius* Manual Procedural by laboratories.
8. Comments received support the revocation of the Standard for General Methods of Analysis for Contaminants (CXS 228-2001), including the methods for copper, iron and zinc, and the inclusion of methods performance criteria in CXS 234-1999.

¹ REP21/CF14, para. 9

² CL 2022/22-CF

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9. One country mentioned that Appendix I. Section II, Elaboration of Codex texts, in the Procedural Manual states that numeric values for LOD and LOQ could be used as an alternative to a minimum applicable range. As Appendix 1 in CX/CF 22/15/15 includes both LOD/LOQ values and a minimum applicable range, it may cause confusion since the LOD/LOQs are below the minimum applicable range. In this sense, the country suggests that i) the table in Appendix 1 is modified to clarify that either approach ("minimum applicable range" OR "LOD/LOQs") can be used to set method performance criteria, but that a method does not need to meet both approaches. ii) CCMAS be asked to comment on which approach ("minimum applicable range" OR "LOD/LOQs"), if any, is preferred.
10. 40th session of the Codex Committee on Methods of Analysis and Sampling approved³ the draft preamble and document structure for the *General Standard on Methods of Analysis and Sampling* (CXS 234-1999). Section II for provisions for which there are method performance criteria lists both minimum applicable range and LOD/LOQ. Additionally, performance criteria listed in CXS 234-1999 contains all the proposed criteria.
11. Regarding the possible inconsistency pointed out in paragraph 9, LOD/LOQ should be below the ML. On the other hand, the minimum applicable range should include the ML. In this sense, it is expected that LOD/LOQ are below the minimum applicable range.
12. One member country pondered about the method used to calculate precision. The performance criteria established in Appendix I of CX/CF 22/15/15 were calculated considering 'Section II: Elaboration of Codex texts, Principles for the Establishment of Codex Methods of Analysis', of the 27th edition of the Codex Alimentarius Commission Procedural Manual.
13. There was general support to request CCMAS to suggest examples of applicable analytical methods that meet the established performance criteria for lead and cadmium. There was also support to transfer analytical methods listed in Appendix II for lead from CXS 234 to the column of "example of applicable methods" in the table with specified the matrix or food grouping, if the methods meet the performance criteria. The observer mentioned that it is the responsibility of CCMAS to assess if the existing methods in CXS 234 are meeting the proposed criteria.
13. One country suggested adding AOAC, 2015.01 (heavy metals in food by ICPMS) and EN 15763:2009 as examples of applicable analytical methods. AOAC method is to analyse the following commodities: Foods/Beverages and Beverage Materials, Cacao Bean and Its Products/Chocolate, Beverages and Beverage Materials/Fruit Juice, Foods/Fish, Baby Foods/Infant Formula, Grains/Rice-Based Food Products. Regarding EN method, it is necessary to identify for which commodity the method is applicable as the method is for "Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion".
14. Comments received on the CL 2022/22-CF supported 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.

OBJECTIVES OF THE PRE-SESSION WORKING GROUP

15. The objective of the pre-session WG is to allow participants to consider the proposals for the review of methods of analysis for contaminants as listed in the *Standard for General Methods of Analysis for Contaminants* (CXS 228-2001) with a view to transfer them to the *General Standard for Methods of Analysis and Sampling* (CXS 234-1999). It is also an opportunity to make recommendations for consideration by the plenary at CCCF15.

RECOMMENDATIONS FOR THE PRE-SESSION WORKING GROUP, AND IF AGREEMENT, CCCF15

16. CCCF15 is invited to consider the following:
 - a. To consider the performance criteria listed on Annex 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.

³ REP19/MAS Appendix III

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- c. To consider whether to request CCMAS to:
 - 1. remove analytical methods listed in Annex II for lead from CXS 234; and
 - 2. transfer these methods to the column of “example of applicable methods that meet the criteria” in Annex I, if they met the performance criteria established, and
 - 3. identify for which commodities the methods AOAC, 2015.01 (heavy metals in food by ICPMS) and EN 15763:2009 are applicable considering the performance criteria and include them in the column of “example of applicable methods that meet the criteria” in Annex I
 - d. To request CCMAS to identify and suggest other examples of applicable analytical methods meeting performance criteria in Annex 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 Annex I.

ANNEX 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 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Cranberry	lead	0.2	0.078 <u>to</u> 0.322	0.02	0.04	41	80-110%		
Currants	lead	0.2	0.078 <u>to</u> 0.322	0.02	0.04	41	80-110%		
Elderberry	lead	0.2	0.078 <u>to</u> 0.322	0.02	0.04	41	80-110%		
Fruits, except cranberry, currants and elderberry	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Bulb vegetables	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 <u>to</u> 0.072	0.01	0.02	44	60-115%		
Leafy vegetables, except spinach	lead	0.3	0.127 <u>to</u> 0.473	0.03	0.06	38	80-110%		
Legume vegetables	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Fresh farmed mushrooms (common mushrooms (<i>Agaricus bisporous</i>), shiitake mushrooms (<i>Lentinula edodes</i>), and oyster mushrooms (<i>Pleurotus ostreatus</i>))	lead	0.3	0.127 <u>to</u> 0.473	0.03	0.06	38	80-110%		
Pulses	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Root and tuber vegetables	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Canned fruits	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Jams, jellies and marmalades	lead	0.4	0.180 <u>to</u> 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 <u>to</u> 0.620	0.04	0.08	37	80-110%		
Canned vegetables	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Preserved tomatoes	lead	0.05	0.028 <u>to</u> 0.072	0.01	0.02	44	60-115%		
Table olives	lead	0.4	0.180 <u>to</u> 0.620	0.04	0.08	37	80-110%		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 <u>to</u> 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 <u>to</u> 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 <u>to</u> 0.072	0.01	0.02	44	60-115%		
Grape juice	lead	0.04	0.022 <u>to</u> 0.058	0.008	0.016	44	60-115%		
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.078 <u>to</u> 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 <u>to</u> 0.014	0.002	0.004	44	60-115%		
Fish	lead	0.3	0.127 <u>to</u> 0.473	0.03	0.06	38	80-110%		
Meat of cattle, pigs and sheep	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Meat and fat of poultry	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Cattle, edible offal of	lead	0.2	0.078 <u>to</u> 0.322	0.02	0.04	41	80-110%		
Pig, edible offal of	lead	0.15	0.054 <u>to</u> 0.246	0.015	0.03	43	80-110%		
Poultry, edible offal of	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Edible fats and oils	lead	0.08	0.045 <u>to</u> 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 <u>to</u> 0.058	0.008	0.016	44	60-115%		
Milk	lead	0.02	0.011 <u>to</u> 0.029	0.004	0.008	44	60-115%		
Secondary milk products ⁴	lead	0.02	0.011 <u>to</u> 0.029	0.004	0.008	44	60-115%		
Natural mineral waters	lead	0.01	0.006 <u>to</u> 0.014	0.002	0.004	44	60-115%		
Salt, food grade	lead	1	0.52 <u>to</u> 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 <u>to</u> 0.322	0.02	0.0400	41	80-110%		
Wine from grapes harvested after July 2019	lead	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.054 <u>to</u> 0.246	0.015	0.03	43	80-110%		
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.028 <u>to</u> 0.072	0.01	0.02	44	60-115%		
Bulb vegetables	cadmium	0.05	0.028 <u>to</u> 0.072	0.01	0.02	44	60-115%		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.028 <u>to</u> 0.072	0.01	0.02	44	60-115%		
Leafy vegetables	cadmium	0.2	0.078 <u>to</u> 0.322	0.02	0.04	41	80-110%		
Legume vegetables	cadmium	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Pulses, except soya bean (dry)	cadmium	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac	cadmium	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Stalk and stem vegetables	cadmium	0.1	0.032 <u>to</u> 0.168	0.01	0.02	44	80-110%		

⁴ 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 <u>to</u> 0.168	0.01	0.02	44	80-110%		
Rice, polished	cadmium	0.4	0.180 <u>to</u> 0.620	0.04	0.08	37	80-110%		
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.078 <u>to</u> 0.322	0.02	0.04	41	80-110%		
Marine bivalve mollusks (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.135 <u>to</u> 2,865	0.2	0.4	29	80-110%		
Cephalopods	cadmium	2	1.135 <u>to</u> 2,865	0.2	0.4	29	80-110%		
Natural mineral waters	cadmium	0.003	0.002 <u>to</u> 0.004	0.0006	0.0012	44	40-120%		
Salt, food grade	cadmium	0.5	0.234 <u>to</u> 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 <u>to</u> 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 <u>to</u> 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 <u>to</u> 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 <u>to</u> 1.054	0.07	0.14	34	80-110%		

ANNEX 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