

# CODEX ALIMENTARIUS COMMISSION



Food and Agriculture  
Organization of the  
United Nations



World Health  
Organization

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## Agenda Item 16

CX/CF 22/15/15-Add.1

April 2022

ORIGINAL LANGUAGE ONLY

### JOINT FAO/WHO FOOD STANDARDS PROGRAMME

### CODEX COMMITTEE ON CONTAMINANTS IN FOODS

15th Session

Virtual

9-13 and 24 May 2022

### REVIEW OF METHODS OF ANALYSIS FOR CONTAMINANTS

#### Comments in reply to CL 2022/22-CF

*Comments of Canada, Chile, Cuba, Ecuador, Iraq, Peru, Saudi Arabia, Singapore,  
United States of America (USA)  
and FoodDrinkEurope*

#### Background

1. This document compiles comments received through the Codex Online Commenting System (OCS) in response to CL 2022/22-CF<sup>1</sup> issued in March 2022. 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 appendix

2. The comments submitted through the OCS are hereby attached in the [Annex](#) and are presented in table format.

<sup>1</sup> Codex circular letter, including CL 2021/87-CF, are available on the Codex webpage/Circular Letters:

<http://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/>

or on the dedicated Codex webpage/CCCF/Circular Letters:

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

**GENERAL AND SPECIFIC COMMENTS**

COMMENT	MEMBER/ OBSERVER
<p>(1) <u>Recommendations:</u></p> <p>a. <u>Comment</u> #1: Canada supports the values of the performance criteria in Appendix I of CX/CF 22/15/15 but has a question about the specific criteria presented in Appendix I.</p> <p>Section II, Elaboration of Codex texts, in the Procedural Manual (p90 of 27th edition) states that numeric values for LOD and LOQ could be used as an alternative to a minimum applicable range. The criteria in Appendix 1 includes both LOD/LOQ values and a minimum applicable range. Since the LOD/LOQs are below the minimum applicable range, this apparent inconsistency could cause confusion. Suggest that:</p> <ul style="list-style-type: none"> <li>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.</li> <li>ii) CCMAS be asked to comment on which approach ("minimum applicable range" OR "LOD/LOQs"), if any, is preferred.</li> </ul> <p>a. <u>Comment</u> #2: Canada supports the use of numeric performance criteria for lead, cadmium, and other food contaminant standards in general, that are 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.</p> <p>(2) <u>Recommendations:</u></p> <p>b. <u>Comment</u>: Canada supports the revocation of the Standard for General Methods of Analysis for Contaminants (CXS 228-2001), including the methods for copper, iron and zinc, if this standard would not house any additional, relevant information to that which will be in the General Standard for Recommended Methods of Analysis and Sampling (CXS 234-1999), once required updates to the CXS-234 are made following any recommendations from CCCF15.</p> <p>(3) <u>Recommendations:</u></p> <p>c. <u>Comment</u>: Canada supports providing examples of applicable analytical methods that meet the established performance criteria. Recommending numeric performance criteria for food contaminant standards and providing examples of suitable methods that meet the numeric criteria, rather than solely providing specific analytical methods, is consistent with the approach that CCMAS has already started moving towards for lead in certain food commodities.</p> <p>(4) <u>Recommendations:</u></p> <p>d. <u>Comment</u>: Canada supports requesting CCMAS to suggest examples of applicable analytical methods that meet the established performance criteria for lead and cadmium.</p> <p>Codex member countries and observers may also have suggestions that could be considered.</p> <p>(5) <u>Recommendations:</u></p> <p>e. <u>Comment</u>: Canada supports updating the numeric criteria for cadmium and lead in natural mineral water so that they are calculated in accordance with the guidelines in the '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.</p>	Canada
<p><u>Appendix</u></p> <p><u>Comment</u>: The 'a' in all cells in the "Minimum applicable range (mg/kg)" column should be replaced with the word 'to' in the table titled 'Numeric performance criteria for lead and cadmium in foods' in Appendix I.</p>	Canada

COMMENT	MEMBER/ OBSERVER
<p>Chile está de acuerdo con enviar al CCMAS el cuadro expuesto sobre criterios de rendimiento para el plomo y el cadmio en el Apéndice I del documento CX/CF 22/15/15, para considerar su inclusión en la Norma general para los métodos de análisis y de muestreo (CXS 234-1999). Chile quisiera destacar que la forma de expresar los criterios utilizada en el Apéndice I facilitaría la interpretación de lo indicado en el Manual de Procedimientos del Codex Alimentarius por parte de los laboratorios de ensayo, y por lo tanto cree muy conveniente enviar este cuadro al CCMAS para su revisión.</p>	Chile
<p>Chile está de acuerdo con recomendar al CCMAS la revocación del documento Métodos de análisis generales para los contaminantes (CXS 228-2001), incluidos los métodos para el cobre, el hierro y el zinc. Chile considera que revocar el documento citado es necesario para evitar confusiones, tomando en cuenta que los métodos serán reemplazados por criterios.</p>	
<p>Chile está de acuerdo con solicitar al CCMAS eliminar los métodos analíticos enumerados en el Apéndice II para el plomo de CXS 234, si estos son reemplazados por criterios de rendimiento. Chile está de acuerdo con transferir estos métodos a la columna de «Ejemplo de métodos aplicables que cumplen los criterios» en el Apéndice I, en caso de que cumplan los criterios de rendimiento establecidos.</p>	
<p>Chile considera que sería muy útil solicitar al CCMAS que identifique y sugiera ejemplos de métodos analíticos aplicables que cumplan los criterios de rendimiento del Apéndice I, siempre considerando que estos sean viables de implementar tanto tecnológicamente como económicamente por los países en desarrollo.</p>	
<p>Chile considera oportuno y necesario solicitar al CCMAS que evalúe la idoneidad de sustituir los criterios de rendimiento existentes en CXS 234 para el plomo y el cadmio en las aguas minerales naturales de conformidad con el Apéndice I.</p>	
<p>Cuba agradece la oportunidad de emitir sus criterios sobre la CL 2022/22-CF Solicitud de observaciones sobre el examen de métodos de análisis de contaminantes: Propuesta de criterios numéricos de rendimiento de los métodos para el plomo y el cadmio en los alimentos:</p> <p>En el documento de referencia citado para análisis CX/CF 22/15/15, en el Cuadro 3: Directrices para establecer valores numéricos para los criterios (Manual de procedimiento de la Comisión del Codex Alimentarius) del documento CX/CF 22/15/15 en Aplicabilidad/Recuperación/Concentración se hace referencia a las concentraciones siguientes: concentración <math>\geq 10</math> (razón 10-1), <math>\geq 1</math> (razón 10-3), <math>\geq 0.1</math> (razón 10-1). Se sugiere considerar definir las concentraciones de la siguiente manera (<math>\geq 10 &lt; 100</math>; <math>\geq 1 &lt; 10</math>; <math>\geq 0.1 &lt; 1</math>).</p>	Cuba
<p>Cuba está de acuerdo con examinar los criterios de rendimiento enumerados en el Apéndice I para el plomo y el cadmio y decidir si el cuadro se debe enviar al CCMAS para considerar su inclusión en la Norma general para los métodos de análisis y de muestreo (CXS 234-1999).</p>	
<p>Ecuador considera que es durante la plenaria del CCCF15 que deberían examinarse los criterios de rendimiento enumerados en el Apéndice I para el plomo y el cadmio y posteriormente decidir si el cuadro se debe enviar al CCMAS para considerar su inclusión en la Norma general para los métodos de análisis y de muestreo (CXS 234-1999).</p>	Ecuador
<p>Agree</p>	Iraq
<p>En esta ocasión, el Perú considera que los criterios numéricos de funcionamiento de los métodos para el plomo y el cadmio en los alimentos deben ser remitidos al CCMAS para considerar su inclusión en la Norma general para los métodos de análisis y de muestreo (CVS 234-1999).</p>	Peru

COMMENT	MEMBER/ OBSERVER
<p>Response to</p> <p>a) <u>Appendix I</u></p> <ul style="list-style-type: none"> <li>Precision is usually expressed as absolute standard deviation (<math>s_r</math>, <math>s_R</math>), relative standard deviation (RSD), variance (<math>s^2</math>), or the coefficient of variation (CV). Though <math>s_r</math> and <math>s_R</math> are the most important precision types, the relative standard deviations (<math>RSD_r\% = 100s_r/\text{mean}</math> and <math>RSD_R\% = 100s_R/\text{mean}</math>) are the most useful precision measures in analytical chemistry because the RSD values are usually independent of concentration. Therefore, using the RSD values facilitates the comparison of variabilities at different concentrations. When the RSD increases rapidly with decreasing concentration or amount, this delineates the limit of usefulness of the method (limit of reliable measurement). The HorRat value is a very rough but useful summary of the precision in analytical chemistry. The HorRat value is used as a guide to determine the acceptability of the precision of a method which is the ratio between the relative standard deviations of reproducibility found (<math>RSD_R\%</math>) and the predicted reproducibility relative standard deviation (<math>PRSD_R\%</math>). <math>HORRAT_R = RSD_R(\text{found}, \%) / RSD_R(\text{calculated}, \%)</math>. The limits for performance acceptability are 0.5–2; however, <math>0.5 &lt; HORRAT \leq 1.5</math>—Method reproducibility is normally expected while <math>HORRAT &gt; 1.5</math>—method reproducibility is higher than normally expected for some analyte in a specific matrix, and further investigation needed (Appendix D: Guidelines for Collaborative Study Procedures To Validate Characteristics of a Method of Analysis). The criteria of precision (<math>RSD_R\%</math>) listed in Appendix I for lead and cadmium is equivalent to <math>HORRAT &gt; 1.5</math>, which is slightly higher for this kind of analyte.</li> <li>We recommend submitting the performance criteria table to CCMAS, and we suggest adding AOAS, 2015.01 (heavy metals in food by ICPMS) and EN 15763:2009 as examples of applicable analytical methods.</li> <li>We support transferee 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. The matrix plays an important role in the validity of the method, so by using the performance criteria listed in Appendix I for lead and cadmium, some methods listed in CXS 228 RSD<sub>r</sub> and RSD<sub>R</sub> are overestimated. For example, AOAC 991.10 RSD<sub>r</sub> and RSD<sub>R</sub> of lead in wheat bean detected by GFAAS are above 55%, while the rest of the matrixes included in the method were in accordance with the accepted guidelines of acceptance RSD<sub>R</sub>.</li> <li>We agree to request CCMAS to identify and suggest examples of applicable analytical methods meeting performance criteria in Appendix I.</li> </ul>	Saudi Arabia
Singapore supports the recommendations provided in CX/CF 22/15/15.	Singapore
The United States supports the recommendations in CX/CF 22/15/15	USA
<p>FoodDrinkEurope thanks the (EWG) lead by Brazil, the USA and Japan, and the working group members for the opportunity to provide comments on the document CL 2022/22-CF (March 2022).</p> <p>The recommendations proposed by the Working Group (WG) are fully aligned with those initiated at CCMAS via AOAC.</p> <p>We support the revocation of the Standard for General Methods of Analysis for Contaminants (CXS 228-2001), including the methods for copper, iron and zinc. We strongly believe that the Recommended methods of analysing and sampling (CXS 234) shall become the central point for recommendations of methods.</p> <p>We support the proposals of the WG to request CCMAS to:</p>	FoodDrinkEurope

COMMENT	MEMBER/ OBSERVER
<p>a) remove analytical methods listed in Appendix II for lead from CXS 234. The methods will be replaced by the proposed criteria.</p> <p>b) 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. It is the responsibility of CCMAS to assess if the existing methods in CXS 234 are meeting the proposed criteria.</p> <p>c) to identify and suggest examples of applicable analytical methods meeting performance criteria in Appendix I. CCMAS might face with a situation that existing methods in CXS 234 do not meet the proposed criteria. Then CCMAS should propose other suitable methods, if available.</p> <p>d) 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. There are different criteria for Natural mineral waters in CXS-234, hence there is a need in the revision.</p> <p>We thank the Committee for taking these points into account and look forward to further discussion at the CCCF15 session.</p>	