

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda Item 4.1

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX ALIMENTARIUS COMMISSION
Forty-fourth Session

**COMMENTS ON DRAFT STANDARDS AND RELATED TEXTS SUBMITTED BY THE 41ST SESSION OF
THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING (CCMAS41)
TO THE COMMISSION FOR FINAL OR STEP 5 ADOPTION¹**

BACKGROUND

This document compiles the comments on the draft standards submitted at Step 8 or Step 5/8 and the proposed draft standards submitted at Step 5 of the Procedure. The comments are those received through the Codex Online Commenting Systems (OCS), or via email by the time this document was issued. The comments are as shown in Appendix I.

OCS is an online tool that enables Codex Contact Points to submit comments on draft texts in a standardised way, thus providing more transparency and better management of comments on different Codex texts as requested through Circular Letters. Since its launching at CAC39 (2016), the OCS has been used for different Codex Committees.

EXPLANATORY NOTES ON APPENDIX I

The comments received are presented in a table format, with two columns as follows:

First column – Presents the comments with the rationale.

Second column – Presents the provider of the comments (name of country or observer)

¹ This document compiles comments submitted through OCS, or via email by the time this document was issued, in reply to CL 2021/60/OCS - MAS

Appendix I

Comments in regard to (i) Methods of analysis / performance criteria; (ii) The revised *Guidelines on Measurement Uncertainty* (CXG 54-2004) (at Step 8); and (iii) The *General Guidelines for Sampling* (CXG 50-2004) (at Step 5)

In reply to CL 2021/60/OCS - MAS

Comments of: Australia, Brazil, Canada, Colombia, Costa Rica, Cuba, Egypt, Iran, Iraq, Morocco, Norway, Peru, Philippines, United Kingdom, Uruguay, EURACHEM, GOED, IDF/FIL

GENERAL COMMENTS	MEMBER / OBSERVER
<p>General Comments Brazil appreciates the excellent work done by the United States, New Zealand and Germany and thanks for the opportunity to present the following comments:</p> <p>(i) Methods of analysis / performance criteria Brazil agrees to submit the methods and numeric criteria for adoption/revocation by CAC44 as agreed by CCMAS in para. 24 and 42 of REP21/MAS.</p> <p>(ii) The revised <i>Guidelines on Measurement Uncertainty</i> (CXG54-2004) (at Step 8); and Brazil agrees to advance the revised <i>Guidelines</i> to Step 8 for adoption by CAC44.</p> <p>(iii) The <i>General Guidelines for Sampling</i> (CXG50-2004) (at Step 5) Brazil agrees to forward the revised <i>General Guidelines on Sampling</i> (CXG 50-2004) to CAC 44 for adoption at Step 5.</p> <p>In addition to that, Brazil would like to recall that CCMAS41 could not reach consensus on method ISO 5537/IDF 26 for determination of moisture content in dried milk and the Committee agreed to consider this matter at its next session. That is the reason Brazil cannot agree that the method ISO 5537/IDF 26 be submitted for adoption by CAC for the following products: 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.</p>	Brazil
<p>Cuba agradece la oportunidad de responder la carta circular CL 2021/60/OCS-MAS, y apoyamos en principio los documentos: a. métodos de análisis/criterios de rendimiento (Apéndice II); b. el Proyecto de revisión de las Directrices sobre la incertidumbre en la medición (CXG 54–2004) (Apéndice III) (en el trámite 8), y c. el Anteproyecto de revisión de las Directrices generales sobre muestreo (CXG 50-2004) (Apéndice IV) (en el trámite 5)</p>	Cuba

<p>Le Maroc n'a pas d'objection pour :</p> <ul style="list-style-type: none"> - Partie 1 <p>L'adoption des méthodes d'analyses par CAC44 (CCNFSDU/CCASIA/CCAFRICA/CCNASWP/CCNE/CCPFV et de lait et produits laitiers).</p> <ul style="list-style-type: none"> - Partie 2 <p>La révocation des méthodes d'analyses CCNFSDU/CCPFV et de lait et produits laitiers.</p> <ul style="list-style-type: none"> - Partie 3 <p>L'amendement du CXS 234 pour adoption par le CAC44.</p> <ul style="list-style-type: none"> - Partie 4 <p>4.1 Le Renvoi à CCAFRICA méthodes d'analyses des dispositions du projet de norme pour la viande séchée.</p> <p>4.2 Le renvoi au CCNASWP méthodes d'analyses des dispositions de la norme régionale pour les produits de kava à utiliser comme boisson lorsque mélangé avec de l'eau.</p> <p>4.3 Le renvoi au CCFO (approuvé par le CCMAS, pour examen par le CCFO).</p> <p>4.4 Le renvoi au CCFO : pour examen et réponse par le CCFO.</p> <ul style="list-style-type: none"> - Partie 5 <p>- l'examen des méthodes du paquet graisses et huiles pour examen par le groupe de travail électronique sur le paquet réalisable des graisses et des huiles.</p> <p>- proposer le projet de révision des directives sur l'incertitude de mesure (CXG 54-2004) (Annexe III) à l'étape 8.</p> <p>- proposer l'avant-projet de lignes directives générales sur l'échantillonnage (CXG 50-2004) (Annexe IV) à l'étape 5, bien que cette révision n'a pas répondu au souci exprimé de rendre les directives plus faciles à utiliser. En parallèle le Maroc propose d'élaborer l'e-book et le guide de sélection et de conception des plans d'échantillonnage,</p>	Morocco
<p>The following comments are on the proposed draft revised General Guidelines on Sampling (CXG 50-2004) (Appendix IV) (at Step 5). We are reluctant to support the adoption at step 5 The reason for this is that we cannot see that the revised Guide is more user-friendly for the target group, however, the subsequently apps and e-book will hopefully not require statistical knowledge to apply, and hence we welcome further development.</p> <p>It may seem that the purpose of the revision of this Guide has not been entirely clear. Our understanding was that the aim of the revision was to make the current Guide more user-friendly for the target group; bearing in mind that they do not necessarily possess knowledge and interest in statistics and may not be familiar with the different ISO standards for sampling plans. According to our understanding, the aim was to simplify the structure and provide better guidance for the Codex committees and member countries. The revised Guide focus on applying risks in the design of a sampling plan and thereby includes even more statistics. As it has become a guide for designing sampling plans, this could preferably be reflected in the title of the Guide.</p>	Norway
<p>CL 2021/60/OCS-MAS - Solicitud de observaciones (ii) la revisión de las Directrices sobre la incertidumbre en la medición (CXG 54-2004) (en el trámite 8)</p>	Peru

<p>El Perú agradece al COMITÉ DEL CODEX SOBRE MÉTODOS DE ANÁLISIS Y TOMA DE MUESTRA, por el esfuerzo emprendido en su 41° sesión plenaria para la revisión de las Directrices sobre la incertidumbre en la medición (CXG 54-2004) (en el trámite 8), que nos da la oportunidad para presentar los siguientes comentarios.</p> <p>El Perú en respuesta a la carta circular CL 2020/31/OCS-MAS en el 2020 presentó comentarios al Anteproyecto de las Directrices sobre la incertidumbre en la medición (CXG 54-2004), los que han sido considerados por el CCMAS en la revisión del Anteproyecto.</p> <p>El Perú se muestra a favor de la aprobación de las Directrices sobre la incertidumbre en la medición (CXG 54-2004) (en el trámite 8).</p>	
<p>(i) The Philippines supports the adoption of methods of analysis /performance criteria for provisions in <i>Recommended Methods of Analysis and Sampling</i> (CXS 234-1999).</p> <p><i>Rationale:</i> The methods of analysis/performance criteria proposed met the criteria stated in Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS234 (MAS/40 CRD/27) – 3.2 Acceptance of Methods of Analysis.</p> <p>(ii) The Philippines supports to advance the proposed Draft Revised <i>Guidelines on Measurement Uncertainty</i> (CXG 54-2004) to step 8.</p> <p><i>Rationale:</i> The revision of the Guidelines was to make it simpler and to provide overarching principles and guidance on measurement uncertainty. All the concerns and comments raised by members and observers were addressed and these were noted during the 41st Session of CCMAS.</p> <p>(iii) Proposed Draft revised <i>General Guidelines on Sampling</i> (CXG 50-2004)</p> <p>The Philippines supports the revised CXG 50 package (the revised CXG 50 and its supporting documents) for adoption at Step 5.</p> <p>The Philippines agrees to re-establish the EWG to continue to revise the <i>General Guidelines on Sampling</i> CXG 50-2004 and to further develop the documents in support of CXG 50 taking into account the comments received to CL2021/10-MAS with intention that they are part of the CXG 50 package</p> <p><i>Rationale:</i> The revised guidelines will have a provision of a wider range of sampling plan options that enables different types of sampling plans to be designed and evaluated, providing a wider consideration of cost and fairness as well as sampling, testing and a decision on acceptance or rejection of the food commodity. The revised guideline is also much simpler and useful appended sections.</p> <p>Other issues:</p> <p>1. The Philippines supports the Adoption of amendments to methods of analysis / performance criteria for provisions in <i>Recommended Methods of Analysis and Sampling</i> (CXS 234-1999) from “Water” to “Water (Moisture)”</p> <p><i>Rationale:</i> “Water (Moisture)” is more specific description of the provision. It is the determination of the amount of water vapour and other volatile components present in a sample whereas “Water” content determines the amount of water in a sample.</p>	<p>Philippines</p>

<p>2. The Philippines supports the editorial amendment to the provision in Section 3.3 of the <i>Standard for Edible Casein Products</i> (CXS 290-195) from “maximum free acid” to “maximum free acidity”.</p> <p><i>Rationale:</i> “Free acidity” is more appropriate description of the provision rather than “free acid”. “Free acidity” describes the amount of acid in a substance and it is an important parameter that defines the quality of a sample.</p> <p>3. The Philippines supports the revocation of methods of analysis in <i>Recommended Methods of Analysis and Sampling</i> (CXS 234-1999).</p> <p><i>Rationale:</i> The information captured in the commodity “milk products” was already captured for the specific commodity listings and that the removal of this category from CXS234 would therefore not affect availability of methods of analysis.</p>	
<p>(i) UK supports the Methods of Analysis and Performance Criteria for adoption at CAC44</p> <p>(ii) UK supports the Proposed Draft Guidance on Measurement Uncertainty (CXG54-2004), as presented, for adoption at Step 8; however, para 19 needs to make clear what level of validation is required and should reference the different types of method validation covered in para 13.</p> <p>(iii) The UK supports the proposed draft revised General Guidelines on Sampling.</p>	United Kingdom

SPECIFIC COMMENTS	MEMBER /OBSERVER
<p>The following comments are provided with respect to each part of the circular letter, being editorial changes and needing modification prior to the text appearing in CXS 234 or being reference to another committee, including:</p> <p>- Under METHODS OF ANALYSIS FOR ADOPTION BY CAC44 1.1. CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES - Infant formula - Potassium - AOAC 2011.14 / ISO 15151 IDF Please add '229' following IDF</p> <p>- Infant formula - Sodium - AOAC 2011.14 / ISO 15151 IDF Please add '229' following IDF</p> <p>- Infant formula - Zinc - AOAC 2011.14 / ISO 15151 IDF Please add '229' following IDF</p> <p>1.5 FAO/WHO COORDINATING COMMITTEE FOR NEAR EAST (CCNE) - Mixed Zaatar - Insects/ /Insect Fragments Please remove the second forward slash (/)</p>	Australia

- Mixed Zaatar - Mould damage - Method V-8 Spices, Condiments,..Number 5) - Visual Examination (IV
 Please remove the open bracket before IV

4.3 FOR REFERRAL TO CCFO (endorsed by CCMAS, for consideration by CCFO) on page 18
 Named Vegetable Oils - Apparent density - ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95 Pycnometry - I
 Please remove 'with the appropriate conversion factor /'

- Under Proposed Draft Revised Guidelines for Sampling (CXG50-2004) (at Step 5)
 In the CL 2021/60/OCS – MAS(iii), the section heading number hierarchy has been modified and needs to be returned to how it appears in that agreed in REP21/MAS - Appendix IV, as this has a number of implications, including making all the documents cross-references incorrect, e.g. cross reference in Section 2 paragraph 1 starting with 'In Section 2' on page 30 should now be 'In Section 4-8' if the section heading number hierarchy is retained.

The Symbol '©' in Section 31 paragraph 1, second sentence, should be a 'c'.

Canada would like to provide the following comments on REP21/MAS – Appendix II, Appendix III and Appendix IV

APPENDIX II
 PART 1, METHODS OF ANALYSIS FOR ADOPTION BY CAC44
 1.1 CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Commodity	Provision	Method	Principle	Type
Infant Formula	Thiamine	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
		EN 14122	HPLC with pre- or post-column derivatization to thiochrom	# III
		AOAC 986.27	Fluorometric method	III
	Riboflavin	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
		EN 14152	HPLC	# III
		AOAC 982.31	Fluorometric method	III

Canada

Each of the following methods should have "229" after the IDF as below (see page 38)

Infant formula	Potassium	<u>AOAC 2011.14 / ISO 15151</u> <u>IDF 229</u>	<u>ICP emission spectroscopy</u>	<u>III</u>
Infant formula	Sodium	<u>AOAC 2011.14 / ISO 15151</u> <u>IDF 229</u>	<u>ICP emission spectroscopy</u>	<u>III</u>
Infant formula	Zinc	<u>AOAC 2011.14 / ISO 15151</u> <u>IDF 229</u>	<u>ICP emission spectroscopy</u>	<u>III</u>

1.5 FAO/WHO COORDINATING COMMITTEE FOR NEAR EAST (CCNE)

I have a question as to whether ISO 939 should be coupled with ISO 930 and if AOAC 941.12 requires ISO 939

Mixed Zaatar	Acid-insoluble ash (dry weight basis)	<u>ISO 939</u> and ISO 930 (corrected for moisture by ISO 939) <u>AOAC 941.12 (corrected for moisture by ISO 939)</u>	<u>Calculation by moisture and ash</u> <u>Distillation and</u> Gravimetry, Furnace, 550°C	I
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Part 4

Part 4.4 Referral to CCFO: For consideration and reply by CCFO

Suggest removal of comments *as indicated below*, shared as part of EWG

Named Animal Fatty acid composition

Fats *Canada: Replace AOCS Ce 1f-96 with Ce 1j-07. Retype to Type III, including the ISO methods. Suggest AOCS Ce 2-66 and Ce1j-07 as Type II.

Additional notes for the Appendices:

Appendix III

Terms and definitions: It was suggested that the years be removed from the reference to guidelines, etc.

For examples, CXG 72-2009 should read CXG 72, etc.

<p><u>Appendix IV</u></p> <p>Section 31, para 1, last line: ©appears, but text should be (c)</p> <p>Section 31, para 2: AQL and LQL are mentioned, but are not defined earlier</p> <p>Section 42: As above, AQL and LQL are mentioned, but are not defined earlier</p>																															
<p>PART 2. METHODS OF ANALYSIS FOR ADOPTION BY CAC44 1.8 MILK AND MILK PRODUCTS Incluir técnica para Leche Fermentada, Sólidos Totales el método AOAC 990.20 – AOAC 990.19 – AOAC 925.23 e ISO 13580 IDF 151 Los métodos sugeridos no están incluidos y constituyen un método validado de referencia.</p> <p>PART 2. METHODS OF ANALYSIS FOR ADOPTION BY CAC44 1.8 MILK AND MILK PRODUCTS Incluir técnica de Grasa para Leche Fermentada</p> <table border="0"> <thead> <tr> <th>Commodity</th> <th>Provisión</th> <th>Method</th> <th>Principle</th> <th>Type</th> </tr> </thead> <tbody> <tr> <td>Fermentad Milks</td> <td>Total Fat</td> <td>AOAC 2000.18</td> <td>Gravimetry (Gerber)</td> <td>I</td> </tr> </tbody> </table> <p>El método no está incluido y constituye una parte importante en la verificación de leche fermentada y leches en general</p> <p>PART 2. METHODS OF ANALYSIS FOR ADOPTION BY CAC44 1.8 MILK AND MILK PRODUCTS Incluir técnica de pH para Leche Fermentada</p> <table border="0"> <thead> <tr> <th>Commodity</th> <th>Provision</th> <th>Method</th> <th>Principle</th> <th>Type</th> </tr> </thead> <tbody> <tr> <td>Fermented Milks</td> <td>pH</td> <td>AOAC 945.27</td> <td>Potenciometric</td> <td>II</td> </tr> </tbody> </table> <p>El método no está incluido y constituye una parte importante en la verificación de leche fermentada y leches en general.</p> <p>PART 2. METHODS OF ANALYSIS FOR ADOPTION BY CAC44 1.8 MILK AND MILK PRODUCTS Incluir técnica de proteína para Leche Fermentada</p> <table border="0"> <thead> <tr> <th>Commodity</th> <th>Provision</th> <th>Method</th> <th>Principle</th> <th>Type</th> </tr> </thead> <tbody> <tr> <td>Fermented Milks</td> <td>Protein</td> <td>AOAC 991.20</td> <td>Kjeldahl</td> <td>I</td> </tr> </tbody> </table> <p>El método no está incluido y constituye una parte importante en la verificación de leche fermentada y leches en general.</p> <p>Se sugiere incluir los métodos planteados , por tratarse de métodos analíticos aprobados por AOAC ya que al incluir diversas metodologías para medir un mismo parámetro fisicoquímico, otorga libertad para que los laboratorios puedan adaptar sus técnicas según las condiciones de cada empresa en cuanto a capacidad de adquirir los recursos como materiales, reactivos, equipos.</p>	Commodity	Provisión	Method	Principle	Type	Fermentad Milks	Total Fat	AOAC 2000.18	Gravimetry (Gerber)	I	Commodity	Provision	Method	Principle	Type	Fermented Milks	pH	AOAC 945.27	Potenciometric	II	Commodity	Provision	Method	Principle	Type	Fermented Milks	Protein	AOAC 991.20	Kjeldahl	I	<p>Colombia</p>
Commodity	Provisión	Method	Principle	Type																											
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<p>Así mismo, se soporta la rigurosidad técnica de estas normas y su forma de validación se resume en el siguiente párrafo:</p> <p>Según (Narizano,2001) “Las organizaciones como AOAC y otras que actúan en conjunto con AOAC (ISO, FIL-IDF, NMKL, OIV) coordinan el desarrollo de métodos y su validación y proceden luego a la publicación de los mismos.</p> <p>En el desarrollo y validación del método se establecen claramente el alcance (producto, matriz), la selectividad y especificidad, los rangos de aplicación y la incertidumbre estimada en el resultado (repetibilidad, reproducibilidad, exactitud). Todos estos parámetros que se establecen en los centros de desarrollo de los métodos mediante los estudios colaborativos son los que caracterizan el desempeño del método y su aptitud para el uso propuesto.</p> <p>En efecto, es necesario que la incertidumbre del resultado sea adecuada para poder verificar el cumplimiento de las especificaciones que se han fijado como requisitos para la conformidad con las normas de los productos que estamos evaluando mediante el ensayo.</p> <p>En el caso de los métodos de AOAC, así como los métodos adoptados por el Codex Alimentarius para la evaluación de conformidad de productos , es necesario que se cumpla con el proceso completo de validación en un estudio colaborativo que involucre un número mínimo de laboratorios y que dé como resultado la estimación de repetibilidad, reproducibilidad, sesgo, límite de detección, límite de cuantificación, selectividad, rango de aplicación, linealidad y robustez.</p> <p>Es imprescindible que cada laboratorio demuestre que es capaz de producir resultados con la incertidumbre que corresponde al método validado. Solo de esta forma y con el cumplimiento de los demás requisitos de la ISO 17025 podrá asegurarse la calidad de los resultados emitidos e informados”</p> <p>Part 2 METHODS OF ANALYSIS FOR REVOCATION BY CAC44 2.3 MILK AND MILK PRODUCTS Aunque son métodos para revocación, no es claro en qué casos aplica el análisis de hierro para productos lácteos y el análisis de cobre en productos lácteos grasos.</p>	
<p>Costa Rica considers that the document is ready for adoption. In that sense, Costa Rica supports the advance.</p>	<p>Costa Rica</p>
<p>Egypt appreciates the approach taken by CCMAS on the documents no CL2021/60-OCS-MAS related to the following:</p> <p>(i) Methods of analysis / performance criteria;</p> <p>(ii) The revised Guidelines on Measurement Uncertainty (CXG54-2004) (at Step 8);</p> <p>and</p> <p>(iii) The General Guidelines for Sampling (CXG50-2004) (at Step 5) .</p> <p>In this regard, Egypt would like to confirm agreement on the mentioned document without comments.</p>	<p>Egypt</p>

Agree	Iraq
<p>For Appendix iii- REVISION OF THE GUIDELINES ON MEASUREMENT UNCERTAINTY: It is recommended Uncertainty measurement in microbiology tests according to the ISO standard 19036 will be added to the scope</p> <p>For Appendix IV - PROPOSED DRAFT REVISED GENERAL GUIDELINES ON SAMPLING: In scope section is recommended, two below items will be excluded from the scope for clarification:</p> <p>1- This Sampling method is not applicable for moving lots.</p> <p>2- This Sampling method is not applicable for heterogeneous lots.</p> <p>For Appendix IV - PROPOSED DRAFT REVISED GENERAL GUIDELINES ON SAMPLING: In 6-1-1 random sampling: Using the Random table or application to identify the package is recommended.</p>	Iran
<p>Uruguay desea recordar con respecto al punto a. métodos de análisis/criterios de rendimiento (Apéndice II), PART 3. AMENDMENTS to CXS 234 for adoption by CAC44, que de acuerdo con el REP21/MAS (párrafos 36 a 42) el método IDF 5537/IDF 26 para los siguientes productos: 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</p> <p>no debe pasar a aprobación por parte de la CAC ya que no hubo consenso en relación a dicho método para determinar humedad y la decisión se diferió para el CCMAS 42.</p> <p>En párrafos 36 al 42 del REP 21/MAS quedó establecido que:</p> <p>"Moisture</p> <p>36. CCMAS could not reach consensus on method ISO 5537 IDF 26 for determination of moisture content in dried milk.</p> <p>37. Proposals were made for AOAC 927.05 as the preferred Type I method, noting that:</p> <ul style="list-style-type: none"> • this was a standard method widely used for determination of moisture in dried milk in many countries around the world; and • the ISO IDF method had limitations for use especially since the equipment and utensils were not widely available, were costly and led to environmental waste, and was therefore not accessible to many countries. <p>38. Those members supporting the AOAC 927.05 also reminded the Committee that not only should CCMAS consider performance data but also should look into applicability, availability and cost of methods in line with the criteria for selection of methods set out in the Procedural Manual.REP21/MAS 5</p> <p>39. The EWG Chair explained, that it was necessary to consider performance data to evaluate replacement of a Type I method which was already listed in CXS234 for many years, and reminded CCMAS that according to its own rules in the Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS234, performance / validation data should be submitted in the template provided 60 days prior to a Session of CCMAS. He acknowledged that when evaluating methods for inclusion in CXS234, consideration should also be given to accessibility and cost implications.</p> <p>40. The observer from IDF, drawing attention to CRD6, provided a history of the updating of the ISO method over time to allow better precision and presented some of their research into the use of the ISO IDF method. It had been shown that equipment was available on the market, and some laboratories had built equipment for application of the method in-house, and supported the retention of the current method as Type I and proposed</p>	Uruguay

<p>that AOAC 927.05 could be endorsed as Type IV.</p> <p>Conclusion</p> <p>41. CCMAS agreed to consider this matter at its next session.</p> <p>Conclusion</p> <p>42. CCMAS agreed to:</p> <ul style="list-style-type: none"> i. submit the methods and numeric criteria as endorsed to CAC44 for adoption and inclusion in CXS234 (Appendix II, Part 1 and Part 3) and request revocation of the methods for milk products (Appendix II, Part 2); ii. defer decision on the methods for moisture content to CCMAS42; and agreed: <ul style="list-style-type: none"> o to request the PWG on endorsement to consider this matter; o to assess the data to support if AOAC 927.05 is fit for purpose and that such data should be submitted according to the template in Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS234; and o consideration should also be given to the accessibility and cost of the methods recommended for endorsement." <p>En relación al punto a. métodos de análisis/criterios de rendimiento (Apéndice II), para las siguientes partes:</p> <p>PART 1. METHODS OF ANALYSIS FOR ADOPTION BY CAC44</p> <p>PART 2. METHODS OF ANALYSIS FOR REVOCATION BY CAC44</p> <p>PART 4. METHODS OF ANALYSIS REFERRED</p> <p>PART 5. FATS AND OILS PACKAGE FOR CONSIDERATION BY EWG</p> <p>Uruguay no tiene observaciones.</p> <p>En referencia a los puntos b. el Proyecto de revisión de las Directrices sobre la incertidumbre en la medición (CXG 54-2004) (Apéndice III) (en el trámite 8), y c. el Anteproyecto de revisión de las Directrices generales sobre muestreo (CXG 50-2004) (Apéndice IV) (en el trámite 5), Uruguay no tiene observaciones.</p>	
<p>10. 'A minimum of ten (10) lots and ten individual subsamples per segment is needed to estimate the within segment variation to allow design of a sampling plan. Laboratory samples must be tested at least in duplicate to allow estimation of the component of variation due to measurement error, unless estimates are available from other sources such as test method validation.' (Section 53, p50).</p> <p>This is similar to validation method described in Eurachem UfS Guide (2019)iii, where the minimum is 8 lots (sampling targets), again with duplicated analytical measurements. It gives an estimate of the uncertainty from sampling, which is also include in line 2 of the tree-nut examples in the box below (page 51). The caption for this box states: Codex Standard 193 shows the breakdown of the total variation for aflatoxins in tree-nuts, with a focus on the sample preparation and testing; the variation due to sampling includes both between and within segment variation'.</p> <p>Your definition of 'segment' is 'A portion of the lot to which inference will be made' (page 48) which is equivalent to 'sampling target' in the Eurachem UfS Guide* (i.e. 'Portion of material, at a particular time, that the sample is intended to represent'). It follows that the 'within-segment variation' should be included in MU (as UfS), but the 'between-segment variation' should be excluded from MU (*Equations 1 & 2 in Section 9.3). It would be helpful to explain this in your Guidance.</p>	<p>EURACH EM</p>

* M H Ramsey, S L R Ellison and P Rostron (eds.) Eurachem/EUROLAB/ CITAC/Nordtest/AMC Guide: Measurement uncertainty arising from sampling: a guide to methods and approaches. Second Edition, Eurachem (2019). ISBN (978-0-948926-35-8). Available from <http://www.eurachem.org> (Section 9.3)

In addition, defining the role of FNC as “If the characteristic does not follow a normal distribution in the lot” and stating “The main advantage of FNC inspection plans is that they can be used even when the underlying quality characteristic is not normally distributed” is a duplication of the same statement. The second statement is not an advantage but is a characteristic intrinsic to this definition of FNC. The advantages of the FNC approach have therefore been overstated.

Page 55, section 67: Why not recommending a data transformation before compliance assessment? Once transformed the data would be following a normal or approximately normal distribution and FNC approach would not be necessary. Is this not a simpler option?

‘an additional allowance is required to compensate for variation in the lot to enable such assessments to be made’ (Section 47, p48). The meaning of this text is unclear. Does it refer to the random error (i.e. part of MU) that remains even when the sample is assumed to be unbiased?

Definitions: ‘Laboratory sample = A portion of the sub-sample that is measured’. (Table in Section 48. P49.) This term is more usually defined as: Sample as prepared for sending to the laboratory and intended for inspection or testing. ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999).

Add Definition that is more usual for what was described for ‘Laboratory sample’: Test sample = Sample, prepared from the laboratory sample, from which the test portions are removed for testing or analysis. (IUPAC (1990) Nomenclature for sampling in analytical chemistry (Recommendations 1990), prepared for publication by Horwitz W, Pure and Applied Chemistry, 62, 1193–1208).

‘The aim of acceptance sampling inspection is to make good decisions about a lot given when measurement errors are present whereas the purpose of conformity assessment is to say something about the true values of the samples tested, allowing for measurement uncertainty’ (Section 58, p52). The description of Conformity Assessment (CA) is inaccurate, as it enables a decision to be made about a whole lot (not just about the samples measured). Furthermore, CA can allow for the fact that the samples are never fully representative of the lot, as there is uncertainty in the measurement values that arises from both the sampling and the chemical analysis. *

* A. Williams and B. Magnusson (eds.) Eurachem/CITAC Guide: Use of uncertainty information in compliance assessment (2nd ed. 2021). ISBN 978-0-948926-38-9. Available from www.eurachem.org

“the focus lies on the identification and evaluation of the main components of measurement uncertainty.” (page 26 section 10). This is correct and in line with ISO GUM. However, in Section 11, there is a contradictory statement “While performing a measurement, it is important to consider all

possible uncertainty components which will influence the result of the measurement.” These Codex Guidelines should avoid providing such lack of consistency on advising or recommending for best procedures.

‘The classification of uncertainty contribution as either ‘significant’ or ‘negligible’ based upon the error-variance ratio exceeding 10% is arbitrary, or indeed ‘subjective’ as you state. A more satisfactory approach is to always include MU, as it will always be present. Furthermore, it is essential to combine the analytical and sampling* sources of uncertainty into the total measurement uncertainty (MU), (*within-segment, not between-segment, see point #20 above). If MU is too large to give FFP and reliable conformity assessment, then it can be reduced most effectively by decreasing the MU from the dominant component (Section 16.5 of Eurachem UfS Guide).**

**M H Ramsey, S L R Ellison and P Rostron (eds.) Eurachem/EUROLAB/ CITAC/Nordtest/AMC Guide: Measurement uncertainty arising from sampling: a guide to methods and approaches. Second Edition, Eurachem (2019). ISBN (978-0-948926-35-8). Available from <http://www.eurachem.org> (Section 9.3)

Page 54, section 64: Any actual variability (expressed as a variance) while following a given measurement procedure cannot be smaller than the random variability while following the same measurement procedure, although the estimated variance can be. The equation is correct but the text therefore needs some correction.

Page 54, Figure 6: Bias can be acting on the same side as the specification limit (as this figure illustrates) or on the opposite side to the specification limit. A note should be added informing readers of this possibility. In the second case, the probability of nonconforming would be reduced after accounting for the bias.

Page 52, section 58: Using “good” associated with a conformity assessment about a lot is not adequate. Replace by “correct decision”. “Good” or “bad” compliance decisions for whom (consumer or producer)?

On page 46 section 41 one should read “standard uncertainty, u ” not “standard deviation, s ”. Most commonly all sources of uncertainty are not included in a simple standard deviation, which includes only random variability. The equation should read $x \pm ku$

‘As measurement uncertainty has the potential to affect both producer’s and consumer’s risks it is necessary to consider both measurement and sampling uncertainty in the design of sampling plans’ (Section 67, p55). It is hopefully true, but not explicit, that both aspects (MU and UfS) are being considered, but their separate listing suggests that UfS is in not being included within MU.

Definitions: ‘Sub-sample = A portion of the composite sample that is sent to the laboratory’ (Table in Section 48. P49). This term is more usually defined as: Selected part of a sample. Note: The subsample can be selected by the same method as was used in selecting the original sample, but need not be so. ISO 3534-2: 2006 Statistics – Vocabulary and symbols - Part 2: Applied statistics. International Organization for Standardization, Geneva (2006).

<p>'..is the precision parameter for the beta distribution' (Section 43, p47) – the printed symbol does not match that used for theta in the equation above.</p> <p>For variables plans, information about the measurement error, specifically the repeatability, reproducibility and possibly bias is required to enable the effect of measurement errors on the performance of sampling plans to be investigated and adjustments to be made if required' (Section 21, p40). Measurement uncertainty (MU) includes all of these, and should also include UfS, to assess the performance of sampling plans. The ME (and MU) considered still seem to ignore the contribution from sampling to the measurement process. In a previous reply from CCMAS it was argued that UfS is being allowed for in the Guidelines, but this was questioned by Eurachem (and no reply has been received to clarify this).</p> <p>There is now a clearer explanation of Measurement Uncertainty (MU) and Measurement Error (ME), but the Acceptance Sampling (AS) method is still based upon ME rather than MU.</p>	
<p>Please find here several comments by the Global Organization for EPA and DHA Omega-3s (GOED)</p> <p>(1) Methods of analysis / performance criteria (Appendix II)</p> <p>4.3 Commodity, Fish oils. Current status indicated as “endorsed by CCMAS, for consideration by CCFO”</p> <p>Comments:</p> <ul style="list-style-type: none"> - Fish oils, Acidity: Acid Value – GOED supports - Fish oils, Peroxide Value – GOED supports - Fish oils, Phospholipids – GOED supports - Fish oils, Triglycerides – GOED supports <p>4.4 Commodity, Fish oils. Current status indicated as “Referral to CCFO: For consideration and reply by CCFO”</p> <ul style="list-style-type: none"> - Fish oils, Fatty acid composition, AOCS Ce 2-66 and AOCS Ce 1a-13 – GOED supports - Fish oils, Fatty acid composition, AOCS Ce 1b-89, Type III selection – GOED supports - Fish oils, Fatty acid composition, AOCS Ce 2b-11 and AOCS Ce 1i-07, Type III selection – GOED supports - Fish oils, Fatty acid composition, AOCS Ce 2b-11 and AOCS Ce 1j-07 – GOED supports - Fish oils, Fatty acid composition, ISO 12966-2 and AOCS Ce 1i-07 – GOED supports - Fish oils, Fatty acid composition, AOCS Ce 2-66 and AOCS Ce 1i-07 – GOED supports - Fish oil, vitamin A (all-E retinol and 13-Z-retinol) – GOED supports - Fish oil, vitamin A (all-E retinol) – GOED supports - Fish oil, vitamin D (vitamin D2 and D3) – GOED supports <p>Not reflected in the Circular Letter are the suggestions GOED made in CCMAS40 through the submission of a conference room document. We like to bring these comments back to attention, in case CCMAS would find these of interest:</p>	GOED

1. Moisture and volatile matter in Fish oils

Comment – The method ISO 662 “Moisture and volatile matter” (listed in Appendix I, Part A – Method “ISO 662”) for the determination of moisture and volatile matter in fish oils is a very old method and is not suitable for newer type of fish oils that are commercialized today. Such oils today consist of refined fish oils and concentrates of EPA and DHA that are very sensitive to oxidation, and for sure will rapidly oxidize (if not handled under an inert atmosphere) under the conditions specified in this method which involving drying at 105oC. Such refined and concentrated fish oils will gain weight due to a very fast oxidation instead of losing weight as expected by the loss of moisture and volatile matter, and this method is therefore not universally useful for fish oil anymore today. It is possible that at the time the method was developed and adopted by Codex its suitability was limited to crude fish oils, for which ISO 662 remains in use today.

For this reason, GOED recommends the inclusion of suitable methods for the commodity “Fish Oils” for the determination of water/moisture content that are based on Karl Fischer titration, notably AOCS Official Method Ca 2e-84 (“Moisture, Karl Fischer Method”), European Pharmacopoeia method 2.5.12 (“Water: Semi-Micro Determination”), and the United States Pharmacopoeia method 921 (“Water Determination”).

In addition, CCMAS may want to consider addressing the suitability of ISO 662 for “Fats and Oil (all)”. It is probably more correct to limit the recommended use of this method only for specific named oils, for example:

The recommendation to use ISO 662 for the determination of “Moisture and volatile matter” should be maintained for “Named Vegetable Oils.”

The recommendation to use ISO 662 for the determination of “Moisture and volatile matter” should be maintained for “Olive Oils and Olive Pomace Oils.”

2. Methods for the quantification of omega-3 fatty acids in Fish oils

Comment – GOED provided suggestions for suitable methods for the quantification of omega-3 fatty acids, including EPA and DHA, in fish oils that should be included. Although these were discussed during CCMAS40, we cannot find the need to their inclusion reflected in the document that this Circular Letter refers to. It is important to understand that methods for “Fatty Acid Profile” in fish oils are not quantitative methods, and only express fatty acid levels in area percent (the detector response) for a quick assessment of fatty acid profiles for trading purposes. Specific methods are needed however to make an accurate quantification of the fatty acids of interest in fish oils, which are the omega-3 fatty acids, in particular EPA and DHA. For your reference, here are our suggestions for CCMAS:

For the category “Fish oils,” a number of methods for the determination of “Fatty acid composition” are listed. In our opinion, suitable methods for the quantification of the omega-3 fatty acids, EPA, DHA and the Total Omega-3 Fatty Acids in fish oils should be added (in addition to AOCS Method Ce 1i-07 which is already provided). These are:

- European Pharmacopoeia method 2.4.29 “Composition of Fatty Acids in Oils rich in Omega-3 Acids”
- United States Pharmacopoeia method USP401 “Fats and Fixed Oils.”

Whereas we support elevating method AOCS Ce 1i-07 to a Type II method status, both mentioned pharmacopoeial methods are considered equally suitable for the quantification of EPA, DHA and Total Omega-3 fatty acids in fish oils (composed of triglycerides, as well as omega-3 ethyl ester concentrates prepared from fish oils). These methods are used on par with the AOCS Ce 1i-07 method in the Laboratory Proficiency Program that

<p>AOCS organizes annually for laboratories to measure EPA, DHA and Total Omega-3 Fatty Acids. Both pharmacopeial methods could be considered a Type II method, and method validation details are retained by the respective pharmacopoeial organizations.</p> <p>3. Arsenic, under the category "Fats and Oils (all)"</p> <p>The DIN EN 1557 method should be listed for the Commodity Fats and Oils (all), as a type II or type III method, in order to include a method for inorganic arsenic in oils, such as fish oils, which need to abide by a maximum limit for inorganic arsenic. This is the information we provided for CCMAS40:</p> <p>Codex has adapted the following requirement for arsenic** in edible oils, in CXS 193-1995 (General Standard for Contaminants and Toxins in Food and Feed, see page 45); "If the As-tot concentration is below the maximum levels (ML) for As-in, no further testing is required, and the sample is determined to be compliant with the ML. If the As-tot concentration is above the ML for As-in, follow-up testing shall be conducted to determine if the As-in concentration is above the ML."</p> <p>For fish oils covered by CXS 329-2017, the ML is for (As-in). Hence, we suggest including a recommended method for the analysis of inorganic arsenic (As-in) that is suitable for fish oils (including krill oil):</p> <ul style="list-style-type: none"> • Analysis of foodstuffs - Determination of inorganic arsenic in algae - Atomic absorption spectrometry-hydride technique (HGASS) after acid extraction (adoption of the standard of the same name, DIN EN 15517, September 2008 edition) - DIN EN 15517 <p>** Definition of Arsenic: total (As-tot) when not otherwise mentioned; inorganic arsenic (As-in); or other specification.</p> <p>Further comments:</p> <p>(2) The revised Guidelines on Measurement Uncertainty (CXG54-2004) (Appendix III) (at step 8) GOED has no comments</p> <p>(3) The General Guidelines for Sampling (CXG50-2004) (Appendix IV) (at step 5) GOED has no comments</p>	
<p>IDF support the adoption of methods of analysis as recorded in the Appendix II of this circular letter with the following corrections or remarks:</p> <ul style="list-style-type: none"> - Part 1.1: at the end of the table the IDF reference number '229' is missing in the last three lines. The correct reference to the method should read: AOAC 2011.14 / ISO 15151 IDF 229 - Part 1.8: Numeric performance criteria for methods of analysis for copper and iron in milkfat products. The report may not reflect clearly that CCMAS agreed to the numeric criteria for iron and copper and to include examples of applicable methods including those currently listed in CXS 234 as examples for further review at its next session. IDF suggest adding a note to the list of example 'To be discussed at the next CCMAS session'. - IDF will share with the Codex Secretariat a number of editorial suggestions for consistency, and corrections in particular in relation with the footnotes. 	IDF/FIL