

# CODEX ALIMENTARIUS COMMISSION



Food and Agriculture  
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World Health  
Organization

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Agenda Item 14

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## JOINT FAO/WHO FOOD STANDARDS PROGRAMME

### CODEX COMMITTEE ON PESTICIDE RESIDUES

53<sup>rd</sup> Session

(Virtual)

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#### DISCUSSION PAPER ON THE REVIEW OF MASS SPECTROMETRY PROVISIONS IN THE GUIDELINES ON THE USE OF MASS SPECTROMETRY FOR THE IDENTIFICATION, CONFIRMATION AND QUANTITATIVE DETERMINATION OF PESTICIDE RESIDUES 9CXG 56-2005) AND THE GUIDELINES ON PERFORMANCE CRITERIA OF PESTICIDE RESIDUES IN FOOD AND FOODED (CXG 90-2017)

(Prepared by the Electronic Working Group  
chaired by the Islamic Republic of Iran and co-chaired by India)

#### Background

1. The *Guidelines on the use of mass spectrometry (MS) for identification, confirmation and quantitative determination of residues* (CXG 56-2005) were adopted by the 28<sup>th</sup> Session of the Codex Alimentarius Commission (CAC28, 2005)<sup>1</sup>. Since then there have been many improvements in MS and separation techniques, liquid chromatography (LC) and gas chromatography (GC) that are often used with MS which are not included in CXG56.
2. On the other hand, the *Guidelines on performance criteria for methods of analysis for the determination of pesticide residues in food and feed* (CXG 90-2017) were adopted by CAC40 (2017)<sup>2</sup> and includes criteria for the use of mass spectrometry in identification and confirmation of pesticide residues.
3. At 50<sup>th</sup> Session of the Codex Committee on Pesticide Residues (CCPR50, 2018), Iran presented a proposal for new work on the revision of CXG56 and highlighted the gaps in the Guidelines that required addressing e.g. the title does not match the content of the Guidelines; the Guidelines focus on confirmation test only; the Guidelines cover mass spectrometry in general which requires more detail guidance; apparent editorial mistakes in the text; etc. The Committee acknowledged the relevance of the issue and agreed that a discussion paper could look into this need to revise CXG56 and their harmonization with CXG90 and other relevant Codex documents.<sup>3</sup>
4. CCPR51 (2019) considered the proposal on exploring the possibility to merge CXG56 and CXG90 into one single document, and if feasible and appropriate, to proceed with the withdrawal of CXG56. The Committee noted general support for the proposal. It was also noted that CXG90 was developed recently and covers not only MS but also other modern techniques for the determination of pesticide residues while taking into account the needs and capacities of developing countries. This spirit should be maintained when considering the possible merging of the two guidelines to avoid overlapping of documents. The first step would so be to explore whether the provisions on MS in CXG90 are sufficient to meet the needs of members and to examine the need and room for improvement CXG90, if appropriate by taking into account relevant information from CXG56.
5. CCPR51 thus agreed to re-establish the Electronic Working Group (EWG), chaired by Iran and co-chaired by Costa Rica with the following Terms of Reference (TOR):<sup>4</sup>
  - (i) To determine if CXG90 adequately covers mass spectrometry and if so, to propose revocation of CXG56.
  - (ii) If there are provisions from CXG56 that could be relevant but not included in CXG90, to look into the feasibility to merge the two documents and
    - if appropriate to present a proposal for new work, and

<sup>1</sup> ALINORM 05/28/41, Appendix V

<sup>2</sup> REP17/CAC40, Appendix III

<sup>3</sup> REP18/PR50, paras. 164 - 166

<sup>4</sup> REP19/PR51, paras. 180 - 186

- if possible to present an outline of the merged Guidelines for consideration at CCPR52.2
6. CCPR52 (2021) noted general support to continue working on this matter. In general, delegations supported revocation of CXG56 and the transfer of relevant provisions to CXG90 if appropriate to avoid duplication and expressed various views for further consideration by the EWG.
7. CCPR52 therefore agreed to re-establish the EWG, chaired by Iran, and co-chaired by India with the following TOR:<sup>5</sup>
- (i) To determine if CXG 90-2017 adequately cover mass spectrometry and if so, to propose revocation of the CXG 56-2005.
  - (ii) If there are provisions from CXG 56-2005 that could be relevant but not included in CXG 90-2017, to look into the feasibility to merge the two documents, and:
    - If appropriate to present a proposal for new work, and
    - If possible, to present an outline of the merged guidelines for consideration at CCPR53.
8. This document sets out the discussion in the EWG and the recommendations of the EWG.

#### **PROCESS FOLLOWED BY AND DISCUSSION IN THE EWG**

9. The EWG was established and worked through the online forum. The EWG members are listed in Appendix I to this document.
10. A draft document was circulated to the EWG members to seek their comments on the following questions:
- (i) revoke CXG 56-2005 and reach a consensus on CXG 90-2017 covering all parts of CXG 56-2005 or
  - (ii) there are provisions from CXG 56-2005 that could be relevant but not included in CXG 90-2017.
11. In response to these questions, comments from 7 member countries were received i.e. Australia, Chile, Canada, Costa Rica, Uruguay, United States and Thailand. Most of the member countries believed that the mandate of the EWG had not been properly discussed and correctly followed. USA requested that the EWG engaged in discussion before proposing to revoke CXG56 or to merge the two documents if some relevant provisions of CXG-56 are to be included in CXG-90.
12. Costa Rica while agreeing with Australian considered that a complementary confirmation of identity could be achieved by: (i) using an alternative chromatographic column; (ii) another ionization technique (e.g. chemical ionization); (iii) controlling other reaction products of certain ions by double mass spectrometry (MS/MS or MS<sub>n</sub>) or (iv) controlling other ions with a higher mass of resolution. (page 2 of guideline CXG 56-2005, fifth paragraph).
13. Chile noted that CXG90 is robust and updated document and those techniques which are not considered in CXG90 could be omitted, since the current use of this technique could be low. Uruguay agreed that CXG90 adequately covers MS and that CXG 56-2005 should be revoked. Australia noted that, if CXG56 should be revoked, the EWG should then consider if there are provisions from CXG 56-2005 that could be transferred into CXG90. These points should be agreed upon before the development of a new work proposal.
14. In order to achieve consensus about the revocation of CXG 56-2005 or merging both the documents together, a questionnaire was circulated to the EWG member as presented in Appendix I to collect EWG members' comments and suggestions
15. In response, to the second round of comments (replies to the questionnaire), comments from five country members were received i.e. USA, Chile, Canada, Uruguay and China. Majority of the member countries believe that there are some extra instrumentation and derivatization techniques used in pesticide analyses in CXG56 that are not included in CXG-90 while CXG90-2017 adequately covers method performance criteria related to mass spectrometry.

#### **CONCLUSIONS**

16. The revocation of CXG-56 could be proposed due to the lack of enough information about mass techniques related to identification, confirmation and quantitative determination of pesticide residues. In addition new techniques such as tandem mass spectrometry as well as high resolution mass spectrometry (HRMS) are not covered in this guideline.

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<sup>5</sup> REP21/PR52, paras. 195 - 197

17. CXG 90-2017 adequately covers this information and contain:
  - General principles of confirmatory tests in determination of pesticide residues especially in multi-residue methods and demonstrating advances of MS technique among other confirmative techniques both for GC and HPLC applicable pesticides.
  - Criteria for selection of recognition ions for identification, confirmation and quantitative detection
  - Interpretation of results and Identification and Confirmation of residues.
  - Advances and limitations of quantification of identified residues.
18. Considering that, CXG 90-2017 covers not only MS but also other modern techniques for the determination of pesticide residues revocation of CXG 56 is proposed to avoid overlapped documents.
19. There are some provisions from CXG 56-2005 that could be relevant but not included in CXG 90-2017. Suggestions of some member countries are worth consideration and need to be included in the existing guideline CXG 90-2017.

#### **RECOMMENDATIONS**

CCPR is invited to consider:

- i) Revocation of CXG 56-2005, and
- ii) Those provisions from CXG 56-2005 that some members found relevant but not included in CXG 90-2017, might be considered in the next revision of CXG90.

**APPENDIX I**

**Comments on the proposed revision of the Guidelines on performance criteria of pesticide residues in food and feed (CXG 90-2017) and Guidelines on the use of mass spectrometry for the identification, confirmation and quantitative determination of pesticide residues (CXG 56-2005)**

The comments may be offered in the following format:

Name of the member and country: \_\_\_\_\_

No	Content of Document CAC/GL 56-2005	Page no. of CAC/GL 56- 2005	Whether already addressed in CXG 90-2017 (Yes/No)	If yes, Para No. of CXG 90-2017	If no, whether required to be included in CXG 90-2017	Comments/ Remarks
1	Confirmatory Tests	1				
2	Gas Chromatography/ Mass Spectrometry (GC/MS)	2				
3	HPLC And HPLC-MS	2,3				
4	THIN LAYER CHROMATOGRAPHY (TLC)	3				
5	DERIVATISATION (a) Chemical reactions	3				
	(b) Physical reactions	3				
	(c) Other methods	4				
6	Table 6. Detection methods suitable for screening (Phase 1) and confirmation (Phase 2) of residues	5				
7	Figure 2. Schematic Representation of Screening and Confirmation (Phase 1 and Phase 2) for Pesticide Residues	6				

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