

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

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

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PROPOSAL FOR NEW WORK ON GUIDANCE FOR MONITORING THE PURITY AND STABILITY OF REFERENCE MATERIAL OF MULTI-CLASS PESTICIDES DURING PROLONGED STORAGE

(Prepared by India as Chair of the Electronic Working Group)

KEY COMMENTS RECEIVED – Subsequent changes in the project document

1. In Section 1 (Purpose and scope of new work), few countries sought clarification about the scope of guidelines whether these are applicable to Certified Reference Materials (CRMs) or Reference materials (RMs) or both. It has been clarified that the scope of the new work will focus on the Reference Material purity specified by the Reference Material Provider (RMP). The new work includes monitoring the stability of RMs (solids/liquids) of known purity, and the guidelines are proposed to be applicable to all categories of pesticides.
2. Suggestions made by different countries regarding extending the use of RMs beyond RMPs indicated expiry dates for pesticide residue analysis in food and environment samples have been incorporated in Sections 2 (Relevance and timeliness of the work) and 3 (Main aspects to be covered). After the expiration date, the RMs retaining the purity specified in the CoA can be used as RMs or as quality control materials (QCMS) for the analysis of pesticides provided that these are stored at $\leq -18\text{ }^{\circ}\text{C}$
3. Changes have been incorporated in Section 4.1 (General criterion), Section 5 (The relevance to the Codex strategic objectives), and Section 6 (Information on the relation between the proposal and other existing Codex documents as well as other ongoing work) to include **stock solutions** in the scope of the new work.
4. Changes have been made in section 4.2.1 (Scope of work and establishment of priorities between the various sections of the work) to prioritize work step-wise (i-v) to ensure that the stability of RM purity (concentration accuracy) of the individual pesticide specified by the RMP in the CoA remain valid within and beyond the expiry dates. The revised section clearly states that the proposed storage conditions ($\leq -18\text{ }^{\circ}\text{C}$) for RMs can also be used to monitor the stability of the stock solutions of RMs.
5. Certain sentences in the document have been reframed or modified. General and specific comments/suggestions offered by the various member countries have been included to improve the project document.

CONCLUSIONS

Comments received have enhanced the clarity of the project document. The proposed new work on monitoring the stability of RMs purity will provide guidance to the pesticide residues analysis laboratories for extended use of pesticide RMs beyond their expiry dates. These guidelines will be applicable to pesticide RMs of known purity determined by the RMP.

RECOMMENDATIONS

The EWG recommends CCPR54 to:

1. consider the proposal for new work on monitoring the stability of reference material purity of pesticides during prolonged storage.
2. Review the outline of the proposed new work, if agreement is reached to proceed with the new work
3. Establish an EWG, chaired by India, co-chaired by Iran and Argentina, working in English and Spanish, to develop guidelines for monitoring the stability of reference materials purity of pesticides during; prolonged storage based on the outline provided for consideration by CCPR55

The EWG will base its discussion on the outline of the guidelines as contained in Appendix III to CX/PR 23/54/14 while taking into account comments submitted in reply to CL 2023/38-PR and key points raised in plenary in relation to this document. The EWG would present a proposed guidelines to CCPR55 for consideration.

APPENDIX I
PROJECT DOCUMENT
PROPOSAL FOR NEW WORK ON
GUIDANCE FOR MONITORING THE STABILITY OF
REFERENCE MATERIAL PURITY OF PESTICIDES DURING PROLONGED STORAGE
(For consideration by CCPR)

1) Purpose and scope of new work

Pesticide residues in food commodities have become a worldwide agricultural trade-concern which has led to enforcement of strict pesticide regulations. More than 1200 pesticides are used to control the pests on different food commodities. Analyses of multi-class pesticides in the food chain ~~with reliable measurement and accuracy~~ requires their specific Reference Materials (RMs) ~~of known chemical purity~~ specified by the Reference Material Producers (RMPs) to ensure safe food, safety, food quality and the safe environment. However, limited shelf life based on short expiry period, and high recurring cost of RMs act as major impediments for pesticide residue analysis. These problems are magnified for multi-pesticide residue analysis by testing laboratories situated in developing countries as they are required to allocate a large part of their funds to the frequent procurement of expensive RMs as their use is restricted by the date of expiry recommended by the RMPs in the certificate of analysis. Further, due to supply chain constraints, some laboratories receive RMs close to their expiration date as per the CoA. In such situations the laboratories are forced to buy new standards and prepare new stock solutions more frequently than necessary. This leads to insurmountable extra work and increased laboratory costs, especially for compounds for which stability is normally not questionable. Additionally, shipping of RMs by the suppliers to laboratories increase the acquisition time for procurement creating hurdles in sustainable pesticide residue control program. Many times, countries cannot afford frequent purchase of high-cost RMs for their pesticide residue and food safety work. Many RMs stay stable even after the date of expiry recommended by the RMPs as mentioned in the CoA and continue to retain their valid purity as per CoA. Some studies have also reported that if RM's are stored at better storage conditions than recommended by the manufacturer, the RM's are stable for much longer than the expiry date mentioned by the RMP's. Such RMs may therefore be allowed to be used in the laboratories even after the RMPs' indicated expiry date if laboratory checks are in place to demonstrate that they are stable and continue meeting the purity requirements.

Purity, characteristic of a reference material, when stored under specified conditions, refers to the measurement of the quantity of a prevalent component of a substance when only that component is present. In a measurement quality system, RMs with certified purity are required to determine the amount of reference chemical in the sample.

The lack of data on the purity of RM's during prolonged storage and absence of SOP's for their analysis prevent their use beyond the expiry period. ~~More than 1200 pesticides are used to control the pests on different food commodities. For quantitative determination of the residues of these pesticides on food matrices, their specific RMs are required by the testing laboratories. However, limited shelf life and short expiry date typically recommended by Reference Material Producers (RMPs), hence, and high recurring cost of RMs act as major impediments for pesticide residue analysis.~~

~~These problems are magnified for multi-pesticide residue analysis laboratories situated in developing countries as they are required to allocate a large part of their funds to the frequent procurement of expensive RMs whose use is restricted by the date of expiry recommended by the RMPs in their CoAs.~~

~~Further, due to supply chain constraints, some laboratories receive RMs close to their expiration date as per the CoA. In such situations the laboratories are forced to buy new standards and prepare new stock solutions more frequently than necessary. This leads to insurmountable extra work and increased laboratory costs, especially for compounds for which stability is normally not questionable. Additionally, shipping of RMs by the suppliers to laboratories increase the acquisition time for procurement creating hurdles in sustainable pesticide residue control program. Many times, countries cannot afford frequent purchase of high-cost RMs for their pesticide residue and food safety work. Many RMs stay stable even after the date of expiry recommended by the RMPs as mentioned in the CoA and continue to retain their valid purity as per CoA. RMs are therefore entitled for continuous use in the laboratories even after RMPs' indicated expiry date as valid RMs as long as proper laboratory checks are in place to demonstrate that they continue meeting they meet the purity requirements.~~

The proposed guidelines on monitoring the stability of purity of RMs will provide guidance to the pesticide residues analysis laboratories for extended use of the RMs with which are stable and have acceptable purity beyond their expiry date. These guidelines will be applicable to RMs (solids/liquids) of ~~multi-class~~ pesticides of known purity specified by the RMP and their stock solutions. ~~in the certificate of analysis (CoA)~~

~~2) — The guidelines will also be applicable to stock solutions of RMs of individual pesticides.~~

3)2) Relevance and timeliness of the work

The accurate determination of pesticide residues in food commodities is required for food safety, fixation of MRLs for pesticides, overcoming barriers due to pesticide residues in traded commodities and various other applications. RMs with specified purity are required for purity assessment and accurate qualitative and quantitative analysis (trueness and/or precision) of pesticide active ingredient(s) in technical materials and formulations, stock solutions, working solutions, and for the analysis of pesticide residues in food commodities and in soil and water samples.

Qualitative and quantitative determination of pesticide residues in food can be achieved by using high quality RMs, validated analytical protocols, comprehensive quality systems, and competent staff engaged in operating the analytical equipment. RMs that retain their purity even after their expiry period as recommended by the RMPs, may be continued to be used after verification of their purity as specified in the CoA.

At present no guidelines on extending the validity of RMs beyond the expiry period are provided by regulatory agencies of different countries.

The proposed work on guidance for monitoring the stability of RM purity of ~~multi-class~~ pesticides before and after RMPs' indicated expiry dates for extended use of RMs is thus relevant and timely for consideration by the Codex Committee on Pesticide Residues (CCPR).

4)3) Main aspects to be covered

The central objective is to use the RMs beyond their specified expiry dates for pesticide residue analysis in food and environment samples. The main aspect to be covered is to develop comprehensive harmonized guidelines which enable the laboratories to monitor the stability of RM purity of pesticides during prolonged storage (before and after expiry). If the purity of the RMs is found acceptable, their use as RMs after expiry may be continued to be allowed.

5)4) Assessment against the *Criteria for the Establishment of Work Priorities*

4.1 General criterion

General criterion of the proposed new work is to verify the purity of RMs as specified by RMP before and after expiration by standardized analytical protocols so that such materials that retain their purity as per the CoA even after expiry are continued to be used as valid RMs.

Another criteria aspect of the proposed work is to monitor the stability of stock solutions used for multiple pesticide residue analysis to ensure that the concentrations of individual pesticides continue to be accurate within the indicated expiry date, so that the stock solutions continue to be valid for the accurate and reliable determination of pesticide residue levels.

4.2 Criteria applicable to general subjects

4.2.1 Scope of work and establishment of priorities between the various sections of the work

The CCPR recognizes the significance of RMs in the analysis of pesticide residues in food commodities and in the soil and aquatic environment. However, the lack of data on purity of RMs during prolonged storage, and absence of SOPs for their analysis prevent their use beyond the expiry period as recommended by RMPs. Scope of the work shall therefore be prioritized stepwise as (i) developing SOP including a set of procedures along with storage conditions for monitoring the purity of the RMs ~~and, (ii) determining their purity~~ at different time intervals within the expiry period, and beyond the expiry period, (iii) ascertaining whether the purity of the RMs is acceptable for its use beyond the expiry date recommended by the RMP, ~~and~~ (iv) developing guidelines for use of RMs by laboratories beyond their RMP indicated expiry date, ~~and v) developing guidelines for monitoring the stability of stock solutions used for multiple pesticide residues analysis to ensure the concentration accuracy of the individual pesticides remain valid within the expiry dates.~~

4.2.2 Amenability of the subject of the proposal to standardization

The expiry period of the pesticide RM is determined based on their type, class, chemical structure, and storage conditions like temperature, humidity etc. Pesticides are used globally, and these guidelines would be applicable to all the laboratories with varying levels of technology. There are certain European Reference Materials (ERM) Application Notes that describe some practical aspects associated with the handling and use of RMs. The proposal is thus considered amenable to standardization.

4.2.3 Consideration of the global magnitude of the problem or issue

Since pesticides are used globally, the development of standard analytical protocols for monitoring the purity of RM of pesticides for extended use beyond their expiry is of global relevance to ensure food safety and fair-trade practices for agricultural commodities moving in international trade.

No impediment to international trade is foreseen by having an international agreed guidance on the use of verified expired RMs in pesticide residue analysis in exportable food commodities.

5. The relevance to the Codex strategic objectives

The Codex Strategic Plan 2020-2025 underpins the high priority that continues to be placed on food safety and quality by FAO and WHO and guides the Codex Alimentarius Commission in carrying out its responsibilities to fulfil the mandate of protecting consumer health and ensuring fair practices in the food trade. The use of RMs for pesticides is important for the establishment and implementation of Codex maximum residue limits (CXLs) and achieving the goals of the Codex. The development of guidance on monitoring the purity of RM of pesticides during prolonged storage [and the monitoring of stability of stock solutions used for multiple pesticide residue analysis](#) is relevant to the Codex strategic objectives.

6. Information on the relation between the proposal and other existing Codex documents as well as other ongoing work

No guidance document related to monitoring the stability of purity of RMs [and their stock solutions](#) during prolonged storage is either available or being currently considered by the Codex.

7. Identification of any requirement for and availability of expert scientific advice

No provision of scientific advice is required for the development of the guidelines.

8. Identification of any need for technical input to the standard from external bodies

For the elaboration of this document, the advice from FAO, WHO and the JMPR Secretariat will be taken as and when required. Other documents issued by international organizations such as the relevant SANTE, ISO guidelines and research reports in literature have been used as a reference to develop the guidance document.

9. The proposed timeline for completion of the new work, including the start date and the proposed date for adoption by the Commission

Subject to approval by the Codex Alimentarius Commission, the Guidelines will be considered at CCPR54 (2023) and finalized for adoption by CAC in 2026 or earlier.