CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 7 **CRD08**

June 2024

Original language only

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PESTICIDE RESIDUES

55th Session
Chengdu, Sichuan province, People's Republic of China
3-8 June 2024

Comments submitted by Brazil

Agenda Item 7 **CX/PR 24/55/6 – CL 2024/45-PR**

Guidelines for monitoring the purity and stability of reference materials and related stock solutions of pesticides during prolonged storage (at Step 4)

CCPR55 - Distribution of CL 2024/45-PR - Guidelines for monitoring the purity and stability of reference materials and related stock solutions of pesticides during prolonged storage

Brazil would like to provide general and specific comments on this document.

GENERAL COMMENTS

This document is important for laboratories analyzing pesticide residues in food, the publication of that will be very useful, as it will be a reference for the proposed procedure.

SPECIFIC COMMENTS

Brazil suggests the need for stability studies to also involve the evaluation of mixed standard stock solutions, these solutions are Certified Reference Materials (CRMs) in which multiple pesticides are present.

This comparison of mixed stock standard solutions can perfectly be carried out according to the procedure described in Paragraph 9 in "approach 1".

The need to consider the possibility of carrying out the procedure with mixed solutions arises because, usually, most pesticide residue analysis laboratories use multi-residue methods in their scope that analyze a few hundred pesticides simultaneously, for this reason it is used the acquisition of CRM of mixed solutions for routine analyzes (as is the case of laboratories that analyze official programs PNCRC and PARA in Brazil).

APPENDIX I

SCOPE AND OBJECTIVE

7. This document is applicable to reference materials (RMs) of pesticides and their individual stock solutions of known purity specified by a reference material producer (RMP).

Brazil would like to make it clear that stock solutions prepared by the laboratory itself are also included in this document, and not just isolated standard solutions sold as CRMs.

8. These guidelines will enable the pesticide residue laboratories to overcome the shortcomings associated with RMs and use them beyond their expiry dates mentioned in the product information sheet. After the expiration date, the RMs retaining the purity specified in the product information sheet can be used as RMs or as quality control materials (QCM) for the analysis of pesticides provided that these are stored under conditions specified in the guidelines.

Brazil would like to make it clear that these standards can be used as RMs or CRMs, as long as the conditions specified in the certificates are guaranteed. They would be the same standards, only with expanded validity as long as the premises of this document are proven (in General Criteria).

8. To avoid any cross contamination or degradation of RMs, the vials must be placed in airtight capped tube/sealed pouch and immediately stored in the freezer at conditions better than those recommended by RMPs; preferably at subzero temperature. The stock solutions must also be stored in airtight capped glassware (preferably volumetric flask). Storage conditions should be monitored with appropriately calibrated equipment and should be controlled and recorded.

Volumetric flask is not glassware for storage and storage of solution. Brazil suggests that this "preference" be removed from the document.

Approach 2: Verification of purity of neat standards of pesticide reference materials during prolonged storage (not suitable for verification of stock solutions)

18. To verify the purity of the RM, chromatographic assay should be performed as per the analytical conditions mentioned in the product information sheet/CoA by the RMP. The verification of RM purity is performed by considering the purity (in terms of percent peak area) mentioned in the product information sheet/CoA as the reference value.

19. Prepare fresh stock solution of the newly acquired neat standards of RMs and internal standard (a different unexpired RM) of appropriate concentration in a suitable organic solvent. Appropriate concentration will depend on the response of the RM in the detector. Generally, for HPLC-DAD/GC-FID, good response is obtained between 10 ppm to 100 ppm. Higher concentration of the RM may lead to saturation of the detector.

20. The standard solution of the RM prepared at an appropriate concentration from the stock solution is injected into the instrument (HPLC-DAD /HPLC-UV /GC-FID/ LC-MS and GC-MS in full scan mode) as per the analytical conditions mentioned in the product information sheet/CoA and the percent peak area so obtained is recorded as percent purity. A minimum of five replicate measurements should be performed to obtain a mean value of percent purity and the %RSD should be ≤ 10%. The instrument should be calibrated as per the conditions recommended by the manufacturer.

Brazil considers that the change to the requirement that the chromatographic test must be carried out following the analytical conditions mentioned in the product information sheet/CoA. Most producers use chromatographic techniques with conventional detectors (DAD or FID, for example and described in items 19 and 20) and pesticide analysis laboratories use techniques coupled to mass spectrometry. It should then allow the use of the chromatographic technique already used by the laboratory on a routine basis, even if, in this case, analysis is carried out by FullScan, as suggested by the document. Also in this part, it would be interesting to leave it open that any other validity standard could be uses for comparison (facilitating the routine of laboratories, which should only acquire these "internal standards" with a certain frequency).

Definitions

Brazil considers it would be good to have internal standard defined as part of the Definitions.

Approach 2

Brazil considers that Approach 2 might need some additional details (ex. how is peak area calculated).