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

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PESTICIDE RESIDUES

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Comments on Agenda Item 8: Draft Guidance on performance criteria for methods of analysis for the determination of pesticide residues at Step 7, submitted by Brazil, Kenya, Peru, Uganda and African Union

Brazil

Item 12.c - *"range of sample matrices covered by the validation (e.g. "cucurbits, root vegetables, citrus")."*

Adequacy of the sentence

Matrices used on the validation (e.g. "citrus, soybeans, potato").

Reason

The range of sample matrices covered by the validation could be defined per representative commodity categories (e.g. High water content, high oil content,...). For example, a validation in potato does not cover only root vegetables analysis, but could be used for all samples from high starch group instead.

Item 13. - *"Ideally, selectivity should be evaluated to demonstrate that no interferences occur which detrimentally affect the analysis. It is impractical to test the method against every potential interferent, but it is recommended that common interferences are checked by analyzing a blank for every batch of samples and reagents."*

"Matrix interferences are evaluated by analyses of samples known to be free of the analytes."

Adequacy of the sentence

Ideally, selectivity should be evaluated to demonstrate that no interferences occur which detrimentally affect the analysis. It is impractical to test the method against every potential interferent, but it is recommended that common interferences are checked by analyzing a reagent blank per validation. In case of reagents and/or solvents are changed between batches of samples, additional reagent blank could be performed.

Matrix interferences are evaluated using samples known to be free of the analytes.

Reason

- If no reagents and solvents are changed between batches, so only one reagent blank could be enough to prove that no interferences are found using the current procedure.

- Better writing.

Clarifications:

- It is important to clarify if the 'blank' is the control sample or the reagents blank.

- It is important to clarify how to conduct the 'analyte-to-analyte interferences' check in case it is required.

Item 14. - *"To minimally estimate rates of false positives and negatives during method validation, an adequate number (suggested >5 each) of diverse matrix blanks (not from the same source) should be analyzed along with spiked matrices at the analyte reporting level."*

Adequacy of the sentence

To estimate rates of false positives and negatives during method validation, at least one control sample (duplicate) for each validated matrix and one blank of reagents should be analyzed along with spiked matrices at the analyte reporting level.

Reason

Based on the instruments used for residue analysis, which have high selectivity, and considering that confirmatory techniques are also required, the controls described would be enough to prove the selectivity.

Clarifications:

- It is not clear the number of samples and replicates are required. It is important to clarify the number of control/spiked samples and if any replicates are required.

Item 16.c - *"If individual residuals deviate by more than ±20%, statistical consideration of outliers should be made, possibly leading to re-analysis of the sequence if quality control criteria are not met."*

Adequacy of the sentence

If individual residuals deviate by more than ±30%, statistical consideration of outliers should be made, possibly leading to re-analysis of the sequence if quality control criteria are not met.

Reason

Especially for calibration levels near the instrumental LOQ, deviates higher than 20% could be found due to the instrument variation at these levels.

Besides that, as we also consider other parameters in order to prove the linearity, such as, the correlation coefficient (r2) that should be ≥ 0.98 (r ≥ 0.9875), the calculated concentration of each standard injected might be checked and should not differ by more than 30% from the respective nominal value especially in the range of the found residues.

Clarifications:

- It is important to clarify the number of replicates per point are required?

Item 18. - "*The value of the intercept should be close to zero".*

Clarifications:

Is this really required? For some compounds, this is not possible.

In theory, close to zero is the "ideal" scenario, but there're deviations that shift the curve from its ideal condition and, despite all careful work, it's difficult to avoid random errors. I think this paragraph could be considered as a suggestion/ recommendation and not a requirement.

Item 23.

Clarifications:

- What would be the acceptable parameters and variability values?

Item 24. – "*In both cases, the assumption needs to be checked if the analyte level is expected to vary substantially"*

Clarifications:

- Please clarify what means "*substantially".*

Item 38. e - "*If a method is being used for compliance testing (i.e. if a commodity is complaint with an established MRL) the MRL (or CXL) must be one of the spiking levels".*

Change of Criteria

"If a method is being used for compliance testing (i.e. if a commodity is complaint with an established MRL) the MRL (or CXL) must be included on analytical range validated".

Reason

The method would need to re-validated every time the MRL changes.

Item 40. - "*Analysis of incurred matrix to support method validation is strongly encouraged."*

Clarifications:

Is required analysis of incurred residue in a method validation? How this should be done?

Item 47. e - "*All measured reagent and matrix blank samples should be shown to be free of carry-over, contamination, and/or interferences above 20% of the LOQ*".

Change of Criteria

All measured reagent blank should be free of interferences above 20% of the LOQ, nevertheless for matrix blank samples it is considered 30% of the LOQ.

Reason

According to international guidelines (e.g. SANCO 3029, SANCO 825 and ENV/JM/MONO(2007)17), blank values in the area of analytical interest (untreated samples and procedural blanks) have to be determined from the matrices used in fortification experiments and should not be higher than 30% of the LOQ. If this is exceeded, detailed justification should be provided.

Kenya

Background:

During the 48th session of the CCPR, there was a general arrangement on the proposed draft on Guidelines on Performance Criteria for Methods of Analysis for the Determination of Pesticide Residues in Food (REP 16/PR – Appendix XI). However, it was noted that in light of the changes that have been made in the draft delegations reconsider the document at the nest session CCPR49 for completion and final adoption at Codex Alimentarius Commission.

Position:

Kenya thanks the EWG led by USA and co-chaired by China and India.

We note that the paper has improved substantially. However, we propose the following amendments as tabulated:

Document Paragraph / Line	Specific comment	Rationale
Title and subsequent body of the document	We propose the use of "Guidelines" consistently in the document; as in the title of the document	There is lack of consistency in the use of both "Guidelines" and "Guidance" interchangeably it the document. This is confusing to the user of the document.
Paragraph 2	We propose the removal of the word "degradants". We propose the inclusion of "residue definition" in the definitions that would be used throughout the document without giving reference to "…degradants, products during formed during analysis, isomers.." The use of the "residue definition" should be maintained consistently throughout the document (Paragraph 2, 5 and 12)	The use of many terms brings confusion and inconsistency to the intended residue definition. It is recalled that although metabolites, degradation products and impurities are included in the general definition of pesticide residues, this does not necessarily mean that metabolites or degradation products should always be included in the residue definition for enforcement (MRLs) purposes or for estimation of dietary intake (STMR, HR). (FAO Manual on the Submission and Evaluation of Pesticide Residues Data, Third Edition; 2016)

Document Paragraph / Line	Specific comment	Rationale
Paragraph 10, Line 6	We propose to delete the word "rates" and replace with "frequency"	It is more statistically relevant to use frequency here.
Paragraph 13, Line 1	We propose the deletion of the word "detrimentally" with "significantly"	This is a more scientific way of describing the matter in discussion.
Paragraph 33, Line 3	We propose the deletion of the word "not as great as" with "less than"	The change offers clarity in a scientific way of expressing the issue.
Paragraph 43, Line 2	We propose the word "affordable" be deleted from the sentence.	The affordability of proficiency test programs is relative and hence should be deleted.
General comment	We propose the inclusion of "Abbreviations and Glossary of terms" in the document, to include terms used within the document e.g. TOF (Time of Flight), found in Table 1.	This will improve on the easy use of the document, especially for first time readers of the document.

Other general comment:

Given the importance of the testing of pesticide residues in agricultural commodities in trade, we feel that the Guideline needs to be taken up for laboratories undertaking pesticide residue analysis. In view of this, it is important to take into consideration the needs for developing countries and provide a simplified guidance on the implementation of these Guidelines. After evaluating the relevance of this document for the determination of pesticide residues and its impact on regulatory practices for enforcement of MRLs for pesticides, a practical approach on how to implement the guidance is needed to assist developing countries. We hope that other international agencies such as the International Atomic Energy Agency (IAEA) would design practical training programs to target especially developing countries to ease the implementation of these Guidelines.

Perú

La Comisión Técnica sobre Residuos de Plaguicidas del Perú, inicialmente acordó de manera colegiada, respaldar el Anteproyecto de directrices sobre criterios de rendimiento para métodos de análisis para la determinación de residuos de plaguicidas, el cual proporcionará a los miembros del Codex un documento en el que se expongan los métodos y criterios de rendimiento de las prueba de análisis para los residuos de plaguicidas en los alimentos.

Sin embargo, al documento se introdujo una serie de cambios de redacción para mejorar la precisión y claridad del documento y eliminar referencias a otros documentos adoptados por la Comisión del Codex Alimentarius o desarrollados por otras organizaciones internacionales. En este sentido se está consultando internamente con nuestros expertos y otras partes interesadas pertinentes a fin de evaluar plenamente los requisitos técnicos de las Directrices; debido que se trata de una cuestión particularmente delicada para los países en desarrollo, por lo que las Directrices no deberían poner en peligro la capacidad de los laboratorios para determinar los residuos de plaguicidas en esos países.

Al respecto, los miembros de la Comisión Técnica identificaríamos con algunos expertos de laboratorios, los cambios propuestos y las observaciones o justificaciones pertinentes sobre un párrafo específico (en las categorías de cuestiones de redacción, cuestiones de fondo, cuestiones técnicas y traducción) o a nivel del documento (observaciones generales) antes del 17 de abril del presente y tener una posición más clara sobre este anteproyecto.

Uganda

Position: Uganda appreciates the work of the EWG over the years. We would like to make the following general and specific comments on the draft guidelines for Performance Criteria for the Methods of Analysis for the Determination of Pesticides Residues in Food

General comment:

We propose the addition of abbreviations and Glossary to the guidelines; this will enhance the easy use of these guidelines

Specific comment:

Title and body of the document: We suggest the use of the word Guidelines should be applied throughout the document.

Rationale: The words guidelines and guidance are being used interchangeably

African Union

Issue: During the 48th session of the CCPR, there was a general arrangement on the proposed draft Guidelines on Performance Criteria for Methods of Analysis for the Determination of Pesticide Residues in Food (REP 16/PR – Appendix XI). Considering the changes that had been made, CCPR48 agreed to reconsider the document during CCPR49 with the view of completing the work and recommending its adoption at step 8.

General Comments:

AU thanks the Electronic Working Group led by USA and Co-chaired by China and India.

The guidelines on method performance is key in helping to demonstrate compliance of laboratories to the quality management system.

Given the sensitivity and importance of testing pesticide residues in agricultural commodities in trade, the guidelines must be consistent with existing laboratory capacities in developing countries. In advancing these guidelines, consideration should be given to the "relevance of these guidelines to the determination of pesticide residues and their impact on regulatory practices for enforcement of MRLs for pesticides".

Specific comments:

Document Paragraph / Line	Specific comment	Rationale
Title and subsequent body of the document	We propose the use of "Guidelines" consistently in the document; as in the title of the document	There is lack of consistency in the use of the terms "Guidelines" and "Guidance"
Paragraph 2	We propose the removal of the word "degradants". We propose the inclusion of "residue definition" in the definitions that would be used throughout the document without giving reference to "…degradants, products during formed during analysis, isomers.." The use of the "residue definition" should be maintained consistently throughout the document (Paragraph 2, 5 and 12)	The use of many terms could led to confusion and inconsistency to the intended residue definition.
Paragraph 10	Replace the word "rates" with "frequency"	It is more statistically relevant to use frequency here.
Paragraph 13, Line 1	Replace the word "detrimentally" with "significantly"	This is a more scientific way of describing the matter under discussion.
Paragraph 17,	More clarification is needed on the use of $1/x$ and $1/x^2$	Further clarification is needed to understand the need for the use of this approach in calibration.
Paragraph 33	Replace the word "not as great as" with "less than"	To conform with accepted terminology
Paragraph 43, Line 2	Delete the word "affordable" from the sentence.	The affordability of proficiency test programs is relative and hence should be deleted.