CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 9 **CRD23**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON PESTICIDE RESIDUES

48th Session
Chongqing, P.R. China, 25-30 April 2016

PROPOSED DRAFT GUIDELINES ON PERFORMANCE CRITERIA FOR METHODS OF ANALYSIS FOR THE DETERMINATION OF PESTICIDE RESIDUES - CX/PR 16/48/13

Comments of 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 potatodoes 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 reagent blank in every batch of samples."*

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 blankper validation. In case of reagents/solvents are changed between batches additional reagent blank could be performed.

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.

Item 14. –"*To minimally estimate rates of false positives and negatives during method validation, an adequate number (suggested >20 each (SANTE/11945/2015)) 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 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.

Item 16.b – "T*he calibration standards should be evenly spaced over the concentration range of interest"*

Adequacy of the sentence

The calibration standards should be evenly spaced over the concentration range of **interest and the calibration range should encompass the entire concentration range likely to be encountered**

Item 18 –*"Forcing calibration curves through zero is also worth considering or may be warranted to reduce bias at low concentrations."*

Change of criteria

The curve should not be forced to pass through the origin (through zero).

Reason

All instruments and analytes are associated with background signal noise, which is expected to be non-zero.

Item 27 line 10 and 11 – "*The analytical method must be sensitive enough so that the LVL for each analyte is at or below the current CXL."*

Adequacy of the sentence

The analytical method must be sensitive enough so that the LVL for each analyte is at or below the current CXL. The validation range should cover theexisting CXL.

Item 38 line 5 – "*to check the recovery and **precision"***

Adequacy of the sentence

to check the recovery and **intermediate precision condition of measurement**

Item 39 -*"...or by determination of the recovery of analyte fortified into known blank sample material"*

Adequacy of the sentence

(...) or by determination of the recovery of analyte fortified into known blank sample material (**analysis must be performed in repeatability and intermediate precision conditions).**

Item 39 -*"Furthermore, recoveries >120% can only be explained through an interferent or bias that should be addressed in the method, including re-assessment of calibration"*

Adequacy of the sentence and Change of criteria

Furthermore, recoveries >120% must not be acceptable and a more accurate method must be used.

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

Question

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

Item 45 e – "*All measured reagent and matrix blank samples must be shown to be free of carry-over, contamination, and/or interferences above 20% of the LOQ".*

Change of Criteria

All measured reagent blank must 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.

Item 45 e – "*The retention time of the analyte in the extract should correspond to that of the reference value (point a.) within ±0.2 min or 0.2% relative retention time, for both gas chromatography and liquid chromatography".*

Adequacy of the sentence

Examine the retention time for each analyte in each sample. If a peak response is detectable, the retention time of that peak should not have shifted by more than +/- 5% from the average retention time of the bracketing standards injected prior to and after the sample.

If the retention time appears to have shifted by more than +/- 5%, steps must be made to confirm the identity of the analyte in the chromatogram. The effects of matrix may be evaluated by examining the procedural or instrument recovery. If the procedural or instrument recovery has a similar retention time shift, and if the retention time shift has been confirmed, the analyte identification can be accepted. If the procedural or instrument recovery does not exhibit a retention time shift, it can be assumed that the peak identification is incorrect unless steps are taken to confirm the peak identification.

Reason

The retention time should not shift by more than +/- 5% between consecutive standard injections. Not only within ±0.2 min because it depends on other factors, such as shape of the peak and run time for each method.

Item 46 –*"Examples of analytical techniques that may be suitable to meet criteria for confirmatory analytical methods are summarized in Table 3."*

Correction

It should be Table 2 instead of Table 3.