

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
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Agenda Item 9

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

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COMMENTS on the proposed draft Guidelines on performance criteria specific for methods of analysis for determination of pesticides residues in food at Step 3 submitted by El Salvador, European Union, Kenya and African Union

El Salvador

We support the development of the proposed draft submitted by the electronic Working Group led by the United States. The development of this guidance will facilitate the selection of methods of analysis for the different matrices used by each country and at the same time it will strengthen the technical criteria for the selection of appropriate methods and implementation of guidelines to reduce trade barriers.

European Union

The European Union wishes to provide the following comments:

The EU doesn't support the advancement of the document in its current state and is therefore in favour of the re-establishment of the electronic working group. In general it is proposed to re-structure the document, re-formulate the definitions and remove the redundancies.

Since feed is mentioned in the text of the document (page 2, MRL definition and page 3, point 1 of scope) it should also be included in the title (Food and Feed).

Although the focus of the document is on pesticide residues in food, in some parts of the document references or examples are made from analytical fields other than pesticide residues. For example: the use of LC-DAD (page 10, Table 4), the reference to soil (page 6, point 10.i), the reference to microbiological growth inhibition (page 7, paragraph 12), "total arsenic" (page 4, point 10.a) or ammonia (page 4, point 10.b).

The definition of 'Determination' (page 2) could be deleted as it does not match in all cases with the use of this word in the text. The word determination is also used in the document title but probably with a different meaning than that used under 'Definitions'.

The definition of 'Limit of detection' (page 2) includes the concept of 'beta error', which is rather used in the veterinary drugs field and not in the pesticide residues field.

Paragraph 10 (items A-M, pages 4-6) is based on the 'IUPAC's Harmonized guidelines for single-laboratory validation of methods of analysis'. This section should be harmonised with the specific guidelines in the other sections.

In paragraph 10.a on 'Applicability' (page 4), also a list of commodity groups and representative commodities should be mentioned.

As regards 'Calibration and linearity' (paragraph 10.c page 4), the draft guideline should also consider calibration models other than the linear one. Furthermore the EUMS are of the view that multi-level calibration should have three or more levels¹.

'Limit of quantification' (paragraph 10.h page 6) should be defined as the signal or measurement value that will produce estimates having a specified relative standard deviation (RSD), commonly 10% (or 6%). It is proposed to only include this definition under the section 'Definitions' (page 2). The wording 'Limit of determination' should be removed from the text.

For the 'Within-laboratory reproducibility' (paragraph 17, page 7) a criterion of $\leq 20\%$ is proposed.

As regards 'Mean recovery and precision criteria for plant and animal matrices' (Table 1, page 8), the EUMS suggest using the criteria as defined in EU Guideline SANCO/12571/2013 G5.

¹ EU Guideline SANCO/12571/2013 C17

'A quantitative analytical method should be demonstrated at both initial and extended validation stages, as being capable of providing acceptable mean recovery values at each spiking level and for at least one representative commodity from each relevant group (see Annex 1). Acceptable mean recoveries are those within the range 70–120%, with an associated repeatability $RSDr \leq 20\%$, for all compounds within the scope of a method. The method-LOQ is the lowest spike level of the validation meeting these method performance acceptability criteria. In certain cases and typically with multi-residue methods, recoveries outside this range may be accepted. Exceptionally, where recovery is low but consistent (i.e. demonstrating good precision) and the basis for this is well established (e.g. due to analyte distribution in a partitioning step), a mean recovery below 70% may be acceptable. However, a more accurate method should be used, if practicable. Within-laboratory reproducibility ($RSDwR$), which may be determined from on-going QC-data in routine analyses, should be $\leq 20\%$, excluding any contribution due to sample heterogeneity.'

As regards the identification criteria that must be met for regulatory purposes in the case of chromatography-MS/MS, the retention time of the detected analyte peak must be within 0.1 min of the contemporaneously analyzed analyte reference standard peak (paragraph 23, page 9), but in paragraph 27 of page 10 the criterion is different: 'the retention time of the analyte in the extract should correspond to that of the calibration standard (may need to be matrix-matched) with a tolerance of ± 0.2 min, for both gas chromatography and liquid chromatography'. The EUMS support a tolerance of 0.2 minutes.²

The values of the tolerances of ion ratios of ion transitions given in Table 3 differ from those in Paragraph 23, page 9: 'the ratios of peak areas for each ion transition must match the ratios of the reference standard(s) within $\pm 10\%$ absolute for one transition or $\pm 20\%$ absolute for two transitions'. The EUMS support the values given in Table 3.³

Paragraph 29 (p 10) is not relevant for the item 'performance characteristics of confirmatory methods'.

Table 4 (p 10) is proposed to be deleted since it is in contradiction with the information on confirmatory methods in the text.

It would be useful to add a paragraph on the stability of standard solutions to the document.

Kenya

Definition

G. DETECTION LIMIT (LOD)

Comment

We have observed that the abbreviation (LOD) does not reflect the word 'detection limit' and has different meaning. We propose to use the word LOD and delete the word detection limit [DL]

In broad terms, the LOD is the smallest amount or concentration of analyte in the test sample that can be reliably distinguished from zero. For analytical systems where the validation range does not include or approach it, the detection limit does not need to be part of a validation.

H. LIMIT OF DETERMINATION OR LIMIT OF QUANTIFICATION (LOQ)

Comment:

The two are different and should be separated and the definition should be aligned with guidance document SANCO/12571/2013.

There is need to distinguish the limit of detection from the limit of determination and limit of quantification

African Union

Position: AU agrees with the proposed guidance on performance criteria specific for methods of analysis for determination of pesticide residues. In view of the fact that the proposed comments have been incorporated in the draft (references from SANCO/12571/2013). However, AU would also like to propose that due attention should be given to the progress of work by the Codex Committee on Methods of Analysis (CCMAS) in the development of guidance on performance criteria for methods of analysis.

Rationale: This document is very important and meet all the requirements for performance criteria. However, it should be harmonise with the guidance document on performance criteria being developed by CCMAS.

² EU Guideline SANCO/12571/2013 D2

³ EU Guideline SANCO/12571/2013 D9.