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European Union Comments

CODEX COMMITTEE ON PESTICIDE RESIDUES

48th Session

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AGENDA ITEM 9

PROPOSED DRAFT GUIDELINES ON PERFORMANCE CRITERIA SPECIFIC FOR METHODS OF ANALYSIS FOR DETERMINATION OF PESTICIDES RESIDUES IN FOOD

(CX/PR 16/48/13)

European Union Competence
European Union Vote

The European Union (EU) would like to thank the electronic working group chaired by the United States and co-chaired by China and India for the preparation of the document on 'Proposed draft Guidelines on performance criteria specific for methods of analysis for determination of pesticides residues in food.'
The EU wishes to provide the following specific comments:

Page	Paragraph	Comment	Rationale
2	Index	~~A.~~ Defining the Purpose of the Method and Scope ~~B.~~ Supplementing other Codex Alimentarius Commission Guidelines ~~C.~~ Method Validation **Annex I:** Definitions **Annex II:** References	Editorial change: adapt any title in the index as it is reported in the text
3	5	~~Ideally r~~**Residue** analytical methods should be able to measure al components of the residue definition.	In all cases analytical methods should be able to measure al components of the residue definition.
4	14	(suggested > 20 each (~~SANTE/11945/2015~~)	The reference to SANTE/11945/2015 should be deleted as it doesn't suggest analysing 20 matrix blanks, only 20 samples of different commodities spiked at the SDL.
4	14	Validations of screening methods (presence/absence analyses) are discussed in paragraphs ~~31-33~~ **32-34**.	
5	16a	replicate determinations at **three concentrations or single determinations at** five or more concentrations should be performed.	It is not a prerequisite to perform double injections at each level in case of a calibration at five concentration levels.

Page	Paragraph	Comment	Rationale
5	16d	~~the calibration by interpolation between two levels is acceptable providing the difference between the 2 levels is not greater than a factor of 10 and providing the response factors of the bracketing calibration standards are within acceptable limits. The response factor of bracketing calibration standards at each level should not differ by more than 20% (taking the higher response as 100%).~~	This contradicts with point a. Initial validation criteria are mixed with calibration for quantification. Bullet points a to d within paragraph 16 apply, as specified in the document, for the initial method validation (for univariate linear calibration). For that reason, bullet point d should be removed from paragraph 16.
7	25	The initial validation should be carried **out** at the targeted limit of quantification (LOQ) or reporting limit of the method, and at least one other higher level, for example, 2-10x the targeted LOQ or the MRL.	Editorial change
7	26	By long-standing definition among analytical chemists, the LOQ is the concentration at which the average signal/noise ratio (S/N) equals 10 in the analysis. ~~At the LOQ in a normal (Gaussian) statistical distribution, the analyte will be determined 95% of the time in the sample using the method.~~	It is proposed to delete the sentence because it is confusing.
7	~~27~~	~~0.1 mg/kg~~ **0.01 mg/kg**	Cfr. Previous version.
8	31	~~SANCO/12571/2013 Appendix C~~ **SANTE/11945/2015 Appendix C**	The most up to date version of the document.
8	34	~~SANCO/12571/2013 Annex A~~ **SANTE/11945/2015 Annex A**	The most up to date version of the document.
9	36	The requirement to recover a range of different pesticide residues in one extraction increases the potential for compromised selectivity in MRMs compared to single ~~analyte~~ **residue** methods.	According to the glossary in Annex I, it should be quoted as "single residue method".
9	37	(i.e. trueness - see F ~~p.7~~ and precision - see G ~~p.7~~).	Editorial change. Both chapters can be found on p. 6. However, it is not necessary to quote the pages.
9	38	When the residue defintion includes two or more analytes, ~~then whenever possible,~~ the method should be validated for all analytes	It is sufficient to say that the method "should" be validated for all analytes. Residue definitions are established to be applied in monitoring, a guidance should not suggest that it is an option.
9	40	**Analysis of incurred matrix to support method validation is strongly encouraged.** For interpreting recoveries, it is necessary to recognize that analyte**s** spiked into a test sample may not behave in the same manner as the biologically incurred analyte (pesticide residue). In many	There is a contradiction because recoveries cannot be calculated when working with incurred matrix. Therefore, it is recommended to move the sentence "Analysis of incurred matrix to support method validation is strongly encouraged." to the beginning of the

Page	Paragraph	Comment	Rationale
		situations, the amount of an extracted incurred residue is less than the total incurred residues actually present. This may be due to losses during extraction, intra-cellular binding of residues, the presence of conjugates, or other factors that are not fully represented by recovery experiments using analyte-fortified blank matrices. ~~Analysis of incurred matrix to support method validation is strongly encouraged.~~ At relatively high concentrations, analytical recoveries are expected to approach one hundred percent. At lower concentrations, particularly with methods involving extensive extraction, isolation, and concentration steps, recoveries may be lower **than at higher concentrations.** Regardless of what average recoveries are observed, recovery with low variability is desirable so that a reliable correction for recovery can be made to the final result, when required. Recovery corrections should be made consistent with the guidance provided by the CAC/GL 37-2001.	paragraph.
10	45	~~SANCO/12571/2015~~ **SANTE/11945/2015**	The most up to date version of the document.
10	45	**The following identification criteria should be met:** a……. b…… etc.	From the previous version a sentence to introduce the list of points is missing.
10	45 b.	Ion ratio reference values are to be set in the same way as in Section 45 a. The different ions used for identification must co-elute and have similar peak shapes. The ion from the calibration standard with the higher average intensity is to be used as the denominator in the ion ratio, expressed in % (due to signal fluctuations, **matrix effects, etc …, deviations of** ion ratios up to **30%** ~~130%~~ **are acceptable**) ~~before the ions should be reversed in setting the ion ratio).~~	The use of 130% is confusing; it is proposed to refer to the percentage of deviation. The parameter 30% is also specified in Table 1.
11	46	Methods based on high-resolution mass spectrometry are considered to provide improved reliability through precise measurement of the mass/charge of the ion that can be obtained using unit-resolution mass spectrometry techniques. Different types and models of mass spectrometric detectors provide different degrees of selectivity, which relates to the confidence in	The last three lines are not specifically related to high resolution mass spectrometry, so in order to ensure that no possible confusion could be created, it is suggested to delete those lines. The most up to date version of the document SANCO/12745/2015 is SANTE/11945/2015.

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		identification. The criteria for identification based on ~~SANCO/12745/2015~~ **SANTE/11945/2015** are provided in Table 1. They should only be regarded as guidance criteria for identification, not as absolute criteria to prove presence or absence of a compound. ~~For example, other acceptable regulatory criteria for analyte identification based on ion ratios entail ±10% or ±20% absolute differences (not relative) for one or two sets of ions, respectively, vs. the reference ion ratios for the analyte(s).~~	
12	Table 1	~~SANCO/12745/2015~~ **SANTE/11945/2015**	The most up to date version of the document.
14	Annex I	**Analyte protectant:** Compounds that strongly interacts to fill active sites in the gas chromatographic system, thereby reducing the analyte ~~interaction s~~ **interactions** with those active sites and yielding less peak tailing or losses, thus a higher analyte response.1	Editorial change.
16	Annex II: References	**2** CAC PM 24 2015 1pageEN	The document "Codex Alimentarius Commission Procedural Manual, 24th edition" quoted at line 2 of the table has no reference number. All the following numbering should be adjusted.
17	Annex II: References	~~19 - Miskolc, Hungary Nov 1999~~	Reference 19 could be deleted: it is a section of the book quoted in reference 20. It seems redundant to quote both 19 and 20.
18	Annex II: References	~~25 - Lehotay,S.J., Sapozhnikova, Y., Mol, H.G.J.~~ **~~Lehotay, S.J., Mastovska, K., Amirav, A., Fialkov, A.B., Martos, P.A., Kok, A. d., Fernández-Alba, A.R.~~**	The article has been already quoted as reference 13. The reference 25 should be deleted (moreover, the authors are also incorrect).
18	Annex II: References	**ENV/JM/MONO(2014)20 "OECD Guidance Document for Single Laboratory Validation of Quantitative Analytical Method-Guidance used in support of pre-and post-registration data requirements for plant protection and biocidal products."**	This document should be added to the reference list. It is quoted in paragraphs 11 and 20.