codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 7 (a) **CX/PR 07/39/5**
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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PESTICIDE RESIDUES
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DISCUSSION PAPER ON APPLICATION OF PRACTICES ON THE ESTIMATION OF UNCERTAINTY OF RESULTS

(Prepared by IAEA)

INTRODUCTION

1. While finalizing the Guidelines on the Estimation of Uncertainty of Results, the Committee at its 39th Session had agreed that a circular letter would be issued requesting information from governments on application practices currently in use at the national or regional level on measurement uncertainty in reporting test results and its application in relation to the risk management of pesticide residues in food (ALINORM 06/29/24, para. 177).

2. By the Circular Letter (CL 2006/9-PR) member governments were requested to provide their comments and information on the above subject matter, however reply was received only from Australia.

BACKGROUND

3. In many laboratories the application of the principles of measurement uncertainty continues to be a major obstacle towards meeting the technical requirements laid out in ISO Standard 17025.

4. There is still insufficient understanding of the measurement uncertainty concept. Different from other mathematical and statistical concepts like the setting of MRLs, it has to be elaborated individually causing considerable work for each laboratory.

5. The complexity of residue analytical procedures incurs a multitude of influencing steps and factors which are diverse in nature, mostly difficult to define and to integrate into the estimation/ mathematical calculation of measurement uncertainty values.

6. Problems encountered in the calculation of uncertainty budgets are related to thousands of relevant pesticide/ crop combinations, as well as dozens of analytical methods used in pesticide residue analysis. The large number of combinations makes the individual estimation of all uncertainty budgets practically impossible[1].

7. A comprehensive concept on measurement uncertainty including sampling, the kind of commodity/ sample size and the analytical procedure itself, is not covered by existing guidelines in terms of the practical application of the concept, particularly for residue analysis in foodstuffs.

8. There is no single or unified approach for estimating measurement uncertainty to make the application of the concept simple and easily applicable for relevant residue analytical methods.

9. There are continuous reservations against applying the metrology based “bottom up” approach due to the difficulties encountered in this approach in residue analysis. However, the need for expressions of ranges around analytical results is widely recognized, but alternative approaches are deemed necessary towards feasible solutions in this matter.

SOLUTION APPROACHES

10. Harmonization of the application of measurement uncertainty principles in the analysis of pesticide residues should be sought. An easy-to-use and practical-oriented guidance to be applied straight forward for the determination of pesticide residues in foodstuffs should be available.

11. Alternatively the integration of theoretical and practical approaches, “bottom up” and “top down”, including simple estimation of common figures for universal steps, for weighing, volumetric steps etc could be promoted, e.g. by designing spreadsheet based templates that could be used by laboratories to calculate their uncertainty budget. Experiences of institutions going in that direction show to date that this does not really simplify the complexity of the matter due to the huge number of factors and variables.

12. The application of a radically simplified supporting concept, like the utilization of results of Proficiency Testing schemes in conjunction with intralaboratory method validation data looks promising in the interest of a simplified concept which then would be easily applicable in any laboratory.

13. Using a generalized “top down” approach would in many cases mean larger related standard deviations than if calculated “bottom up” for each pesticide-commodity combination individually. Larger uncertainty values[2], e.g. ± 25% or ± 50%, in some cases would impose a lower action level with regard to compliance with MRLs. In the vast majority of cases a ± 50% approach will cover the uncertainty to be expected. On the other hand for the laboratories this simplification would mean enormous savings in terms of efforts, time and resources.

RECOMMENDATIONS

14. A simplified alternative to calculating uncertainty values “bottom up” such as a specific and practical-oriented guideline tailored for the estimation of measurement uncertainty and specifically applicable for the determination of pesticide residues in foodstuffs should be made available.

15. A generalized approach of measurement uncertainty values should be utilized as calculated from the results of Proficiency Testing schemes, e.g. as laid out in the publication of L. Alder et al., based on the Horwitz approach of empirical Relative Standard Deviations.

16. In case laboratories feel uncomfortable with larger ranges, or if narrower uncertainty ranges would be justifiable the conventional “bottom up” calculation may be used to specifically generate more distinct uncertainty values as needed.

PROPOSAL

17. It is proposed to tailor the application of the measurement uncertainty concept particularly for the field of pesticide residue analysis of foodstuffs from “bottom up” towards “top down”. Empirical standard deviations based on the results of Proficiency Testing schemes and the Horwitz formula should be taken as the basis for the measurement uncertainty budget. Laboratories in that case would not need to eventually determine thousands of individual values of measurement uncertainties for any individual active ingredient to be determined.

18. Method validation for verifying the recovery values and associated standard deviations of analytical methods complying with the quality requirements will need to be performed. Once such analytical parameters are verified by validation studies, the respective methods can be regularly used in the context of the existing laboratory quality assurance and management environment.

REFERENCES

[1] Lutz Alder et al. Estimation of Measurement Uncertainty in Pesticide Residue Analysis. Journal of AOAC International. Vol 84, No 5, 2001, 1569-1577

[2] Document No SANCO/10232/2006. QUALITY CONTROL PROCEDURES FOR PESTICIDE RESIDUE ANALYSIS. 24/March/2006