

codex alimentarius commission



FOOD AND AGRICULTURE
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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PESTICIDE RESIDUES

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RECOMMENDATIONS FOR METHODS OF ANALYSIS

PROPOSED DRAFT AMENDMENTS TO THE INTRODUCTION SECTION OF THE RECOMMENDED METHODS OF ANALYSIS FOR PESTICIDE RESIDUES

(Prepared by the Netherlands)

BACKGROUND

1. The Codex Committee on Pesticide Residues in its thirty-second session discussed recent developments of principles and practices of method validation. The Committee agreed that methods appropriately validated in a single laboratory can be considered suitable for the determination of compliance with Codex MRLs in addition to methods that have been validated to through collaborative trial. It was noted that the current texts in Volume II of the *Codex Alimentarius* would need an up-date to accommodate more appropriately for single laboratory validated methods. The Executive Committee in its 47th session approved new work on amendments to the introduction section of the Recommended methods of analysis for Pesticide Residues.

PROPOSED AMMENDMENTS

2. Amendments are based on the report of a Joint AOAC/FAO/IAEA/IUPAC Expert Consultation on Single Laboratory Validation on Analytical Methods for Trace-level Concentrations of Organic Chemicals¹ held in Miskolc, November 1999. The process of method validation will be described in the guidelines on Good Laboratory Practice in Pesticide Residue Analysis, while the elements relevant to the choice of a method with respect to its suitability for checking compliance with Codex MRLs can be inserted in the Introduction section of the List of Recommended Methods of Analysis for Pesticide Residues.

3. The Committee is invited to comment on the Proposed Draft Amendments given in Annex, especially on the element of single laboratory validated methods and the criteria and explanatory text on the hierarchy of methods with respect to their scope.

¹ Report of the AOAC/FAO/IAEA/IUPAC Expert Consultation on Single Laboratory Validation on Analytical Methods for Trace-level Concentrations of Organic Chemicals, in Principles and Practices of Method Validation, A.Fajgelj and A. Ambrus (editors), The Royal Society of Chemistry, London, 2000, ISBN 0-85404-783-2, pages 179-252 also available at this URL: http://www.iaea.org/trc/pest-qa_val2.htm

ANNEX

PROPOSED DRAFT AMMENDMENTS FOR THE INTRODUCTION SECTION OF THE LIST OF RECOMMENDED METHODS OF ANALYSIS FOR PESTICIDE RESIDUES**1. INTRODUCTION****1.1 Scope**

Hereunder are given analytical methods which can be applied to the determination of pesticide residues for regulatory purposes. The list, given in para.2, is not exhaustive and methods not mentioned in the list can also be applied, provided that they can be shown to produce valid results, by the analyst using them.

1.2 Criteria for the selection of analytical methods

Whenever possible, the the following criteria were used when selecting analytical methods:

- (i) Available through national or international standards organizations, books, manuals, open literature, on the internet or GSPF;
- (ii) collaboratively studied or known to have been validated in a number of laboratories. For single laboratory validated methods validation must have taken place according chapter 4 of Volume 2 of the *Codex Alimentarius*, as a minimum;
- (iii) capable of determining more than one residue, i.e. multi-residue methods;
- (iv) suitable for as many commodities as possible at concentrations at or below the specified MRLs;
- (v) applicable in a regulatory laboratory equipped with routine analytical instrumentation. [This differs according to the lab and the country]

Preference was given to gas chromatography or high performance liquid chromatography as the separation step for the methods. Under certain conditions however, methods using less sophisticated procedures, such as thin-layer chromatography or spectrophotometry, may be applicable. This may be the case, for example, when an exporting country wants to check whether or not a commodity produced in that country complies with an Codex MRL. In this case, the treatment history of the commodity may be known or assumed, so that the method used need not be as elaborate as in cases where samples of unknown treatment history are under investigation. Also, when the MRL is high compared to the limit of determination, simpler methodology may be applied in order to arrive at a "pass/no pass" decision or for quick screening purposes.

1.3 Application of methods

It will always be necessary for the analyst to validate a method before it is first applied in a practical situation. There is a further need for regular checks on the performance of the method in use at or above the MRL. For all new pesticide/commodity combinations the method must be validated following Good Practice in Residue Analysis. Confirmation of the identity of an indicated residue by an independent technique is also to be regarded as an essential part of Good Practice in Residue Analysis, especially when the initial result suggests that an MRL is exceeded.

Mass spectrometry has become for many residues the method of choice for confirmatory purposes, but the ultimate choice of a confirmatory test depends upon the technique used in the initial determination and upon the available instrumentation and necessary expertise.