

# codex alimentarius commission



FOOD AND AGRICULTURE  
ORGANIZATION  
OF THE UNITED NATIONS

WORLD  
HEALTH  
ORGANIZATION



JOINT OFFICE: Viale delle Terme di Caracalla 00100 ROME Tel: 39 06 57051 www.codexalimentarius.net Email: codex@fao.org Facsimile: 39 06 5705 4593

Agenda Item 8b)

CX/MAS 02/11

## JOINT FAO/WHO FOOD STANDARDS PROGRAMME

### CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-fourth Session

Budapest, Hungary, 18-22 November 2002

#### SINGLE-LABORATORY VALIDATION

#### REQUIREMENTS FOR SINGLE-LABORATORY VALIDATION FOR CODEX PURPOSES<sup>1</sup>

##### INTRODUCTION

1. At its 23<sup>rd</sup> Session the Codex Committee on Methods of Analysis and Sampling discussed the possible use of single-laboratory validation for Codex purposes. It was decided to redraft requirements for such use and to await the appearance of the draft IUPAC guideline on single-laboratory validation for adoption by reference<sup>2</sup>.
2. To evaluate the possible role of single-laboratory validation for Codex purposes it is essential that Codex methods can be used to settle conflicts on composition of foodstuffs. Therefore, mutual acceptance of results is a prerequisite. Traditionally this mutual acceptance was obtained by using the same inter-laboratory validated methods. Recently, the *criteria approach* towards methods of analysis is getting more and more adopted. In this approach more than one method can be used, provided they fulfil certain criteria. An important criterion is the availability of interlaboratory precision data.
3. In the field of residue analysis fully validated methods are not always available. These methods develop rapidly because of the on going marketing of new active substances. Governments must be able to enforce limit on these substances as soon as they come on the market and cannot wait until methods might be validated. Because of the wide scope of these methods both with respect to analytes and substrates it cannot be expected that all combinations will be ever validated in the classical interlaboratory way. Possibly characteristic combinations can be studied, as was done *e.g.* in the EU Standards, Measuring and Testing project on pesticide residues in fruit and vegetables. However, those projects tend to be obsolete by the time of publishing. In this case classical selective detectors were used, whereas now GC-MS is the state of the art.
4. Because of the lack of up to date interlaboratory validated methods a strong need to use single-laboratory validated methods exists in the field of residue analysis. In recent years much effort has been made to elaborate suitable protocols for this single-laboratory validation. As a result an IUPAC protocol has been published.
5. In general the use of single-laboratory validation methods will be especially appropriate for multi-residue methods. In the case of single analytes of great trade importance it is expected that full validation will be the starting point of a Codex specification.
6. The use of single-laboratory validated methods has also been in discussion in the Committee on Residues of Veterinary Drugs in Foods (CCRVDF) and is accepted in the veterinary control in the EU, laying down detailed criteria in "Commission Decision implementing Council Directive 96/23/EC concerning the

<sup>1</sup> Prepared by the Netherlands in cooperation with others

<sup>2</sup> see also CX/MAS 02/10 under Agenda Item 8a).

performance of analytical methods and the interpretation of results" (to be published in September (Doc Sanco/1085/2000 rev 7).

7. Single-laboratory validation also plays a role in the management of new hazards and acute food quality accidents. Usually these incidents are not directly related to Codex specifications but have implications for food control and acceptance of lots. Trade parties will have to negotiate in those cases to accept analytical results mutually.

8. The use of single-laboratory validated methods for Codex purposes is an extension of the acceptance of the criteria approach, that also does not specify a specific method to be used, but sets criteria to methods, leaving choice to the analyst of appropriate methods.

## **REQUIREMENTS FOR USE OF SINGLE-LABORATORY VALIDATED METHODS FOR CODEX PURPOSES**

9. The main issue in use of analytical methods for Codex purposes is the mutual acceptance of laboratory data. One aspect is the comparability of the methods. Another aspect is the proficiency of the laboratory. It is in this field that strong improvements have been made during recent decades. The guidelines on quality assurance, proficiency testing and single-laboratory validation (IUPAC) give important safeguards to the reliability of analytical data in addition to collaboratively validated methods.

10. A main topic in the acceptability of results obtained by single-laboratory validated methods is the presence of data giving an interlaboratory reference. Three methods can give such a reference:

- a. calibration using reference materials;
- b. comparison of results achieved with other methods;
- c. systematic participation in proficiency tests.

11. Taking into account this external reference, conditions can be set up for cases where single-laboratory validated methods can be used:

- a. no inter-laboratory validated method is appropriate;
- b. The single-laboratory validated methods must fulfil the following criteria:
  - i the method is validated according to an internationally recognized protocol (e.g. the IUPAC protocol);
  - ii the use of the method is embedded in a quality assurance system under accreditation;
  - iii external reference is given at least by systematic participation in proficiency schemes. Additional external reference can be obtained by calibration using reference materials and comparison of results with other methods.

## **CONCLUSIONS**

12. CCMAS is invited to propose an addition to the Procedural Manual after the general criteria to the "criteria approach" (page 66), indicating criteria for the use of single-laboratory validated methods for CODEX purposes, as follows:

### *General Criteria for the Selection of Single-Laboratory Validated Methods of Analysis*

Especially in the case of multi-analyte/multi-substrate methods and new hazards, interlaboratory validated methods are not available or appropriate. Criteria used to select a method include the General Criteria for the Selection of Methods of Analysis, where appropriate. In addition the single-laboratory validated methods must fulfil the following criteria:

- i the method is validated according to an internationally recognized protocol (e.g. the IUPAC protocol);
- ii the use of the method is embedded in a quality assurance system under accreditation;
- iii external reference is given at least by systematic participation in proficiency schemes. Additional external reference can be obtained by calibration using reference materials and comparison of result with other methods.