

# codex alimentarius commission



FOOD AND AGRICULTURE  
ORGANIZATION  
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**Agenda Item 9**

**CX/PR 05/37/9**

## JOINT FAO/WHO FOOD STANDARDS PROGRAMME

### CODEX COMMITTEE ON PESTICIDE RESIDUES

**Thirty-seventh Session**

**The Hague, The Netherlands, 18-23 April 2005.**

#### **Comments on the Proposed Draft Guidelines on the Use of Mass Spectrometry for Identification Confirmation and Qualitative Determination of Residues at Step 3 submitted by Australia and the United States of America**

The 36<sup>th</sup> Session of the Committee on Pesticide Residues decided to circulate the Proposed Draft Guidelines on the Use of Mass Spectrometry for Identification Confirmation and Qualitative Determination of Residues at Step 3 of the Codex Procedure. Comments were received from Australia and the USA, both showing general support for the document, but recommending some editorial and technical changes. The detailed comments are given below:

#### **Australia:**

1. It is recommended that the Commission Decision 2002/657/EC implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results in *Official Journal of the European Communities* L221/8, August 17, 2002 be referenced in the Guideline document. It is further proposed that the criteria point system from CD 2002/657/EC for identification of unknown peaks in mass spectrometric techniques be considered for adoption or incorporation into the Guide.
2. It is viewed that the usefulness of the draft document could be improved by incorporating a brief discussion on the comparative value of mass spectrometric techniques such as MS/MS, High Resolution Mass Spectrometers, and semi-high resolution instruments such as time-of-flight mass spectrometers which provide enhanced sensitivities as well as improvements in the assignment of peak identity.
3. It is suggested that the title of the Guideline be either amended to "The Use of Chromatography and Mass Spectrometry for Identification, Confirmation and Quantitative Determination of Residues" or, more concisely as "Guidelines on the Confirmation of Residues" to reflect more accurately the contents of the Guide.
4. It is proposed that the opening two sentences of paragraph 3 under the Gas Chromatography/Mass Spectrometry heading be amended to "If the ions detected still indicate the possible presence of residues of a particular compound, further confirmation is required before reporting the result".

#### **United States of America:**

Paragraph 2, seventh sentence: They are based [,] on either further interpretation of chromatographic and mass spectrometric data, [or] alternative methods using different physico-chemical properties of the compound, *or a* [the] combination of various separation....

Paragraph 3, first sentence: Insert comma after “confirmation.”

Paragraph 3, second sentence: Care should be taken that the instrument is adjusted correctly before starting the analysis [,] insert ; in place of comma

Paragraph 3, fourth sentence: Insert comma after “phase 1.”

Paragraph 3, fifth sentence: Insert comma after “retention time.”

Paragraph 3, seventh sentence: Change “an suitable interval” to “*a* suitable interval.”

Paragraph 4, second sentence: Particular problems [but], although it is *sometimes* difficult.

Paragraph 5, third sentence: Insert comma after “sample material.”

Paragraph 5: The last sentence is rewritten. “Blank” untreated samples should be available to the laboratory to check the occurrence of possible interfering substances.

Paragraph 6, first sentence: Third word, change [to] to *for*. Insert comma after “analyst’s part.”

Paragraph 6, second sentence: Change [apparatus] to *instrumentation*.

### **Page 73, Gas Chromatography/Mass Spectrometry (GC/MS)**

Paragraph 1, first sentence: Omit [can], change [to represent] to *to represents* and change [equipment] to *instrumentation*.

Paragraph 2, first sentence: Change [close to critical level] to *close to the critical level*.

Paragraph 3, first sentence: Insert comma after “presence of a residue.”

Paragraph 3, third sentence: Change [equipment] to *instrumentation*. Insert comma after “retention time.”

Paragraph 3, fourth sentence: Insert comma after “In this case.” Change [has to be made] to *have to be made*.

Paragraph 3, last sentence: [Rations] misspelling, change to *ratios*.

Paragraph 3, last sentence: Change [the use of alternative analytical techniques, examples are listed in Table 6] to *the use of alternative analytical techniques. Examples are listed in Table 6*

Paragraph 4, first sentence: Corrected sentence—Further confirmation by mass spectrometry can be accomplished by acquisition of the complete electron-impact mass spectrum (in practice generally from  $m/z$  50 to beyond the molecular ion region).

Paragraph 4, third sentence: Change [eg] to *e.g.*

### **Page 73, HPLC and HPLC-MS**

Paragraph 1, first sentence omit [where] and [is].

### **Page 74, HPLC and HPLC-MS**

Paragraph 1, second sentence:

Change [certain type of compounds] to *certain types of compounds*.

Paragraph 1, last sentence: Change [and need for more efficient cleanup] to *and the need for more efficient cleanup*.

#### **Page 74, Derivatisation**

Paragraph 1, first sentence change [not only] to *none must*.

(a) Chemical Reactions: third sentence: Change [so that the results from each maybe directly compared] to *so that the results from each may be directly compared*.

The Committee is invited to review the above recommended changes to the draft Guidelines through its *Ad-Hoc* Working Group on Methods of Analysis. The Netherlands will prepare a modified version of the document taking into account the written comments received as well as observations from the floor for further advancement in the Codex Procedure.