LIVSMEDELSVERKET

NATIONAL FOOD ADMINISTRATION

Quality Control Procedures for Pesticide Residue Analysis SANCO/10232/2006

Fifth review

Tuija Pihlström

National Food Administration, Sweden

Outline of the presentation

- Goal and background of document
- Procedure for the revision (advisory group, WG, NRL, WS,)
- Suggested issues/topics in guidelines

Goal for the preparation:

To reach a complete harmonisation of quality assurance measures for pesticide residue analysis within Europe

- The reported result is adequate for its purpose and consistent with other similar results
- To define the minimum criteria which are required
- To find an optimum between cost and output (low cost –high efficiency/quality)

Background of the document

Initiated and prepared by Alan Hill (U.K.) and the organising committee for the EU Workshop on Coordinated Analytical Quality Control 1997:

J. Santos (PT), C. Lentza-Rizos (GR), A. De Kok (NL), M-A. Piedallu (FR), L. Alder (DE), A. Andersson (SE)

On behalf of European Commission

(F. Hinsley, B. Drukker)

Reviews:

1. Doc. 7826/VI/97
   -discussed at 1st EU AQC,1997, in Portugal
2. Doc. SANCO/3103/2000
   -discussed at 2nd EU AQC,1999, in Greece
3. Doc. SANCO/10476/2003
   -discussed at 3rd EU AQC, 2003 in U.K.
4. Doc. SANCO/10232/2006
   -discussed at 4th EU AQC, 2005 in Sweden

Advisory group

 A de Kok

 A Fernandez-Alba

 M Gamon

 M Anastassiades

 A Andersson

 P Cuhra

 S Reynolds

 R Lippold

 P Pelozi

 P Ravio

 A Valverde

 W Zacharae

 M Paulsen

 J Durcanska

Procedure of the revision

Suggested issues/topics

- Inclusion of food of animal origin and cereals (representative matrices, validation criteria etc)
- Issues/topics in guidelines that need updating
- Document - harmonization considering the history, other rules (e.g. CODEX, OECD, CEN)

Selection of the paragraphs

- **§42-43** Frequencies and minimum number of analytes for calibration including Table 1
- **§60 -62** Frequencies and minimum number of analytes for routine recovery inclusive Table 2
- **§55** Validation, inclusion of animal products and cereals
- **§83** Common interpretation of analytical results in respect of the correction of results for recovery

LIVSMEDELSVERKET

NATIONAL FOOD ADMINISTRATION

Procedure for the revision NRL and the official laboratories

The draft document was sent to the NRLs and the official laboratories for comments

Response 22 July- 31 August

Member states 2007	Comments on
Belgium	42,43,55,56,58,60,83,Annex
Cyprus	83
France	13,42,43,53,56,57,58,60,72,83
Greece	Agrees (NRL of animal origin)
Ireland	15,18,22,38,58,83,93
The Netherlands	12,13,23,32,56,83,Appendix,Annex
Austria	83,86
Portugal	42,43,58,83,86,92
Slovakia	34,42,43,55,56,65,83,94,Annex
Slovenia	See separate comments
Spain	42,43,55,56, Table 1 and 2, Glossary
U.K.	42,43,60,93

Comments from NRL and Off. Labs

Paragraph	no	Frequency
Reporting of results (Recovery correction)	83 and 93	10
Representative analytes (Calibration, recovery tests)	42-43, 60	6
Method Validation (Represenative matrices)	55,56,57	5
Others		1 to 3

§83 Reporting results
Correction for recovery

Purpose and an intended effect of the correction:

1. Correction should improve the results- closer to the "true value"
2. The result can be compensated for the incomplete extraction of the analyte from sample
3. Common interpretation in regulation of contaminants/VMPs takes account correction for recovery
4. Conclusion from 4th AQC in Stockholm 2005

§83 Reporting of results Proposal

Residue data exceeding an MRL must be corrected for recovery. The adjustment should be stated based either using the mean value from three recoveries performed in same matrix and analysed in the same batch or using two standard additions e.g. at two and five times the residue in the sample. In general, residues below MRLs are not to be adjusted for recovery, when the batch recoveries fall within the acceptable range. If residue data are adjusted for recovery this should be done as described above and must be stated.

§ 42 Representative analytes Calibration…….

Purpose of the calibration:

To avoid false negatives

To test the sensitivity of the detection system

The detection system should be *calibrated (=checked) with all analytes for every batch of analyses.

*at the lowest level =calibration

§ 42 Representative analytes Calibration…….

If impractical or unreasonably large number of calibrations, the minimum number of analytes for calibration has been suggested as follows:

.

§ 43 Frequency for calibration
Proposal:

The minimum number of analytes for calibration (frequently found and occasionally found analytes) must be 15 plus 25% of the total number of analytes included in the scope of the analysis. If the scope includes 1 to 20 analytes, all analytes must be calibrated in every batch.

§ 43 Frequency for calibration Proposal:

e.g. Scope 100 analytes →15+25% of 100 =40

Minimum number of representative analytes for calibration

Table 1. Minimum frequencies for calibration -Proposal

	Frequently found analytes	Occasionally found analytes	Rarely found analytes
Minimum frequency of calibration	Calibration in each batch of analyses. At least at the level corresponding to the reporting limit.	A rolling programme at least every third batch . At least at the level corresponding to the reporting limit.	A rolling programme at least every third month At least at the level corresponding to the reporting limit

§60 Method validation
Frequency for routine recovery

In a perfect world-recovery of all analytes measured with each batch

If not possible the minimum of acceptable frequency of recovery is given

Purpose:

Acceptable screening and method at the time of analysis for all analytes searched

Table 2. Frequency for routine recovery- Proposal

	Indicators of representative analytes	All other representative analytes	All other analytes
Minimum frequency of recovery	In each batch of analyses Preferably at different levels and covering different commodities each time	At least every third batch Preferably at different levels and covering different commodities each time	A rolling programme to include all other analytes at least every third month Preferably at different levels and covering different commodities each time

§55 Method validation

- Inclusion of animal products and cereals
- Selection of representative matrices- Annex1
- Validation- selection of matrices-one of the each main group ?

The Pareto principle

" A Pareto optimal allocation is one where there is no feasible reallocation that would be strictly preferred by all agents".

- i.e. there should not be any changes which do not promote all laboratories

http://europa.eu.int/comm/food/plant/protection/resources/qualcontrol_en.pdf