codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 9 (c) **CX/PR 05/37/11**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PESTICIDE RESIDUES
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The Use and Implications of Measurement Uncertainty

(prepared by the Netherlands)

The 36th Session of the Committee on Pesticide Residues decided to circulate the Proposed Draft Guidelines on the Estimation of the Uncertainty of Results at Step 3 of the Codex Procedure. The Guidelines currently do cover the estimation of the measurement uncertainty but at this moment do not cover its use. In para 190 of the report of the 36th session the Committee noted that these is a general consensus about the estimation of uncertainty while there are widely different views and practices among members concerning the use of measurement in compliance testing. The Codex Committee on Methods of Analysis and Sampling advanced Draft Guidelines on Measurement Uncertainty for adoption to the Commission on its last session. The Commission adopted the Guidelines with amendments. In the last session of the Commission New Zealand expressed the view that it is not clear how the information on measurement uncertainty would be used, and that barriers to trade might result from misuse or misunderstanding. They also pointed out that matters related to measurement uncertainty should be considered in relation with other relevant Codex texts. This position was supported by other delegation. The Guidance on the estimation of measurement uncertainty for Pesticide Residues is to be included in the Guidelines on Good Practice in Pesticide Residue Analysis and therefore well embedded in relevant Codex texts, nevertheless the present document considered at step 4 of the Codex Procedure also gives little guidance on the use of uncertainty data.

A Consultants meeting convened by the FAO/IAEA Division (Vienna 22-26 March 2004) recommended the guidance that follows below:

Use of Uncertainty Information

If required, the result should be reported together with the expanded uncertainty, U, as follows

Result = $x \pm U$ (units)

The expanded uncertainty, U, may be calculated from the standard combined uncertainty (S_{Res}) with a coverage factor of 2 as recommended by EURACHEM or with the Student ***t*** value for the level of confidence required (normally 95%) where the effective degree of freedom is less than 20. The respective calculations for the expanded uncertainty are as follows

$U = 2S_{Res}$ or $U = t_{v,0.95}S_{Res}$

The numerical value of the reported results should follow the general rule that the last digit can be uncertain. Rounding the results should be done only when the final result is quoted since rounding at the initial stages of calculation may introduce unnecessary bias in the calculated values.

The interpretation of a residue value followed by the decision on the compliance of a lot with the MRL depends on how the number of reported significant figures, the uncertainty of the result and the recovery correction are used.

For the purpose of explication, it is assumed that the best estimate of the residue content is reported for a sample. How the results are interpreted depends upon the purpose of the testing. Typical reasons include testing compliance with the national MRL, certifying compliance with the Codex MRL of a commodity for

export, and generating dietary intake estimates of residues. The first two purposes are routinely encountered in residue testing environments and are examined further.

5.1 Testing compliance with an MRL at national level

The expanded uncertainty should be calculated using S_L from equation 1 as $U = kS_L$.where $S_L=CV_L$ x residue.

Figure 1 shows how the testing results can be displayed in terms of the measured value of the residue, the corresponding uncertainty interval, and the MRL.

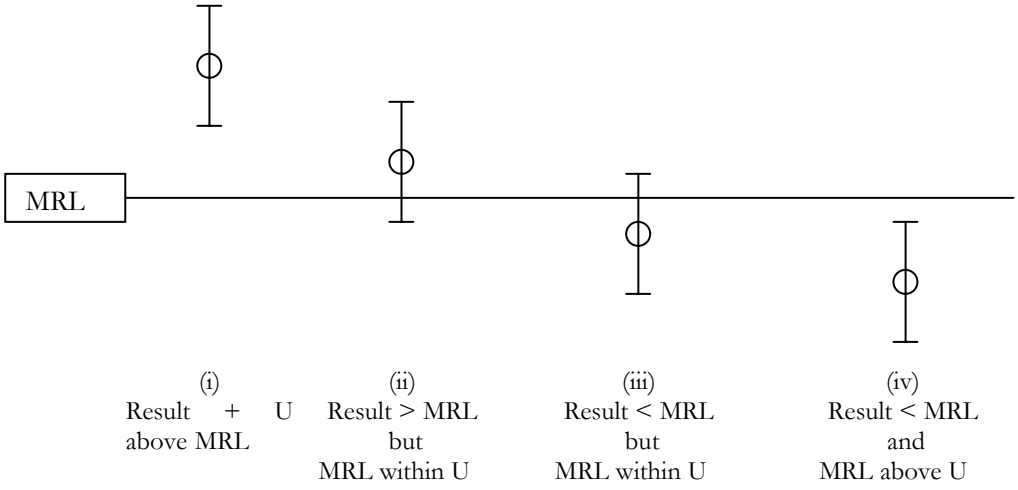

Figure 1. Illustration of the relationship of measured value expded uncertainty and MRL

Situation (i)

The analytical result bounded by the measurement uncertainty endpoints is greater than the MRL. The result indicates that the residue in the sampled lot is above the MRL.

Situation (ii)

The analytical result is greater than the MRL with the lower endpoint of the measurement uncertainty less than the MRL

Situation (iii)

The analytical result is less than the MRL with the upper endpoint of the measurement uncertainty being greater than the MRL.

Situation (iv)

The analytical result bounded by the expanded measurement uncertainty endpoints is less than the MRL.

5.1.1 Decision Environment

The decision-making in Situation (i) is clear. In order to avoid lengthy explanation of the uncertainty in a court case involving the performance of the analysis for testing compliance with the MRL at the national level in locally produced or imported commodities, the laboratory may report the results as the sample contains "not less than 'x – U' residues." Hence, any enforcement action is only taken after the analyst is certain that the specification has been significantly exceeded. This satisfies the requirement to prove beyond reasonable doubt that a limit has been exceeded if the case should come to court.

The same clarity is observed in Situation (iv). The sample would be considered compliant by all Enforcement Authorities**.**

The middle situations are problematic for decision-makers. If the uncertainty of the result is not used in Situation (ii), the lot would be declared noncompliant which is an incorrect decision. Since the deviation from the MRL is within the uncertainty of the measurement, the sampled lot should be declared as being

compliant with the MRL. In Situation (iii), the sampled lot would be considered as being compliant with the MRL by Enforcement Authorities in general, but some Enforcement Authorities could incorrectly decide otherwise.

5.2 Certifying compliance of a lot to be exported

The certification of any composite sample of a specified size complying with the MRL by the laboratory requires that the uncertainty of sampling is specified and the compliance is stated at a specified probability level with a given confidence level.

There is a basic problem in that as there is no internationally agreed or nationally declared value for the acceptable violation rate other than the USA where β_p = 99% compliance is required at β_t = 99% confidence level.

The coverage factors required for the calculation of the expanded uncertainty depend on the number of effective degrees of freedom of the estimated standard uncertainty. They are given in Table 6.

Table 6 Coverage factors for the calculation of expanded uncertainty $U= kS_{Res}$ [a]

Degree of freedom	t at 95%[b]	k at β_p=0.95,β_t=0.95[c]	k at β_p=0.99,β_t=0.99[c]
5	2.6	3.7	7.3
15	2.1	2.6	4.3
20	2.1	2.4	3.9
∞	2	1.65	2.3

Notes: (a) The expanded uncertainty uses S_{Res} from equation 1.
(b) This is recommended by EURACHEM.
(c) The coverage is important on the upper end of the distribution: one sided tolerance factors are included in the table.

The tested lot is compliant if the analytical result, X, plus the upper bound of the measurement uncertainty limit is less than the MRL. That is,

$X+kS_{Res} < MRL$

For a commodity to be exported to the USA, the upper endpoint of measurement uncertainty must be less than the MRL. This criterion implies that the measured residue must be significantly lower.

For instance, let the MRL be 1 mg/kg, the combined relative standard uncertainty of the pesticide result be 0.33 based on 21 observations, and the measured residue be 0.55 mg/kg.

(i) It follows that residues in 95% of a samples taken from the lot may be expected to be lower than the MRL (0.55 + 2.4 * 0.33* 0.55 = 0.99 mg/kg). Where a 95% compliance is acceptable the sampled lot would satisfy the requirements of the importing country.

(ii) However, when the commodity is intended for export to the USA, the residue must be less than 1 mg/kg in 99% of the samples. Based on the 0.55 mg/kg measured residue it may be expected that residues up to 1.3 mg/kg can occur in 99% of the samples (0.55 + 3.9*0.33*0.55 = 1.258). Therefore, the residue measured in one sample must be ≤0.43 mg/kg to certify compliance (0.43 + 3.9*0.43*0.33 = 0.983; 0.44+3.9*0.33*0.44=1.006 mg/kg).