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Case study theme:
Equivalence of compliance evaluation

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Overview

- 1 Pesticide residues in foodstuffs**
- 2 Establishment of MRL**
- 3 Monitoring of foodstuffs**
- 4 Discussion**



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1 Pesticide residues in foodstuffs

- Needed for agricultural productivity;
- Safe to environment and public health if correctly used:

➤➤ Pesticide residues checked considering MRL (Maximum Residue Level)

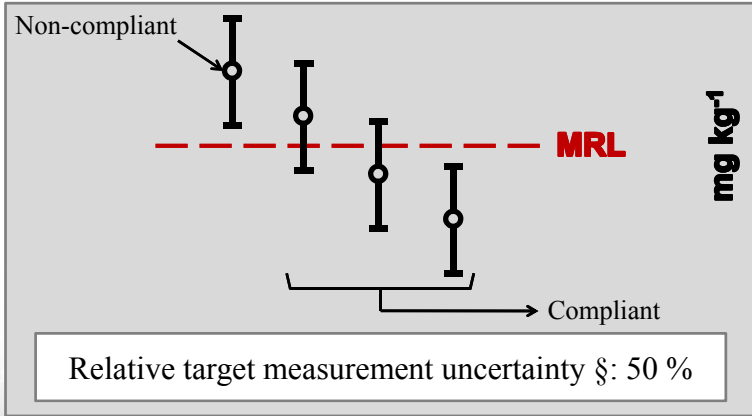
MRL – Safe limits that establish trade rules



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1 Pesticide residues in foodstuffs

(...) Compliance evaluation criterion:
Non-compliant if $> \text{MRL}$;



Relative target measurement uncertainty $\leq 50\%$

§ JCGM, International vocabulary of metrology — Basic and general concepts and associated terms (VIM), JCGM 200:2008 (2008) (http://www.bipm.org/utis/common/documents/jcgm/JCGM_200_2008.pdf).

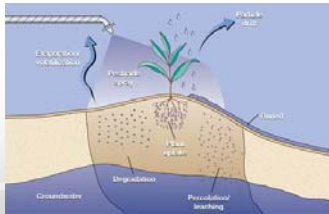
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2 Establishment of MRL

Supported on “supervised residue studies” (...)

Supervised residue studies:


- Assist the evaluation of the safety and efficacy of the product;
- Obtain the data for establishing maximum limits for residues of the pesticide in food or agricultural commodities.



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2 Establishment of MRL

“Supervised residue studies” involve the use of measurement procedures optimised for specific analytes;




- Measurements results are not corrected for observed analyte recovery.

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2 Establishment of MRL

“Supervised residue studies” involve the use of measurement procedures optimised for specific analytes;



- Measurements results are not corrected for observed analyte recovery.

Observed high analyte recovery (makes residues comparable with toxicological limits)

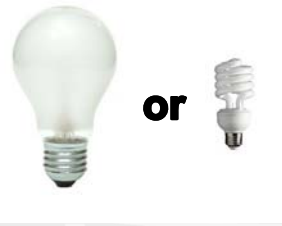


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3 Monitoring of foodstuffs

3.1 European Union Monitoring:

- Multi-analyte and multi-matrix measurement procedures are used (some affected by low recovery);
- Measurement results are reported uncorrected for mean recovery observed on spiked samples, R_{Spk} , [if R_{Spk} is within 70-120 % §]:



§ SANCO, Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed; SANCO/10684/2009 (2009) (http://ec.europa.eu/food/plant/protection/resources/qualcontrol_en.pdf).

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3 Monitoring of foodstuffs

3.1 European Union Monitoring:

- Multi-analyte and multi-matrix measurement procedures are used (some affected by low recovery);
- Measurement results are reported uncorrected for mean recovery observed on spiked samples, R_{Spk} :

Case Study I

Actual mass fraction [mg kg^{-1}]: 25; MRL [mg kg^{-1}]: 10;

Measurement procedure A, MP-A (●):
 $R_{Spk} = 90\%$;
 U' (relative expanded uncertainty) = 50 %;

Measurement procedure B, MP-B (○):
 $R_{Spk} = 70\%$;
 $U' = 50\%$.

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3 Monitoring of foodstuffs

3.1 European Union Monitoring:

- Multi-analyte and multi-matrix measurement procedures are used (some affected by low recovery);
- Measurement results are reported uncorrected for mean recovery observed on spiked samples, R_{Spk} :

Case Study I

Actual mass fraction [mg kg^{-1}]: 25; MRL [mg kg^{-1}]: 10;

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 $R_{Spk} = 90\%$;
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Measurement procedure B, MP-B (○):
 $R_{Spk} = 70\%$;
 $U' = 50\%$.

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3 Monitoring of foodstuffs

3.1 European Union Monitoring:

(...)

- Measurement results uncertainty (MU) are not compensated for observed bias (would not reach target MU).

Case Study II
 Actual mass fraction [mg kg⁻¹]: 25;
 MRL [mg kg⁻¹]: 10;
 Mean analyte recovery: 70 %;
 Relative expanded uncertainty: 50 %.

§ M. Thompson, S. Ellison, A. Fajgelj, P. Willetts and R. Wood, Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement, J. Pure & Applied Chemistry, 71(2), 337-348 (1999).

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3 Monitoring of foodstuffs

3.1 European Union Monitoring:

(...)

Bias compensated MU:

- It does no guarantee equivalence of evaluations;
- It overestimates measurement results uncertainty.

Case Study II
 Actual mass fraction [mg kg⁻¹]: 25;
 MRL [mg kg⁻¹]: 10;
 Mean analyte recovery: 70 %;
 Relative expanded uncertainty: 50 %.


§ M. Thompson, S. Ellison, A. Fajgelj, P. Willetts and R. Wood, Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement, J. Pure & Applied Chemistry, 71(2), 337-348 (1999).

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3 Monitoring of foodstuffs

3.1 European Union Monitoring:
Metrological characterization of the problem (VIM3) :

- Measurement results are traceable to the value estimated by different operationally defined measurement procedures;
- Measurement results are not metrologically comparable and can reach different compliance evaluation conclusions.




JCGM, International vocabulary of metrology — Basic and general concepts and associated terms (VIM), JCGM 200:2008 (2008) (http://www.bipm.org/utls/common/documents/jcgm/JCGM_200_2008.pdf).

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3 Monitoring of foodstuffs


3.2 Australian Monitoring:

- Measurements results are reported corrected for analyte recovery observed on spiked samples (R_{Spk}).



Spiked

Vs.



Incurred

[(...) but measurement procedure performance (in particular analyte recovery) can be different for spiked and incurred residues]

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3 Monitoring of foodstuffs

3.2 Australian Monitoring:

- Measurements results are reported corrected for analyte recovery observed on spiked samples (R_{Spk}):

Case Study III

Actual mass fraction [mg kg^{-1}]: 25; MRL [mg kg^{-1}]: 10;

Measurement procedure A, $MP-A$ [●]:
 $R_{Spk} = 90\%$;
 R_{Inc} (R for incurred) = 88%;

Measurement procedure B, $MP-B$ [○]:
 $R_{Spk} = 70\%$;
 R_{Inc} (R for incurred) = 50%.

$[R_{Spk}/R_{Inc}](I) \neq [R_{Spk}/R_{Inc}](II)$

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3 Monitoring of foodstuffs

3.2 Australian Monitoring:

Metrological characterization of the problem (VIM3) :

- Measurement results are traceable to the value of the spiked sample;
- Measurement results can be not compatible due to differences in $[R_{Spk}/R_{Inc}]$ ratios (...)

JCGM, International vocabulary of metrology — Basic and general concepts and associated terms (VIM), JCGM 200:2008 (2008) (http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2008.pdf).

4 Discussion



How should we deal with this?

- Is this an important issue?
- How can we metrologically characterise the problem (VIM3) ?
- Which is the ideal solutions to the problem?
- What are the economically viable solutions to the problem?

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