



Updates on Testing of Pesticide Residues in Food

29 October 2015

Food Testing – Pesticide Residues

- Previous briefs available on websites of
 - Government Laboratory
 - Centre for Food Safety



Government Laboratory
The Government of the Hong Kong Special Administrative Region

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Training and Development Collaborations

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Training and Development Collaborations

The Government Laboratory (GL) is committed to contributing to the sustainable development of measurement science and scientific infrastructure in Hong Kong by fostering collaborations and positive partnerships with counterparts, professional organizations and client departments involving in scientific investigations.

Technical exchange

GL often organizes talks and seminars to facilitate the local testing sector in building their testing capabilities and preparing for laboratory accreditation to ISO/IEC 17025:2005.

Briefing sessions

Over the years, GL continued organizing technical seminar for the commercial sector to provide a platform for technical exchange. Among others, more than 10 briefing sessions on food testing covering determination of preservatives, veterinary drug residues, pesticide residues, food additives, food dyes, toxins and etc had been organized. These briefing sessions provided opportunities for GL to share its experience with local commercial laboratories and enhance their interests in the provision of food testing service.

Technical seminar for commercial testing sector



Centre for Food Safety
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Pesticide Residues in Food Regulation

Pesticide Residues in Food Regulation

The Legislative Council has completed its scrutiny of the Pesticide Residues in Food Regulation (Cap. 132CM) ("the Regulation") in June 2012 and the Regulation comes into operation on 1 August 2014.

- [Pesticide Residues in Food Regulation](#)
- [Hong Kong Pesticide MRL Database](#)

<http://www.govtlab.gov.hk/>

<http://www.cfs.gov.hk/>



Pesticide Residues in Food

- **To avoid common mistakes in calculations**
- **To improve ruggedness of the analytical method**
- **To speed-up the result reporting cycle**



Pesticide Residues in Food

- **To avoid common mistakes in calculations**
- **Residue definition, MRL, Reporting Limit (RL)**
- **Portion of samples for laboratory analysis**
- **Data integration**
- **Quantitative Calibration**
- **Measurement Uncertainty**



Pesticide Residues in Food

- To avoid common mistakes in calculations
- Residue definition, MRL, Reporting Limit (RL)
- Portion of samples for laboratory analysis

Portion of Commodities to which CODEX Maximum Residue Limits Apply and which is Analyzed, CAC/GL 41-1993 (Revision 1993, Amendment 2010), CODEX Alimentarius, 2010.

➔ Refer to previous presentation files for examples of calculation involved



Data integration – Indoxacarb as Example

- **What is Indoxacarb ?**
- **ISO 1750 Amendment 3 (1981) → Indoxacarb is the S-isomer, the only stereoisomer (CAS 173584-44-6, DPX-KNI28)**
- **Residue study data uses:**
- **DPX-MP062 (3 + 1 of S + R isomer)**
- **DPX-JW062 (1 : 1 of S : R isomer)**
- **Consider any possible R / S isomer separation in GC or LC**
- **Consider the isomer ratio of reference standard / sample**
- **Consider the residue definition: Sum of indoxacarb and its R enantiomer**
- **Consider detector response factor of S- and R- isomers**
- **Consider PAM, JMPR and CODEX publications**
- **→ Integrate peaks from all isomeric components**



Quantitative Calibration - Overview

- **Multi-level Calibration – Linear Regression**
- **Multi-level Calibration – Relative Response Factor (RRF)**
- **Multi-level Calibration – Standard Addition**
- **Bracketing – by interpolation between two levels**
- **Single-level Calibration**

Plotting concentration of target analyte with ...

- **Analyte Area**
- **Area Ratio from analyte / internal standard (IS)**
- ➔ **Linearity of the plot / calculation**
- ➔ **Relative or absolute acceptable calibration range**

- **SANCO/I2571/2013**
- **ISO 11095:1996 – Linear Calibration Using Reference Materials**



Matrix-matched Calibration

- Compensate matrix effect measured as detector response
- Matrix co-elution during chromatographic separation
- Ion-source effects

Solution-based Calibration



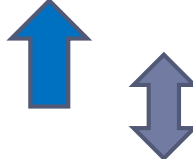
- Select relatively “**interference free**” m/z or SRM
- Ion-ratio of target analyte should be similar among:
 Solution-based <> Sample extract <> Matrix-matched
- Some analytes have less than three reliable MRM in some matrices-detector combinations

Procedural Standard Calibration

- Method dependent
- e.g. Analytes require derivatisation before analysis



Compensation by adding Internal Standard vs “Matrix-Cal”

- **Isotopically labelled internal standard** 
 - **Compensates matrix effect within sample extract, improve method performance (recovery, repeatability)**
 - **Insufficient clean-up / chromatographic separation would widen the RRT between labelled IS and native analyte**
- **Matrix-based calibration** 
 - **Compensates matrix effect between sample and standard extract**
- **Other internal standard** 
 - **IS and analyte may respond differently**



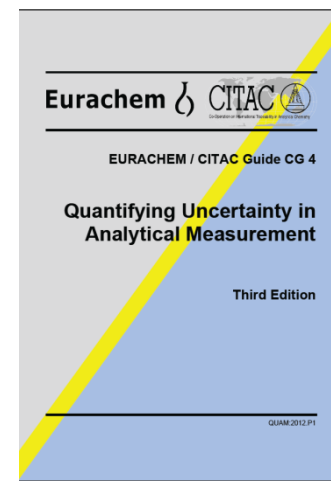
Measurement Uncertainty - Overview

Type A – Statistics

Type B – From other information



IUPAC





Measurement Uncertainty – Reference Documents

EU - Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed, SANCO/12571/2013

- **Paragraphs E8 – E15**

- **Within-lab data**

- **“Inter-laboratory bias”, evaluated by PT results**

- **Prerequisite to use of default MU (50%)**

- **Reporting limit vs lowest spike level**

- **Appendix C – Worked examples on MU estimation**

- **Participation of EUPT**

- **Within-lab data**



Measurement Uncertainty – Reference Documents

CODEX - Guidelines on Estimation of Uncertainty of Results, CAC/GL 59-2006 (Amendment 2011)

- **Approaches –**

- **EUPT participation → 20-25% RSD from EUPTs → 50% (k=2)**

- **Precision data – Horwitz approach**

- **Precision data – Inter-laboratory (collaboration / PT studies)**

- **In-house validation (including PT)**

- **Worked examples in Annex**



Pesticide Residues in Food

- **To improve ruggedness of the analytical method**
 - **Homogeneity of laboratory sub-sampling**
 - ➔ **Suitable blender & food processor**
 - **Benefits:**
 - ➔ **Blend all sample “in one go”**
 - ➔ **Shorter blending time, avoid degradation of analytes**
 - ➔ **Improve within-lab repeatability**



Pesticide Residues in Food

- **To improve ruggedness of the analytical method**
- **Clean-up materials**
 - ➔ **dispersive SPE for GC and LC analysis**
 - ➔ **SPE for GC analysis**
 - ➔ **Choice of extraction solvent and pH**
- **Examples of International published methods**
 - ➔ **EURL : <http://www.eurl-pesticides.eu/>**
 - ➔ **USA : CLG-PST5, USDA method**
 - ➔ **AOAC : 2007.1, Official Methods of Analysis**
 - ➔ **GB, BS, Japan, CODEX... etc**



Pesticide Residues in Food

- **To improve ruggedness of the analytical method**
- **Automation of laborious steps**
 - ➔ **Example: vertical shaker**
 - **Benefits:**
 - ➔ **Reproducible shaking conditions**
 - ➔ **Avoid degradation of acid sensitive analytes**
 - ➔ **Improve work efficiency:**
Batch shaking of extraction and clean-up tubes



Pesticide Residues in Food

- **To speed-up the result reporting cycle**
- **Maximize the analytes in multi-residue analysis**
- **Group-up analytical methods**
- **Screening → Confirmation → Quantitation**
- **Chromatographic run time**
- **Detector selection**



Pesticide Residues in Food

- **To speed-up the result reporting cycle**
- **Maximize the analytes in multi-residue analysis**
- **Group-up analytical methods**
- ➔ **Minimize number of injection and release instrument time**

- **Screening → Confirmation → Quantitation**
- ➔ **Most of the reported values are “Not Detected”**
- ➔ **Quantitative analysis of results below Reporting Limit (RL)**



Pesticide Residues in Food

- **To speed-up the result reporting cycle**
- **Detector selection**
 - **Limitation of traditional detectors, such as GC-ECD / FPD / PFPD / NPD and LC-DAD / FLD**
 - **“Limited specificity”**
 - **“Does not provide unambiguous identification”**
 - **Refer to paragraph C13 of SANCO/12571/2013**



Pesticide Residues in Food

- **To speed-up the result reporting cycle**
- **Fast GC / LC separation**
- **Detector selection - MS/MS, TOF, Orbitrap**
- **Benefits:**
 - ➔ **Maximize the analyte per chromatographic run**
 - ➔ **Remove matrix interferences**
 - ➔ **Reduce repeating sample preparation and injections for confirmation**



Thank You