Updates on Testing of Pesticide Residues in Foods

18 October 2012
Pesticide Residues in Food Regulation

Public Health and Municipal Services Ordinance (Cap. 132)

- Passed the negative vetting in the Legislative Council on 6 June 2012.

- Enacted on 1 August 2012 with 2 years grace period.

- Grace period
  To allow sufficient time for the trade to comply with the Regulation, there would be a grace period of about two years prior to the commencement of the Regulation. The Regulation will commence on 1 August 2014.
Pesticide Residues in Food Regulation

- Number of pesticides to be included:
  - 360 pesticides

- Number of Maximum Residue Limit (MRL) /Extraneous Maximum Residue Limit (EMRL)
  - Approximate 7000 limits

- Food types:
  - Include different foods from plant and animal origins
  - Include foods of high fiber, high protein, high fat, high carbohydrate, high water content, etc.
Pesticide Residues in Food Regulation

http://www.gld.gov.hk/cgi-bin/gld/egazette/gazettefiles.cgi?lang=e&year=2012&month=5&day=4&vol=16&no=18&gn=73&header=1&acurrentpage=12&df=1&nt=s2&agree=1&gaz_type=ls2&part=1&newfile=1&pid=

食物內除害劑殘餘規例

http://www.gld.gov.hk/cgi-bin/gld/egazette/gazettefiles.cgi?lang=c&year=2012&month=5&day=4&vol=16&no=18&gn=73&header=1&part=2&move=2&df=1&nt=s2&agree=1&acurrentpage=12&gaz_type=ls2
### Schedule 1

#### Part 1

**Maximum Residue Limit (MRL)**

<table>
<thead>
<tr>
<th>Item</th>
<th>Pesticide</th>
<th>Residue definition</th>
<th>Description of food</th>
<th>Maximum residue limit (MRL) (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>1-Naphthaleneacetic acid</td>
<td>Sum of 1-naphthaleneacetic acid and its conjugates, expressed as 1-naphthaleneacetic acid</td>
<td>Orange, Sweet</td>
<td>0.1</td>
</tr>
<tr>
<td>1.2</td>
<td>1-Naphthaleneacetic acid</td>
<td>Sum of 1-naphthaleneacetic acid and its conjugates, expressed as 1-naphthaleneacetic acid</td>
<td>Tangerine</td>
<td>0.1</td>
</tr>
</tbody>
</table>
### Part 2

#### Extraneous Maximum Residue Limit (EMRL)

<table>
<thead>
<tr>
<th>Item</th>
<th>Pesticide</th>
<th>Residue definition</th>
<th>Description of food</th>
<th>Extraneous maximum residue limit (EMRL) (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Aldrin and Dieldrin</td>
<td>Sum of HHDN and HEOD</td>
<td>Berries and other small fruits</td>
<td>0.05</td>
</tr>
<tr>
<td>1.2</td>
<td>Aldrin and Dieldrin</td>
<td>Sum of HHDN and HEOD</td>
<td>Citrus fruits</td>
<td>0.05</td>
</tr>
<tr>
<td>1.3</td>
<td>Aldrin and Dieldrin</td>
<td>Sum of HHDN and HEOD</td>
<td>“Assorted tropical and subtropical fruits—inedible peel”, except banana, mango and pineapple</td>
<td>0.05</td>
</tr>
<tr>
<td>1.4</td>
<td>Aldrin and Dieldrin</td>
<td>Sum of HHDN and HEOD</td>
<td>Banana</td>
<td>0.02</td>
</tr>
<tr>
<td>1.5</td>
<td>Aldrin and Dieldrin</td>
<td>Sum of HHDN and HEOD</td>
<td>Mango</td>
<td>0.03</td>
</tr>
</tbody>
</table>
Testing Objectives

- The Testing Method(s) shall be able to analyse the pesticide residues according to the stated residue definition.
- The Reporting Limit of an appropriate testing method shall be at least equal to or lower than the regulatory limit.
- According to the regulation, some foods may require lower reporting limits (<0.1mg/kg)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Food</th>
<th>MRL (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diazinon</td>
<td>Maize</td>
<td>0.02</td>
</tr>
<tr>
<td>Chlorpyrifos</td>
<td>Mango</td>
<td>0.05</td>
</tr>
<tr>
<td>Azinphos Methyl</td>
<td>Apple</td>
<td>0.05</td>
</tr>
</tbody>
</table>
Residue definition means the combination of the pesticide and its metabolites, derivatives and related compounds to which an MRL or EMRL applies.

Examples of different types of residue definitions

1) Parent pesticide (mostly by multi-residues method)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Residue Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acephate</td>
<td>Acephate</td>
</tr>
</tbody>
</table>

2) Sum of compounds (mostly by multi-residues method)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Residue Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>DDT</td>
<td>Sum of p,p'-DDT, o,p'-DDT, p,p'-DDE and p,p'-TDE (DDD)</td>
</tr>
</tbody>
</table>
Pesticide Residue Definition

3) Expression of parent compounds (by multi- or single residue method)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Residue Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methiocarb</td>
<td>Sum of methiocarb, and its sulphoxide and sulphone, expressed as methiocarb</td>
</tr>
</tbody>
</table>

4) Conversion of compounds (by single residue method)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Residue Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dithiocarbamates</td>
<td>Sum of all dithiocarbamates, determined as CS$_2$ evolved during acid digestion, expressed as mg CS$_2$/kg</td>
</tr>
</tbody>
</table>

5) Definition involving moiety (by single residue method)

<table>
<thead>
<tr>
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<th>Residue Definition</th>
</tr>
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<tbody>
<tr>
<td>Vinclozolin</td>
<td>Sum of vinclozolin and all metabolites containing the 3,5-dichloroaniline moiety, expressed as vinclozolin</td>
</tr>
</tbody>
</table>
Sampling - Portion of Commodity for Analysis

Codex Alimentarius Commission (Codex) Standard

- Portion of Commodities to which is analysed

CAC/GL 41-1993 (Amendment 2010)
Sampling - Portion of Commodity for Analysis

Codex Alimentarius Commission (Codex) Standard

- Codex Classification of Foods and Animal Feeds

- The document is now under revision by the CODEX committee of pesticide residues (CCPR).
Trend of Development for Pesticide Residues Analysis

Challenges:

- Use of the number of pesticides is continuously increasing internationally.
- Need of speeding up analytical process
- Need of improving data quality and analytical results comparability from different laboratories
Trend of Development for Pesticide Residues Analysis

The direction of method development has also been changed:

- Traditionally, international organizations and different national / regional institutions would publish standards or reference test methods for specific pesticides.

- Regarding the test method recognition and quality control, the international trend has gradually changed from developing prescribed test methods to the establishment of analytical performance criteria so that satisfactory comparability of results can be achieved by employing different analytical methods but with performance meeting the same requirements under internationally recognized protocols.
For the above reasons:

Different laboratories can consider their testing needs and intended purposes, facilities and resources, and use relevant criteria protocols or reference methods compiled by international or national organizations, or adopt appropriate test methods and fulfill the quality requirements being specified.

Comparability can further be demonstrated by participating proficiency tests or interlaboratory comparisons.
Key Elements on Method Performance

- Accuracy / Trueness
- Precision
- Specificity & selectivity
- Limit of Detection (LOD)
- Limit of Quantitation (LOQ)
# Examples of General Guidelines

<table>
<thead>
<tr>
<th>ORGANIZATION</th>
<th>DOCUMENT REFERENCE</th>
<th>TITLE</th>
</tr>
</thead>
<tbody>
<tr>
<td>EU</td>
<td>SANCO/12495/2011 Implemented by 01/01/2012 Supersedes Document No. SANCO/10684/2009</td>
<td>Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed</td>
</tr>
</tbody>
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# Examples of General Guidelines

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<tbody>
<tr>
<td>PRC</td>
<td>GB/T 27404-2008</td>
<td>Criterion on Quality Control of Laboratories – Chemical Testing of Food 實驗室質量控制規范 食品理化檢測</td>
</tr>
<tr>
<td>HKAS (HKSAR)</td>
<td>HOKLAS Supplementary Criteria No. 37</td>
<td>&quot;Food&quot; Test Category - Chemical Testing</td>
</tr>
</tbody>
</table>
Considerations on Selection of Test Method

- Quality
- Speed
- Cost
Considerations on Selection of Test Method

Instrumentation Specificity

ECD, FPD → MSD → MS/MS, HRMS, TOF-MS

Quality & Speed

GPC → SPE → Dispersive SPE (QuEChERS)

Cleanup Speed
Test Method References

   - Document is cancelled

Since the rapid development and application of new methods makes it very difficult continually update a hard copy Standard of listed methods. Maintaining the currency of the Standard would be resource intensive and problematic particularly when relying on the expertise and resources of volunteers.

The Committee: considered how to address methods of analysis for pesticide residues in relation to the request of the 34th Session of the Commission to develop criteria as opposed to a list of methods of analysis and reasserted its previous decision to recommend revocation of the Standard of Analysis of Pesticide Residues: Recommended Methods (CODEX STAN 229-1993) by the Commission and agreed to develop performance criteria for suitability assessment of methods of analysis (para. 185);
2) 中華人民共和國國家標準
“常用農藥殘留量檢測方法標準選編(上下冊)” 2009版,

3) United States Environmental Protection Agency (USEPA) Residue Analytical Methods (RAM)
Test Method References

4) EU Reference Laboratories for Residues of Pesticides
   Multi-residues method
   Single residue methods

5) Department of Food Safety, Ministry of Health, Labour and Welfare, Japan Analytical Methods for Residual Compositional Substances of Agricultural Chemicals, Feed Additives, and Veterinary Drugs in Food.
Test Method References

6) Food and Drug Administration, Washington, D.C., USA
Pesticide Analytical Manual (PAM) PAM Volume I, 3rd Edition
(Updated October, 1999)

PAM Volume II (Updated January, 2002)
(Index only)

7) Official Methods of Analysis of AOAC INTERNATIONAL (OMA), 18th Edition (AOAC国际官方分析方法)
(Method search only)
8) British Standards

**BS EN 15662:2008**
Foods of plant origin. “Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE. QuEChERS-method”.

**BS EN 15637:2008**
Foods of plant origin. “Determination of pesticide residues using LC-MS/MS following methanol extraction and clean-up using diatomaceous earth”.
Thank you!