



Proposed Regulatory Framework for Pesticide Residues in Food

Analytical Techniques and Development in Pesticide Residues Analysis

1 March 2011



Proposed Regulatory Framework for Pesticide Residues in Food

- Number of pesticides to be included:
 - About 400
- 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.



Trend of Method Development (1)

The use of the number of pesticides is continuously increasing internationally. In order to fulfill the needs of speeding up analytical time and improving the quality, 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 recognised protocols.



Trend of Method Development (2)

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.



Trend of Method Development (3)

Commonly used international analytical performance criteria approaches for pesticides:

- Codex Alimentarius Commission (Codex)

Guidelines on Good Laboratory Practice in
Residue Analysis

CAC/GL 40-1993, Rev.1-2003

- European Union

Method Validation and Quality Control
Procedures for Pesticide Residues Analysis in Food and
Feed

SANCO/10684/2009



Development in Analytical Techniques (1)

Test methods for pesticides can be generally divided into two categories:

➤ Single residue test methods

➤ Based on the characteristics of pesticide residue or the definition of the pesticide, the pesticide needs to be analyzed by individual test method.

➤ Multi-residue test methods

➤ Pesticide residues are suitable for analysis by multi-residue test method which can analyze more than a hundred kinds of pesticide residues under the same testing procedure.

Due to the rapid research and development of the multi-residue test methods, many pesticides, which are used to be analyzed by single residue test methods in the past, are now able to be analyzed by multi-residue test methods.



Development in Analytical Techniques (2)

The advancement of analytical techniques and equipment for pesticide are continuing :

➤ Testing procedure automation, manual work is replaced by employing automated equipment, such as processes in the extraction and cleanup procedure.

➤ Equipment advancement:

- Chromatography : from the use of gas chromatography (GC) to the use liquid chromatography (LC)

- Detectors : from the use of electron capture detector (ECD), flame photometric detector (FPD), etc. to the use of mass spectrometer (MS), tandem mass spectrometer (MS/MS) and time of flight mass spectrometer (TOF-MS), etc.



Preparation for Pesticide Residues Testing

- Instrument acquisition
- Procurement of pesticide standard reference materials and other related chemicals
- Laboratory staff training
- Selection or development of appropriate test methods based on the needs and intended purposes, facilities and resources



Test Method References (1)

- Reference test methods from national institutions or international organizations related to the proposed regulation framework
 - Codex Alimentarius Commission (**Codex**) Standard – Analysis of Pesticide Residues : Recommended Methods
 - National Standards of the People’s Republic of China “Compilation of Official Methods of Analysis for Pesticide Residues” (Vol. 1 & II), 2009.
 - United States Environmental Protection Agency (**USEPA**) - Residue Analytical Method(**RAM**)



Test Method References (2)

- Other reference methods, such as:
 - AOAC Official Methods of Analysis
 - United States Food and Drug Administration (FDA) - Pesticide Analytical Manual (PAM)
 - European Union Reference Laboratories (EURL)
 - Test method published by the Department of Food Safety, Ministry of Health, Labour and Welfare, Japan.
 - British Standards



Pesticide Multi-residue Test Method (1)

Currently, the sample preparation procedure for pesticide multi-residue analysis is mainly based on QuEChERS method or its modifications.

QuEChERS is the abbreviation of Quick, Easy, Cheap, Effective, Rugged, Safe.



Pesticide Multi-residue Test Method (2)

Main steps for multi-residue analysis

- ✓ Sampling and Homogenization
- ✓ Extraction
- ✓ Cleanup
- ✓ Instrumental Analysis

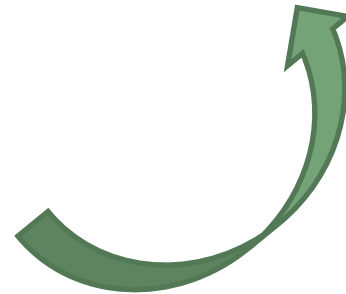
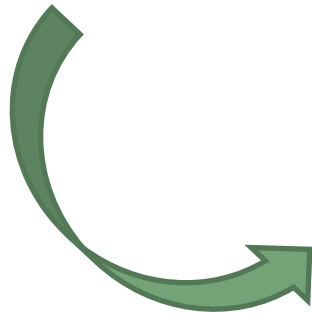
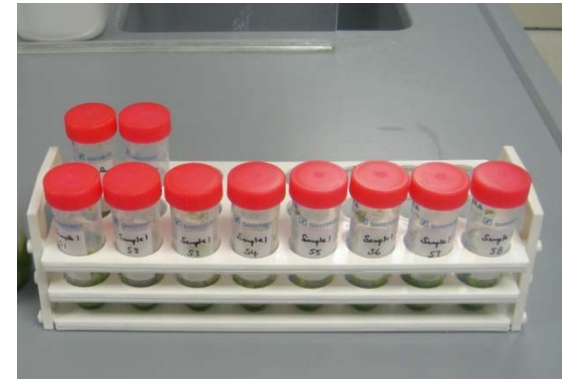


Pesticide Multi-residue Test Method (3)

Sampling and Homogenization:

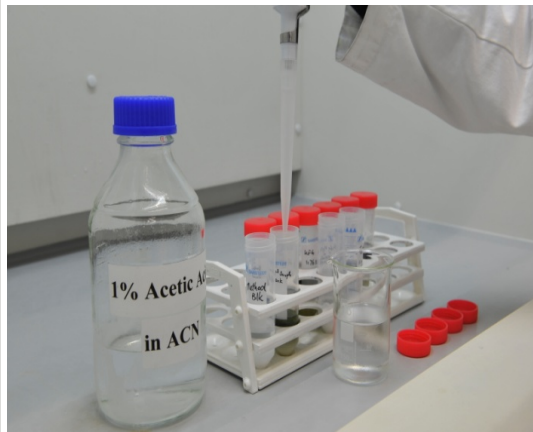


After sampling, chopping and homogenization, weigh the necessary amount of sample into the centrifuge tube.

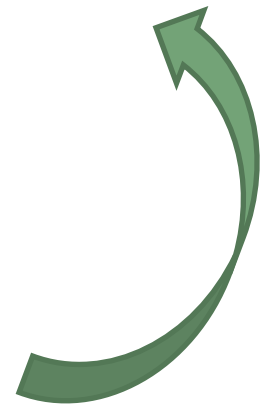
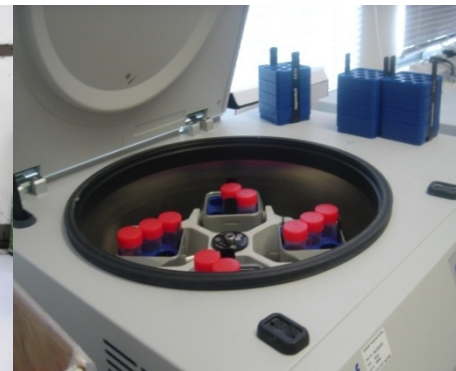
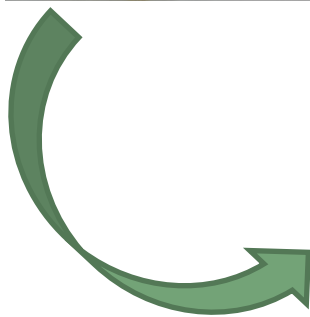
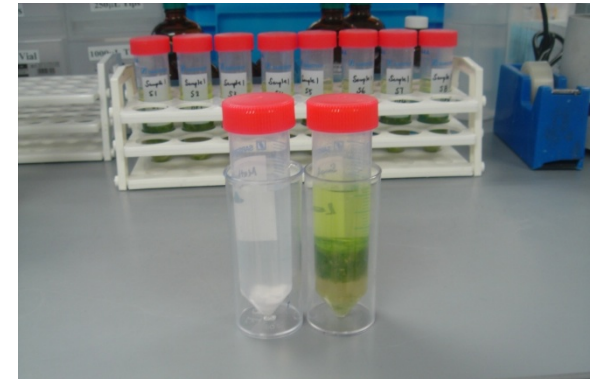


Pesticide Multi-residue Test Method (4)

Extraction:



- Add acetonitrile and shake.
- Add magnesium sulfate and sodium chloride, shake and centrifuge.



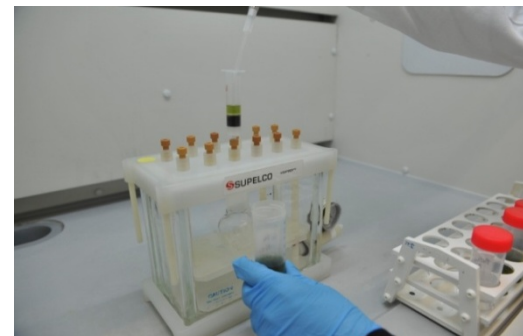
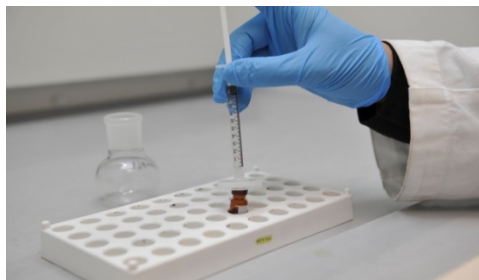
Pesticide Multi-residue Test Method (5)

Cleanup:

Add sample extract from the upper acetonitrile layer to a PSA/GCB dispersive solid phase extraction cleanup tube. Shake and centrifuge.



Cleanup completed



Alternatively, cleanup can be performed by using the dual bed GCB/NH₂ solid phase extraction cartridge.

Other cleanup methods such as C18 column or gel permeation chromatography can assist in the cleanup of meat and seafood matrices.



Pesticide Multi-residue Test Method (6)

Instrumental Analysis:

The Instrument analysis is mainly divided into **gas chromatography (GC)** and **liquid chromatography (LC)** analyses.

For GC analysis, gas chromatograph - mass spectrometer (**GC-MS**) and gas chromatograph - tandem mass spectrometer (**GC-MS/MS**) are mainly employed.

For LC analysis, liquid chromatograph - tandem mass spectrometer (**LC-MS/MS**) is mainly used.



Process of Pesticide Multi-residue Test Method

Traditionally, direct use of quantitative method was usually employed for determination of concentration of pesticides. With the increasing number of pesticides in testing, the multi-residue test method can firstly screen out the pesticides in the samples with strong signal and subsequently confirmation and quantitation analysis are performed.

Analysis can be divided into three steps:

- 1) Screening
- 2) Confirmation
- 3) Quantitation

- This method can reduce work in data analysis.



Thank you!

