

POPs Analysis in Central Lab of Residue Analysis of Pesticides and Heavy Metals in Food

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LAB OF RESIDUE ANALYSIS OF PETROLEUM
AND N.M.C. CENTER

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Historical Background

- Central Lab of Residue Analysis of Pesticides and Heavy Metals in Food is the main output of the project “ Quality Control of Agricultural Products **QCAP**”. This project is a model for successful collaboration between Finland (FINIDA) and Egypt (Ministry of Agriculture) .
- Central Lab of Residue Analysis of Pesticides and Heavy Metals in Food was established in **1995** under **Egyptian Agricultural Research Center (ARC)** which belong to **Ministry of Agriculture**

Laboratory Accreditation

- The lab has been accredited for first time in **1996** by Finnish Accreditation Services (**FINAS-ILAC**) on basis of **EN45001 & ISO Guide 25**
- On **2002** The lab has been Re-Accredited on the new international quality system **ISO 17025**

Laboratory Functions

1. Establishment an effective certification system for Egyptian exports and imports of agricultural products to ensure their conformity with national and international regulations.
2. Organizing and carrying out the national monitoring programs of pesticides, heavy metals, mycotoxins, persistent organic pollutants, and micro-organisms in domestic and imported food products.

Lab Analytical Resources

- 9 Gas Chromatograph (3 ECD, 3 NPD, 2 FPD, 1 FID)
- 1 GC-MS (Low Resolution)
- 1 GC-MS (High Resolution)
- 4 HPLC
- 1 Atomic Absorption Spectrometer
- 1 UV Spectrophotometer

In addition to the technical and mechanical equipments

Analysis Groups

1. Pesticide Residue Analysis (1995)
2. Heavy Metals Analysis (1995)
3. Mycotoxins Analysis (1995)
4. Microbiological Examination (1999)
5. Persistent organic Pollutants (1999)
6. Non Routine Analysis (1999)

Persistent Organic Pollutants Group

- Established in **1999**
- 8 Chemists and 3 technicians
- Analysis of **PCDDs** , **PCDFs**, **DL-PCBs** using **HRGC/HRMS (AutoSpec NT - Micromass)**
- Waiting for Accreditation on analysis of **PCDDs** , **PCDFs**, **DL-PCBs** in Animal-Origin food (**August 2004**)

PCDDs, PCDFs analyzed in the Lab

PCDDs

1. 2,3,7,8-TCDD
2. 1,2,3,7,8-PCDD
3. 1,2,3,4,7,8-HxCDD
4. 1,2,3,6,7,8-HxCDD
5. 1,2,3,7,8,9-HxCDD
6. 1,2,3,4,6,7,8-HpCDD
7. 1,2,3,4,6,7,8,9-OCDD

PCDFs

1. 2,3,7,8-TCDF
2. 1,2,3,7,8-PCDF
3. 2,3,4,7,8-PCDF
4. 1,2,3,4,7,8-HxCDF
5. 1,2,3,6,7,8-HxCDF
6. 2,3,4,6,7,8-HxCDF
7. 1,2,3,7,8,9-HxCDF
8. 1,2,3,4,6,7,8-HpCDF
9. 1,2,3,4,7,8,9-HpCDF
10. 1,2,3,4,6,7,8,9-OCDF

PCBs analyzed in the Lab

DL-PCBS

I-Planar (Non ortho) PCBs

1. PCB 77
2. PCB 81
3. PCB 126
4. PCB 169

II-Non Planar (mono ortho) PCBs

1. PCB 105
2. PCB 114
3. PCB 118
4. PCB 123
5. PCB 156
6. PCB 157
7. PCB 167
8. PCB 189

Other PCBs

1. PCB 28
2. PCB 52
3. PCB 101
4. PCB 138
5. PCB 153
6. PCB 180

Persistent Organic Pollutants Group

Future Trends

- Validation of analytical methods for analysis of PCDDs , PCDFs, DL-PCBs in Water and Soil (and may be in Air) to be accredited in 2005.
- Studying levels of POPs in fish (Bio-indicator) in Egypt (River Nile and lakes) will start in next few weeks (3 M.Sc studies)
- Analysis of Poly Aromatic Hydrocarbons (PAHs), and Poly Brominated Diphenyl Ethers (PBDEs)

Analysis of POPs

- Very complicated Micro-Analysis (ppt, ppq level)
- Long Time
- Expensive
- Highly sophisticated analytical instrumentation
(**HRGC/HRMS**)
- **Isotope Dilution & Internal Standard Technique**
- **Sample Matrix (e.g Water, Soil, Tissue)**

Analytical Procedure

Sample Preparation

Extraction

Cleanup

Quantification

Quality Assurance / Quality Control

Sample Preparation

- Involves modifying the physical form of the sample so that the analytes can be extracted efficiently
- Samples must be in a liquid form or in the form of finely divided solids in order for efficient extraction to take place

Extraction

- Extraction of different analytes depends on type of sample matrix
- Addition of C13-labeled standard before starting extraction
- Soxhelt extraction is the most common extraction procedure

Extract Cleanup

- Cleanup procedures depend on type of sample matrix

1-Gel Permeation Chromatography (GPC)

Removal of high molecular weight interferences that cause GC column performance to degrade

2-Anthropogenic isolation column

Removal of lipids

Extract Cleanup

3-Acid, Neutral, and Basic Silica gel, Alumina, and Florisil

Removal of non-polar and polar interferences

4-Carbopack/Celite

Removal of non-polar interferences

SEPARATION AND IDENTIFICATION

- The analytes are separated by GC and detected by high-resolution (> 10000) mass spectrometer
- Each individual analyte is identified by comparing the GC retention time and ion abundance ratio of two exact m/z 's with the corresponding retention time of an authentic standard and the theoretical ion abundance ratio of two exact m/z 's
- Each individual analyte is identified when retention times and ion abundance ratios agree within predefined limits

Quantification

- Quantification is performed using Selected Ion Recording (SIR) mode
- The GC-MS system is calibrated and the concentration of each native compound is determined using the isotope dilution technique
- The GC-MS system is calibrated and the concentration of each labeled compound is determined using the internal standard technique

HRGC-HRMS (AutoSpec-Ultima)



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Quality Assurance/Quality Control

- The lab has to have a formal quality assurance program consists (at least) of :

1- An initial demonstration of lab capability

2-Analysis of samples spiked with labeled compounds to evaluate and document data quality

Quality Assurance/Quality Control

- 3- Analysis of standards and blanks as tests of continued performance
- 4- Lab performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the analytical method

THANK YOU



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