

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

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

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

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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
- The lab has been accredited in **1996** by **FINAS** on basis of **EN45001 & ISO Guide 25** and on **2002** the quality system has been updated on basis of **ISO 17025**

# Historical Background

- Persistent Organic Pollutants Section has been established in September 1999
- Technical Assistance from Technical Research Center of Finland (VTT)
- High Resolution GC-MS (AutoSpec-Ultima) in January 2003

# Laboratory Functions

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

# Organization structure

## I-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)

# Organization structure (Cont.)

## II-Supporting Sections

1. Supporting staff (Maintenance, Computer services, and security)
2. Administration (Accounting, Purchasing, Stores, Personnel)
3. Client Services
4. Reporting
5. Information Center

# Organization structure (Cont.)

## III- Assisting Groups

1. Sample reception
2. Sample preparation
3. Glassware washing

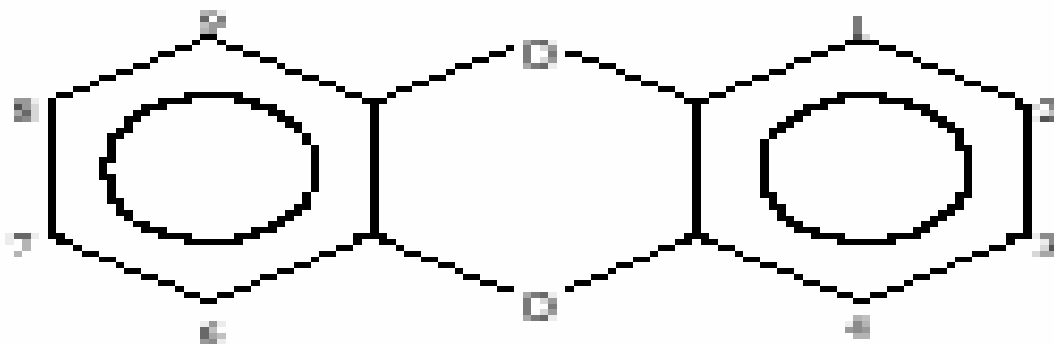
# Persistent Organic Pollutants (POPs)

- 1. Pesticides (e.g HCB, DDT, HCH, Toxaphene)**
- 2. Industrial Products : PolyChlorinated Biphenyls (PCBs)**
- 3. By-Product of combustion and industrial Process :**
  - 3.1 PolyChlorinated Dibenzo-p-Dioxins (PCDDs)**
  - 3.2 PolyChlorinated Dibenzofurans (PCDFs)**

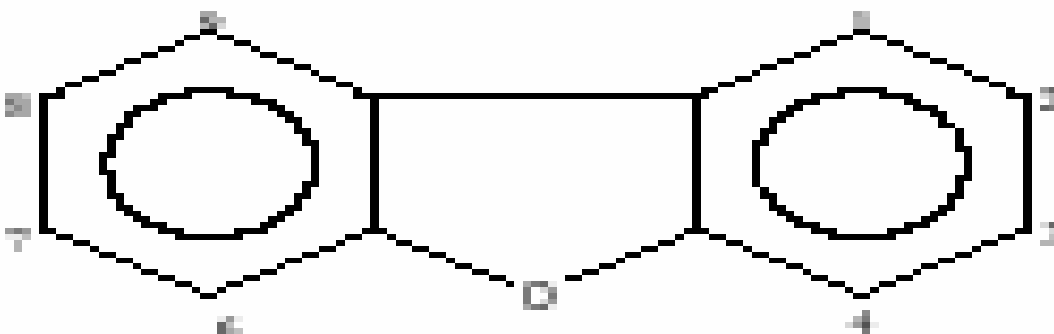
# Characteristic Features of POPs

1. Organic Compounds
2. Highly Resistant to photolytic, chemical, and biological degradation
3. Low water solubility
4. High lipid solubility
5. Bioaccumulation
6. Semi-Volatile

# Chemical Structure of Dioxins and Furans



Dibenzo-p-dioxin



Dibenzofuran

# Dioxins and Furans

- Dibenzo-*p*-dioxin and dibenzofuran, when chlorinated are collectively known as dioxins. There are 75 possible congeners of chlorinated dibenzo-*p*-dioxin and 135 of dibenzofuran. Only those containing chlorine at the 2,3,7 and 8 positions are toxic. There are 17 of these toxic congeners (often called the “dirty seventeen”).

# Persistent Organic Pollutants Group

- Established in 1999
- 8 Chemists and 3 technicians
- Analysis of PCDDs , PCDFs, DL-PCBs using HRGC/HRMS (AutoSpec NT – Micromass - Water)
- Waiting for Accreditation on analysis of PCDDs , PCDFs, DL-PCBs in Animal-Origin food (Sep 2005)

# 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

## Other 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

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

# Laboratory Functions

- Organizing the national monitoring programs of persistent organic pollutants in domestic and imported food products.

# 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 2006.
- Studying levels of POPs in fish (Bio-indicator) in Egypt (Mediterranean Sea, Red Sea, River Nile and lakes) by 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)



# 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

# Laboratory Authorization

1. The Prime minister decree no. 1202/1999 authorized the laboratory to analyze Poly Chlorinated Dibenzo Dioxins (PCDDs), Poly Chlorinated Dibenzo Furans (PCDFs), and Poly Chlorinated Biphenyls (PCBs) compounds in animal-origin food shipments imported to Egypt from all countries.

# Laboratory Authorization

2. Ministerial Decree No. 711,798/2000 prohibit the importing of the animal-origin food and feed from any country in case of rejection of five imported animal-origin food and feed shipments from this country as a result of contamination of these five shipments with Poly Chlorinated Dibenzo Dioxins (PCDDs), Poly Chlorinated Dibenzo Furans (PCDFs), and/or Poly Chlorinated Biphenyls (PCBs) compounds according to the analysis carried out in the Central Laboratory of Residue Analysis of Pesticides and Heavy Metals in Food

# THANK YOU

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