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ANALYSIS OF ORGANOCHLORINE PESTICIDE RESIDUES AND RELATED COMPOUNDS

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The problem of environmental contamination by persistent organochlorine pesticides and related compounds still evokes a major concern due to the presence of their residues in the environment and in human tissues. Polychlorinated biphenyls (PCBs), Polychlorinated dibenzo-p-dioxins (PCDDs), Polychlorinated dibenzo-furans (PCDFs), Aldrin, Isodrin, Dieldrin, Endrin, Hexachloro cyclohexane (HCH), DDT etc. members of a family of persistent organochlorines are considered as the most toxic chemical group among the man-made organic contaminants of great concern. In developing countries like India, organochlorine pesticides especially DDT are extremely being used in agriculture and vector control programmes. High concentration of DDT found in the marine environment has led to a total ban on its use in USA, Europe and Japan. On an average, 55000 tons of pesticides are used every year in India for agricultural purposes. The world consumption of pesticide during the period, 1980-95, is around 4571-6445 thousand tons. The cumulative effects of these pesticides can be expected to be considerable on the aquatic living beings of the coastal environment. Attempt is therefore, made all over the world to assess the levels of contamination of the environment by the chlorinated hydrocarbons. Before going into the details of the methods of a analysis of pesticides it would be justified to discuss about the following points:

1. What is pesticide ?
2. Classification of the pesticide.
3. Why the analysis of organochlorine pesticides and related compounds is so important.
4. What are the source of pollution ?
5. What are the steps to be followed for the analysis of pesticides and related compounds.

Discussion:

Pesticide:

It is a generic name of the chemical compounds which control the pests. The pests are of different types. The pesticides are being named according to the mode of action of each class of compounds, eg. insecticides are those which can control the insects, acaricides are ment for controlling the acarides, so also the weedicides for weeds, miticides for mites, germicide for germs, fungicide for fungus and so on and so forth.

Classifications of pesticides:

Pesticides are classified according to the purposes for which it is being used. Broadly, pesticides are classified as inorganic pesticides and Organic pesticides. Inorganic pesticides are of different types, insecticide,

fungicides, herbicides, rodenticides. Organic pesticides are classified as insecticides, herbicides, fungicides, acaricide, miticides, bactericides, germicides, etc. Organochlorines, organophosphates, carbamates, pyrethroids and Nitrophenols, chlorophenoxy, dipyridyls, ureas, uracils, triazines, thiocarbamates, etc. are belonged to the class herbicides whereas organotins, organomercurials, dithiocarbamates, organophosphates, and other organics are belonged to the class of fungicides.

Importance of Organochlorines:

Organochlorine pesticides are in general, fat soluble and highly persistent for which they are accumulated in the food chain easily posing a great threat to the human health. So it becomes an urgent need to assess the level of contamination in the environment due to organochlorine pesticides.

Sources of Pollution:

The first and foremost source of pollution is the industrial discharge, secondly the land drainage from the agricultural fields and also the river run off. Besides, the atmospheric weathering is one of the major path of contamination of the environment.

Steps to be followed for the analysis of the pesticides:

1. Collection of samples.
2. Extraction of pesticides and related compounds.
3. Purification and clean up of the samples.
4. Analysis of the purified extracts.

Collection of samples:

Different types of apparatus are used to collect different types of samples.

- a) Van Veen grab, Peterson grab for the sediment samples.
- b) Box corer, Gravity corer and Piston corer for coring sediment samples.
- c) Nansen bottle, Niskin bottle for water samples.
- d) In-situ sampler for concentration of organic from water.
- e) Vacuum pump filter for atmospheric samples.

Materials required for the extraction:

- a) Solvents : Acetone, Dichloromethane, Methanol, Hexane, Pentane, Isopentane.
- b) Electrolytes: Amm. chloride, Sodium chloride, Potassium chloride, etc.
- c) Dehydrating agent: Anhydrous sodium sulfate.
- d) Acids : Conc. sulphuric acid.
- e) Chromatographic columns for purification : Florisil, Silica, activated charcoal, activated copper.

- f) Concentrator: Kuderna danish evaporator.
- g) Glass apparatus: Soxhlet apparatus, Beakers, glass bottles, round bottom flasks, graduated cylinders with stopper, volumetric flasks, Highly sensitive balance.

Methods of Extraction

a) Sediment sample :

About 100-150 gm sediment sample is to be treated first with 15 ml 1% Amm. chloride and then kept for over night. The moistened sediment will have to be shaken thoroughly with 200 ml acetone thrice, acetone and hexane mixture (1:1) and finally with 200 ml hexane for about eight hours. The combined extract will have to be evaporated almost to dryness and re-dissolved in double distilled hexane which is subsequently to be purified with conc. sulphuric acid. The acid will be washed out by 1% sodium bicarbonate solution. The moistened extract will be dehydrated with anhydrous sodium sulfate powder. The anhydrous extract will then be treated with activated copper powder in order to remove any sulphurated compounds. Finally the purified extract will be evaporated to reduce the volume up to 5 ml and kept ready for analysis by gas chromatography coupled with electron capture detector.

b) Water sample:

Organochlorine pesticides and related compounds can be extracted from the water sample by passing a large volume of water (100-200 l) through XAD-2 resin column or polyurethane foam. The resin will then be eluted by different solvents, eg. methanol, acetone, dichloromethane, hexane, etc. The combined extract will be evaporated to dryness and redissolved in hexane which is subsequently purified by stepwise treatment with conc. sulfuric acid, 1% sodium bicarbonate solution, anhydrous sodium sulfate, activated copper powder. The purified extract will be reduced to a volume of 5 ml and kept ready for analysis by GC. coupled with electron capture detector.

c) Biological sample:

The soxhlet method of extraction is generally recommended for the biological samples. The purification and clean up procedure is same as above.

Techniques of operation of Gas Chromatograph:

- 1) Selection of chromatographic column.
- 2) Selection of the carrier gas to be used.
- 3) Selection of the detector to be used.
- 4) Conditioning of the column.
- 5) Conditioning of the instrument.
- 6) Calibration of the bleeding of the column.
- 7) Calibration of the optimum temperature of the column.
- 8) Calibration of the optimum temperature of the detector.
- 9) Calibration of the flow of the carrier gas.
- 10) Selection of the attenuation of the instrument.

- 11) Selection of the chart speed of the printer.
- 12) Determination of the column efficiency.
- 13) Standardization of the method of quantitation of the compounds with the help of standard samples.
- 14) Reproducibility.
- 15) Identification of the peaks with respect to the reference peak.
- 16) Determination of the relative retention time.