17.0 ESTIMATION OF ORGANOCHLORINE PESTICIDE RESIDUES – GC METHOD

Reagents

- Acetonitrile
- Distilled water
- Petroleum ether (40-60ºC)
- Sodium chloride (AR grade)
- All standards from Sigma.

Apparatus

- Conical flask
- Separating funnel
- Graduated cylinder
- Florisil column
- Blender

Fig. 17.1 Pesticide residues estimation by GC

Procedure

Extraction

- Blend about 50 g sample with 350 ml acetonitrile: water mixture (65:35) for 5 minutes at high speed, (the addition of water to acetonitrile increases the extractability of organochlorine pesticide residues in dry fodders and straws) and filter with the help of filter paper (Whatman No.1) into 500 ml conical flask.
- Transfer the filtrate to 250 ml graduated cylinder and record the volume.
- Transfer the filtrate into 1000 ml separating funnel.
- Measure 100 ml petroleum ether and pour into the separating funnel.
- Shake vigorously (1-2 mts) and add about 10 ml saturated sodium chloride solution and 600 ml of water mixed gently and thoroughly.
- Allow to separate.
- Discard aqueous layer and washed the solvent layer twice with 100 ml portions of water. Discard washings.
- Transfer the solvents to 100 ml stoppered graduated cylinder and recorded the volume.
- This solvent is concentrated to 5-10 ml before it is passed through the column.
- Extract the concentrated mixture and feed ingredients for OCPRs along with fat by the soxlet extraction. About 0.5 g extracted fat was dissolved in 5 ml portion of petroleum ether and passed through a column containing florisil for separation.

Clean up of the residue

The extract is ready for column chromatography, on partially deactivated florisil cartridges (Bond elut @ Varian).

Gas chromatographic analysis

The cleaned up residues were measured using a gas chromatograph equipped with ECD (Fig. 17.1).

Column - Equity TM -5, 30 m x 0.25 mm ID x 0.25 micro m

Operating conditions

Injection port temperature	- 225ºC
Column oven temperature	-100ºC (2 min), 15-160ºC/min, 5-300ºC/min (10min)
Detector temperature	- 310ºC
Carrier gas	- Helium, 30 cm/sec @ 100ºC
Total run time	- 35 min.

$$\text{Pesticide residues (µg/l)} = \frac{A \times B \times C \times D}{E \times F \times G}$$

Where,

A - ng of standard pesticide
B - Peak area of sample
C - Extracted volume (µl)
D - Dilution factor
E - Peak area of standard
F - Volume of extract injected (µl)
G - Volume of sample extracted (ml)

Reference: AOAC (1997) 16th edition.