

Searching for Endrin residue on cotton seed, by Gas

chromatography method.

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Experimental procedure:

50 gr. of sample (cotton seed, three weeks after spraying) was weighed and crinded in a Blender until a homogenous powder was obtained. The sample was transferred into a suitable stoppered glass jar and 300 ml. n-Hexan and 50 gr. anhydrous Na_2SO_4 was added, and 1/2 hour shaken in shaker and left overnight in Refrigerator. The sample was filtered and the filtered phase evaporated in Rotation Distillation apparatus.

The remainder oily layer was transferred into an Erlenmeyer flask. 1 ml of 50 % KOH and 6 ml of isopropyl alcohol was added per gram of initial sample into the flask. (50 ml of 50 % KOH and 300 ml isopropyl alcohol). The flask was attached to a reflux condenser and was gently boiled on a hot plate for 1 hour. The solution was left to cool and when cold, through the top of the condenser a volume of distilled water equal to the volume of solution was added to the flask. The mixture was transferred to a separatory funnel and 150 ml of n-Hexan was added, and shaken vigorously for a minute. After the two phases were separated, the lower water phase was drained. The n-Hexan phase was transferred to a 1-liter separatory funnel and the water phase was returned to the original separatory funnel. This aqueous phase was reextracted 5 times with 50 ml portions of n-Hexan, each time the n-Hexan portions were combined with the total extract.

The combined n-Hexan extract was washed with 300 ml portions of distilled water until the water layer was colorless to phenolphthalein. To the n-Hexan extract a sufficient quantity of anhydrous Na_2SO_4 was evaporated until dryness, the remainder was collected with 4-5 ml n-Hexan and passed through a 3 x 25 cm chromatography column joined to a 250 ml Erlenmeyer flask at the bottom. The bottom of the column was filled with the following absorbents:

1. Anhydrous Na_2SO_4 to give a height of 0.5 cm.
2. 300-mesh. Florisil to a height of 10 cm.
3. Magnesium silicate (activity grade I) to a height of 5 cm.

Then the side arm of column was attached to vacuum pump to press the absorbents and a top layer of 3 cm of anhydrous Na_2SO_4 and a pad of glass wool was added. The column was washed with 100 ml n-Hexan and vacuumed.

The concentrated extract was poured on the top of column and 10 ml n-Hexan was poured 9 times on the top of column. The n-Hexan elution was evaporated (R.D) until dryness and the remainder was collected with about 8 ml n-Hexan and evaporated with water jet vacuum to 1 ml. The remainder was transferred into a 10 ml. volume-trique flask and made up to the mark with n-Hexan 1 ml. of the fival solution at this stage was equivalent to 5 gr. of cotton seed.

Specification of Gaschromatografy:

Model: H4-FI 600-D
Support: 5% SE30 60/80
Celounm Clrom.W5×0.8
Detecfor: E.C.
Recoroer: Honeywell
Chart Speed: 1

Specification of injected samples:

Each test had four treatments consisting of:

1. Blank (Extractions from 50 grs. of untreated cotton seed).
2. Sample. (50 grs. cotton seed three weeks after spraying).
3. Recovery (20γ Endrin was added to 50 grs of cotton seed).
4. Standard solution (20γ Endrin 1 ml n-Hexan).

10 l of sample extract, recovery and blank and 1 l of standard solution was injected.

The resicue of Endrin was calculated by comparison of the area of sample peak with that of the standard and shown as p.p.m.

Test conditions:

Temp. of G.L.C 170 o C.
Flow of Nitrogen 60 ml/minute
Range switch on 1
Attenator switch on 4

The following table shows the P.P.m. quantities of Endrin:

Sample	Recovery	Blank	Recovery per cent
0.343	0.350	0	87.5
0.112	0.370	0	92.5
0.408	0.330	0	82.5
0.170	0.350	0	87.5

The average residue of Endrin was found to be 0.258 P.P.m in the seeds of sprayed cotton.

Note: In n.a.c Journal from U.S.A. the accepted tolerance for Endrin residue on cotton seeds is established as zero.