# DETERMINATION OF RESIDUES OF TRANSFLUTHRIN AND PERMETHRIN IN AIR WHILE USING AN AEROSOL FORMULATION

BY

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# OBJECTIVES

- To develop an analytical method to quantify the residues of Permethrin and Transfluthin in air samples

- To establish an effective procedure to collect air samples following the use of Transfluthrin 0.6% w/w + Permethrin 0.8% w/w Aerosol in a closed chamber

# AEROSOL DISPENSER SYSTEM

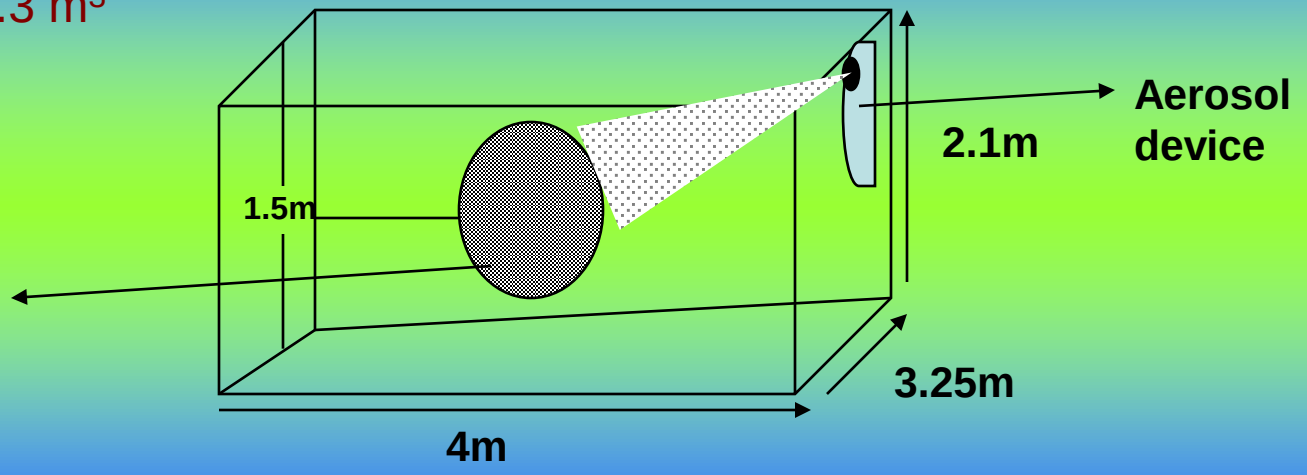

- Dispenser  works on 15 minutes interval
- Declared volume in the can is 154 grams
- Life of the can is 28.9 ~ 30 days
- Spray Rate is 0.055 gram/spray

# STUDY DESCRIPTION

- This residue study was conducted using an automatic aerosol dispenser
- The sprayer was switched on for 24 hours at a spray rate of 0.055g/spray once every 15 minutes
- The residues of permethrin and transfluthirun in air were estimated for a period of 24hrs in a pre-determined intervals (0, 2, 4, 6, 10 and 24 hours)
- Air Samples were collected at 1.5 m height from the floor using cartridge

# SCHEMATIC DIAGRAM - AEROSOL SPRAY

Height – 2.1 m
Floor area – 13 $m^2$
Total Volume – 27.3 $m^3$




- 500 mL of air sample was withdrawn from the chamber approx. 3-5 minutes after the spray
- The collected air sample was pre-concentrated and analyzed by a validated GC-ECD method

# TEST ITEM DETAILS

| | | |
|---|---|---|
| 1. Name of the compounds: | | i. Transfluthrin<br>ii. Permethrin |
| 2. Nature of the compound : | | Insecticide |
| 3. Type of formulation | : | Aerosol |
| 4. Percentage of active ingredient | : | i. Transfluthrin – 0.6%<br>ii. Permethrin – 0.8% |

# INSTRUMENTS/EQUIPMENTS

- Shimadzu GC-17A GC-ECD, Auto injector AOC - 20i with GC-solution software. M/s Shimadzu Corop., Japan
- Mettler Toledo analytical balance AG-245, capable of weighing 0.01 mg supplied by M/s.Mettler Toledo, Switzerland.
- Buchi temperature controlled vacuum rotary evaporator supplied by M/s.Buchi Rotovapour, Switzerland.

# GAS CHROMATOGRAPHY ANALYSIS PARAMETERS

Detector -$^{63}$Ni Electron Capture Detector (ECD)

Column used -DB-210 Megabore (30m length x 0.53 mm I.D., 1.0 µm film thickness).

Temperature conditions

Oven -150°C

Injector -240°C

Detector -260°C

Gas flow rate

Nitrogen -10 mL/min

Makeup -30 mL/min

Column temperature program

| Rate | Temperature °C | Hold time (min) |
|---|---|---|
| - | 150 | 5 |
| 20 | 220 | 6.5 |

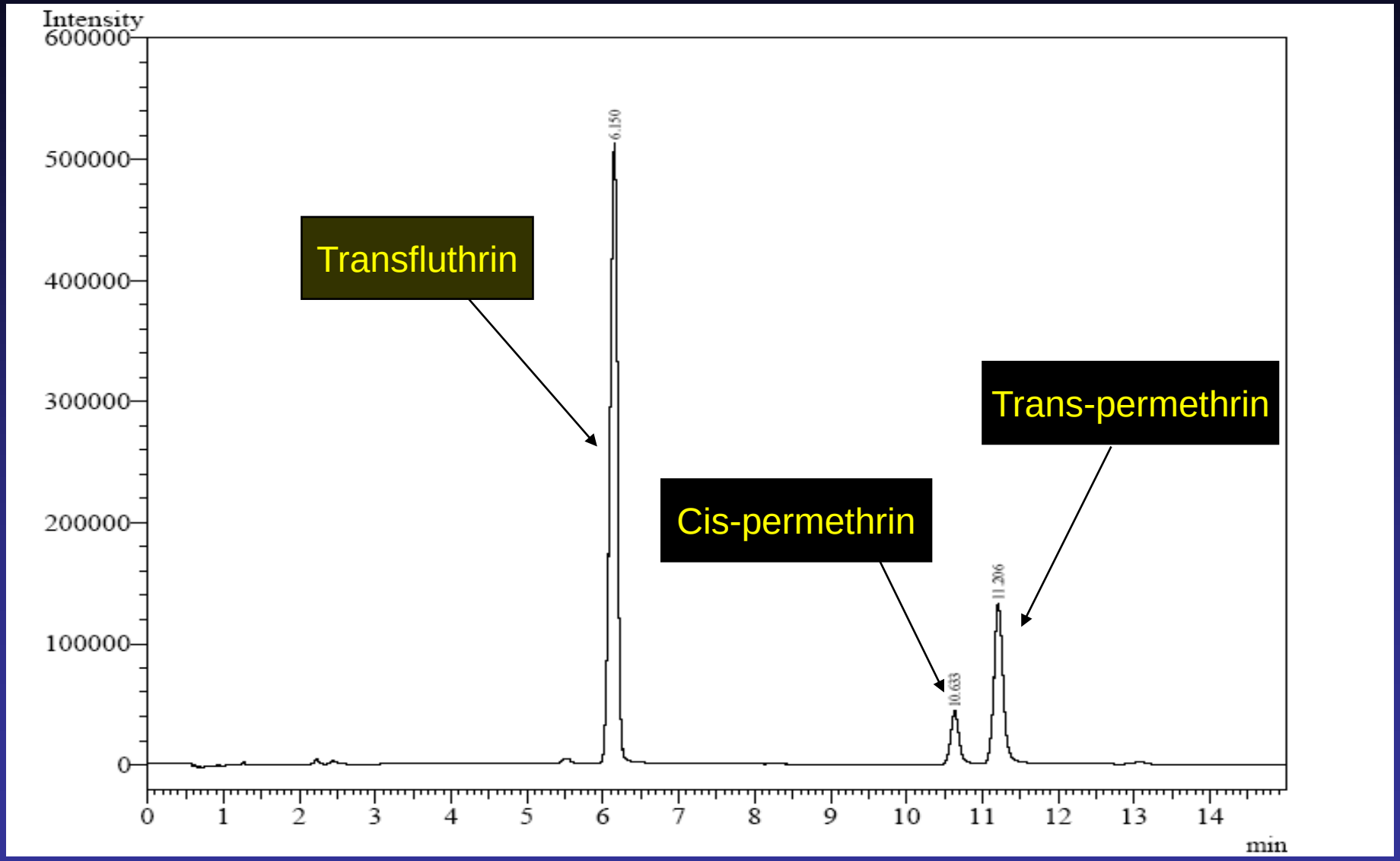
Retention time (approximate)

Transfluthrin (Standard) -6.1 minutes

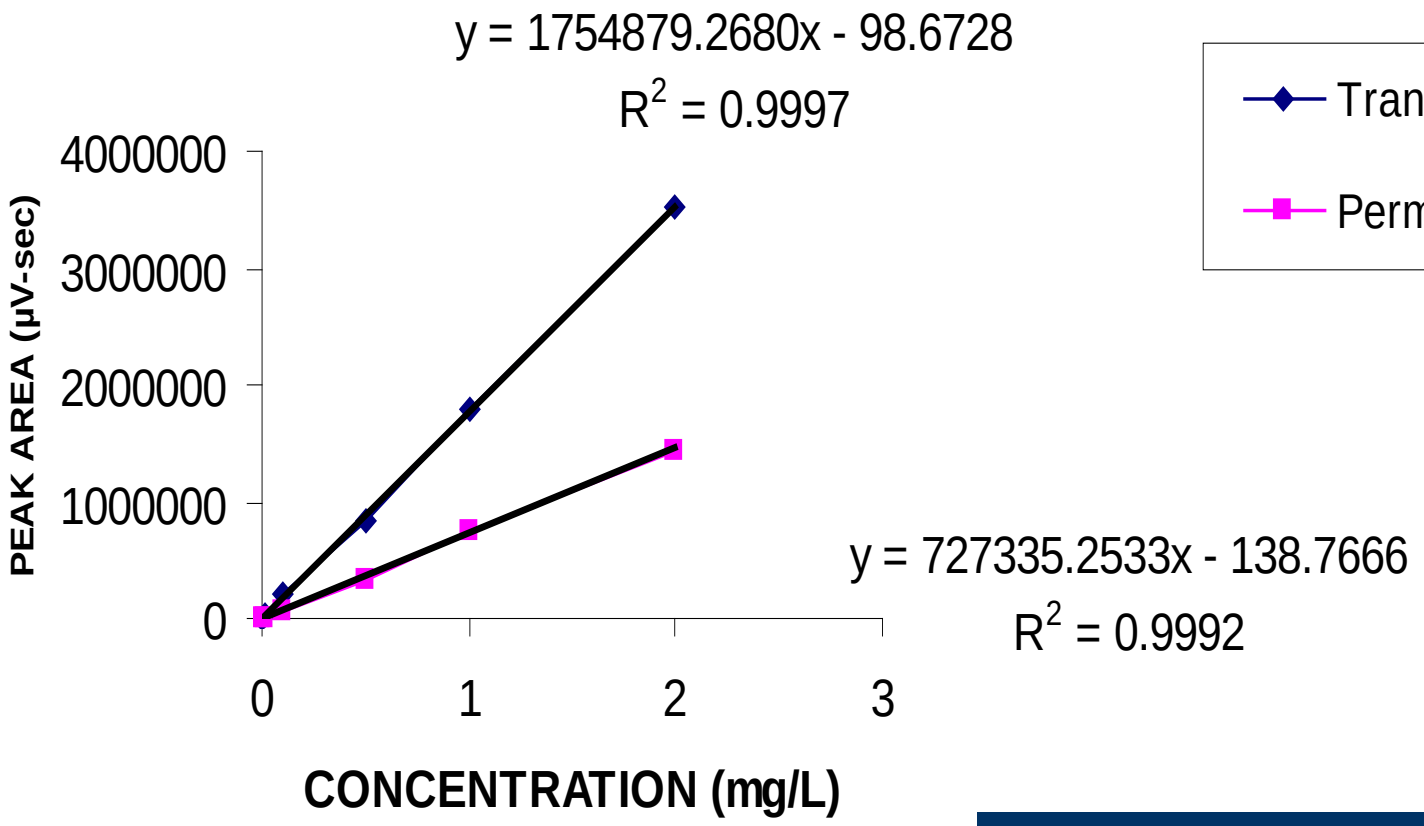
Permethrin (Standard) Cis -10.5 minutes

Trans -11.2 minutes

# REPRESENTATIVE CHROMATOGRAM OF GC-ECD



# METHOD VALIDATION - LINEARITY




**Calibration range 0.001 – 2 mg/L**
**LOQ = 0.001mg/L**

# METHOD VALIDATION - RECOVERY

Spikings: 1, 0.1 and 0.001 mg/L level on Carbon cartridge

↓

Suction of 500mL of Clean air

↓

Eluted with 10mL of Acetone

↓

Analyzed 1µL by GC-ECD

# METHOD VALIDATION - RECOVERY

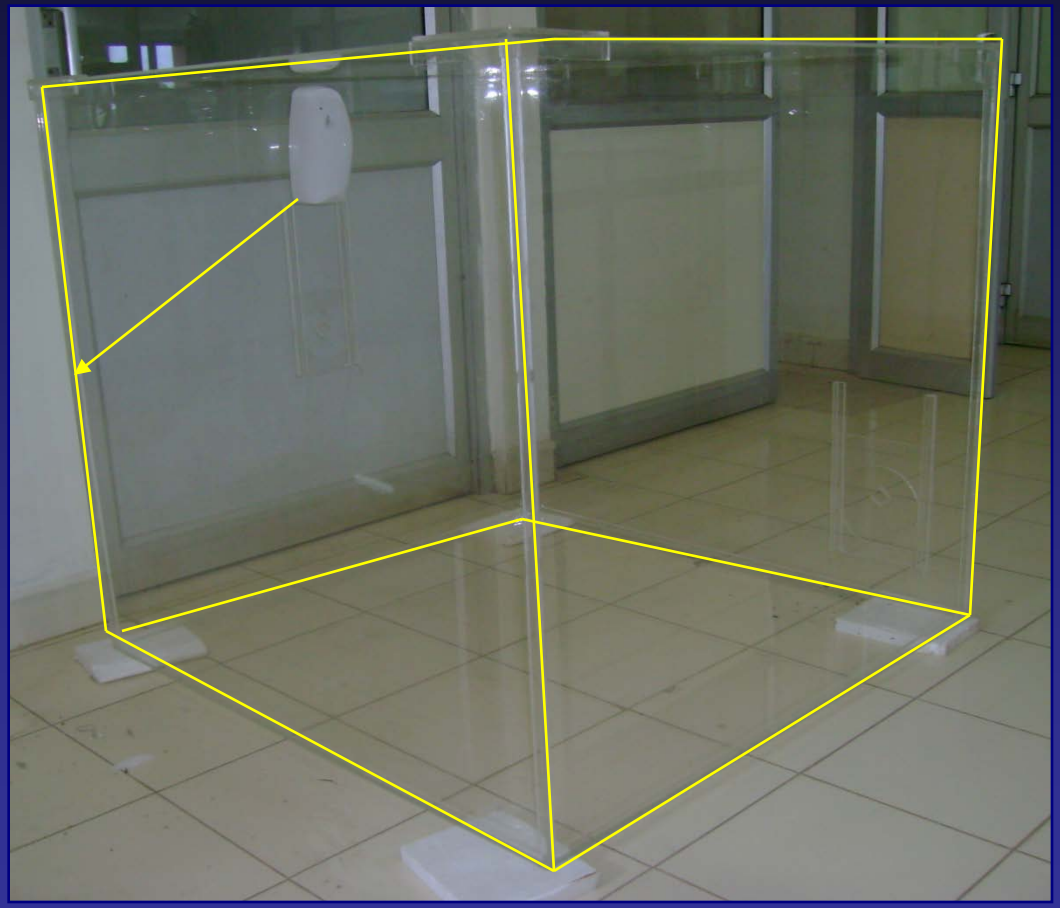
| Fortified concentration (mg/L) | Transfluthrin Recovery (%) | Permethrin Recovery (%) | Transfluthrin mean Recovery (%) ±SD | Permethrin mean recovery (%) ±SD |
|---|---|---|---|---|
| 0.001 | 89.98 | 96.88 | 93.46±3.34 | 93.50±4.04 |
| 0.001 | 93.75 | 89.03 | | |
| 0.001 | 96.65 | 94.59 | | |
| 0.1 | 90.5 | 92.38 | 92.51±2.49 | 94.67±2.00 |
| 0.1 | 95.29 | 95.62 | | |
| 0.1 | 91.74 | 96.02 | | |
| 1 | 94.48 | 93.08 | 94.36±2.26 | 94.38±1.71 |
| 1 | 92.05 | 96.32 | | |
| 1 | 96.56 | 93.75 | | |

SD - Standard deviation

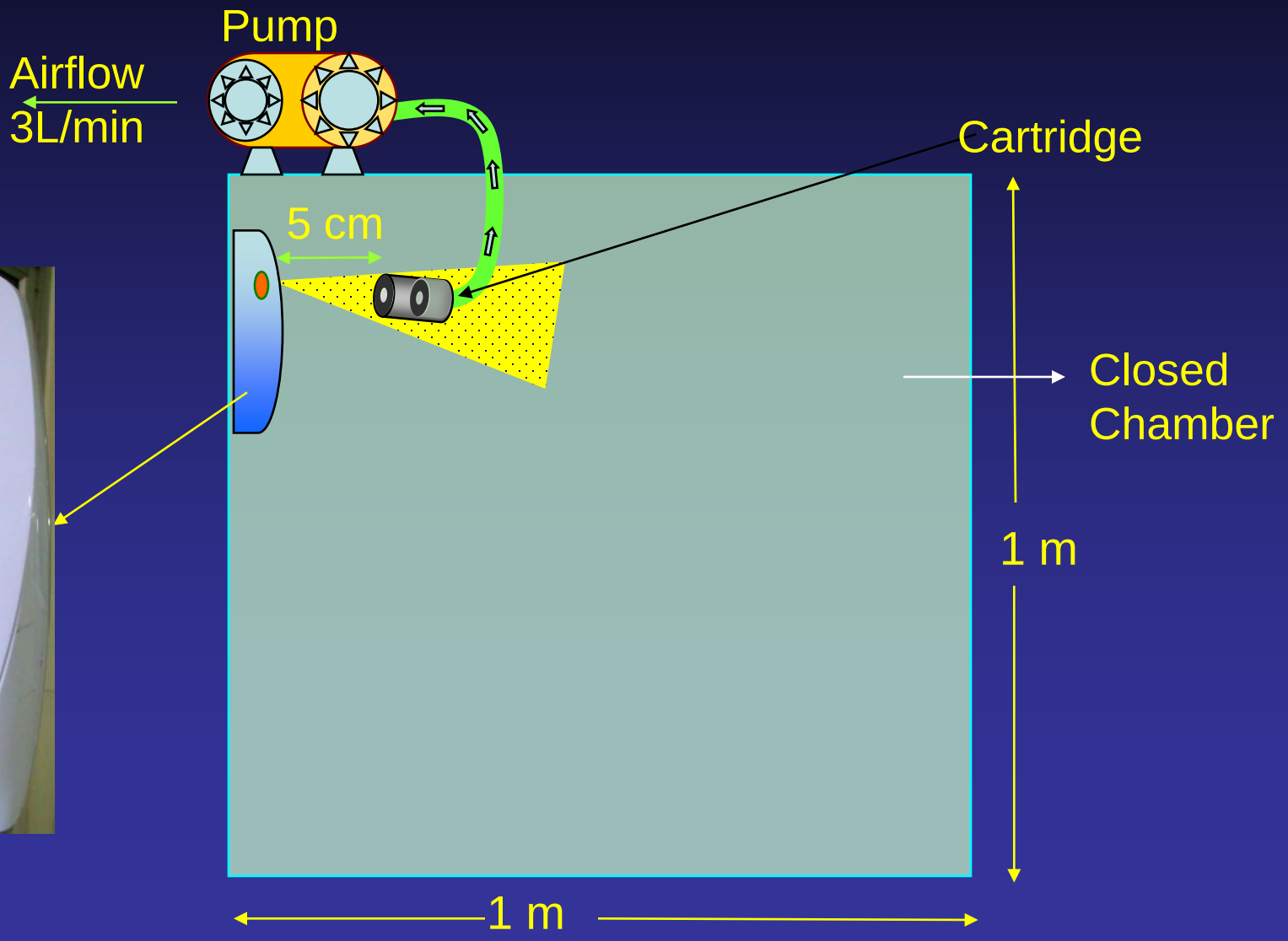
# METHODOLOGY – SAMPLE COLLECTION

- An automatic insect control aerosol system was fixed on wall in one corner of the testing chamber.
- Air samples were collected by placing the carbon cartridge at **5 centimeters** distance from the sprayer nozzle.
- 500 ml of air samples were withdrawn from the chamber at 0-10, 10-20, 30-40 and 60-70 and 90-100 seconds after the spray at a rate of 3 L /min for 10 seconds using a cartridge connected to a vacuum pump equipped with flow regulator
- Trapped residues were eluted using 10 ml acetone twice.
- Evaluated the residues of transfluthrin and permethrin using a validated GC-ECD method.

# AIR CONCENTRATION CHAMBER - SETUP



# STUDY DESIGN – AIR CONCENTRATION RECOVERY TRAP



# PRELIMINARY INVESTIGATIONS

- No residues were found when carbon trap was kept at 20, 30, 45 and 60 cm away from cartridge

- Non-recovery of residues at longer distance can be attributed due to:

  – Low concentration of residues in air samples

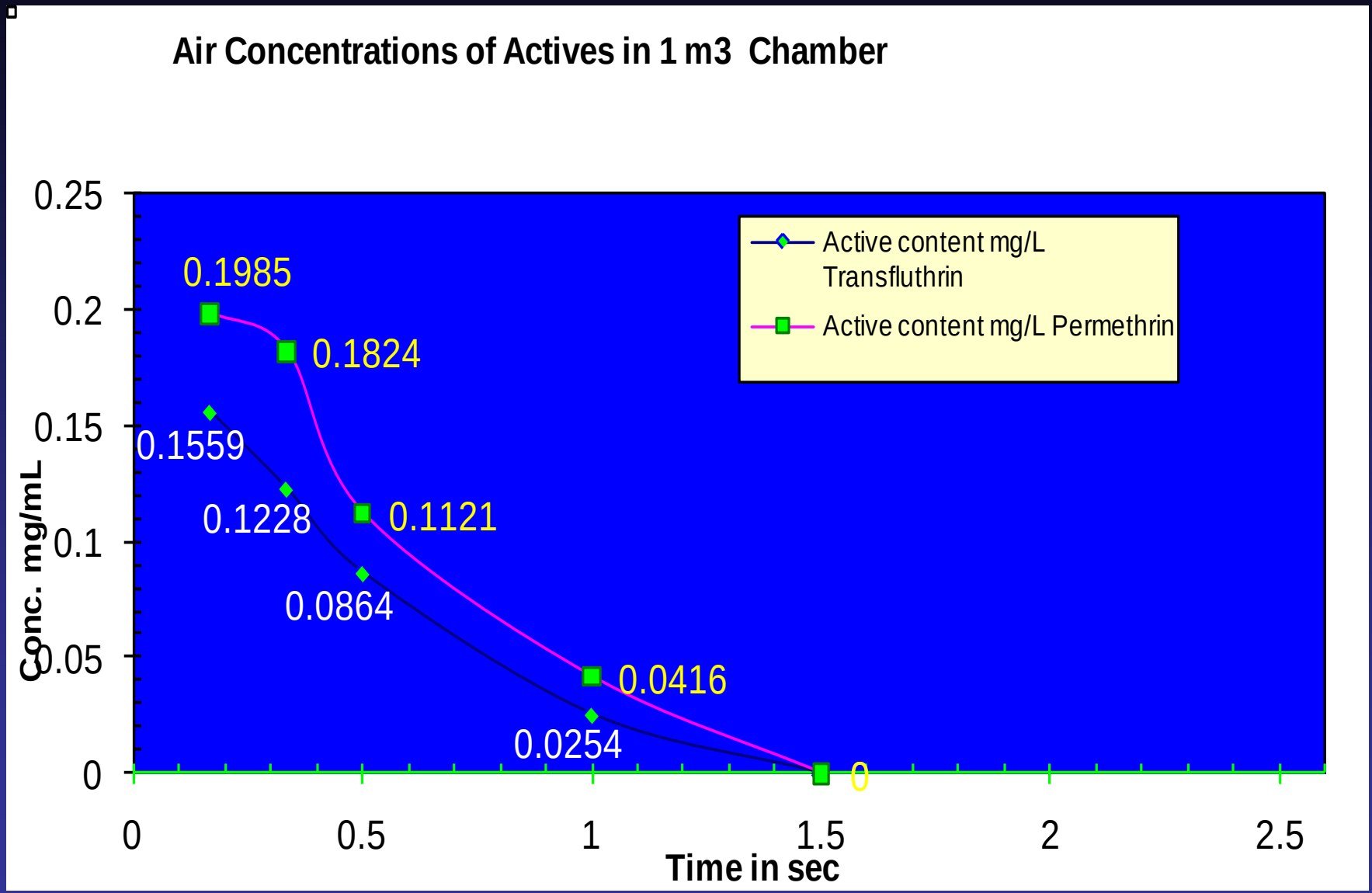
  – Requirement of huge volume air sample to concentrate the residues on the cartridge in a shortest plausible time

  – 5 cm distance between tip of the nozzle and the carbon trap was found optimum.

# PRE CONCENTRATION OF TRANSFLUTHRIN AND PERMETHRIN FOLLOWED BY 0.055 G/SPRAY USING CARBON CARTRIDGE KEPT AT VARIED DISTANCES

| Time gap between Spray and Sample Collection (sec) | Sample Collection Duration (sec) | Sample Collection flow rate (L/min) | Volume of air (mL) | Transfluthrin active content (mg/L) | Permethrin active content (mg/L) |
|---|---|---|---|---|---|
| 0-10 | 10 | 3 | 500 | 0.1559 | 0.1985 |
| 10-20 | 10 | 3 | 500 | 0.1228 | 0.1824 |
| 30-40 | 10 | 3 | 500 | 0.0864 | 0.1121 |
| 60-70 | 10 | 3 | 500 | 0.0254 | 0.0416 |
| 90-100 | 10 | 3 | 500 | BLQ | BLQ |

BLQ - Below Limit of Quantification


Air Concentrations of Actives in 1 m3 Chamber
0.25
0.2
0.15
0.1
0.05
0
Conc. mg/mL
0
0.5
1
1.5
2
2.5
Time in sec
Active content mg/L Transfluthrin
Active content mg/L Permethrin
0.1985
0.1824
0.1559
0.1228
0.1121
0.0864
0.0416
0.0254
0

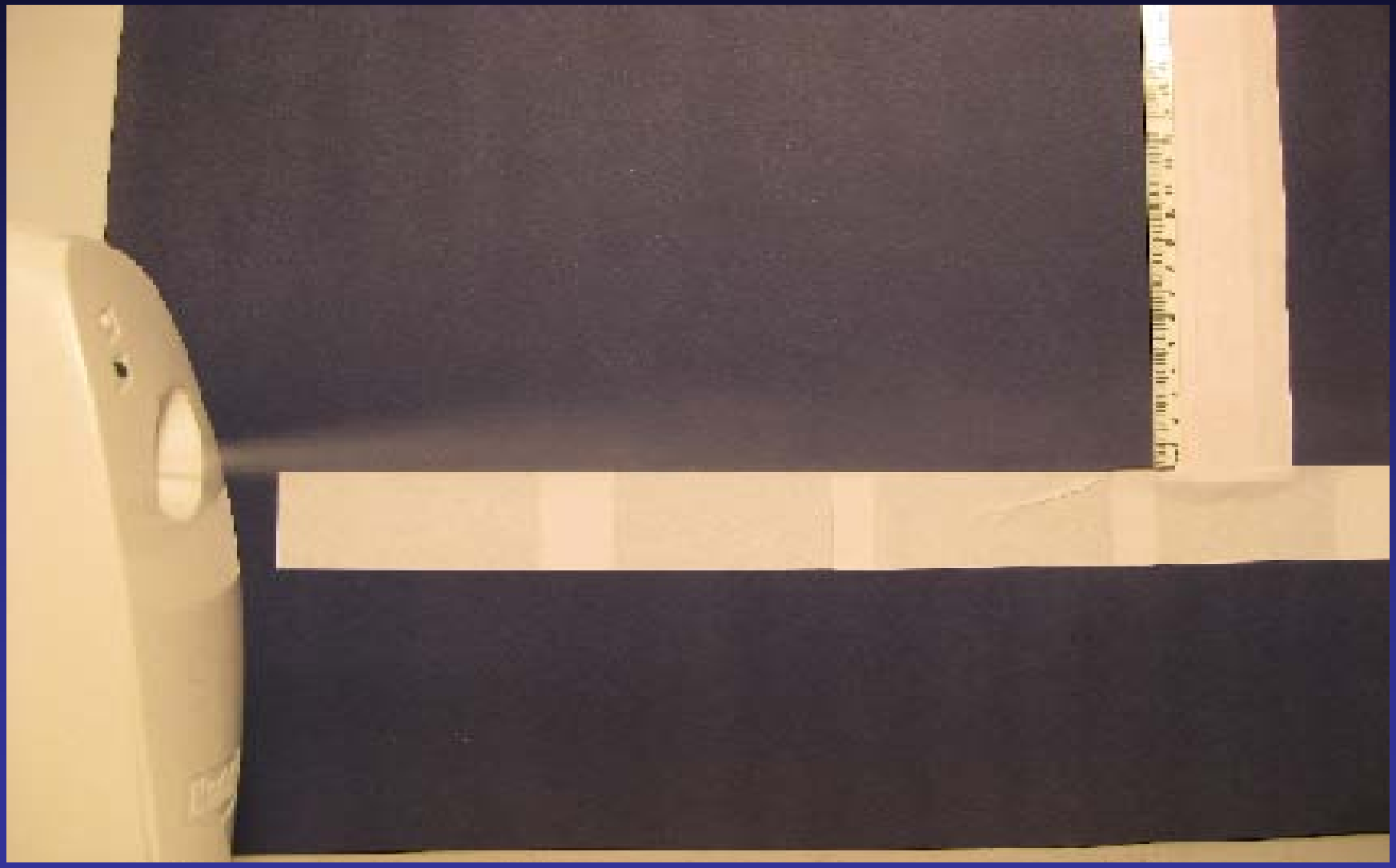
# RESULTS WITH SINGLE SPRAY

- The residues of permethrin and transfluthrin were found to be below detectable level in all sampling occasions

- Non-recovery of residues can be attributed due to:
  - Low concentration of the residues in air
  - The actives may reduces  quit quickly after  the spray
  - Huge volume of air sample may be  required to pre-concentrate the residues on cartridge
  - Sampling and extraction method need to be  optimized

- The present study is designed to minimize such influential  parameters Conclusion

# RESIDUE ACCUMULATION IN A ROOM DUE TO 8 HRS SPRAY



# EXPERIMENTAL DETAILS

- ✓ Aerosol sprayer was switched on for 8 hours in a dark $1m^3$ chamber by fixing the device on wall in one corner.
- ✓ Spray Rate is 0.55g/spray once in 15 minutes time intervals
- ✓ Air sampling was done from different locations of the chamber - Top, Middle & Bottom.

| Insecticide formulation | Time of collection (hours) |
|---|---|
| Permethrin 0.8% w/w + Transfluthrin 0.6% w/w | 0.5, 1, 2, 4, 6 and 8 |

# EXPERIMENTAL DETAILS

- ✓ 500 CC Volume of air samples were collected using cartridge in each location from the room
- ✓ Collected samples were dissolved in 10mL of acetone and quantified by GC-ECD.
- ✓ Once again all the samples were pooled and reduced the volume to 1mL and analyzed by GC-ECD to determine the total residue concentration.
- ✓ Surface concentration was measured after completion of 8 hours
- ✓ Floor of the chamber was covered with poly propylene sheet
- ✓ Poly propylene sheet was cut into pieces and extracted with 250 mL of acetone using end-over-end mechanical shaker.
- ✓ Volume of acetone was reduced to 1 mL using rotary evaporator and analyzed by GC-ECD

# CONCENTRATION OF TRANSFLUTHRIN AND PERMETHRIN IN AIR AT DIFFERENT LOCATIONS IN THE ROOM

| Sampling Occasion (hours) | Locations | | | | | |
|---|---|---|---|---|---|---|
| | Transfluthrin (mg/L) | | | Permethrin (mg/L) | | |
| | Top | Middle | Bottom | Top | Middle | Bottom |
| 0.5* | BLQ | BLQ | BLQ | BLQ | BLQ | BLQ |
| 1* | BLQ | BLQ | BLQ | BLQ | BLQ | BLQ |
| 2 | BLQ | BLQ | 0.0013 | BLQ | BLQ | 0.0016 |
| 4 | 0.0011 | 0.0017 | 0.0019 | 0.0013 | 0.0015 | 0.0021 |
| 6 | 0.0015 | 0.0024 | 0.0036 | 0.0019 | 0.0028 | 0.0047 |
| 8 | 0.0028 | 0.0062 | 0.0081 | 0.0033 | 0.0071 | 0.0093 |

BLQ - Below Limit of Quantification

* - Pooled samples were analysed

# DEPOSITIONS OF TRANSFLUTHRIN AND PERMETHRIN ON FLOOR IN THE CHAMBER

| Sampling Occasion (hours) | Residue (mg/L) | |
|---|---|---|
| | Transfluthrin | Permethrin |
| 8 | 0.282 | 0.683 |

# CONFIRMATION OF RESIDUES BY GC – MS

Instrument - Shimadzu GCMS QP5050A
Column - DB-5 (30 m x 0.25 mm I.D x1.0 µm)

Temperature conditions
Oven -150°C
Injector -240°C
Detector -260°C

Column temperature program

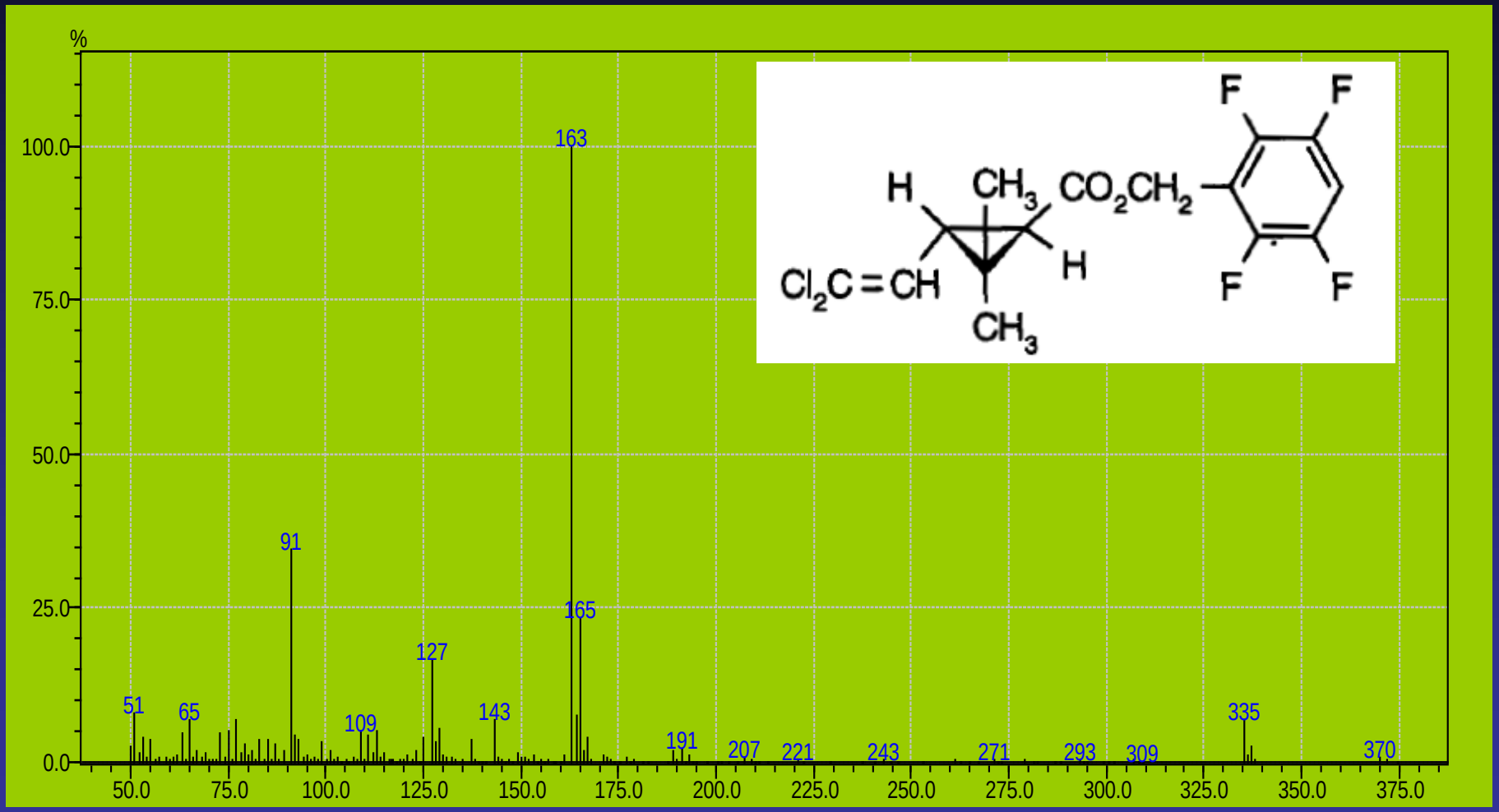
| Rate | Temperature °C | Hold time (min) |
|---|---|---|
| - | 150 | 5 |
| 20 | 220 | 6.5 |

Gas flow rate
Helium -1.0
Spit ratio - 1:20

Ion Monitered
Transfluthrin - 127,163 m/z
Permethrin - 163,183 m/z

Retention time (approximate)
Transfluthrin (Standard) -6.1 minutes
Permethrin (Standard) Cis -14.5 minutes
Trans -15.2 minutes

# MS SPECTRA - TRANSFLUTHRIN



# MS SPECTRA - PERMETHRIN