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ORTHENE AND ORTHO 9006 RESIDUES IN POLLEN SAMPLES

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INTRODUCTION

Acephate (O, S-dimethyl acetylphosphoramidothioate) introduced as an insecticide in 1971 after the field trials by Chevron Chemical Company under the trade mark Orthene is found to be effective in controlling spruce budworm, Choristoneura fumiferana (Clemens). Recently Sundaram and Hopewell (1976) have reviewed its usuage in Canadian forest spray programs. Acephate is partly metabolized to O, S-dimethyl phosphoramidothioate which has similar insecticidal properties. This compound was introduced in 1969 by the same company under the code number "Ortho 9006". This metabolite is the active ingredient in the trade mark material Monitor. for determining Orthene and Monitor residues in forest environmental samples has been developed recently at this Institute (see also Leary 1974). Using this method, acephate and Ortho 9006 residues have determined from twenty-four pollen samples collected from bee-hives exposed to the spray deposit. This report presents the analytical data gathered in this study.

MATERIALS AND METHODS

Pollen Samples

The 24 pollen samples used in the analysis were supplied by the Ecological Impact Group at CCRI. The samples were collected from the Larose Forest by Dr. C.H. Buckner and Mr. B.B. McLeod during the experimental spray operation carried out in June 1975 using Orthene for budworm control and stored in a freezer at 0°C until the analysis.

Analytical Methodology

The methodology used were similar to the one described earlier (1976). Full details of the procedure will be published elsewhere. Spiked pollen samples showed an average recovery of 85% Orthene and 80% of Ortho 9006. The low recovery of the metabolite is due to its loss in the column cleanup. About 20% of Ortho 9006 and about 10% of Orthene were lost in the second elution of the silica column using 100 ml. of 5% methanol in ether.

Gas Chromatography

The GLC conditions and the parameters used are discussed in an earlier publication (Sundaram and Hopewell 1976).

The results are recorded in Table 1.

Analysis of Orthene (R) and Ortho 9006 (R) Residues in Pollen Samples
Received from Larose Forest - Summer 1975 Spray Program
[Ecological Impact Group of CCRI] - Spray Date: June 19, 1975

| ample No. | CCRI Number | Sample Description by EIG, CCRI | Mass (g) | Concentration (ppm) | | |
|-----------|----------------|--|-------------|---------------------|---------|-------|
| | | | | Ortho 9006 | Orthene | Total |
| 1 | 16/76/171-1657 | Pollen - Control, June 15 | 5.0 | N.D. | N.D. | N.D. |
| 2 | 16/76/172-1658 | Pollen - Control, June 17 | 5.0 | N.D. | N.D. | N.D. |
| 3 | 16/76/173-1659 | Pollen - Post spray, Control, June 19 | 5.0 | -T | 0.02 | 0.02 |
| 4 | 16/76/174-1660 | Pollen - Post spray, Control, June 20 | 5.0 | T | Т | Т |
| 5 | 16/76/175-1661 | Pollen - Post spray, Control June 21 | 5.0 | N.D. | T . | T |
| 6 | 16/76/176-1662 | Pollen - Post spray, Control, June 22 | 5.0 | N.D. | T . | T |
| 7. | 16/76/177-1663 | Pollen - Post spray, Control, June 23 | 5.0 | N.D. | N.D. | N.D. |
| 8 | 16/76/178-1664 | Pollen - Post spray, Control, June 24 | . 5.0 | N.D. | N.D. | N.D. |
| 9 | 16/76/179-1665 | Pollen - Bee Range I June 15 | 5.0 | N.D. | N.D. | N.D. |
| 10 | 16/76/180-1666 | Pollen - Bee Range I June 17 | 5.0 | N.D. | N.D. | N.D. |
| 11 | 16/76/181-1667 | Pollen - Bee Range I June 19, 1st post spray | 5.0 | 0.40 | 1.85 | 2.25 |

Table 1. Cont'd

| Sample No. | CCRI Number | Sample Description by EIG, CCRI | Mass (g) | Concentration (ppm) | | | |
|------------|----------------|---|-------------|---------------------|---------|-------|--|
| | | | | Ortho 9006 | Orthene | Total | |
| 12 | 16/76/182-1668 | Pollen - Bee Range I June 20, 2nd post spray | 5.0 | T | 0.19 | 0.19 | |
| 13 | 16/76/183-1669 | Pollen - Bee Range I June 21, 3rd post spray | 8.8 | N.D. | 0.05 | 0.05 | |
| 14 | 16/76/184-1670 | Pollen - Bee Range I June 22, 4th post spray | 4.0 | N.D.º | 0.04 | 0.04 | |
| 15 | 16/76/185-1671 | Pollen - Bee Range I June 23, 5th post spray | 5.0 | Т | 0.03 | 0.03 | |
| - 16 | 16/76/186-1672 | Pollen - Bee Range I June 24, 6th post spray | 5.0 | т | 0.01 | 0.01 | |
| .1.V | 16/76/187-1673 | Pollen - Bee Range II pre spray, June 15 | 5.0 | N.D. | N.D. | N.D. | |
| 18 | 16/76/188-1674 | Pollen - Bee Range II pre spray, June 17 | 5.0 | N.D. | N.D. | N.D. | |
| 19 | 16/76/189-1675 | Pollen - Bee Range II post spray, June 19 | 5.0 | 0.07 | 0.62 | 0.69 | |
| 20 | 16/76/190-1676 | Pollen - Bee Range II post spray, June 20 | . 5.0 | .0.01 | 0.05 | 0.06 | |
| 21 | 16/76/191-1677 | Pollen - Bee Range II post spray, June 21 | 5.0 | N.D. | 0.02 | 0.02 | |
| 22 | 16/76/192-1678 | Pollen - Bee Range II post spray, June 22 | 5.0 | N.D. | Т | T | |

Table 1 Cont'd

| Sample No. | CCRI Number | Sample Description by EIG, CCRI | Mass (g) | Concentration (ppm) | | |
|------------|----------------|--|-------------|---------------------|---------|-------|
| | | | | Ortho 9006 | Orthene | Total |
| 23 | 16/76/193-1679 | Pollen - Bee Range II Post spray, June 23 | 6.5 | T | 0.03 | 0.03 |
| 24 | 16/76/194-1680 | Pollen - Bee Range II Post spray, June 24 | 7.0 | N.D. | Ť | T |

Not detectable N.D.

0.01 ppm MDL =

RESULTS AND DISCUSSION

Amounts of Orthene and Ortho 9006 residues found in the pollen samples are recorded in Table 1. The concentration levels are expressed in ppm units as sampled including the different variables and the data did not contain any correction for the percent recovery from the spiked samples. The limit of detectability of the method is 0.01 ppm for both the compounds.

The results indicate that the prespray pollen samples collected on June 15 and 17, 1975 did not contain any insecticide residues. After spraying on June 19, the control sample contained detectable levels of Orthene (0.02 ppm) which decreased to trace levels (<0.01 ppm) on June 20 to 22, 1975. None was detected on or after 23rd June. The first postspray sample in plots B.R.I and B.R.II contained 0.40 and 1.85 and 0.07 and 0.62 ppm of Ortho 9006 and Orthene respectively which rapidly decreased to trace levels on June 24. The results indicate that the insecticide and the major metabolite dissipated rapidly from the pollen substrate analysed. No interferences either from the contaminants or from the impurities present in the substrate have been encountered.

REFERENCES

- Leary, J.B. 1974. Gas-Liquid Chromatographic Determination of Acephate and Ortho 9006 Residues in Crops. Jour AOAC, 57(1): 189.
- Sundaram, K.M.S. and W.W. Hopewell. 1976. Distribution,

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