

Application Note No. 045

The Analysis of Organophosphorous Pesticides by Large Volume Injection

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Introduction

Organophosphorous pesticides, as a group, contain a number of compounds that are prone to adsorption or thermal degradation in the injector. These compounds represent a challenge to the use of large volume injection (LVI). Care must be taken in the selection of operating conditions and it is essential to use an injection port liner which is very inert.

The Optic 2 programmable injector provides the flexibility required to cope with thermally sensitive compounds and the new multi-capillary liner has proven to be sufficiently inert to permit the analysis of adsorptive compounds, such as azinphos methyl. In addition to being very inert, the multi-capillary liner can be operated to a much higher temperature than conventional packed liners, however, it does have a lower sample capacity (around 40 μ L) when used in the Rapid LVI mode. This limitation can be overcome by using a speed programmed injection. In this case the sample is injected at a rate which matches the evaporation of the solvent to ensure that no more than 40 μ L of liquid is contained within the injector at any one time. In this fashion, larger sample volumes can be injected without the risk of liquid breakthrough in the liner.

The CTC A200S Large Volume Autosampler can inject sample volumes of up to 500 μ L and can be programmed at a sufficiently slow rate to permit speed controlled injection. In this example, a 60 μ L injection proved to be sufficient to enable determinations down to 1 ng/L levels in river water samples and the quality of the data was sufficiently good to permit routine use.

Instrumentation & Conditions

Optic 2-200 Injector

Liner: Multi Capillary liner with a quartz wool plug

- Gas Flows: Split: 50 ml/min
Vent: 150 ml/min
Purge: 5 ml/min
- Equilibration Time: 0:30 m:s
- Initial Temperature: 40 °C
- Vent Time: Auto
- Ramp Rate: 16 °C/s
- Final Temperature: 230 °C
- End Time: 25:00 m:s
- Split Open Time: 1:30 m:s
- Purge Pressure: 10 psi
- Transfer Pressure: 35 psi
- Transfer Time: 1:30 m:s
- Initial Pressure: 10 psi
- Final Pressure: 22 psi
- Solvent Threshold: 95

Gas Chromatograph

Temperature Programme: 70 °C hold 1.5 mins
22 °C/min to 200 °C
0.8 °C/min to 220 °C
30 °C/min to 300 °C hold 4 mins

Autosampler

- Syringe: 500 μ L
- Injection volume: 60 μ L

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Results

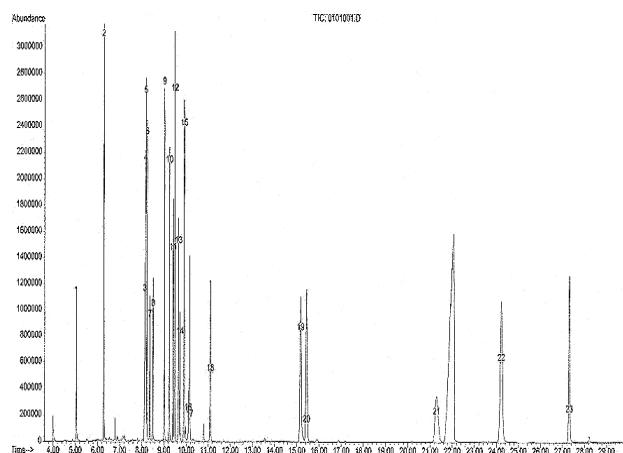


Figure: 60 μ L injection of OP Herbicides

Peak identities for the chromatogram:

- | | | |
|-----------------|----------------------|---------------------|
| 1. Dichlorvos | 9. Desmetryne | 17. Ethyl Parathion |
| 2. Mevinphos | 10. Methyl Parathion | 18. Chlorfenvinphos |
| 3. Simazine | 11. Prometryne | 19. Triazophos |
| 4. D5-Atrazine | 12. Fenchlorphos | 20. Carbophenothion |
| 5. Atrazine | 13. Terbutryne | 21. Azinphos Methyl |
| 6. Propazine | 14. Fenitrothion | 22. Azinphos Ethyl |
| 7. Propetamphos | 15. Malathion | 23. Coumaphos |
| 8. Diazinon | 16. Fenthion | |

Conclusions

The use of a multi-capillary liner with a programmable injector and a CTC large volume autosampler has shown to provide very low detection limits and produce good quality data. The inertness of this liner is particularly good for the analysis of labile organophosphorus pesticides.

Acknowledgements

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