Automated Solid Phase Extraction for Environmental Samples





Figure 1. Instrumentation used for sample preparation. TurboVap* LV for automated sample evaporation. The RapidTrace* for automated SPE.

Introduction

The removal of interferences and sample concentration are essential steps in trace level detection of pesticides, sheep dips, herbicides and PCBs in river water, wastewaters and trade effluents. The following methods were developed by the Environment Agency National Laboratory Service facility at Nottingham using the Biotage RapidTrace for automated solid phase extraction and the TurboVap II for evaporation. The procedures are UKAS accredited and can reach detection limits of 0.001 ug/Litre for some compounds.

Organo Phosphorous Pesticides Clean Up

A 1 Litre sample of Water is extracted with Dichloromethane and evaporated down to approximately 0.5 mL in a TVII. The solvent is then exchanged to hexane and the mixture is again evaporated down to about 1 mL. This dirty extract is then passed through an ISOLUTE* SI 200mg/3 mL column part number 460-0020-B which retains the interferences and elution is achieved using Acetone/Hexane mix to give approximately 11 mL of eluent ready for final evaporation to 0.5mL and analysis by GCMS.

A Typical Range of Components Screened for Using the OPP Method

Triazines	Demeton-s-methyl	Phorate
Fenthion	Carbofenthion	Dichlorovos
Propetamphos	Phenchlorophos	Chlorofeninphos
Azinphos	Dichlorbenil	Diazinon
Fenitrothion	Ethion	Coumaphos
Mevinphos	Parathion	Malathion
Triazophos		

RapidTrace Method

All solvent lines are purged with solvent first and all solvents to run both methods are present (max 8). The organic, cannula and aqueous waste solvent lines are separate (max 4). The Rack has two rows of 10 test tubes 16 mm x 100 mm, approx 12 mL volume max, for the samples and Fraction collection.

Step	Source	Destination	Volume (mL)	Flow (mL/Sec)
Purge - Cannula	Acetone	Cannula Waste	6	0.5
Condition	Acetone	Organic Waste	5	0.25
Condition	Acetone	Organic Waste	5	0.25
Purge - Cannula	Hexane	Cannula Waste	6	0.5
Condition	Hexane	Organic Waste	5	0.25
Load Sample	Fraction 1		1.2	0.08
Purge - Cannula	Acetone/ Hexane	Cannula Waste	6	0.5
Collect	Acetone/ Hexane	Fraction 1	5	0.08
Collect	Acetone/ Hexane	Fraction 1	6	0.5

Common Reagent Table for all Methods

Line No.	Reagent Name	SIP Speed (mL/min)
1	Ether/Hexane	0.5
2	Acetone	0.5
3	Hexane	0.5
4	Acetone/Hexane	0.5

Waste Name	Abbreviation	SIP Speed (mL/min)
Aqueous Waste	Aq W	Air Push = 2mL
Organic Waste	Org W	Air Push Multiplier = 2
Cannula Waste	Cannula	



Organo Chlorinated Pesticides (OCP) Clean Up

A 1 Litre sample of water is solvent extracted with Hexane and evaporated down to approx 1mL which can be carried out using a TurboVap LV. The dirty extract is then cleaned up through an ISOLUTE® FL 200 mg/3 mL column, part number 712-0020-B and elution is done using Ether/Hexane to give approximately 8mL of eluent ready for final evaporation to 0.5 mL and analysis by GCMS.

A Typical Range of Components Screened for Using the OCP

Trichlorobenzenes	Lindane & its isomers	Aldrin & Isodrin
DDT Compounds	Permethrins	Endosulphan
PCBs	1PPB	

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Ordering Information

Part Number	Description	Quantity
C50000	RapidTrace®+ Workstation 1 mL and 3 mL (10 columns)	1
C103198	TurboVap® LV 100/120V	1
C103199	TurboVap® LV 220/240V	1
460-0020-B	ISOLUTE® SI 200 mg/3 mL Sample Volume Columns	50
712-0020-В	ISOLUTE® FL 200 mg/3 mL Sample Volume Columns	50

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