

Pesticides in Soil Using the Dionex ASE™ 200 and the Caliper Life Sciences ASE Compatible TurboVap® LV Concentration Workstation

Introduction

Scientists routinely analyze soils for pesticides, as part of Use/Misuse investigations. Previous to the Dionex ASE 200 and the CaliperLS ASE Compatible TurboVap LV Workstation, samples were extracted by shakeout with the acetone/hexane and then filtered through sodium sulfate. This step was then repeated and the extracts were collected into Kuderna Danish flasks. The sample extracts were concentrated on a steambath and then transferred to a Nitrogen Evaporator for further concentration. This application brief describes how this manual method was recently automated in the past year using technology from Dionex Corporation and Caliper Life Sciences, Inc. and tested with soil samples.

Equipment Specifications and Operating Conditions

Dionex ASE 200

Oven Temperature:	100 °C
Pressure:	1500 psi
Oven heat-up time:	5 minutes
Static time:	5 minutes
Flush Volume:	60% of extraction cell volume
Solvent:	acetone/hexane (1:1, v/v)

CaliperLS ASE Compatible TurboVap LV

Solvent Starting Vol.	Solvent End Vol.	Solvent	Time to Concentrate
30-40mL	dryness	acetone/hexane (1:1, v/v)	30 mins.
Bath Temperature	Gas Pressure	Length of Time*	
55 °C	3 psi	15 minutes	
55 °C	6 psi	7 minutes	
55 °C	15 psi	5 minutes	
60 °C	20 psi	3 minutes	

* Pressure was increased as sample volume decreased

Hewlett Packard 5890 Series II GC Chromatograph

Rtx-5, 0.53mm x 30M column
1µm film
100 °C (0), 8 °C/minute, 260 °C for 25 minutes
Electrolytic Conductivity Detector

1. Reference Dionex Application Note 320.
2. Soil (characterized for organics) obtained from NSI Solutions.
3. Standards for spiking were obtained from ChemService.

Method Summary¹

The extraction was done with 25 grams of clay loam soil (Non-polluted soil#2, CLN Soil-2, Lot#CF002²) weighed into a 22mL stainless steel ASE extraction cell. A cellulose filter was placed at the bottom and the top of the cell. The set consisted of a blank soil and seven spiked soils. Samples were spiked at a 0.20 ppm level³ with the following pesticides: Chlorpyrifos, Endosulfan I, Endosulfan II, and Endosulfan Sulfate. Samples were then extracted on the ASE and evaporated to dryness in the ASE Compatible TurboVap LV. Samples were diluted to 10mL with hexane by using a glass volumetric pipette.

Results

Pesticide Recoveries in Soil Samples

Compound	Mean Recovery
Chlorpyrifos	92%
Endosulfan	88%
Endosulfan II	90%
Endosulfan Sulfate	87%

Note: Mean averages for seven determinations on spiked soil samples.

Summary

In the past year, scientists have found the Dionex ASE 200 to help speed up the extractions and improve recoveries. It has also saved them money on solvents, glassware, and time. With the introduction of the TurboVap LV Workstation, they have optimized their concentration step. Vials are taken directly from the ASE to the TurboVap, thus minimizing sample handling, sample transfer, hood space, and the amount of time necessary for concentration. Recoveries for the pesticides analyzed are comparable to previous manual methods.

Acknowledgments

The data for this Application Brief was provided by a testing laboratory performing Use/Misuse analysis.