Analysis of Organophosphorus Pesticides in Drinking Water Using Solid Phase Extraction

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Why Should We Measure?

- Organophosphorus compounds are a very major class of pesticides and are exported to almost every country in the world, well over 77 million pounds/year in US alone.

- Most intended uses are for row crops, but are also used for mosquito control as well as household and garden pests thereby increasing exposure risks.

- Contamination from these compounds in water must be monitored due to their acetylcholinesterase deactivation potential.

- Monitoring data exists for most pesticides including OP's in drinking water sources.

- Very little monitoring data until recently of OP pestidices under drinking water conditions

- Some OP's are partially removed by DW treatment proceses, but others may be transformed into contaminants which are equally or more toxic than the parent compound.

Method Considerations

- Large numbers of samples require a method which can be run unattended.

- Requires minimum sample prep

- Minimum of sample manipulations

Steps to be Automated:

- 1 Liter samples requiring no pre-extraction
- Large Volume sample through the cartridge in a minimum amount of time
- Drying of Cartridge with no additional manipulation
- Automatic elution and concentration to GC vial
- All surfaces appropriately rinsed to prevent carry-over

An Automated Solution

PrepLinc SPEi System with AccuVap FLX

• Automated, programmable introduction of sample to SPE column

• High-flow drying

• Programmable positive pressure SPE elution

• Inline transfer of elution fraction to AccuVap for concentration

• Concentrated eluate to GC vial for analysis
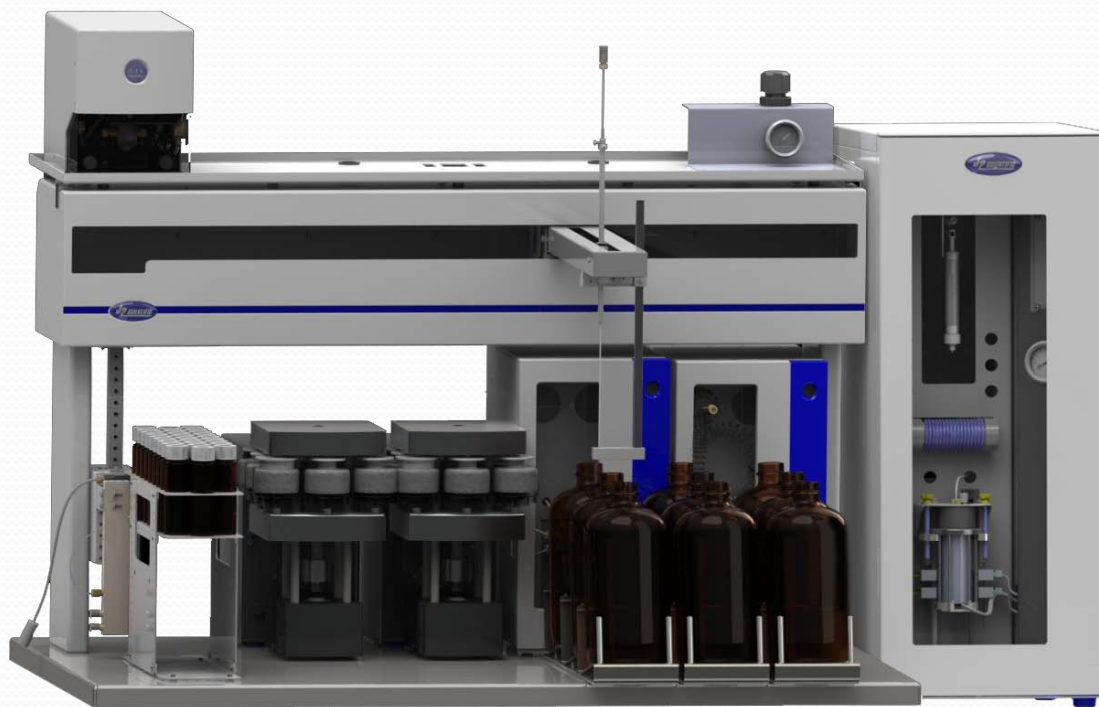
The Experiment

- Automatically process drinking water samples

- Samples spiked with 5 common OP pesticides for evaluation: Ronnel, Chloropyriphos, Chlorfenvinphos, Carbophenothion, Coumaphos

- Principle of Method:

  - 1 Liter water samples are passed directly over Bakerbond C-18 Cartridge

  - Cartridge is dried and eluted with 25 mLs Methylene Chloride

  - DCM extraction solvent concentrated to 1.5 mLs

- DCM concentrate is sent directly to GC vial for analysis by GC/ECD

- Demonstrate the full automation, ease of use and flexibility of the system for these compounds and other OP pesticide samples

Flow through PrepLinc System

- Sample is pumped through the SPE C-18 cartridge at user selectable rate.
- When all sample is pumped through, a sample rinse of container is conducted and also sent through cartridge.
- The cartridge is then dried via air for user selectable time.
- Elution is conducted and sent through to AccuVap for concentration..
- AccuVap concentrate is output to waiting GC vial.

Waste
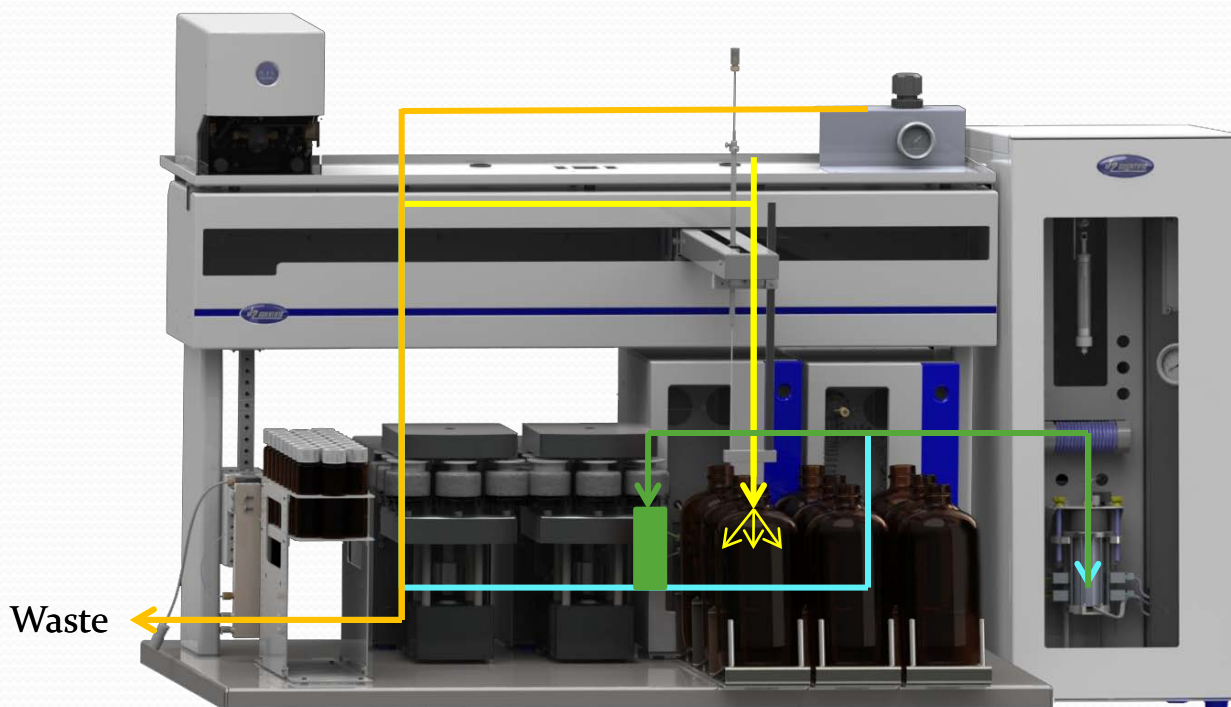
Sequence Programming

- Ease of use; quick system setup

- Up to 24 samples set up to run unattended; no technician intervention

- Increased reproducibility compared to manual or semi-automated equipment
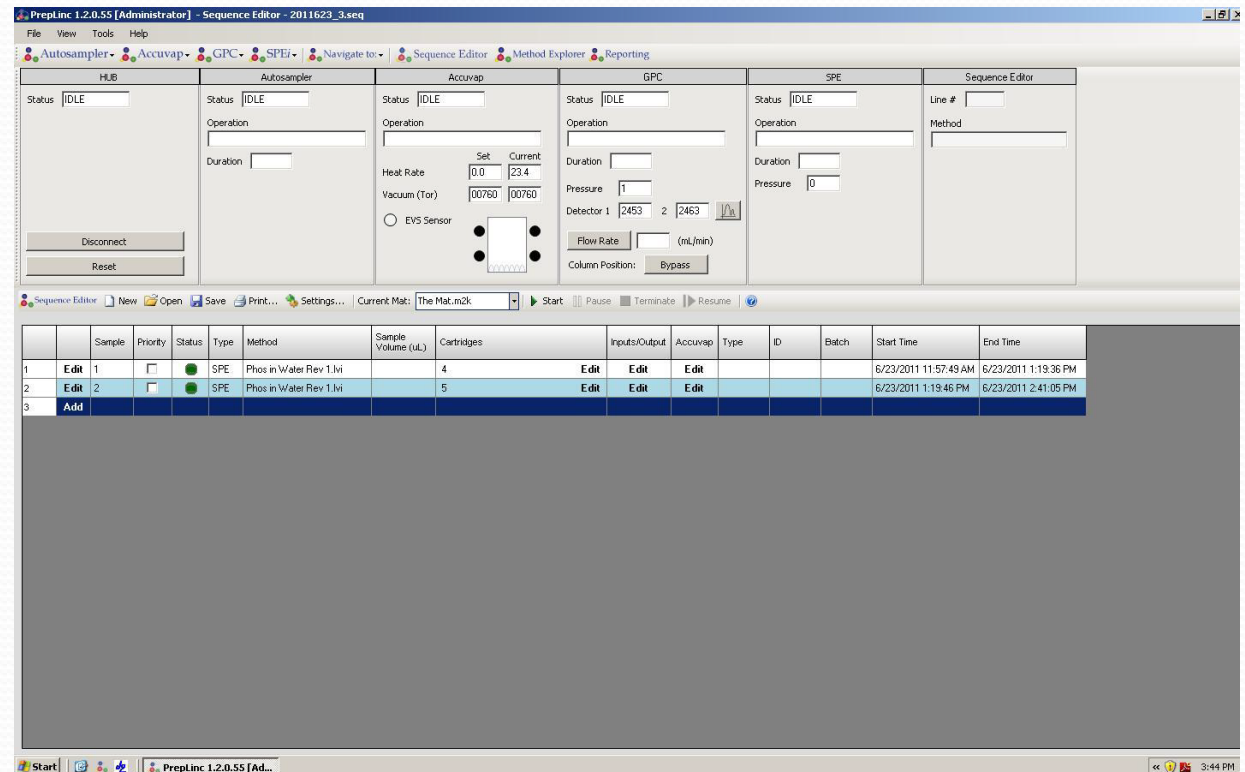
Cartridge Conditioning

- Condition cartridge with choice of solvent or water

- Multiple condition steps with unique parameters

- Program volume and flow rate

- Segregate aqueous and organic waste
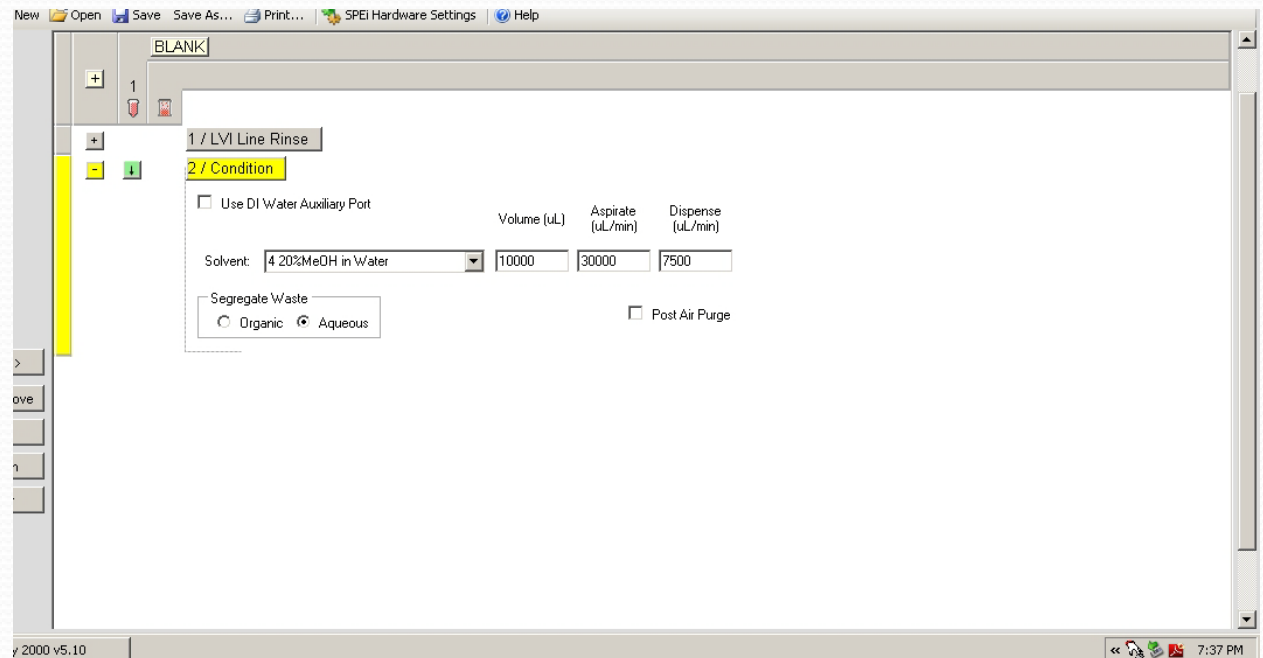
Line Rinsing

• Tubing rinses that are user programmable

• Multiple rinses with choice of solvent or water

• Programmable for volume and flow rate
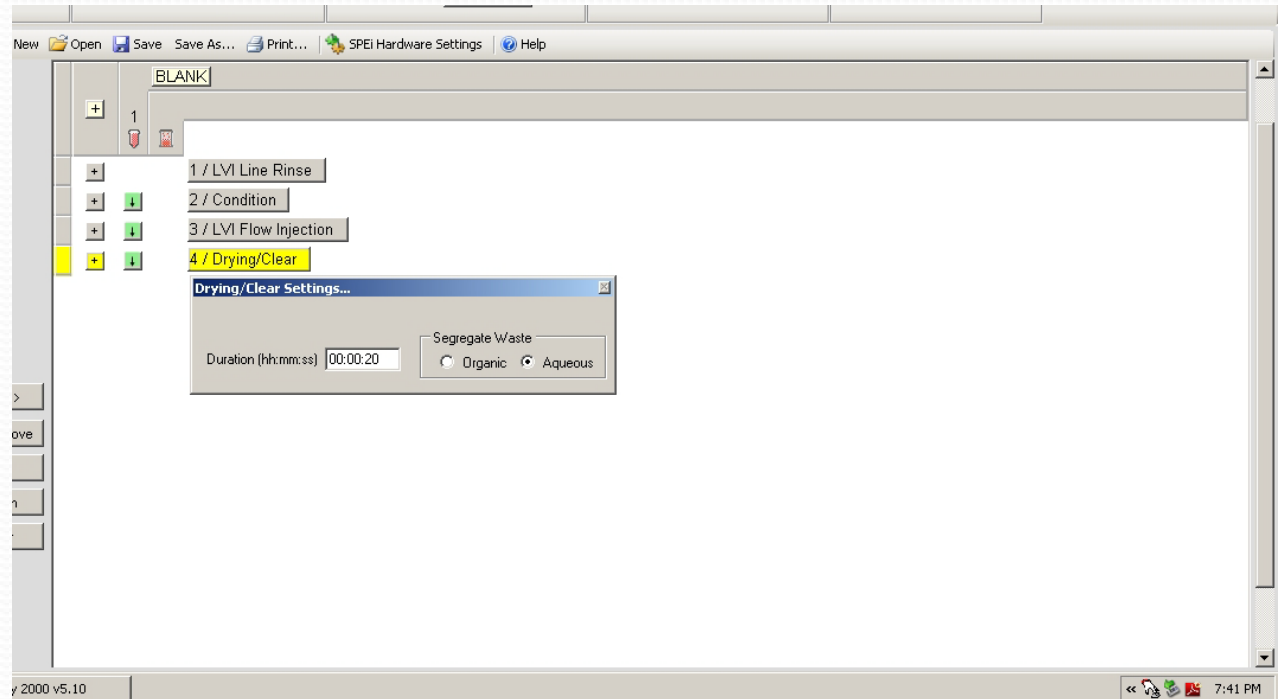
• Segregation of aqueous and organic waste
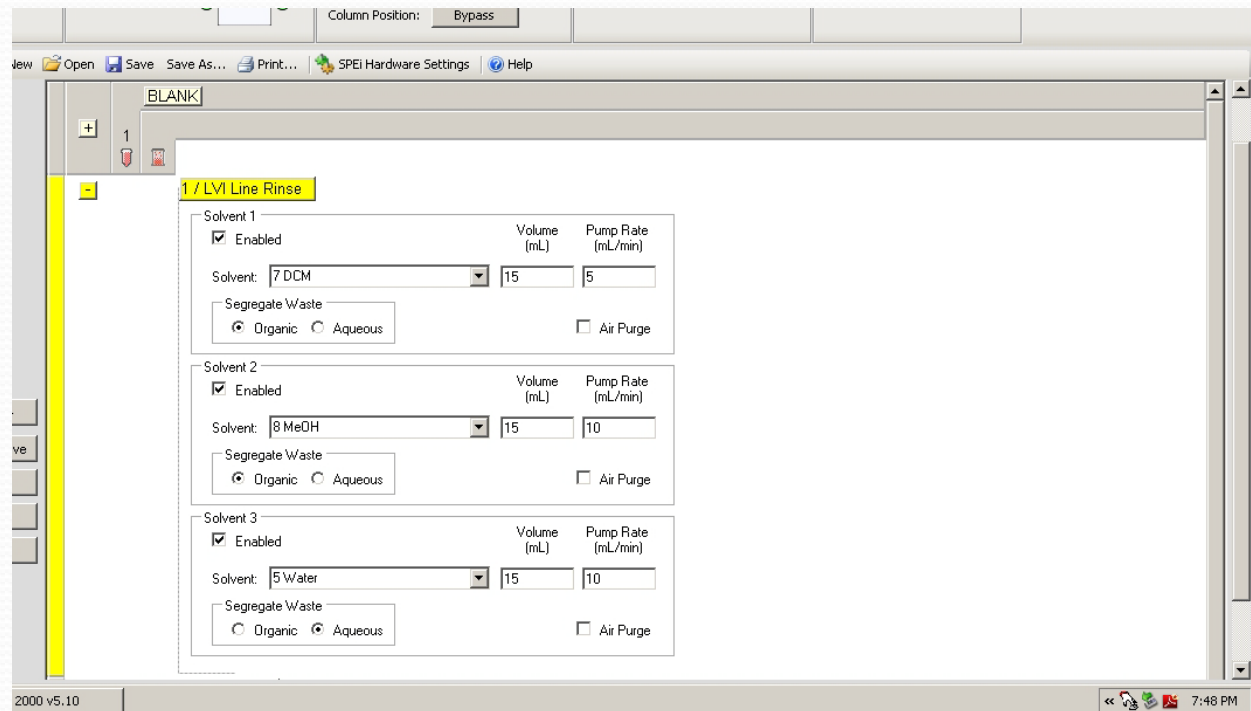
Sample Introduction

•Sample is pumped through the disk or cartridge

•Pumping rates is programmable

•Sample sensing determines end of sample and bottle rinse can be performed

•Waste is segregated
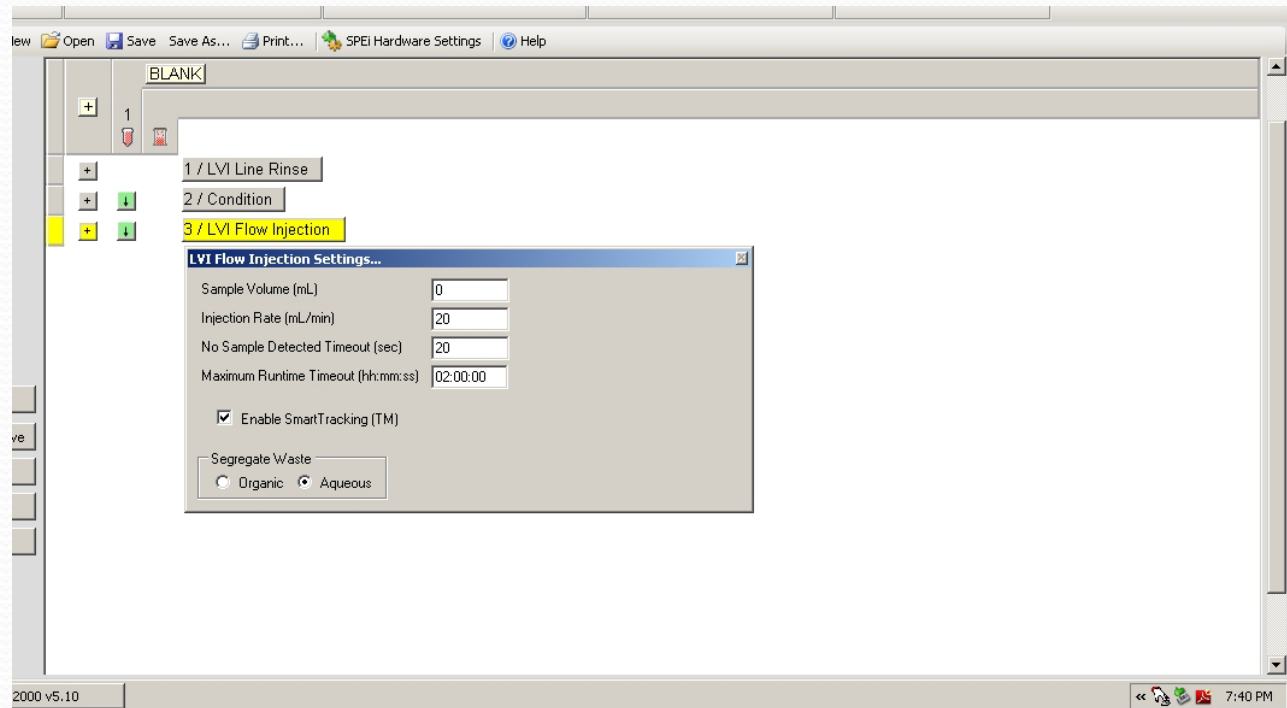
Cartridge/Disk Drying

• High-flow drying to speed process

• Drying time is user programmable

• Waste is segregated

Analyte Elution

• Elution step programmable for solvent, volume and flow rate

• Choose to direct elution to collect vial or AccuVap for concentration

• Purge all tubing lines for complete transfer
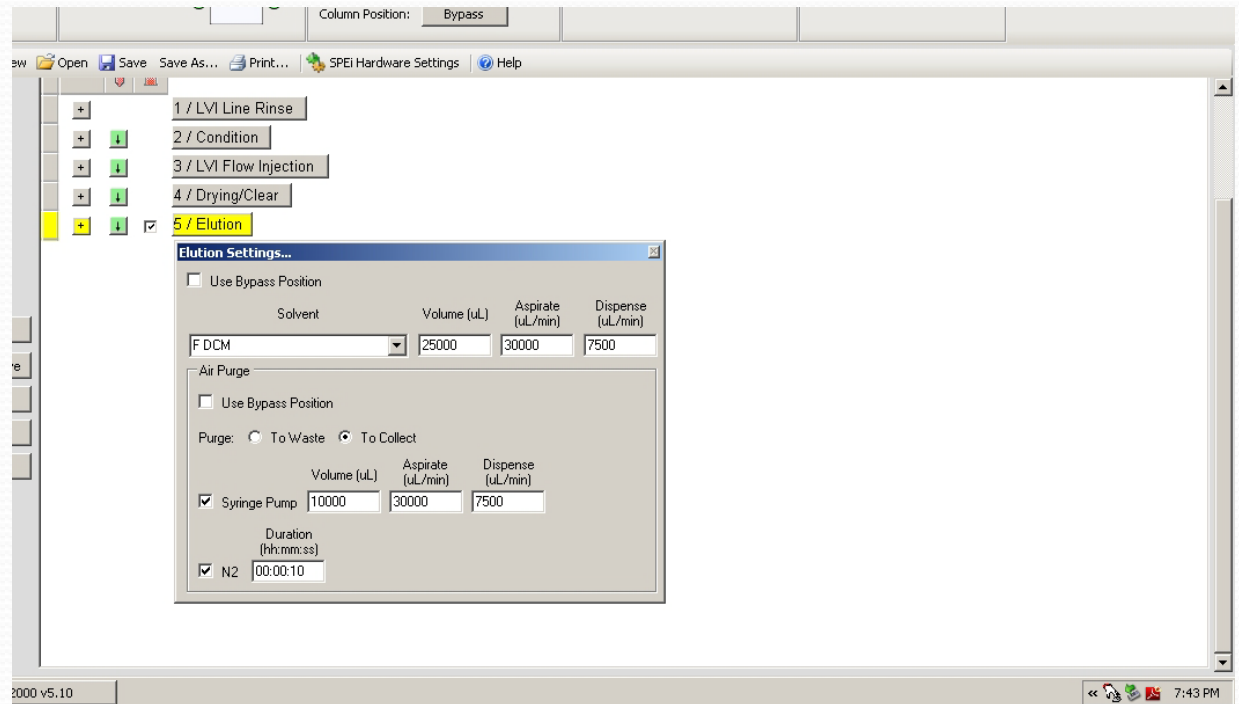
Inline Concentration

- AccuVap provides controlled concentration of samples to final volume

- Heat, vacuum, exchange solvents are programmable for every stage of concentration

- Vigorous programmable rinse between samples

- Delivers sample to a GC vial ready for analysis
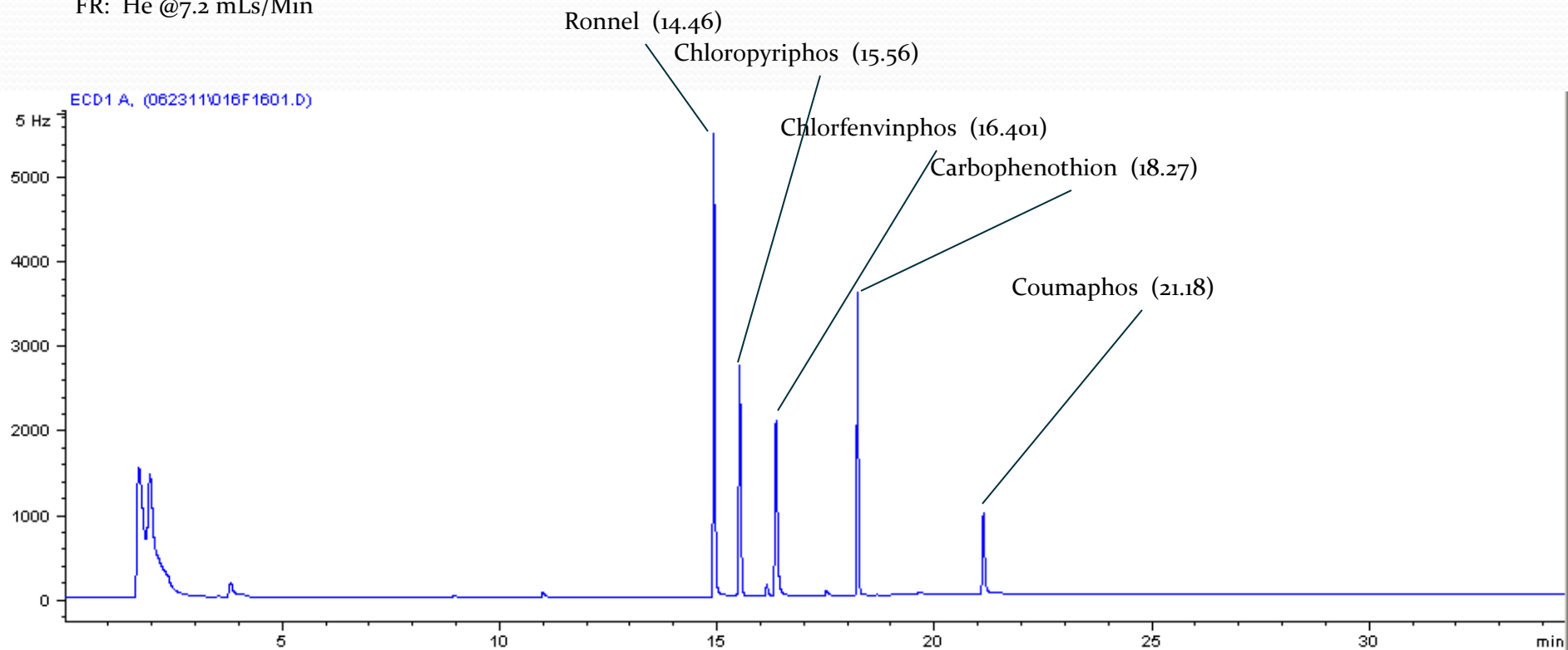
Analysis

Summary of GC conditions

Program Init Oven Temp 110 °C, hold 3 min, then to 275 °C at 10 °C/min
Restek 30 M Rxi-5Sil MS column250 uM diam 0.25 uM film thickness
Det: 330 °C
Inj: 250 °C, Splitless
FR: He @7.2 mLs/Min

ECD1 A, (062311\016F1601.D)

Ronnel (14.46)
Chloropyriphos (15.56)
Chlorfenvinphos (16.401)
Carbophenothion (18.27)
Coumaphos (21.18)

Typical Data Results

Recovery Selected Phosphates						
5.0 PPB	1	2	3	4	Avg	%CV
Ronnel	91.6	90.3	84.2	93.7	87.5	4.6
Chloropyriphos	93.1	93.0	87.3	87.1	90.1	3.7
Chlorfenvinphos	92.8	93.9	87.5	88.6	90.7	3.5
Carbophenothion	92.4	93.8	89.6	90.7	91.6	2.0
Coumaphos	91.8	93.4	90.2	90.8	91.5	1.5
2.5 PPB						
Ronnel	58.3	59.1	84.3	83.5	71.3	20.4
Chloropyriphos	64.3	65.0	92.6	91.9	78.4	10.3
Chlorfenvinphos	59.2	60.5	89.1	89.3	74.5	22.8
Carbophenothion	58.9	59.9	90.2	90.9	75.0	24.0
Coumaphos	66.8	67.7	96.5	98.1	82.3	21.1

Summary

- Organophosphorus pesticides (OPs) are among the most common pesticides used in industrialized countries and are therefore an important source of contamination. These compounds are very toxic when absorbed by human organisms because of acetylcholinesterase deactivation.

- The PrepLinc system provides an easy and convenient sample preparation platform which meets the needs of analytical laboratories by providing:
  - Automated and unattended sample processing
  - Reduction of analytical technician time
  - Fully processed sample requiring no further manipulation

Thank You