Solvent Exchange from Dichloromethane to n-Hexane Using the DryVap® Concentrator System

Introduction

The analysis of pesticides for environmental extracts typically requires a solvent exchange from dichloromethane (DCM) to an ECD-compatible solvent such as n-hexane. The prescribed method of solvent exchange and concentration is given in EPA Method 3510 and uses a Kuderna-Danish (K-D) apparatus achieve the results.

The K-D method is very time consuming and can take up to 1.5 hr to complete. It is also sensitive to human error and mistakes such as evaporating too far or too fast or not conditioning the sodium sulfate correctly can often result in lost samples.

The DryVap® concentrator system can automatically dry and concentrate the extract in as little as 30 min. Solvent exchange can be performed by adding 30 mL hexane to the concentrate and repeating the automated process. The DryVap® concentrator system eliminates all but one of the manual steps in the process and accomplishes the task faster than manual K-D evaporations. Furthermore, the final dried, solvent-exchanged, and concentrated sample is kept at room temperature under a blanket of inert gas and can be allowed to stand upon completion on the DryVap®.

To demonstrate the performance of the DryVap® system, three samples were spiked with a pesticides mixture and were then dried, concentrated, and exchange.

Instrumentation

- DryVap® Concentrator System
- DryDisk®
- Agilent 5890 GC-ECD

Method summary

1. Spike three 200 mL aliquots of DCM with 20 ng of organochlorine pesticides mix.
2. Pour the samples into the DryDisk® station of the DryVap® System.
3. Start the DryVap®, the samples will be dried and concentrated automatically to 0.9 mL.

   Main Vacuum: 10 in. Hg

   Nitrogen Pressure: 20 psi

   Dry Volume: 200 mL

   Heater Power: 5

   Auto Rinse: 0

   Sparge Heater: OFF
4. Add 30 mL of n-hexane to the concentrator tube.
5. Using a Pasteur pipette, mix the concentrated extract in the nipple with the exchange solvent.
6. Set the DryVap® Dry Volume to 0 and start, the samples will be concentrated and exchanged automatically to 0.9 mL.
7. Remove the concentrator tube and rinse the lower sides with 250 uL of n-hexane.
8. Adjust the volume to 1 mL using n-hexane.
9. Vial and analyze by GC-ECD.

Results

Table 1 shows the average recoveries and the relative standard deviation (RSD) for the three completed runs. The automation of the DryVap® system allows for the RSD to be less than 5 % (with the exception of three compounds). Also, the high recovery evident for both 4,4'-DDT and Endrin indicate that there is no degradation of thermally labile compounds.

Table 1. Average Recoveries and RSD for Three Organochlorine Pesticide Spikes

Compound	Average Rec.	RSD
a-BHC	78	4.8
Lindane	81	4.7
b-BHC	85	5.3
d-BHC	85	4.2
Heptachlor	87	4.3
Aldrin	79	3.6
Heptachlor Epoxide	80	3.3
g-Chlordane	87	9.3
a-Chlordane	79	3.2
4,4'-DDE	85	0.7
Endosulfan I	77	2.7
Dieldrin	78	2.0
Endrin	103	2.9
4,4'-DDD	85	1.8
Endosulfan II	82	1.8
4,4'-DDT	92	1.3
Endrin Aldehyde	71	3.7
Methoxychlor	89	3.0
Endosulfan Sulfate	82	3.7
Endrin Ketone	79	3.2

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Conclusions

The DryVap® concentration system allows for controlled drying, concentration, and solvent exchange to be completed in very little time. It removes the human element from the process more than conventional methods. This allows for fewer mistakes to be made by the analyst and a higher rate of reproducibility, both of which can save a company time and money.

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