Less Toxic Solvents for use with EPA 3640a

Application Note

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Introduction

The use of dichloromethane as an eluent for sample clean-up with Agilent EnviroPrep columns is recommended in EPA 3640a. However, due to the toxic nature of this chemical, alternative solvents have been investigated as suitable replacements. This note describes the use of several solvent mixtures with EnviroPrep columns and describes the effect of the eluent on the standard test mixture elution described in the EPA methodology.

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Materials and Methods

The sample used for this application was the mixture of five compounds described in the EPA method, listed below:

Corn oil
Bis(2-ethylhexyl) phthalate
Methoxychlor
Perylene
Sulfur

Conditions

Column:	EnviroPrep, 300 x 25 mm (p/n PL1210-6120EPA)
Eluent:	Various
Flow Rate:	6.0 mL/min
Injection Volume:	200 µL
Pressure:	Various
Detector:	UV
Calibrants:	None
Flow Rate Marker:	None

The samples were prepared at the concentration above in each solvent that was to be tested. Each sample was then diluted by 100% before being injected in to the chromatographic system.

The solvents under investigation were ethyl acetate and cyclohexane, selected as relatively safe chemicals to use which, when combined in appropriate proportions, give a range of eluent polarities. For comparison, dichloromethane was also used as a reference.

Results and Discussion

Pure dichloromethane

In dichloromethane the separation gives five clearly resolved peaks (Figure 1).
This is the recommended eluent for the EPA test and the benchmark separation for this application.

Pure Ethyl Acetate

In this experiment (Figure 2) the dichloromethane was replaced with ethyl acetate. However, using this solvent unusual chromatography was obtained due to the high polarity of ethyl acetate compared to dichloromethane. The broad late eluting peak is the sulfur, and the perylene peak appears to have disappeared, presumably due to strong absorption to the packing material.

To reduce the polarity of the eluent the ethyl acetate was mixed with non-polar cyclohexane in varying ratios and separation repeated.

Ethyl acetate / cyclohexane mixtures

Increasing amounts of cyclohexane in the eluent strongly affected the elution of perylene, with the perylene re-appearing as the cyclohexane content increased. The sulfur peak (Figure 3) was late eluting but was relatively unaffected by changes in cyclohexane content.

Further increases to the cyclohexane content strongly affected the elution of sulfur with increasing cyclohexane resulting in extremely late sulfur elution. However, increasing cyclohexane content improved the perylene peak position (Figure 4).

Conclusion

As a replacement eluent for the separation of the EPA test mixture, ratios of ethyl acetate and cyclohexane were investigated. Most of the peaks of the test mix were unaffected, but the effect on the peak positions of perylene and sulfur was pronounced. It was determined that a 1:1 mixture of ethyl acetate and cyclohexane was a suitable replacement for dichloromethane for the separation of the EPA test mixture. Although the peak positions did show some variation compared to those obtained in dichloromethane, the elution time was reasonable and importantly, the elution order remained the same.

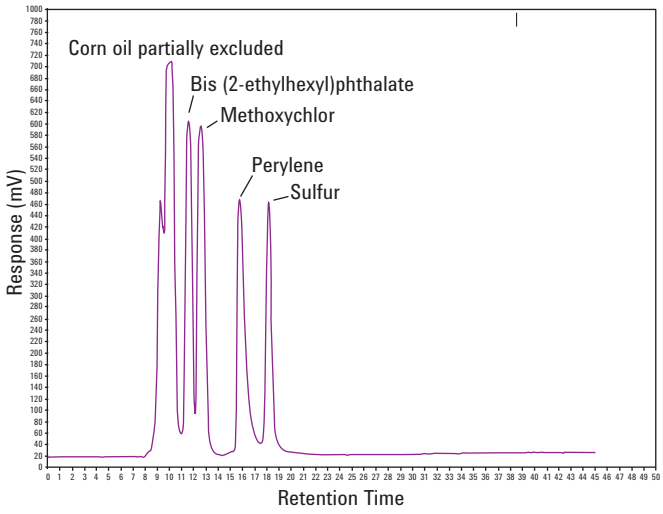

Figure 1. Separation of pure dichloromethane

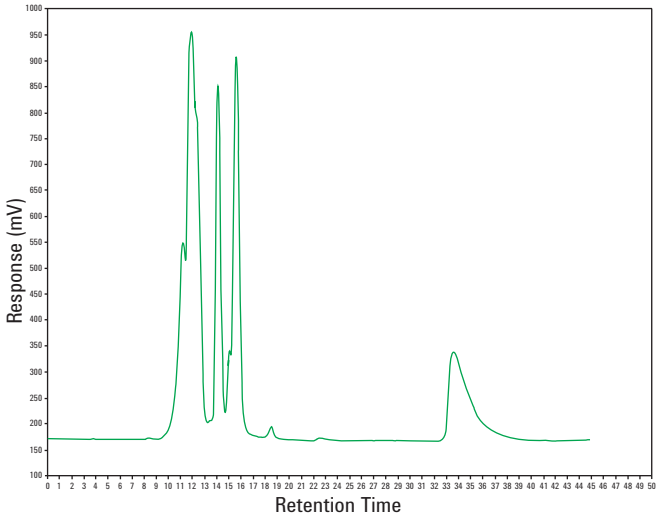

Figure 2. Separation of pure ethyl acetate

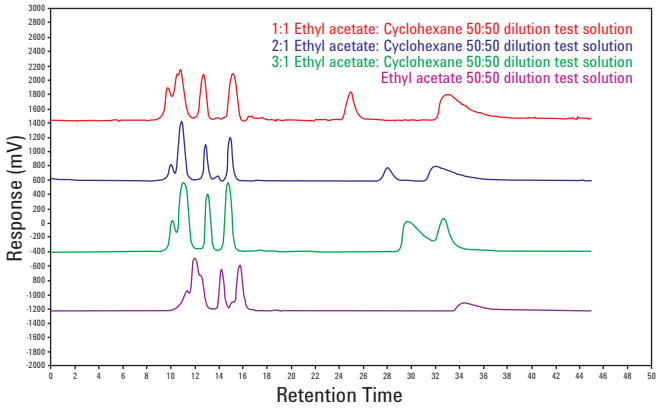

Figure 3. Separation of ethyl acetate / cyclohexane mixtures

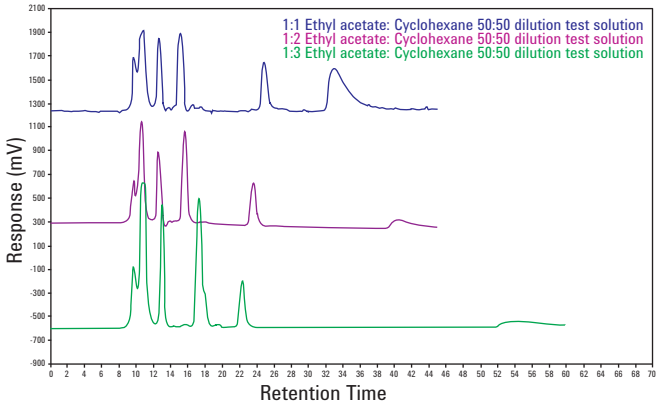

Figure 4. Separation of ethyl acetate / cyclohexane mixtures with increased cycloheaxane content

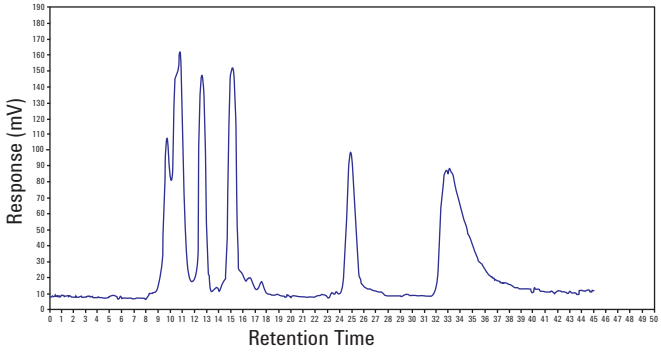

Figure 5. Separation of 1:1 mixture of ethyl acetate cyclohexane mixtures

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