Waters
Lab Highlights
An Internal Communication
of Applications and Techniques
LAH 0273 10/85
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USING THE RCM-100® RADIAL COMPRESSION SYSTEM AND RESOLVE™ C_{18} CARTRIDGE FOR THE ANALYSIS OF DIETHYLENE GLYCOL IN WINE

Considerable attention has been given in the news media recently to the presence of "antifreeze" in some wines of Austrian origin. The original incident in the USA was from wine from the Rust region in Austria. This has sparked concern that such adulteration of wine may be more extensive than the current incident.

Both ethylene glycol and diethylene glycol are used in anti-freeze liquids. The antifreeze reported as being the adulterant in the wine is diethylene glycol, which has the formula:

$$HO\text{-}CH_2\text{-}CH_2\text{-}O\text{-}CH_2\text{-}CH_2\text{-}OH$$

Ethylene glycol and diethylene glycol are clear liquids having a sweet taste. Both are reported as being poisonous to humans and a dose of 100 ml of ethylene glycol is reported as being fatal (Merck Index, 9th Edition).

We were asked whether LC could be used to analyze for this compound in wine. By circumstance, one of our lab staff had purchased (and had been drinking) some of the wine which had been implicated in the incident in the US.

Wehave found that using 100% water as the eluent, a 5µ RESOLVE™ C18 cartridge produced a good separation of diethylene glycol from the other components in wine in a reasonable time. With this column, the wine can be injected directly after filtering. It is advisable to use RESOLVE™ C18 Guard-PAK™ Precolumn Inserts (P/N 85824) inserted directly in the inlet of the cartridge using the Modified Inlet Connector (P/N 85857) in an RCM-100® Radial Compression Module. The purpose of the Guard-PAK Insert is to trap any high molecular weight components which could foul the column when untreated wine is injected.

Using this system, diethylene glycol elutes at about 8 minutes, just after ethanol. We used a Late-harvest Johannisberg Reisling as being a wine which should not contain diethylene glycol. There were tiny amounts of other compounds which could co-elute with the diethylene glycol with some wines but this method would allow direct injection of wines and a rapid analysis within 10 minutes.

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Figure 1 is the chromatogram of a diethylene glycol standard at 665 ppm. Figure 2 is a chromatogram of the wine suspected of being adulterated. This shows a peak where one would expect to see diethylene glycol (at about 8 min). The size of this peak is equivalent to about 3650 mg/l of diethylene glycol. Spiking this Austrian wine with a diethylene glycol sample gave an increase in the size of the peak suspected as being diethylene glycol. Figure 3 is the chromatogram obtained from the unadulterated Reisling.

It is suggested that this method provides a simple, rapid screening LC procedure for diethylene glycol in wine. Since the finding of any glycol in samples could lead to legal action, the confirmation of the identity by some other means, such as the more tedious GC method, would be advisable.

These chromatograms were obtained by using a 410 RI Detector but a 401 Detector would be adequate for this analysis.

Column: 5µ RESOLVE™ C_{18}
8mm X 100mm Cartridge in RCM-100
Mobile Phase: Milli-Q Water
Flow Rate: 1 ml/min
Detector: Model 410 Refractive Index Detector
Sensitivity: X32
Scale Factor: X20
Injection Volume: 10 µl

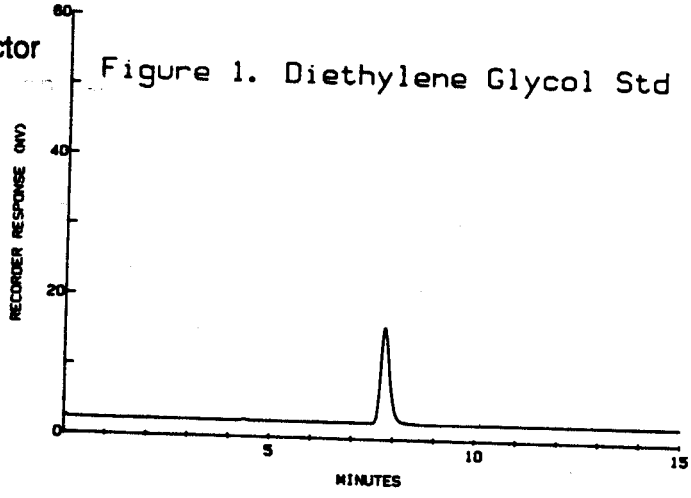

Figure 1. Diethylene Glycol Std

Figure 2. Suspect Adulterated Wine

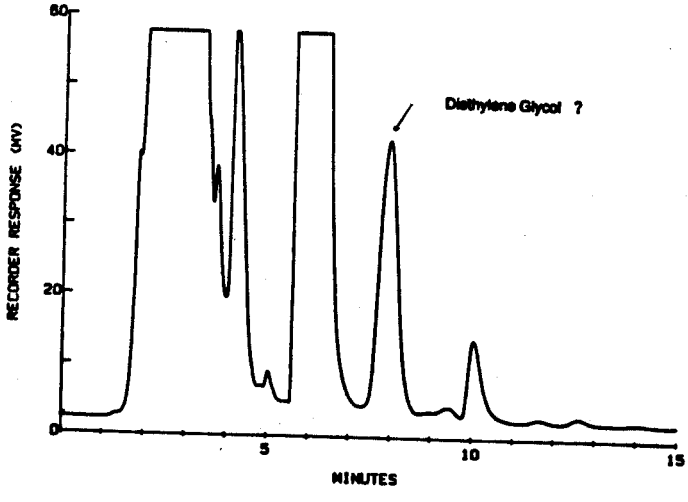

Figure 3. Unadulterated Wine