Optimization of Low-Temperature Evaporative Light-Scattering Detection (LT-ELSD) of Ethylene Glycol

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Ethylene glycol (EG) is a volatile compound that is used in a wide range of applications (e.g., antifreeze and manufacture of polyester fibers and films). EG is readily absorbed by the human body and can lead to kidney damage death, so it is regulated by the FDA as a residual in food and by the EPA as a toxic in air.

A sensitive detection technique is needed to detect and quantify this compound. While an HPLC separation with evaporative light-scattering detection (ELSD) is an ideal method for EG since it does not contain a chromophoric group, its volatility can reduce the sensitivity of the method. The use of the "Low Temperature" features of SEDEX LT-ELSD detectors allows the user to overcome the problem of the volatility of the sample, so that acceptable limits of detection/quantitation can be reached using the LT-ELSD.

Experimental

A SEDEX LT-ELSD Model 75 was used to investigate the sensitivity of detection of ethylene glycol. The mobile phase is water 100% at a flow rate of 1 mL/min. The evaporative tube temperature was set to a very low temperature (30 °C) to minimize solute evaporation. As shown in Figure 1, the water solvent used for the dissolution does not contain pollution, and the signals obtained in the study are only due to ethylene glycol sample.

EG samples were injected (10 µL, eight replicates) thru a narrow bore restrictor to investigate the effect of the evaporation temperature on the detection of ethylene glycol.

Results

The sensitivity of the detector increased as the temperature was decreased. As an example (Figure 1); at 50 °C, the intensities are less than 100 mV, when the temperature was lowered to below 37 °C, the same sample (5 mg/mL, 10-µL injections) amount saturates the detector. The blank signal (no EG) was negligible.

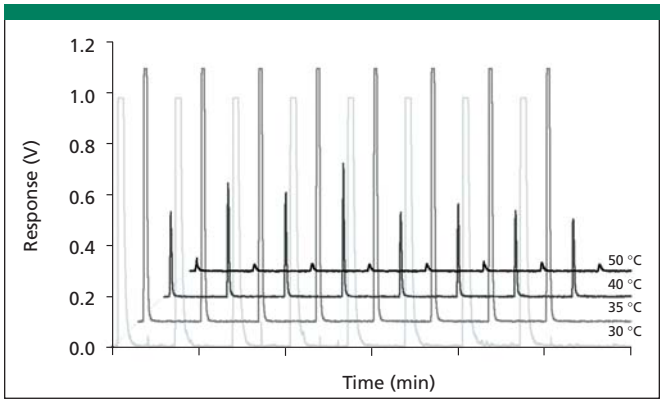

Figure 1: Study of the effect of the evaporation tube temperature on the intensity of ethylene glycol peaks. Eight replicate of ethylene glycol injection (10 µL, 5 mg/mL); mobile phase: water; flow rate: 1 mL/min.

Continuing to reduce the evaporation temperature lead to a more complex situation and a decrease in the S/N is observed. This is due to an increase in noise generated by un-evaporated mobile phase; as an example, we observed that the signal to noise fell as the temperature was lowered from 32 to 30 °C.

We then injected a series of eight replicate injections of 2.5 ppm samples (10 µL) as shown in Figure 2. The S/N was found to be 11.57, and we calculate that the limit of detection (S/N of 3) would be approximately 0.65 ppm, and the limit of quantitation (S/N of 10) would be 2.16 ppm, respectively corresponding to 6.5 ng and 21.6 ng (10-µL injections).

Conclusions

The SEDEX LT-ELSD, which includes a specially designed nebulization cell, allows for extremely efficient low-temperature evaporation of the mobile phase, so that semivolatile solutes can be detected with excellent sensitivity. The experiments described in this note clearly demonstrate that low levels of ethylene glycol, which is quite volatile (this solute is often analyzed by gas chromatography), can be readily detected by HPLC with LT-ELSD. The use of optimized conditions provides satisfactory sensitivity even at low evaporation temperature. This indicates that the SEDEX LT-ELSD should be considered for HPLC detection for semivolatile compounds, especially those that no not include a good chromophore.

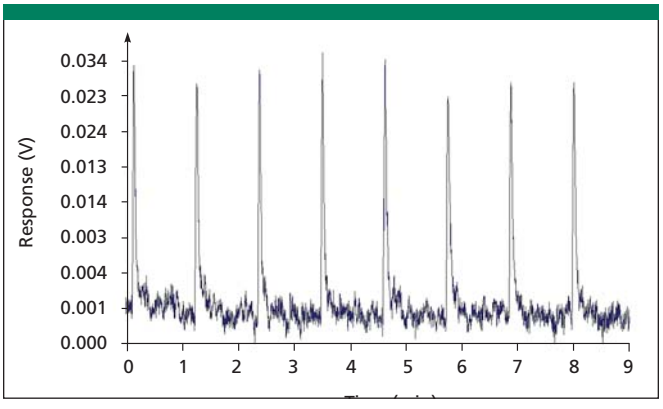

Figure 2: Eight replicates of 2.5 µg/mL injections (10 µL) of ethylene glycol; mobile phase: water, flow rate: 1 mL/min, tube temperature: 32 °C.

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