Benefit of Low Temperature ELSD for the Analysis of Herbicides by HPLC

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Introduction
Phenylurea compounds are used extensively in agriculture as selective herbicides, for pre- and post-emergence, by inhibiting photosynthesis. They are easily taken up from soil solution by the root systems of plants and rapidly translocated into stems and leaves by the transpiration system. Common phenylurea herbicides, such as diuron, have a relatively low tendency to adsorb to soils and sediments, while its degradation products have a relatively long lifetime. Consequently, the mobility and relative persistence of these herbicides increases the chance of migration to ground water via leaching and surface run-off. Developing analytical methods for the analysis of phenylurea herbicides and their metabolites in surface and ground water is vital to the understanding of herbicide fate and degradation pathways in the environment.

Unlike the majority of pesticides, the application of gas chromatography (GC) to the analysis of phenylurea herbicides is difficult because they are thermally unstable and degrade rapidly to isocyanates and amines. In addition, thermal reactions in the GC detector and on column result in a lack of reproducibility preventing the quantitative screening of the degradation products. A better approach for thermally sensitive compounds is to use HPLC, coupled with evaporative light scattering detection.

Evaporative light scattering detectors respond to all compounds that are less volatile than the mobile phase. In the past, high ELSD operating temperatures were required to fully evaporate aqueous eluents, making the detection of semi-volatile analytes difficult or even impossible.

The Varian ELS detector is designed to evaporate highly aqueous mobile phases at ambient temperature. This is achieved by introducing a stream of dry nitrogen during the evaporation stage, which increases the vapor loading capacity of the surrounding gas and enables complete evaporation of the eluent to take place without raising the temperature. The advantage of characterizing samples at ambient temperature is demonstrated by the analysis of a mixture of ten phenylurea herbicides.

Instrumentation
Column: C18 5 µm, 250 x 4.6 mm
Detection: Varian ELSD (neb=25 °C, evap as shown, gas=1.6 SLM)

Materials and Reagents
Eluent A: Water
Eluent B: ACN

Sample Preparation
Sample: Phenylurea herbicide mixture

Conditions
Flow Rate: 0.7 mL/min
Injection Volume: 20 µL
Gradient: 10-80 % B in 40 min

Results and Discussion
The herbicide mixture contains compounds of different volatilities and the highly volatile herbicides are not detected as the ELSD temperature is increased. Only seven of the ten herbicides are detected at 70 °C, compared to all ten at 25 °C. Therefore, by operating the Varian ELS detector at ambient temperature the loss of semi-volatile components is minimized, the sample integrity is preserved and maximum sensitivity is achieved, as shown in Figure 1.
Conclusion
Separation and identification of semi-volatile compounds is straightforward using evaporative light scattering detection at the ambient temperatures available with the Varian ELS detector. The Varian ELS detector surpasses other ELSDs for low temperature HPLC applications with semi-volatile compounds. Its innovative design represents the next generation of ELSD technology, providing optimum performance across a diverse range of HPLC applications. The Varian ELS detector’s unique gas control permits evaporation of high boiling solvents at very low temperatures. For example, 100 % water at a flow rate of 5 mL/min can be removed at 30 °C. The novel design of the Varian ELS detector provides superior performance compared to detectors from other vendors for the analysis of semi-volatile compounds.

References

Figure 1. Varian ELSD reveals the presence of semi-volatile herbicides when operated at ambient temperature.