

Improved Compound Recovery with ELSD Systems

Chromatography Application Note AN91

Abstract

The ability to collect compounds is directly related to the ability to detect them. Many compounds posses weak or non-existent chromophores which causes UV detection to exhibit poor yields. The use of Evaporative Light Scattering Detection (ELSD) allows detection of these compounds; however, there are differences between the Teledyne Isco external ELSD systems and other systems. Compounds that are somewhat volatile can give a reduced response on the ELSD which reduces yield.

Background

Figure 1 below demonstrates how a weak signal reduces the amount of material collected because the material is not "visible" to the detector¹.

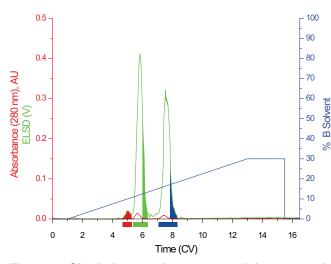


Figure 1: Shaded areas denote material recovered with an ELSD detector, but not detected with a UV detector

Although there are two peaks visible to the UV detector, the minor compound at ~5 column volumes (CV) is not detected by UV absorbance. The shaded areas on the ELSD peaks are also lost if only monitoring the UV signal. The sample is a mixture of tocopherols purified on a 30 g Redi*Sep* Rf Gold[®] diol column with a hexane/ethyl acetate gradient. This example serves to demonstrate how compounds can be lost when a compound is only weakly detected.

Many flash ELSD systems exhibit poor response to semi-volatile compounds compared to the Combi*Flash*[®] these systems also exhibit reduced yield compared to the Teledyne Isco products.

Experimental and Results

A sample of 2,3-isopropylidene-D-ribofuranose was synthesized as previously described¹. The reaction mixture (1.0 mL) was purified on 40g Redi*Sep*[®] Rf silica columns (PN 69-2203-340) using the default gradient on each flash system; a hexanes/ethyl acetate gradient was run.

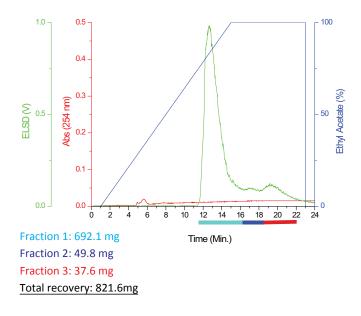


Figure 2: CombiFlash Rf-200i with internal ELSD

Bellinghausen, P; Fowler, N; Lewis, R; Silver, J. Advanced Detection Techniques for Flash Chromatography, Presented at 52nd Spring 2012 meeting of the American Chemical Society, San Diego, California, March 2012

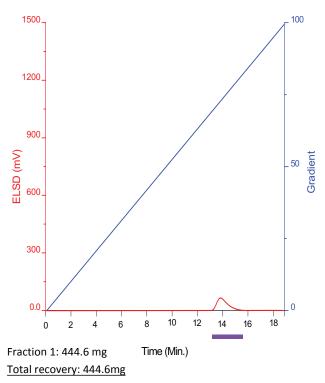


Figure 3: Competitive flash system with ELSD

Conclusion

The Combi*Flash* systems demonstrated much greater recoveries than the competing systems- a yield improvement of nearly two-fold. The competing system detected only 64% of the major peak compared to the Combi*Flash* system, and failed to detect the minor components. For this example, the Combi*Flash* system provided 34% more compound for further synthesis.

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