Improved Compound Recovery with ELSD Systems

TELEDYNE ISCO
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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 detector[1].

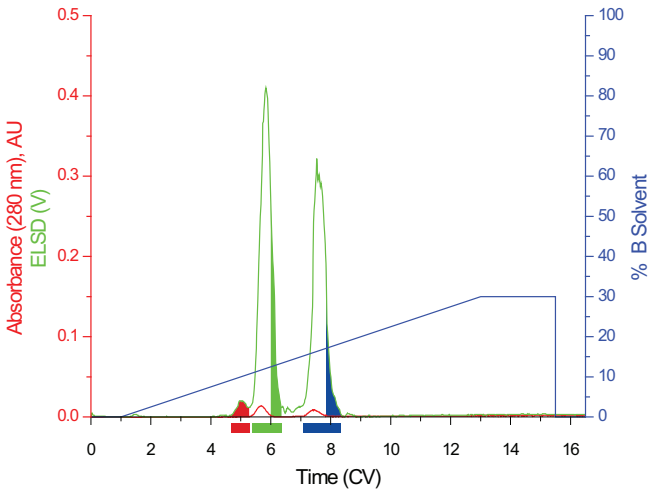


Figure 1: Shaded areas denote material recovered with an ELSD detector, but not detected with a UV detector

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 (PN 69-2203-516) 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*® Rf 200i (PN 68-5230-010) or the Model 340CF external ELSD (PN 68-5237-062); these systems exhibit reduced yield compared to the Teledyne Isco products.

Experimental and Results

A sample of 2,3-isopropylidene-D-ribofuranose was synthesized as previously described[1]. 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.

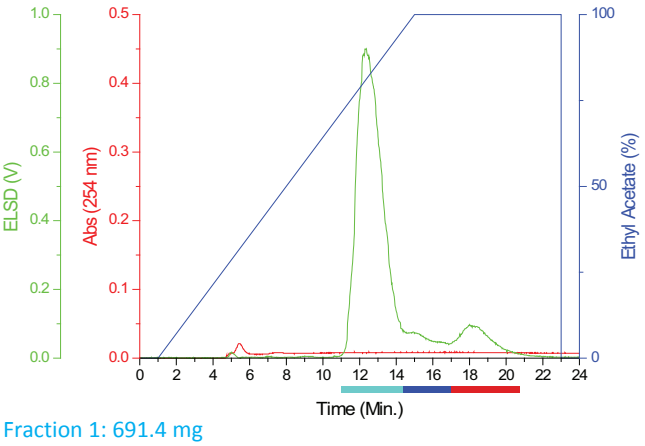
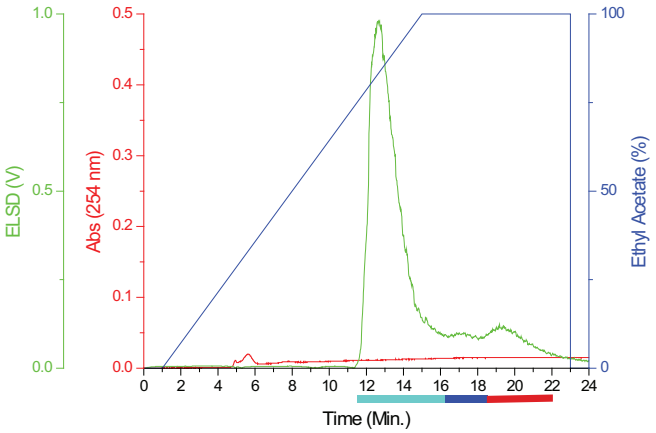


Figure 2: Model 340CF External ELSD

1. 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



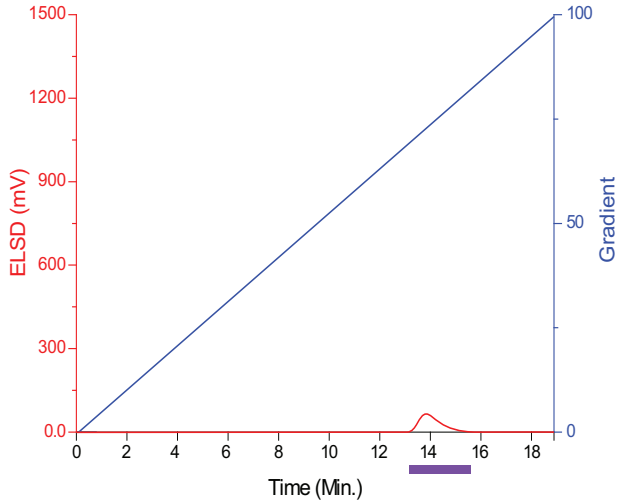
Fraction 1: 692.1 mg

Fraction 2: 49.8 mg

Fraction 3: 37.6 mg

Total recovery: 821.6mg

Figure 3: Combi*Flash* Rf-200i with internal ELSD



Fraction 1: 444.6 mg

Total recovery: 444.6mg

Figure 4: 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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