

Determination of Sample Loss on the Reveleris™ Flash Chromatography System

Introduction

The Reveleris™ system uses RevealX™ Detection Technology (patents pending) to collect fractions from chromophoric and non-chromophoric compounds. This is done by advanced fluidics and signal processing in order to integrate multiple detector signals. This technology involves a proprietary flow splitting design between the UV and ELS detectors within the system. Analysts familiar with ELSD know this is a destructive detector. So the question of sample being lost during this split can be a concern. We tested this for the fractions collected as detected by the ELSD.

Experimental Conditions

Tocopherol was chosen as a difficult test probe, as it generally presents poor peak shape by normal phase flash chromatography, making difficult fraction collection. 2mL of tocopherol was loaded using a 10mL syringe on a Reveleris™ 40g cartridge. The sample concentration was 500mg/mL, so there was a total of 1g on column. The flowrate was 40mL/min and the gradient used was 35% to 100% ethyl acetate in 6 minutes (remaining balance of hexane).

The tubes used to collect the fractions were weighed before collection. After fraction collection, the samples were dried down and the dry tubes were weighed.

Results and Discussion

There were four tubes that contained fractions collected from detection by ELSD. The original tare weight of each empty tube was subtracted from the dry weight of the same tube used after the fraction was dried down. The weights from these four tubes were then added together to determine the total weight of fractions collected. This was a sum of 1.0669g.

The data shows that there was no sample loss based on the weight recovered from the four

dried down fractions. There was actually a slight gain in weight. Additional weight gain can be accounted for by injection syringe accuracy and fraction tube handling.

Conclusion

As the results show, there was no sample loss from the fractions collected as detected by the ELSD detector. This shows that the split to the ELSD does not contribute to significant sample loss. The sophisticated proprietary fluidic and splitting design of the Reveleris™ system enables flash chromatographers to detect previously undetectable non-chromophoric compounds. Sample loss is not a concern.

Figure 1 Conditions

Sample

Concentration: 500mg/mL Tocopherol
Cartridge: Reveleris™ 40 grams
Injection Volume: 2mL using 10mL Syringe

Mobile Phase: A: Hexane

B: Ethyl Acetate

Gradient: (Time, %) (0, 35), (6,100)

Figure 1



ELSD Collection Only				
Fraction #	2	3	4	5
Tare Weight	18.4179g	18.4795g	18.416g	18.4501g
Dry Weight	18.4652g	19.22g	18.6946g	18.4506g
Dry Weight - Tare Weight	0.0473g	0.7405g	0.2786g	0.0005g
Total Weight				1.0669g

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