

Determination of Sample Loss on the Reveleris® X2 Flash Chromatography System

Introduction

The Reveleris® X2 system uses patented* RevealX™ Detection Technology 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® X2 system enables flash chromatographers to detect previously undetectable non-chromophoric compounds. Sample loss is not a concern.



U.S. Patents Nos.
8,115,930
8,305,581
8,305,582
8,314,917

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

www.discoverysciences.com

ALTECH® and REVELERIS® are trademarks, registered in the United States and/or other countries, of Alltech Associates, Inc. REVEALX™ is a trademark of Alltech Associates, Inc. GRACE® is a trademark, registered in the United States and/or other countries, of W. R. Grace & Co.-Conn. This trademark list has been compiled using available published information as of the publication date of this brochure and may not accurately reflect current trademark ownership or status. GRACE DISCOVERY SCIENCES is a product group of W. R. Grace & Co.-Conn, which now includes all product lines formerly sold under the Alltech brand. Alltech Associates, Inc. is a wholly owned subsidiary of W. R. Grace & Co.-Conn. GRACE MATERIALS TECHNOLOGIES is a business segment of W. R. Grace & Co.-Conn., which now includes product lines formerly sold under the GRACE DAVISON brand. © Copyright 2012 W.R. Grace & Co.-Conn. All rights reserved. The information presented herein is derived from our testing and experience. It is offered for your consideration and verification. Since operating conditions vary significantly, and are not under our control, we disclaim all warranties on the results that may be obtained from the use of our products. W. R. Grace & Co.-Conn. and its subsidiaries can not be held responsible for any damage or injury occurring as a result of improper installation or use of its products. Grace reserves the right to change prices and/or specifications without prior notification.

W. R. Grace & Co.-Conn.
7500 Grace Drive, Columbia, MD 21044

Grace Discovery Sciences Regional Headquarters:

AUSTRALIA:
Rowville
Tel: +61 3.9237.6100
Email: discoverysciences.AU@grace.com

BRAZIL:
Sorocaba
Tel: +55 15.3235.4705
Email: discoverysciences.BR@grace.com

CHINA:
Shanghai
Tel: 86 21 5467 4678
Email: dsbiz.asia@grace.com

EUROPE:
Lokeren, Belgium
Tel: +32 (0)9.340.65 65
Email: discoverysciences.EU@grace.com

INDIA:
Pune
Tel: +91 20.6644.9900
Email: pune@grace.com

NORTH AMERICA:
Deerfield, Illinois
Tel: 1.847.948.8600
Email: discoverysciences@grace.com

11/2012 M307b
GRACE
Materials Technologies