APPLICATIONS

Cleanup of Basic Pharmaceutical Drugs from Plasma using Strata® DE Supported Liquid Extraction (SLE)

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Matt Brusius is an avid ice hockey player. He likes skating backwards and taking slapshots from the point.

Introduction

Bioanalytical laboratories within the pharmaceutical industry, such as DMPK labs, are constantly faced with the challenge of isolating proprietary drugs, often basic in nature, from biological samples such as plasma or tissue. With many clean-up techniques available, it is sometimes difficult to choose between speed, which allows for a high-throughput setting, and cleanliness, which results in improved downstream chromatography. In this technical note, supported liquid extraction is carried out in conjunction with LC-MS/MS to effectively extract a basic pharmaceutical drug from plasma, demonstrating the applicability of this clean-up technique for bioanalytical drug analysis.

Experimental Conditions

Sample Pre-treatment

Dilute 150 µL plasma with 140 µL 2 % NH_4OH and 10 µL "Basic Drug A" spiking solution (1 µg/mL).

SLE Protocol

96-Well Plate:	Strata DE 400 µL
Part No.:	8E-S325-5GB
Load:	300 µL Pre-treated sample onto plate
Wait:	5 minutes
Elute:	3x 600 µL Ethyl acetate/Hexane (3:1)
Apply:	Vacuum at 5-10" Hg for 10 seconds
Dry:	Sample under slow stream of Nitrogen at 30 °C
Reconstitute:	100 µL 0.1% Formic acid/Methanol (4:1) with internal standard

LC-MS/MS Conditions

Column: Kinetex® 2.6 µm Biphenyl
Dimensions: 50 x 2.1 mm
Part No.: 00B-4622-AN
Mobile Phase: A: 0.1% Formic acid in Water
B: 0.1% Formic acid in Acetonitrile

Gradient:

Time (min)	% B
0	20
3	70
4	70
4.01	20
5.5	120

Flow Rate: 0.5 mL/min
Injection Volume: 5 µL
Detection: MS/MS (SCIEX API 4000™), ESI+

Results and Discussion

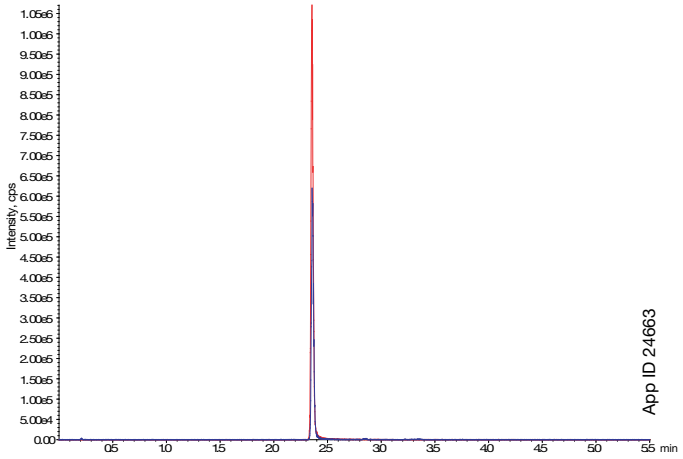
As many drugs within pharmaceutical labs are proprietary, we choose to analyze a representative drug, "Basic Drug A", that contains an amine group and hydrophobic ring structures, both of which are common to most pharmaceutical drugs. When developing our SLE cleanup method, we also chose to use a 96-well plate format that can be automated, making it an ideal format for high-throughput processing.

Table 1 shows the recovery value and %CV for "Basic Drug A". This method shows that it is accurate and precise. The percent recovery is based on a matrix matched standard. **Figure 1** provides a representative chromatogram for an extracted sample.

Table 1. Absolute Recovery and % CV

	% Recovery	%CV (n=8)
Basic Drug A	92	6

Figure 1.
Extracted Chromatogram after Cleanup using Strata DE SLE



Conclusion

The SLE procedure using Strata DE was simple and quick, requiring very little method development other than screening elution solvents. The resulting clean sample indicates that this technique is a great candidate for bioanalytical laboratories as it is fast, scalable for high-throughput, and resulted in a clean sample with little to no interferences.

APPLICATIONS

Ordering Information

Kinetex® Core-Shell LC Columns

Kinetex 2.6 µm Minibore Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	3/pk
Biphenyl	00A-4622-AN	00B-4622-AN	00D-4622-AN	00F-4622-AN	AJ0-9209 for 2.1 mm ID

Kinetex 2.6 µm MidBore™ Columns (mm)				SecurityGuard ULTRA Cartridges‡
Phases	50 x 3.0	100 x 3.0	150 x 3.0	3/pk
Biphenyl	00B-4622-Y0	00D-4622-Y0	00F-4622-Y0	AJ0-9208 for 3.0 mm ID

‡ SecurityGuard ULTRA Cartridges required holder, Part No.: AJ0-9000.

Other particle sizes and dimensions are available. Visit www.phenomenex.com/Kinetex for a complete list of available columns.

Strata® DE Supported Liquid Extraction

Part No.	Description	Unit
8E-S325-FGB	Strata DE SLE 200 µL 96-Well Plate	2/pk
8E-S325-5GB	Strata DE SLE 400 µL 96-Well Plate	2/pk
8B-S325-KDG	Strata DE SLE 12 cc Tube	20/pk
8B-S325-VFF	Strata DE SLE 60 cc Tube	16/pk

Presston™ 100 Positive Pressure Manifold

Part No.	Description
AH0-9334	Presston 100 Positive Pressure Manifold, 96-Well Plate
AH0-9342	Presston 100 Positive Pressure Manifold, 1 mL Tube Complete Assembly
AH0-9347	Presston 100 Positive Pressure Manifold, 3 mL Tube Complete Assembly
AH0-9343	Presston 100 Positive Pressure Manifold, 6 mL Tube Complete Assembly

Presston 100 Tube Adapter Kits (for AH0-9334)

Part No.	Description
AH0-9344	1 mL Tube Adapter Kit
AH0-9345	3 mL Tube Adapter Kit
AH0-9346	6 mL Tube Adapter Kit

WARRANTY Phenomenex warrants that for a period of 12 months following delivery, the Presston 100 Positive Pressure Manifold you have purchased will perform in accordance with the published specifications and will be free from defects in materials or workmanship. In the event that the Presston 100 Positive Pressure Manifold does not meet this warranty, Phenomenex will repair or replace defective parts. Please visit www.phenomenex.com/Presston for complete warranty information.

guarantee

If Phenomenex products in this technical note do not provide at least equivalent separations as compared to other products of the same phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.

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