

APPLICATIONS

Per-Fluoroalkyl Substances (PFAS) from Butter using roQ™ QuEChERS and Strata®-X-AW Solid Phase Extraction by LC-MS/MS

Agustin Pierri¹, PhD and Scott Krepich²

¹Weck Laboratories, Industry, CA 91745 USA

²Phenomenex, Inc., 411 Madrid Ave., Torrance, CA 90501 USA

Overview

Previous analysis has centered around quantitation in drinking water and environmental samples. Presented is a validated two-step extraction procedure of 23 PFAS analytes from butter, making use of QuEChERS followed by Solid Phase Extraction and analyzed by LC-MS/MS. The high resolution and unique mixed-mode selectivity of a Luna® Omega 1.6 µm PS C18 LC column results in excellent chromatography in a very short 4 minute run. In addition, further analysis of milk, eggs, and fish tissue were also validated using the method.

Sample Preparation

Step 1. QuEChERS Extraction and dSPE Protocol

roQ Extraction Kit (Part No. KS0-8910)

- Take 1 g sample* and add into 50 mL centrifuge tube
- *Spike samples with IS/surrogate and/or analytical spike as appropriate

- Add 10 mL Acetonitrile and 10 mL Water

- Add salts in salt packets

- Shake violently to break up clumps and vortex for 30 seconds, and centrifuge

roQ QuEChERS DSPE Kit (Part No. KS0-8920)

- Remove 1 mL of acetonitrile and add to dSPE Kit Tube
- Vortex for 30 seconds and centrifuge
- Remove 1 mL clean Acetonitrile and analyze, or proceed to Step 2 SPE Protocol

Step 2. Solid Phase Extraction (SPE) Protocol

Pre-treatment:

Take 1 mL clean acetonitrile from QuEChERS Protocol and dilute to ~15 mL with water

Solid Phase Extraction

Cartridge: Strata®-X-AW 200 mg, 3 mL

Part No.: 8B-S038-FBJ

Condition: Add 3 aliquots of 0.3% NH₄OH/ACN

Load: ~15 mL of diluted QuEChERS Extract

Wash: 5 mL Water

Elute: 4 mL 0.3% NH₄OH/ACN

Dry: Blow down to 1 mL and Spike with IS and Analyze

LC-MS/MS Conditions

Column: Luna Omega 1.6 µm PS C18

Dimensions: 100 x 2.1 mm

Part No.: 00D-4752-AN

Mobile Phase: A: 5 mM Ammonium Acetate in Water
B: Acetonitrile

Gradient	Time (min)	B (%)
	0	40
	0.5	40
	3	90
	3.1	100
	4	100

Flow Rate: 0.55 mL/min

Injection: 5 µL

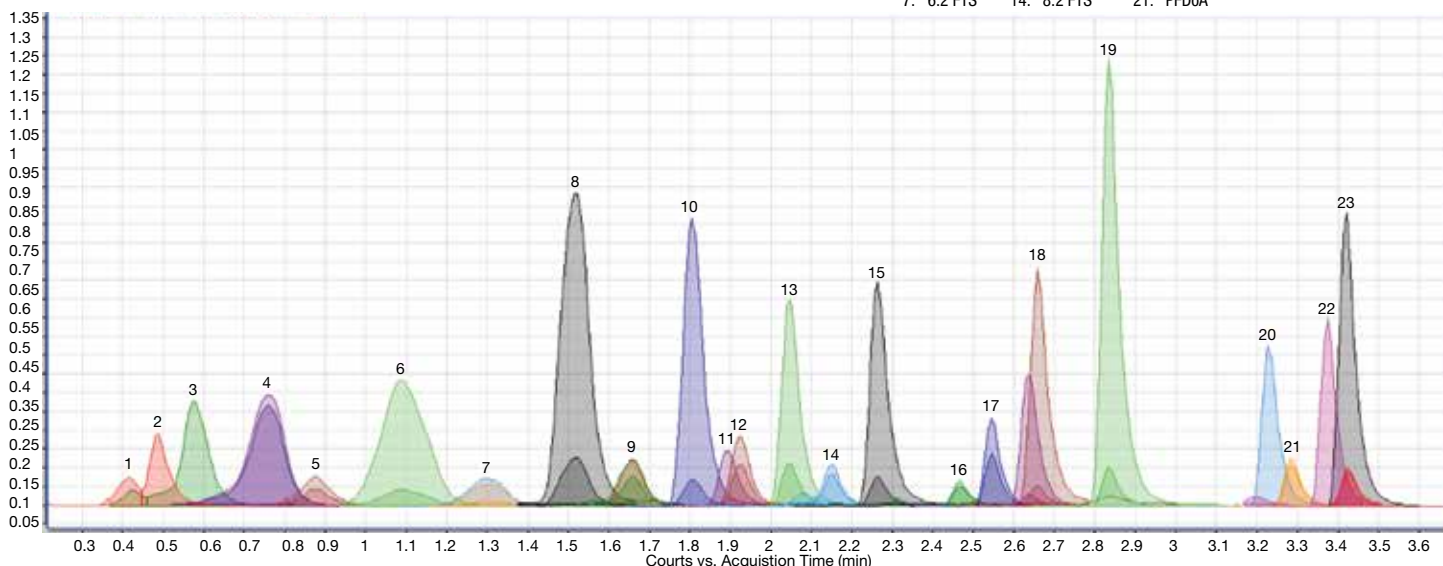
Temperature: 40 °C

System: Agilent® 1290

Detection: Agilent 6460 QQQ

Analytes	1. PFBA	2. PFPeA	3. PFBS	4. PFHxA	5. PFHpA	6. PFHxS	7. 6:2 FTS	8. PFOA	9. PFHpS	10. PFOS	11. PFNA	12. FOSA	13. Me-FOSE	14. 8:2 FTS	15. Me-FOSA	16. PFDA	17. Et-FOSE	18. Et-FOSA	19. PFDS	20. PFUDA	21. PFDoA	22. PFTeDA	23. PFTeDA
----------	---------	----------	---------	----------	----------	----------	------------	---------	----------	----------	----------	----------	-------------	-------------	-------------	----------	-------------	-------------	----------	-----------	-----------	------------	------------

Butter Sample using Two-Step Sample Preparation Method



App ID 24633



APPLICATIONS

Luna[®] Omega Ordering Information

1.6 µm Minibore Columns (mm)		SecurityGuard [™] ULTRA Cartridges [†]				
30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	3/pk		
PS C18	00A-4752-AN	00B-4752-AN	00D-4752-AN	00F-4752-AN	AJ0-9508	for ID: 2.1 mm ID

[†] SecurityGuard ULTRA Cartridges require holder, Part No.: AJ0-9000

5 µm Minibore Columns (mm)		SecurityGuard Cartridges (mm)				
30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	4 x 2.0* (10/pk)		
PS C18	00A-4753-AN	00B-4753-AN	00D-4753-AN	00F-4753-AN	AJ0-7605	for ID: 2.0 - 3.0 mm

5 µm MidBore [™] Columns (mm)		SecurityGuard Cartridges (mm)			
50 x 3.0	100 x 3.0	150 x 3.0	4 x 2.0* (10/pk)		
PS C18	00B-4753-Y0	00D-4753-Y0	00F-4753-Y0	AJ0-7605	for ID: 2.0 - 3.0 mm

5 µm Analytical Columns (mm)		SecurityGuard Cartridges (mm)				
50 x 4.6	100 x 4.6	150 x 4.6	250 x 4.6	4 x 3.0* (10/pk)		
PS C18	00B-4753-E0	00D-4753-E0	00F-4753-E0	00G-4753-E0	AJ0-7606	for ID: 3.1-8.0 mm

[†] SecurityGuard Analytical cartridges require holder, Part No.: KJ0-4282

Australia

t: +61 (0)2-9428-6444
f: +61 (0)2-9428-6445
auinfo@phenomenex.com

Austria

t: +43 (0)1-319-1301
f: +43 (0)1-319-1300
anfrage@phenomenex.com

Belgium

t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
f: +31 (0)30-2383749
beinfo@phenomenex.com

Canada

t: +1 (800) 543-3681
f: +1 (310) 328-7768
info@phenomenex.com

China

t: +86 400-606-8099
f: +86 (0)22 2532-1033
phen@agela.com

Denmark

t: +45 4824 8048
f: +45 4810 6265
nordicinfo@phenomenex.com

Finland

t: +358 (0)9 4789 0063
f: +45 4810 6265
nordicinfo@phenomenex.com

France

t: +33 (0)1 30 09 21 10
f: +33 (0)1 30 09 21 11
franceinfo@phenomenex.com

Germany

t: +49 (0)6021-58830-0
f: +49 (0)6021-58830-11
anfrage@phenomenex.com

India

t: +91 (0)40-3012 2400
f: +91 (0)40-3012 2411
indiainfo@phenomenex.com

Ireland

t: +353 (0)1 247 5405
f: +44 1625-501796
eireinfo@phenomenex.com

Italy

t: +39 051 6327511
f: +39 051 6327555
italiainfo@phenomenex.com

Luxembourg

t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

Mexico

t: 01-800-844-5226
f: 001-310-328-7768
tecnicomx@phenomenex.com

The Netherlands

t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

New Zealand

t: +64 (0)9-4780951
f: +64 (0)9-4780952
nzinfo@phenomenex.com

Norway

t: +47 810 02 005
f: +45 4810 6265
nordicinfo@phenomenex.com

Puerto Rico

t: +1 (800) 541-HPLC
f: +1 (310) 328-7768
info@phenomenex.com

Spain

t: +34 91-413-8613
f: +34 91-413-2290
espinfo@phenomenex.com

Sweden

t: +46 (0)8 611 6950
f: +45 4810 6265
nordicinfo@phenomenex.com

Switzerland

t: +41 61 692 20 20
f: +41 61 692 20 22
swissinfo@phenomenex.com

United Kingdom

t: +44 (0)1625-501367
f: +44 (0)1625-501796
ukinfo@phenomenex.com


USA

t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

www.phenomenex.com

Phenomenex products are available worldwide. For the distributor in your country, contact Phenomenex USA, International Department at international@phenomenex.com

Strata[®]-X-AW Ordering Information

Format	Sorbent Mass	Part Number	Unit
Tube			
	100 mg	8B-S038-EBJ	3 mL (50/box)
	100 mg	8B-S038-ECH	6 mL (30/box)
	200 mg	8B-S038-FBJ	3 mL (50/box)
	200 mg	8B-S038-FCH	6 mL (30/box)
	500 mg	8B-S038-HBJ	3 mL (50/box)
	500 mg	8B-S038-HCH	6 mL (30/box)

roQ[™] Extraction Kits

Extraction kits contain fifty easy-pour salt packets and fifty 50 mL stand-alone centrifuge tubes

Description	Unit	Part No.
AOAC 2007.01 Method Extraction Kits		
6.0 g MgSO ₄ , 1.5 g NaOAc	50/pk	KS0-8911*
EN 15662 Method Extraction Kits		
4.0 g MgSO ₄ , 1.0 g NaCl, 1.0 g SCTD, 0.5 g SCDS	50/pk	KS0-8909*
Original Non-buffered Method Extraction Kits		
4.0 g MgSO ₄ , 1.0 g NaCl	50/pk	KS0-8910
6.0 g MgSO ₄ , 1.5 g NaCl	50/pk	KS0-8912

*AOAC and EN Extraction Kits also available in traditional non-collared 50 mL centrifuge tubes, Part No.: KS0-8911-NC and KS0-8909-NC

roQ dSPE Kits

dSPE kits contain pre-weighed sorbents/salts inside 2 mL or 15 mL centrifuge tubes

Description	Unit	Part No.
2 mL dSPE Kits		
150 mg MgSO ₄ , 25 mg PSA, 25 mg C18E	100/pk	KS0-8913
150 mg MgSO ₄ , 25 mg PSA, 2.5 mg GCB	100/pk	KS0-8914
150 mg, MgSO ₄ , 25 mg PSA, 7.5 mg GCB	100/pk	KS0-8915
150 mg MgSO ₄ , 25 mg PSA	100/pk	KS0-8916
150 mg MgSO ₄ , 50 mg PSA, 50 mg C18E, 50 mg GCB	100/pk	KS0-8917
150 mg MgSO ₄ , 50 mg PSA, 50 mg C18E	100/pk	KS0-8918
150 mg MgSO ₄ , 50 mg PSA, 50 mg GCB	100/pk	KS0-8919
150 mg MgSO ₄ , 50 mg PSA	100/pk	KS0-8920
15 mL dSPE Kits		
900 mg MgSO ₄ , 150 mg PSA, 150 mg C18E	50/pk	KS0-8921
900 mg MgSO ₄ , 150 mg PSA, 15 mg GCB	50/pk	KS0-8922
900 mg MgSO ₄ , 150 mg PSA, 45 mg GCB	50/pk	KS0-8923
900 mg MgSO ₄ , 150 mg PSA	50/pk	KS0-8924
1200 mg MgSO ₄ , 400 mg PSA, 400 mg C18E, 400 mg GCB	50/pk	KS0-8925
1200 mg MgSO ₄ , 400 mg PSA, 400 mg C18E	50/pk	KS0-8926
1200 mg MgSO ₄ , 400 mg PSA, 400 mg GCB	50/pk	KS0-8927
1200 mg MgSO ₄ , 400 mg PSA	50/pk	KS0-8928

Terms and Conditions

Subject to Phenomenex Standard Terms and Conditions which may be viewed at www.phenomenex.com/TermsAndConditions

Trademarks

Luna and Strata are registered trademarks and roQ, MidBore, Giga, and SecurityGuard are trademarks of Phenomenex. Agilent is a registered trademark of Agilent Technologies, Inc.

Disclaimer

Phenomenex is not affiliated with Agilent Technologies.

SecurityGuard is patented by Phenomenex. U.S. Patent No. 6,162,362.

CAUTION: this patent only applies to the analytical-sized guard cartridge holder, and does not apply to SemiPrep, PREP or ULTRA holders, or to any cartridges.

© 2017 Phenomenex, Inc. All rights reserved.



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