

# Sample Preparation Solutions for Environmental Analysis

# **Experience and Expertise**

As the leader in chromatography with over 40 years of experience, Agilent takes extensive measures to ensure that your sample prep results are accurate and reliable every time.



#### Bond Elut Plexa method for polyaromatic hydrocarbons

Twenty-four PAHs in drinking water by automated SPE with fast HPLC-FLD/UV detection (Pub No. 5990-7686EN)

1. 800 mL water sample + 5% isopropanol + internal standard (benzo[a]pyrene-d<sup>12</sup>)

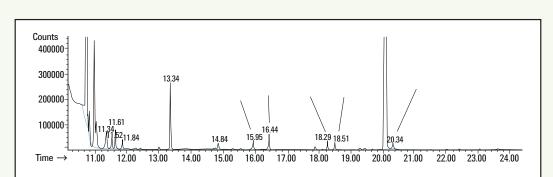
- 2. Condition with 4 mL ethyl acetate + 4 mL dichloromethane + 4 mL methanol + 4 mL water
- 3. Load sample
- 4. Dry for 30 min 5. Elute with 4 mL ethyl acetate + 4 mL dichloromethane 6. Make up to 10 mL with ethyl acetate: dichloromethane (1:1)
- 7. Evaporate off 4 mL 8. Add 0.5 mL acetonitrile



#### Bond Elut Plexa method for phenols

Bond Elut Plexa efficient preparation of phenols in drinking water (Pub No. SI-1549)

- 1. Condition with 10 mL ethyl acetate/10 mL methanol /10 mL water 2. Apply sample at 10-20 mL/min
- 3. Rinse with 10 mL water 4. Dry for 30 min or more with N<sub>2</sub> gas or a vacuum manifold
- 5. Elute with 5 mL ethyl acetate 6. Dry using a few crystals of anhydrous sodium sulfate 7. Gently concentrate 4 mL of eluted solution down to 0.8 mL under N<sub>2</sub> gas
- 8. Add 100 µL N,O-bis (trimethylsilyl) trifluoroacetamide and let stand for an hour 9. Add 20 µL of internal standard 10. Make up to 1 mL with ethyl acetate



8. Methyl 2-bromobutanoate (SS) 11. Methyl dibromochloroacetate

3 4 5 6

12. Methyl tribromoacetate

DB-35ms UI

DB-XLB

Methyl hromodichloroacetate

Figure 1. HPLC/FLD chromatogram of a 5 μL injection of the 20 ppt PAH standard solution on the Agilent Pursuit 3 PAH column.

Figure 2. Gas chromatogram of an extract of phenols from drinking water using Agilent Bond Elut Plexa (phenols at 0.1 ppm).

Methylated HAAs fortified QC sample

Methyl trichloroacetate

6. 1,2,3-Trichloropropane (IS)

2. Methyl bromoacetate

Methyl dichloroacetate

#### Bond Elut SAX method for haloacetic acids

Determination of haloacetic acids in water by GC/µECD using Agilent J&W DB-35ms Ultra Inert and DB-XLB columns (Pub No. 5990-8765EN)

#### **Bond Elut SAX SPE method**

1. Add 0.5 mL aqueous NH<sub>4</sub>CL to 50 mL water sample 2. Adjust pH of sample to pH5  $\pm$  0.5 with sulfuric acid as necessary 3. Add surrogate, and QC spike solution if necessary

4. Assemble glass block manifold and attach SPE cartridges

- 5. Condition Bond Elut SAX SPE cartridges with 10 mL MeOH, followed by 10 mL
- 6. Add the 50 mL water sample to cartridge while drawing under vacuum at 7. Add 10 mL MeOH to cartridge and draw through at 2 mL/min
- 8. Disassemble vacuum manifold and insert 15 mL screw cap centrifuge tubes 9. Add 3 mL 10% H<sub>2</sub>SO<sub>4</sub>/MeOH to cartridge and elute at 1.5 mL/min

#### **Esterification procedure**

- 1. Add 2 mL MTBE w/IS solution to eluent, cap, vortex for 5 seconds 2. Place capped centrifuge tubes in heating block at 50 °C for 2 hrs 3. Remove centrifuge tubes and allow to cool 4. Add 7 mL of 150 g/L aqueous sodium sulfate solution and vortex for
- 30 seconds 5. Allow phases to separate (~5 min)
- 6. Remove and discard lower aqueous layer using long Pasteur pipet 7. Add 1 mL saturated aqueous sodium bicarbonate solution and vortex
- for several seconds

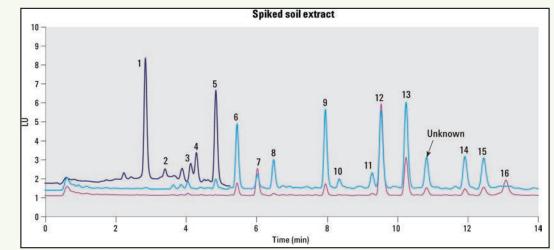
#### 8. Transfer ether layer to GC vial and analyze

Bond Elut QuEChERS method for PAHs

#### Analysis of PAH in soil with Agilent Bond Elut QuEChERS AOAC kit and

#### HPLC-FLD (Pub No. 5990-5452EN)

- QuEChERS AOAC sample prep procedure I. Weigh 5 g soil homogenate into a 50 mL centrifuge tube
- 2. Spike samples with 2000 µL spiking solution and shake vigorously for 1 min 3. Add 5 mL water and shake vigorously for 1 min
- 4. Add 10 mL CH<sub>3</sub>CN 5. Add Bond Elut QuEChERS AOAC salt packet and shake for 1 min, centrifuge
- at 4000 rpm for 5 min 6. Transfer 5 mL aliquot to Bond Elut QuEChERS Dispersive SPE 15 mL tube
- and shake for 1 min, centrifuge at 4000 rpm for 5 min 7. Filter through a 0.45 µm PVDF syringe filter
- 8. Transfer 1 mL extract to an autosampler vial 9. Analyze with HPLC/FLD



8 min

Figure 3. GC/µECD chromatograms for two water samples analyzed using Agilent J&W DB-35ms UI (p/n 122-3832UI) and

DB-XLB (p/n 122-1236) GC columns. Chromatographic conditions are listed in Table 1 in Pub. No. 5990-8765EN.

Figure 3. Overlaid HPLC/FLD chromatograms of a spiked soil sample containing: 1. Nap 2. Acy 3. Ace 4. Flu 5. Phe 6. Ant 7. Fln 8. Pyr 9. BaA 10. Chr 11. BeP 12. BeA 13. BkF 14. DahA 15. BghiP 16. InP. The spiking level for this sample was level 1 (see Table 3 in Pub. No. 5990-5452EN). The blue portion of the chromatogram used the following excitation/emission wavelengths: 260 nm/352 nm; the red portion: 260 nm/420 nm and the light blue portion: 260 nm/440 nm. However, due to lack of a flourophore, UV detection at 230 nm was employed for acenaphthylene. Chromatographic conditions are shown in Table 1 in Pub. No. 5990-5452EN.

## Bond Elut SPE

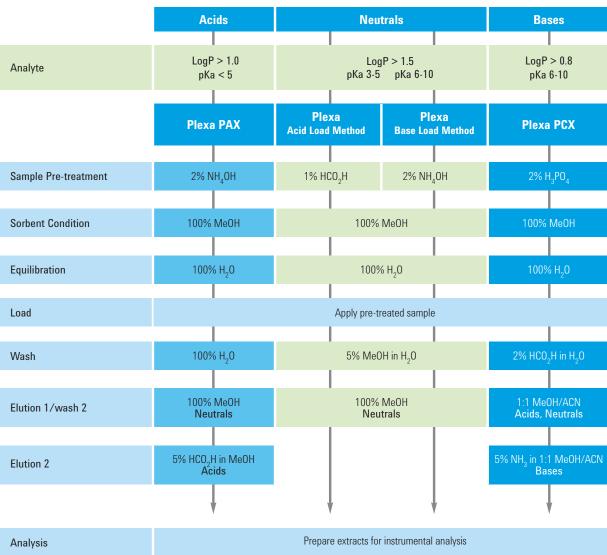
For over 30 years, Bond Elut has been the most trusted name in solid phase extraction (SPE).

Agilent Bond Elut SPE products selectively remove interferences from complex matrices and provide the largest choice of sorbent formats in the market today. Over 40 phase functionalities in more than 30 formats are available.

#### **Bond Elut Polymeric SPE**

- Bond Elut Plexa family is a new generation of polymeric SPE products designed for simplicity, improved analytical performance, and ease-of-use.
- Bond Elut Plexa is a non-polar divinylbenzene-based neutral polymeric sorbent.
- Bond Elut Plexa PCX is a cation exchanger with mixed mode sorbent characteristics.
- Bond Elut Plexa PAX is an anion exchanger with mixed mode sorbent characteristics.

#### General protocol for trouble-free SPE applications with the Bond Elut Plexa family



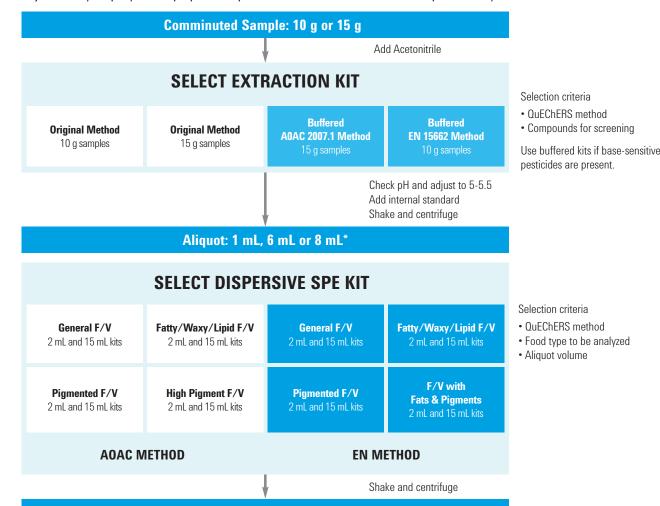
# Bond Elut QuEChERS

Agilent Bond Elut QuEChERS Kits make sample prep easy as 1- 2- 3. Kits are pre-packaged, providing an easy way to capture the time-saving benefits of QuEChERS sample preparation.

- QuEChERS kits are pre-measured and packed in anhydrous salt packets to ensure high recoveries in your pesticide analysis.
- Kits with ceramic homogenizers save sample prep time by reducing shaking steps to a matter of seconds, promoting consistent sample extraction and increased product recovery.
- Universal dispersive kits provide excellent recoveries and reproducibility for soil and other non-food matrices.

#### **Agilent Recommended Standard Operating Procedure for Bond Elut QuEChERS**

In just 3 easy steps, you can prepare sample for multi-class, multi-residue compound analysis.



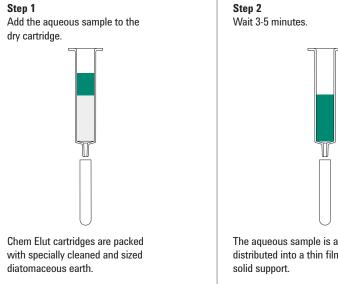
\* Aliguot size is specified by the method, and kits are created for these specific amounts. For pesticides or other compounds with acidic groups (phenoxyalcanoic acids), analyze directly by LC/MS/MS at this point (skip the dispersive SPE stage). These acidic groups interact with the PSA that is part of the dispersive SPE step.

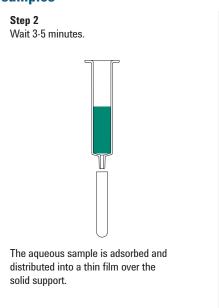
# Chem Elut SLE

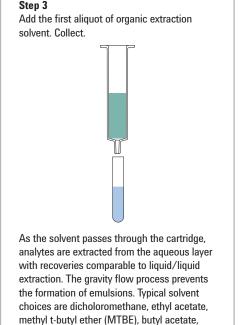
Chem Elut is a high purity, sorbent-supported liquid extraction (SLE) cartridge, available in buffered and unbuffered formats.

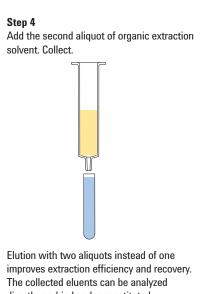
- Chem Elut streamlines methods for all sample types and eliminates phase separation and emulsion problems.
- Easier to automate than manual liquid-liquid method and requires lower volumes of organic (often chlorinated) solvents.
- The base-treated cartridge removes residual acid compounds from a variety of matrices.

## **Extraction procedure for aqueous samples**









directly, or dried and reconstituted

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## **Agilent J&W Ultra Inert GC** columns

Agilent J&W Ultra Inert GC columns push industry standards for consistent column inertness and exceptionally low column bleed, resulting in lower detection limits and more accurate data for difficult analytes. And, Agilent J&W Ultra Inert GC columns are individually tested with the most demanding Ultra Inert test probe mixture in the industry, and a performance summary sheet is shipped with each column. For more information visit www.agilent.com/chem/ultrainert



#### **Agilent LC and** LC/MS columns

With Agilent's ZORBAX and Poroshell 120 LC columns, you'll generate reproducible results across a wide range of applications and conditions. These columns are engineered o deliver superior performance and boost productivity. You get fast LC choices: Rapid Resolution High Throughput RRHT); Eclipse Plus and Poroshell 120, stable to 600 bar; and Rapid Resolution High Definition (RRHD), stable to 1200 bar for ultra-fast separations. For more information visit www.agilent.com/chem/lccolumns



#### Agilent GC, GC/MS, LC and **LC/MS** instruments

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methyl ethyl ketone (MEK).

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### **Bond Elut QuEChERS Extraction Kits**

**Bond Elut QuEChERS Dispersive Kits** 

Fats/Waxy/Lipids F/V: 2 mL

acids, some sugars, more lipids and sterols

Bond Elut SPE, Bond Elut QuEChERS,

and Chem Elut Part Numbers

Unit Part No.

100/pk 12109301

100/pk 12109601

50/pk 12109603

50/pk 12109610

30/pk 12109206

30/pk 12259506

50/pk 12169610B

20/pk 327832

50/pk 167816G

**Bond Elut Plexa (Polymeric SPE)** 

**Straight Barrel Cartridges** 

30 mg, 1 mL

30 mg, 3 mL

60 mg, 1 mL 60 mg, 3 mL

200 mg, 3 mL

200 ma. 6 mL

500 mg, 6 mL

Bond Elut Jr

500 mg, 12 mL

Other Formats

Mega Bond Elut Plexa

60 mg, 3 mL, Gerstel format

200 mg, 3 mL, Gerstel format

			Part No.	Part No. Pac	kets Only
Method	Buffered	Contents	With Tubes	50/pk	200/pk
AOAC 2007.01	Yes	6 g MgSO <sub>4</sub> ; 1.5 g NaAcetate	5982-5755	5982-6755	5982-7755
Original (10 g samples)	No	4 g MgSO <sub>4</sub> ; 1 g NaCl	5982-5550	5982-6550	5982-7550
Original (15 g samples)	No	6 g MgSO <sub>4</sub> ; 1.5 g NaCl	5982-5555	5982-6555	5982-7555
EN 15662	Yes	4 g MgSO <sub>4</sub> ; 1 g NaCl; 1 g NaCitrate; 0.5 g disodium citrate sesquihydrate	5982-5650	5982-6650	5982-7650
Acrylamides*	No		5982-5850		

terina Mastovaka and Steven J. Lehotay have done work to extend the scope of QuEChERS beyond fruits and regetables<sup>2</sup>, using it to extract acrylamides in potato chips and other fried foods. 2: "Rapid Sample Preparation Method for LC/MS/MS or GC/MS Analysis of Acrylamides in Various FoodMatrices" J. Agric. Food Chem, 2006, 54, 7001-7008.

#### **Bond Elut QuEchERS Ceramic Homogenizers** Part No. Ceramic homogenizer for 50 mL tubes 100/pk 5982-9313 5982-9312 5982-9311 Ceramic homogenizer for 2 mL tubes

50 mg PSA

150 mg MgSO<sub>4</sub>

25 mg C18EC

150 mg MgSO<sub>4</sub>

Bond Elut Plexa PCX (Polymeric SPE)				
Description	Unit	Part No.		
Straight Barrel Cartridges				
30 mg, 1 mL	100/pk	12108301		
60 mg, 1 mL	100/pk	12108601		
30 mg, 3 mL	50/pk	12108303		
60 mg, 3 mL	50/pk	12108603		
200 mg, 6 mL	30/pk	12108206		
500 mg, 6 mL	30/pk	12258506		
Other Formats				
Bond Elut Plexa PCX Prospekt	96/pk	12221306		

Bond Elut Plexa Prospekt cartridge, 1 mm 96/pk 12221305

Description	Unit
Straight Barrel Cartridges	
30 mg, 1 mL	100/pk
60 mg, 1 mL	100/pk
30 mg, 3 mL	50/pk
60 mg, 3 mL	50/pk
200 mg, 6 mL	30/pk
500 mg, 6 mL	30/pk

Description	Unit	Pa
Straight Barrel Cartridges		
50 mg, 1 mL	100/pk	12
100 mg, 1 mL	100/pk	12
100 mg, 3 mL	50/pk	12
200 mg, 3 mL	50/pk	12
200 mg, 6 mL	30/pk	12
500 mg, 3 mL	50/pk	12
500 mg, 6 mL	30/pk	12
1 g, 6 mL	30/pk	1:

Bond Elut PPL (Polymeric SPE)				
Description	Unit	Part No.		
Straight Barrel Cartridges				
50 mg, 1 mL	100/pk	1210500		
100 mg, 1 mL	100/pk	1210500		
100 mg, 3 mL	50/pk	1210500		
200 mg, 3 mL	50/pk	1210500		
500 mg, 3 mL	50/pk	1210500		
500 mg, 6 mL	30/pk	1225500		
1 g, 3 mL	50/pk	1210214		
1 g, 6 mL	30/pk	12255002		

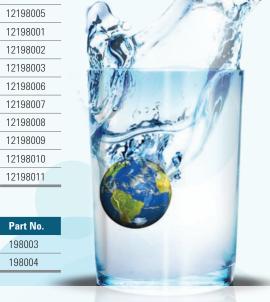
<b>Bond Elut SA</b>	X (Silica	-Based SPE)	
Description	Unit	40 μm Particle Size Part No.	120 µm Particle Size Part No.
LRC Cartridges			
100 mg, 10 mL	50/pk	12113017	14113017
500 mg, 10 mL	50/pk	12113043	14113043
Straight Barrel	Cartridges	3	
50 mg, 1 mL	100/pk	12102079	14102079
100 mg, 1 mL	100/pk	12102017	14102017
100 mg, 3 mL	50/pk	12102125	
500 mg, 3 mL	50/pk	12102044	14102044
500 mg, 6 mL	30/pk	12102144	
1 g, 3 mL	50/pk	12102087	
1 g, 6 mL	30/pk	12256013	14256013
2 g, 6 mL	30/pk	12256051	
2 g, 12 mL	20/pk	12256021	14256021
5 g, 20 mL	20/pk	12256029	14256029
10 g, 60 mL	16/pk	12256037	14256037
Bond Elut Jr			
500 mg	100/pk	12162044B	
1 g	100/pk	12166013B	

Prospekt cartridge, 96/pk 12281022

	15 mL	50/pk	400 mg PSA 400 mg C18EC 1200 mg MgSO <sub>4</sub> 5982-5158 5982-5158CH	150 mg PSA 150 mg C18EC 900 mg MgSO <sub>4</sub> 5982-5156 5982-5156CH
Pigmented F/V: Removes polar organic acids, some sugars and lipids, and carotenoids and chlorophyll; not for use	2 mL	100/pk	50 mg PSA 50 mg GCB 150 mg MgSO <sub>4</sub> 5982-5222 5982-5222CH	25 mg PSA 2.5 mg GCB 150 mg MgSO <sub>4</sub> 5982-5221 5982-5221CH
with planar pesticides	15 mL	50/pk	400 mg PSA 400 mg GCB 1200 mg MgSO <sub>4</sub> 5982-5258 5982-5258CH	150 mg PSA 15 mg GCB 885 mg MgSO <sub>4</sub> 5982-5256 5982-5256CH
F/V with Pigments and Fats: Removes polar organic acids, some sugars and lipids, plus carotenoids and chlorophyll; not for use with planar pesticides	2 mL	100/pk	50 mg PSA 50 mg GCB 150 mg MgSO <sub>4</sub> 50 mg C18EC 5982-5421 5982-5421CH	
	15 mL	50/pk	400 mg PSA 400 mg GCB 1200 mg MgSO <sub>4</sub> 400 mg C18EC 5982-5456 5982-5456CH	
Other Food Methods: Removes biological matrix interferences, including hydrophobic substances (fats, lipids) and proteins	2 mL	100/pk	25 mg C18 150 mg MgSO <sub>4</sub> 5982-4921 5982-4921CH	
	15 mL	50/pk	150 mg C18 900 mg MgSO <sub>4</sub> 5982-4956 5982-4956CH	
All Food Types (Universal): Removes all matrix interfering materials including polar organic acids, lipids, sugars,	2 mL	100/pk	50 mg PSA 50 mg C18 7.5 mg GCB 150 mg MgSO <sub>4</sub> 5982-0028 5982-0028CH	
proteins, carotenoids and chlorophyll	15 mL	50/pk	400 mg PSA 400 mg C18 45 mg GCB 1200 MgSO <sub>4</sub> 5982-0029	

<b>EnvirElut (Sil</b>	ica-Based SPE	<b>:</b> )	
Description		Unit	Part
Straight Barrel	Cartridges		
1 g, 6 mL (PAH)		30/pk	12272
500 mg, 6 mL (Pesticide)		30/pk	12272
Chem Elut			
Buffered pH	Sample Size Volume (mL)	Unit	Part N
4.5	3	100/pk	12198
9.0	3	100/pk	12198
Unbuffered	0.3	100/pk	12198
		100/1	10100

	Volume (mL)		
4.5	3	100/pk	1219800
9.0	3	100/pk	1219800
Unbuffered	0.3	100/pk	1219800
	1	100/pk	1219800
	3	100/pk	1219800
	5	100/pk	1219800
	10	100/pk	1219800
	20	100/pk	1219800
	50	50/pk	1219800
	100	25/pk	1219801
	300	15/pk	1219801
Hydromatrix			
Description			Part No
Hydromatrix bulk, 1	kg		198003



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Hydromatrix bulk, 4 kg

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