

# Sample Preparation Solutions for Food 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 Mycotoxin method for fusarium toxin

New SPE Sorbent for Clean-up of Fusarium Toxin-contaminated Cereals & Cereal-based Foods. Bond Elut Mycotoxin (Pub No. SI-00295)

The optimized SPE conditions are:

1. Extract 2 g of finely ground sample with a solution of 100 mL acetonitrile/water (80/20; v/v) by blending at high speed for 3 minutes. For simultaneous determination of zearalenone (ZEA), spike extract at a level of 50 ng/g sample with zearalenone (ZAN) solution in acetonitrile as internal standard.
2. Filter.
3. Pass 4 mL of the filtrate through a Bond Elut Mycotoxin column (p/n 12195001B).
4. Evaporate 2 mL of the eluate to dryness at 50°C under a gentle stream of nitrogen.
5. Reconstitute in 0.5 mL acetonitrile/water (20/80; v/v). Inject 10 µL into LC/MS/MS for analysis.

Trichothecene	R1	R2	R3	R4	R5
Neosolaniol (NEO)	OH	OH	OH	H	OH
HT-2 toxin (HT-2)	OH	OH	OH	H	OOCH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub>
T-2 toxin (T-2)	OH	OH	OH	H	OOCH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub>
T-2 tetraol	OH	OH	OH	H	OOCH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub>
T-2 tetraol	OH	OH	OH	H	OH
Monoacetoxysolaniol (MAS)	OH	OH	OH	H	H
Diacetoxysolaniol (DAS)	OH	OH	OH	H	H
Deoxynivalenol (DON)	OH	H	OH	OH	-
3-Acetyl-DON (3ADON)	OH	H	OH	OH	-
15-Acetyl-DON (15ADON)	OH	H	OH	OH	-
Nivalenol (NIV)	OH	OH	OH	OH	-
Fusarenon X (FX)	OH	OH	OH	OH	-

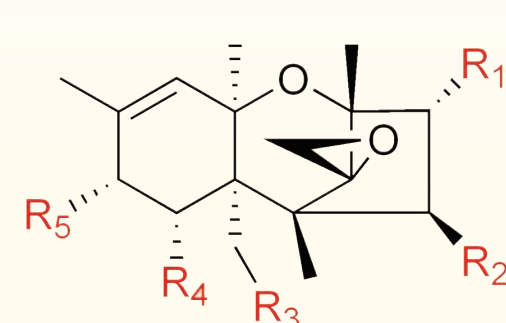


Figure 1. Chemical structure of type A and B trichothecenes, zearalenone (ZEA). Type A trichothecenes have various groups at ring position 8. Type B trichothecenes have a carbonyl function at position 8.

Toxin	Recovery [%] ± RSD [%]	3 concentrations, n = 3
Wheat	90 ± 5.2	93 ± 2.8
Com	98 ± 3.8	96 ± 5.1
Oats	87 ± 1.7	87 ± 3.7
Bread	85 ± 5.7	71 ± 13
Infant Food	88 ± 1.2	88 ± 1.2
DON	67 ± 5.9	74 ± 2.5
3ADON	89 ± 9.3	88 ± 7.6
15ADON	92 ± 13	87 ± 15
FUS	91 ± 1.0	94 ± 2.2
HT-2	87 ± 7.6	88 ± 8.8
T-2	82 ± 7.3	91 ± 3.3
NEO	91 ± 2.6	78 ± 11
DAS	82 ± 8.3	89 ± 3.6
MAS	86 ± 13	85 ± 12
T-2 tetraol	69 ± 1	83 ± 2.8
T-2 tetraol	69 ± 12	75 ± 6.8
ZEA	110 ± 5.9	113 ± 5.0

Table 1. Average recovery and RSD in percentage obtained for 12 trichothecenes and ZEA from spiked wheat, corn, durum, oats, bread, mussels and cereal infant food samples (spiking levels of 50/100, 200/400 and 500/1000 ng/g for trichothecenes, DON and 50 ng/g for ZEA), after clean-up with Bond Elut Mycotoxin columns, (n=3). Data reported by Klitzel et al.<sup>1</sup>

1. M. Klitzel, U. Lauber, H. J. Hummel (2006) "A New Solid Phase Extraction Clean-up Method for the Determination of 12 Type A- and B-Trichothecenes in Cereals and Cereal-based Food by LC/MS/MS". *Mol. Nutr. Food Res.* 20, 261-269

### Chem Elut method for pesticides

Multi-Residue Confirmation of Pesticides in Honey Using Solid Supported Liquid Extraction (Pub No. SI-01002)

SLE procedure on 5 mL Chem Elut cartridge (p/n 12198006)

Method:

1. Spike 1 g honey sample with 20 µL of surrogate standard solution (concentrations listed in Figure 2 in the publication).
2. Mix with 1.25 mL water and 2.5 mL acetone.
3. Add 1.25 mL of NaCl solution (20 g/100 mL).
4. Apply sample to Chem Elut cartridge gravity flow.
5. Allow 15 mins for complete adsorption to take place.
6. Elute twice with 10 mL ethyl acetate.
7. Evaporate at 30 °C.
8. Reconstitute with 200 µL acetonitrile/water (10/90).
9. Inject 20 µL into LC/MS/MS.



Figure 2. Recovery comparison of pesticides between solid supported liquid extraction (SLE) on Chem Elut and classical liquid liquid extraction (LLE).  
Pesticide (ng/mL): Am 0.4, At 0.4, Ca 0.4, Ch 20.0, De 2.0, Di 2.0, Fi 10.0, Im 2.0, Is 2.0, Li 2.0, Mo 2.0, Pi 0.4, Rb 0.4, Si 2.0, TDM 1.0.  
Insecticides: (Ca) Carbaryl (Mb) Methiochlor (Ft) Fenitrothion (Dm) Dimethoate (Ft) Fenprophate (Im) Imidacloprid  
Herbicides: (Am) Amsulfuron (Ri) Rimsulfuron (At) Atrazine (Si) Simazine (Ch) Chlorobutol (Li) Linuron (Is) Isoaflatoxin (Me) Metolachlor  
Fungicides: (Di) Diethofenb  
Metabolites: (Mh) Methocharb sulfide (TOH) 2-Hydroxyterbutylalane

### Bond Elut QuEChERS method for PAHs

Polycyclic Aromatic Hydrocarbon (PAH) Analysis in Fish by GC/MS Using QuEChERS Sample Preparation and High Efficiency DB-5ms Ultra Inert GC Columns (Pub No. 5990-668EN)

1. Weigh 3 g sample (± 0.05g) in 50 mL centrifuge tube.
2. Add surrogate/IS solution, and GC spike solution, if necessary. Vortex 1 min.
3. Add 12 mL of DI water and 2 ceramic homogenizers to the sample (p/n 5982-9313).
4. Add 15 mL of ACN, vortex 1 min.
5. Add Agilent original QuEChERS extraction salt packet for 15 g samples (p/n 5982-8555).
6. Shake vigorously for 1 min in a vertical shaker at 1500 rpm.
7. Centrifuge at 4000 rpm for 5 min.
8. Transfer 8 mL of the ACN layer to Agilent ADAC fatty sample dSPE 15 mL tube (p/n 5982-5158).
9. Vortex 1 min. Centrifuge at 4000 rpm for 5 min.
10. Analyze extract by GC/MS.

GC/MS SIM chromatogram of red snapper fish extracts blank relative to spiked sample after Agilent's QuEChERS extraction and dispersive SPE

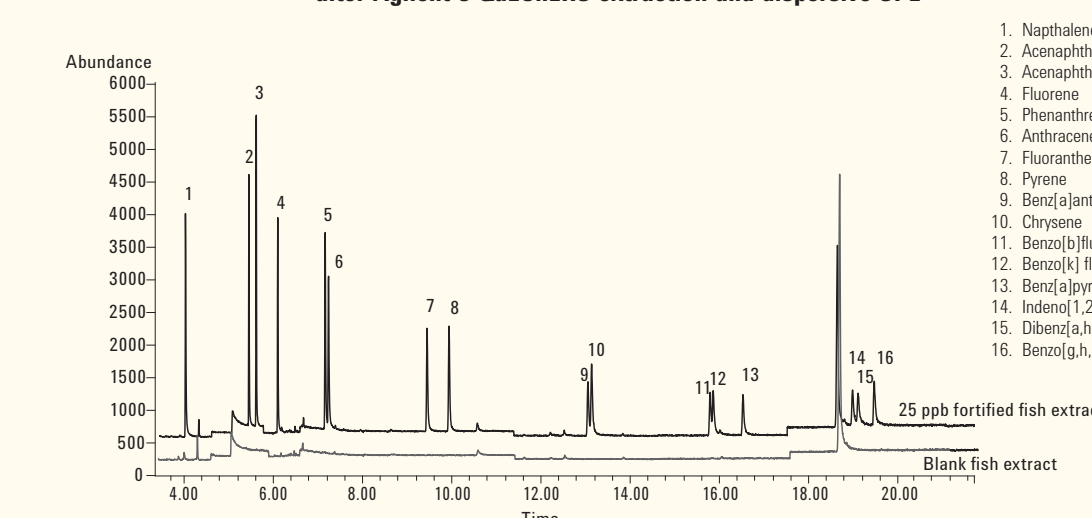


Figure 2. GC/MS SIM chromatogram of the fish extract blank and the 25 ng/mL spiked fish extract analyzed on Agilent J&W DB-5ms UI (p/n 122-5522UI). Chromatographic conditions are listed in Table 1 in the publication.

## Bond Elut Polymeric 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 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.

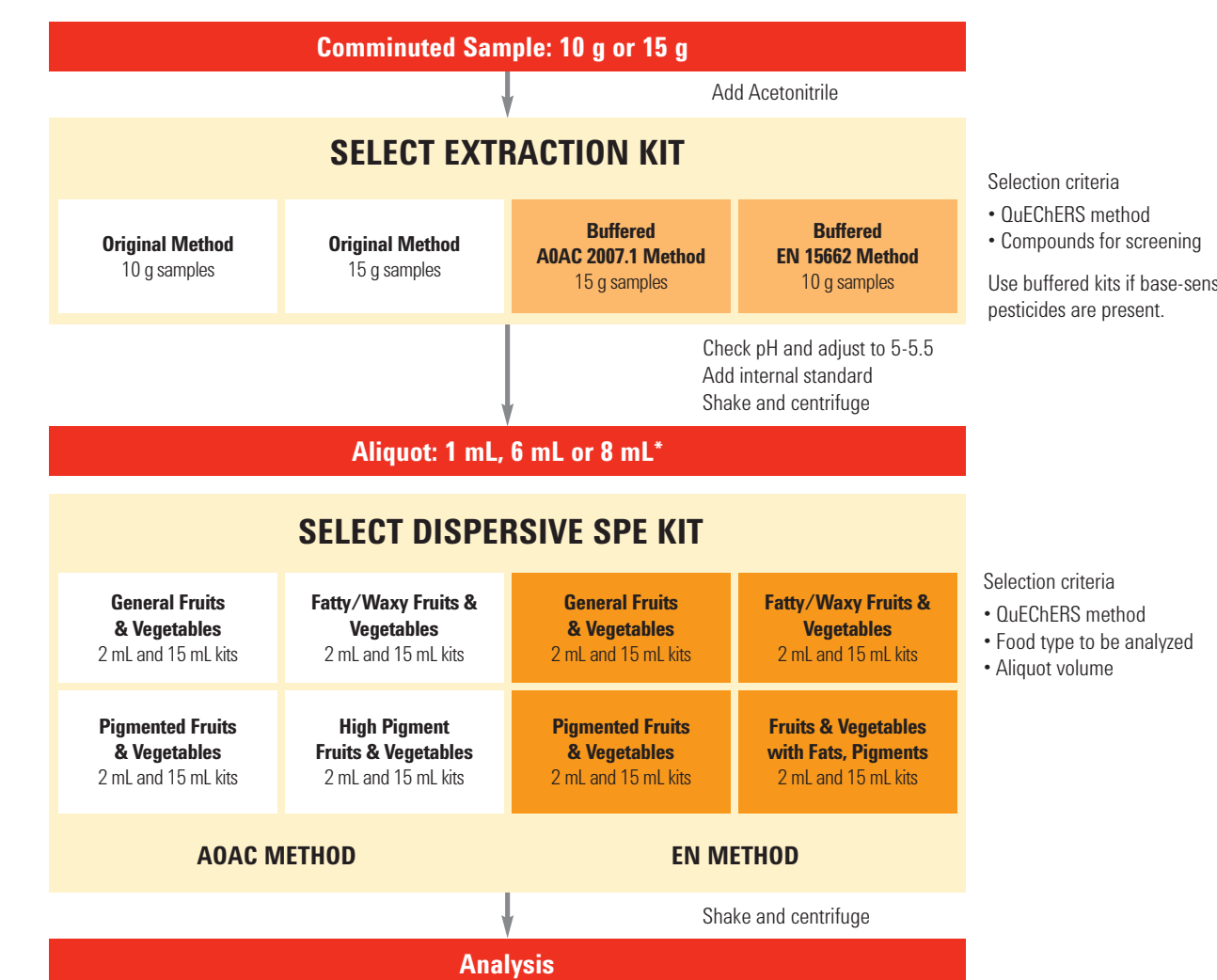
## Bond Elut QuEChERS

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 all types of fruits and vegetables.

### Agilent Recommended Standard Operating Procedure for QuEChERS

In just 3 easy steps, you can prepare any fruit or vegetable sample for multi-class, multi-residue pesticide analysis.



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

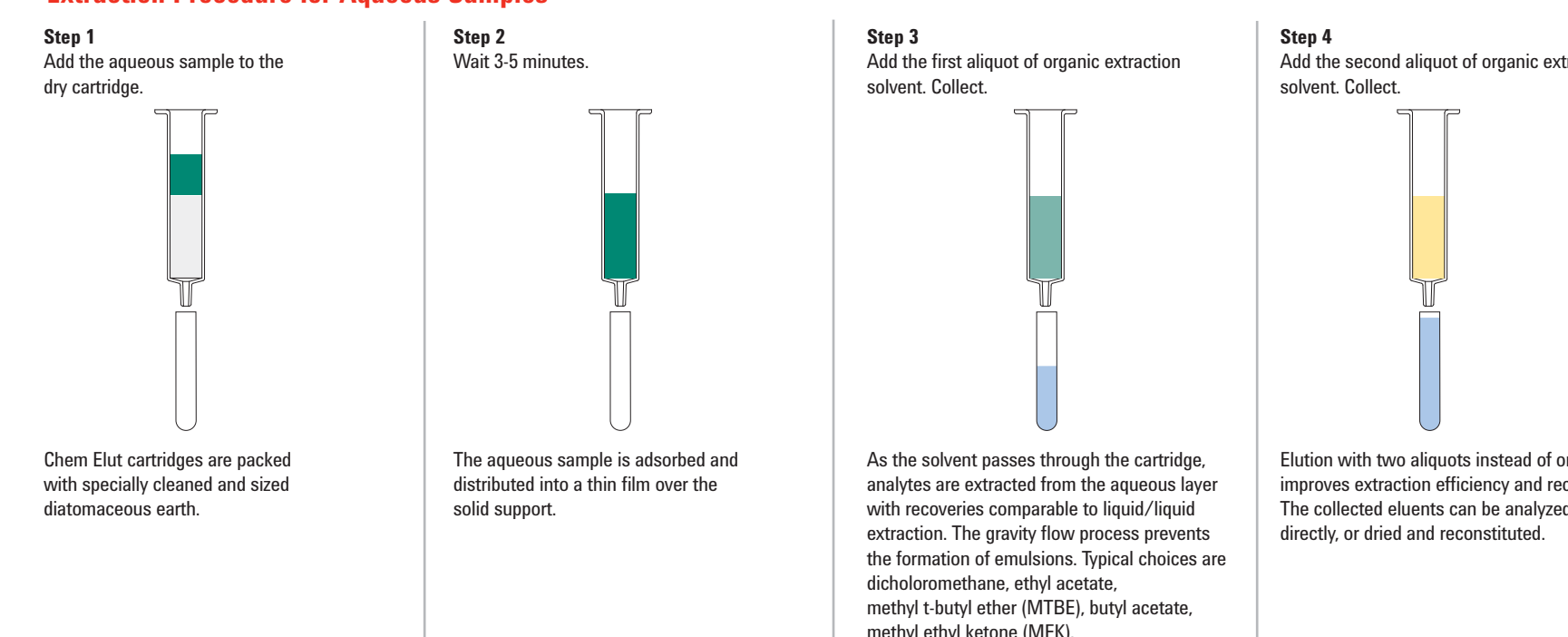
	Acids	Neutrals	Bases
Analyte	LogP > 1.0 pKa < 5	LogP > 1.5 pKa 5-10	LogP > 0.8 pKa 6-10
Sample Pre-treatment	2% NH <sub>4</sub> OH	1% HClO <sub>4</sub>	2% NH <sub>4</sub> OH
Sorbent Condition	100% MeOH	100% MeOH	100% MeOH
Equilibration	100% H <sub>2</sub> O	100% H <sub>2</sub> O	100% H <sub>2</sub> O
Load	Apply pre-treated sample		
Wash	100% H <sub>2</sub> O	5% MeOH in H <sub>2</sub> O	2% HClO <sub>4</sub> in H <sub>2</sub> O
Elution 1/Wash 2	100% MeOH Neutrals	100% MeOH Neutrals	1:1 MeOH/ACN Acids, Neutrals
Elution 2	5% HClO <sub>4</sub> in MeOH Acids		5% NH <sub>4</sub> in 1:1 MeOH/ACN Bases
Analysis	Prepare extracts for instrumental analysis		

## Chem Elut SLE

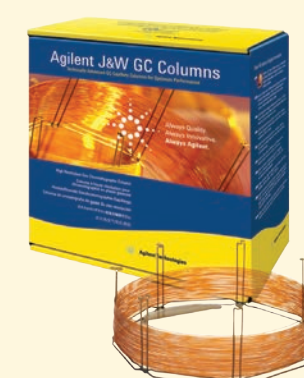
Chem Elut is a high purity sorbent-supported liquid extraction (SLE) cartridge, available in buffered and unbuffered formats.

- 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 components from a variety of matrices.

### Extraction Procedure for Aqueous Samples



Keep your lab functioning at peak efficiency with these Agilent instruments, columns, and supplies



### 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/ultraintert](http://www.agilent.com/chem/ultraintert)



### 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 to deliver superior performance and boost productivity. You get fast LC choices: Rapid Resolution High Throughput (RRHT), Eclipse Plus and Poroshell 120, stable to 800 bar, and Rapid Resolution High Definition (RRHD), stable to 1200 bar for ultra-fast separations. For more information visit [www.agilent.com/chem/lccolumns](http://www.agilent.com/chem/lccolumns)



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

Agilent GC/MS instruments provide higher sensitivity, more reliability and more uptime, with less maintenance. The clearly better Agilent GC/MS portfolio includes GC/MSD, Ion Trap GC/MS, Triple Quadrupole GC/MS, and Q-TOF GC/MS. The infinitely better Agilent LC and LC/MS portfolio includes the 1200 Infinity Series and the 6000 Series. Discover more possibilities for solving separation challenges - from nanoflow to high-throughput, and from amino acid to GPC/SEC analysis. For more information visit [www.agilent.com/chem](http://www.agilent.com/chem)

### Ensuring peak performance and productivity

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## Bond Elut SPE, QuEChERS, and Chem Elut Part Numbers



Description	Unit	Part No.
<b>Straight Barrel Cartridges</b>		
30 mg, 1 mL	100/pk	12109301
30 mg, 3 mL	50/pk	12109303
60 mg, 1 mL	100/pk	12109601
60 mg, 3 mL	50/pk	12109603
200 mg, 3 mL	50/pk	12109610
200 mg, 6 mL	30/pk	12109206
500 mg, 6 mL	30/pk	12295606

Description	Unit	Part No.
<b>Bond Elut Jr</b>		
200 mg	50/pk	12109610B
<b>Mega Bond Elut Plexa</b>		
500 mg, 12 mL	20/pk	327832
<b>Other Formats</b>		
Bond Elut Plexa Prospekt cartridge, 1 mm	96/pk	12221305
60 mg, 3 mL, Gerstel format	50/pk	1678166
200 mg, 3 mL, Gerstel format	50/pk	1678226

Description	Unit	Part No.
<b>Bond Elut Plexa PCX</b>		
<b>Straight Barrel Cartridges</b>		
30 mg, 1 mL	100/pk	12108201
60 mg, 1 mL	100/pk	12108901
30 mg, 3 mL	50/pk	12108303
60 mg, 3 mL	50/pk	12108903
200 mg, 6 mL	30/pk	12108206
500 mg, 6 mL	30/pk	12295606
<b>Other Formats</b>		
Bond Elut Plexa PCX Prospekt cartridge, 2 mm	96/pk	12221308

Description	Unit	Part No.
<b>Bond Elut Plexa PAX</b>		
<b>Straight Barrel Cartridges</b>		
30 mg, 1 mL	100/pk	12107301
60 mg, 1 mL	100/pk	12107601
30 mg, 3 mL	50/pk	12107303
60 mg, 3 mL	50/pk	12107603
200 mg, 6 mL	30/pk	12107206
500 mg, 6 mL	30/pk	12295706

Description	Unit	Part No.
<b>Bond Elut Mycotoxin</b>		
500 mg, 1 mL	50/pk	12102167
<b>Bond Elut Jr</b>		
500 mg	100/pk	12185001B

Buffered pH	Sample Size Volume (mL)	Unit	Part No.
4.5	3	100/pk	12198004
9.0	3	100/pk	12198005
unbuffered	0.3	100/pk	12198001
	1	100/pk	12198002
	3	100/pk	12198003
	5	100/pk	12198006
	10	100/pk	12198007
	20	100/pk	12198008
	50	50/pk	12198009
	100	25/pk	12198010
	300	15/pk	12198011

Hydromatrix	Part No.
(Universal)	
Hydromatrix bulk, 1 kg	198003
Hydromatrix bulk, 4 kg	198004

Method	Buffered	Contents	Part No.	Part No.	Packets Only
ADAC 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 15962	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  
\*Katerina Mastovska and Steven J. Lehotay have done work to extend the scope of QuEChERS beyond fruits and vegetables, using it to extract acrylamides in potato chips and other fried foods.  
2. "Rapid Sample Preparation Method for GC/MS/MS or GC/MS Analysis of Acrylamides in Various Food Matrices". *J. Agric. Food Chem.* 2006, 54, 7001-7008.

Description	Unit	Part No.
<b>Bond Elut QuEChERS Ceramic Homogenizers</b>		
Ceramic homogenizer for 50 mL tubes	100/pk	5982-9313
Ceramic homogenizer for 15 mL tubes	100/pk	5982-9312
Ceramic homogenizer for 2 mL tubes	200/pk	5982-9311

Kit	Size	Unit	ADAC 2007.01 Method Kit Contents Part No.	European Method EN 15962 Kit Contents Part No.
<b>General fruits and vegetables:</b> Removes polar organic acids, some sugars and lipids	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 5982-5022 5982-5022CH	25 mg PSA 150 mg MgSO <sub>4</sub> 5982-5021 5982-5021CH
<b>Fruits and vegetables with fats and waxes:</b> Removes polar organic acids, some sugars, lipon lipids and sterols	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 5982-5122CH	25 mg PSA 150 mg MgSO <sub>4</sub> 5982-5121 5982-5121CH
<b>Pigmented fruits and vegetables:</b> Removes polar organic acids, some sugars and lipids, and carotenoids and chlorophyll, not for use with plantar pesticides	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 5982-5222 5982-5222CH	25 mg PSA 150 mg MgSO <sub>4</sub> 5982-5221 5982-5221CH
<b>Highly pigmented fruits and vegetables:</b> Removes polar organic acids, some sugars and lipids, plus high levels of carotenoids and chlorophyll, not for use with plantar pesticides	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 5982-5252 5982-5252CH	25 mg PSA 150 mg MgSO <sub>4</sub> 5982-5251 5982-5251CH

Kit	Size	Unit	ADAC 2007.01 Method Kit Contents Part No.	European Method EN 15962 Kit Contents Part No.
<b>Fruits and vegetables with pigments and fats:</b> Removes polar organic acids, some sugars and lipids, plus carotenoids and chlorophyll, not for use with plantar pesticides	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 5982-5422 5982-5422CH	25 mg PSA 150 mg MgSO <sub>4</sub> 5982-5421 5982-5421CH
<b>All Food Types (Universal):</b> Removes all matrix interfering materials including polar organic acids, lipids, sugars, proteins, carotenoids and chlorophyll	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 1200 MgSO <sub>4</sub> 5982-0029 5982-0029CH	25 mg PSA 150 mg MgSO <sub>4</sub> 45 mg GCB 5982-0028 5982-0028CH

Kit	Size	Unit	ADAC 2007.01 Method Kit Contents Part No.	European Method EN 15962 Kit Contents Part No.
<b>Fruits and vegetables with pigments and fats:</b> Removes polar organic acids, some sugars and lipids, plus carotenoids and chlorophyll, not for use with plantar pesticides	2 mL	100/pk	400 mg PSA 150 mg MgSO <sub>4</sub> 5982-5452 5982-5452CH	25 mg PSA 150 mg MgSO <sub>4</sub> 5982-5451 5982-5451