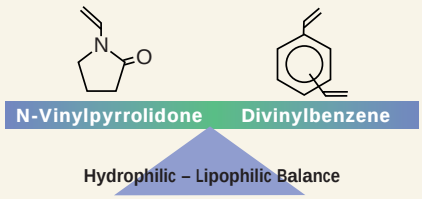
Oasis® Sample Extraction Products for Agrochemical and Environmental Analysis

Superior results, simplicity and speed — whether analyzing water samples, soil, or food extracts.

Waters Oasis® sample extraction products incorporate a water-wettable polymeric sorbent for the determination of polar and nonpolar organic compounds in aqueous samples. Oasis® HLB sorbent is **H**ydrophilic-**L**ipophilic **B**alanced to deliver superior results, whether you're analyzing water samples, soil, or food extracts. The new Oasis® MCX (**M**ixed-mode **C**ation-e**X**change) and Oasis® MAX (**M**ixed-mode **A**nion-e**X**change) enable high recoveries and consistent results whether you're determining herbicides, pesticides, endocrine disruptors, or regulated compounds (such as PAHs) and their metabolites.

Unique water-wettable HLB copolymer



Optimal Properties for Reversed-Phase SPE
Specific Surface Area: 810 m^2/g
Average Pore Diameter: 80 Å
Total Pore Volume: 1.3 cm^3/g
Average Particle Diameter: 30 µm or 60 µm

The Oasis® HLB sorbent is a patented macroporous copolymer made from a balanced ratio of two monomers, the lipophilic divinylbenzene and the hydrophilic N-vinylpyrrolidone.

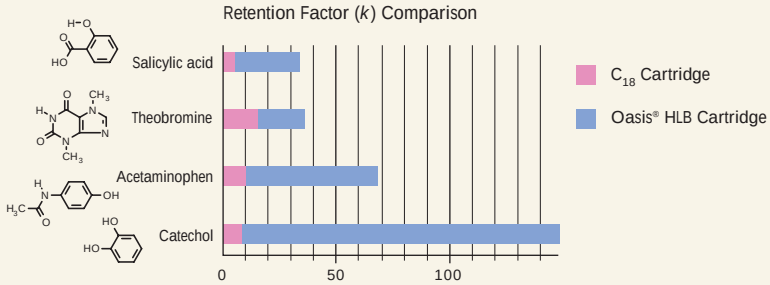
Waters OASIS® SAMPLE EXTRACTION PRODUCTS

Current Oasis® Patents:
Patent No. 5,882,521 (1996), Patent No. 5,976,376 (1998)
Patent No. 6,106,721 (1999), Patent No. 6,254,780 (2001)
Patent No. 6,322,695 (2001), Additional Patents Pending

Waters

Advantages of Oasis® SPE Products for Agrochemical and Environmental Applications

Obtain greater retention and capacity with no breakthrough



Data shown were obtained with two 3.9 mm x 150 mm columns, each packed with one of the sorbents, operated under the same conditions: mobile phase: 20 mM potassium phosphate, pH 7.0/methanol (95/5 v/v); temperature: 30 °C; flow rate: 1.0 mL/min; detection: UV @ 254 nm.

Reduce sample size, processing time and elution volume without sacrificing recovery

Sample: 400 mL drinking water, adjusted to pH 2 with phosphoric acid

Analyte: 100 ng/L phenol with 100 ng/L of 11 other phenols

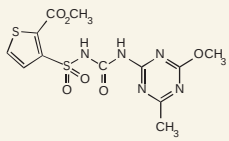
Cartridges	Recovery	Capacity before Breakthrough of phenol*
Oasis® HLB (60 mg)	91%	400 mL
type E (60 mg)	81%	350 mL
type (+) (50 mg)	70%	200 mL

The SPE method used a primary and a secondary 3 cc cartridge in series. Sample was loaded in aliquots to the primary cartridge and the secondary cartridge was checked for breakthrough. Each cartridge was washed with water and then eluted with 100% methanol. Analysis was by GC with FID detector.

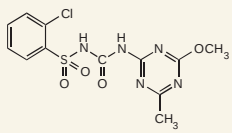
* Sample load resulting in 2% breakthrough of phenol.

Oasis® HLB vs traditional C_{18} silica Extraction of sulfonylurea herbicides

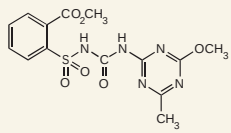
Thifensulfuron methyl



Chlorsulfuron



Metsulfuron methyl



SPE method for sulfonylurea herbicides	Oasis® HLB	C_{18} Silica
Mass sorbent per cartridge	60 mg	1000 mg
Sample volume loaded	100 mL	500 mL
SPE processing time	15 minutes	50 minutes
Elution volume	1 mL	4 mL
% Recovery	90%	85%
Limit of Quantitation (LOQ)	50 ng/L	50 ng/L
Note:	Do not worry if the bed runs dry during conditioning or loading.	Start over if the bed runs dry during conditioning or loading.

Oasis® HLB Methods for LC/GC Analysis

Generic Oasis® HLB method for LC/GC

Conditions for Oasis® HLB cartridge, 3 cc, 60 mg
Part Number WAT094226

Prepare Sample

↓

Condition/Equilibrate: 3 mL solvent*/ 3 mL methanol/ 3 mL water

↓

Load: up to 1000 mL sample

↓

Wash: 3 mL 5% methanol in water

↓

Elute: 6 mL 10% methanol/90% MTBE*

↓

For GC analysis, dry extract over Na_2SO_4, then adjust to 1 mL

For LC analysis, exchange to acetonitrile, then adjust to 1 mL

*methyl t-butyl ether

High recovery of acidic herbicides from aqueous samples*

Compound	Tap Water 2.0 µg/L 5 replicates	Tap Water 400 ng/L 5 replicates	Well Water 2.0 µg/L 5 replicates	Well Water 400 ng/L 5 replicates
picloram	90.9 (7.0)	126 (5.3)	97.5 (3.8)	106 (2.3)
dicamba	85.1 (7.2)	115 (4.4)	98.5 (3.8)	96.3 (8.3)
chloramben	86.7 (7.3)	99.2 (6.9)	95.1 (10)	90.6 (5.6)
4-nitrophenol	83.3 (6.1)	113 (6.0)	90.4 (1.7)	112 (13)
bentazon	89.3 (6.0)	114 (5.6)	91.2 (3.0)	104 (8.8)
2,4-D	92.3 (7.1)	107 (3.1)	86.5 (1.8)	122 (12)
MCPA	97.6 (8.2)	104 (4.5)	80.8 (3.6)	96.7 (5.5)
dichloprop	96.4 (11)	107 (9.0)	87.4 (3.0)	103 (6.0)
2,4,5-T	106 (6.2)	116 (8.8)	95.1 (5.0)	96.6 (12)
MCPP	100 (7.7)	116 (6.6)	93.8 (3.0)	94.7 (2.9)
3,5-dichlorobenzoic	93.3 (6.3)	119 (9.7)	84.3 (2.7)	96.9 (5.9)
2,4-DB	95.4 (5.1)	110 (8.4)	83.7 (5.6)	83.3 (5.2)
2,4,5-TP	89.3 (7.9)	92.5 (6.7)	87.7 (5.3)	82.7 (10)
acifluorfen	94.8 (8.3)	102 (8.5)	70.0 (17)	81.3 (8.2)
dinoseb	71.7 (7.1)	73.8 (6.8)	54.7 (5.2)	88.1 (1.9)

The analysis above was performed using Waters Oasis® HLB extraction cartridges (3 cc/60 mg sorbent). The elution solvent was 10% methanol in methyl t-butyl ether (10% MeOH/MTBE), the solvent specified for the EPA 515.2 analysis method. The high and consistent recovery seen in this experiment (Well Waters, 400 ng/L 5 replicates), was obtained by evaporative concentration of the eluent to 100 µL and dilution to 500 µL with water.

* % RSD

Reference: Michael S. Young, Waters Column VI (5), 1997

Optimized Oasis® HLB method for low ppt analysis for LC/MS and GC/MS

Conditions for Oasis® HLB cartridge, 6 cc, 200 mg
Part Number WAT106202

Prepare Sample

↓

Condition/Equilibrate: 3 mL MTBE/ 3 mL methanol/3 mL water

↓

Load: up to 800 mL sample

↓

Wash 1: 3 mL 40% methanol in water

40% methanol wash removes organic interference

↓

Wash 2: 3 mL water

↓

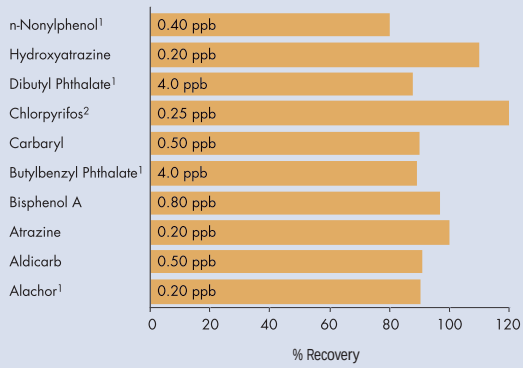
Wash 3: 3 mL 10% methanol/ 2% NH_4OH in water

pH 11 wash removes humic interference

↓

Elute: 6 mL 10% methanol/MTBE

High recovery and reproducibility of endocrine disruptor analysis



Shown are nine compounds considered to be possible endocrine disruptors and a highly polar metabolite, hydroxyatrazine. Each compound was spiked at the indicated concentration into 250 mL of drinking water, then extracted with 6 cc/200 mg Oasis® HLB cartridges using the same SPE method. All recoveries, shown as the mean of 5 replicates, ranged from 80 to 120 %. All RSDs were 11% or lower except for the phthalate esters (RSDs < 17%).

Results were obtained by LC with UV detection except where noted.

1 Similar results were obtained using GC with FID or NPD. Higher recovery possible with methylene chloride.

2 Analysis by GC/NPD

Oasis® MAX Methods for Acidic Compounds

Generic Oasis® MAX method

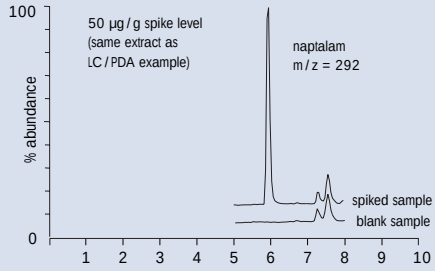
Conditions for Oasis® MAX cartridge, 6 cc, 500 mg
Part Number 186000865

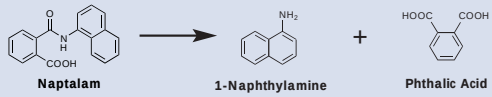
- Prepare Sample: pH 5.5 to 7.5
- Condition: 3 mL methanol / 3 mL water
- Load: up to 300 mL sample
- Wash 1: 3 mL 50 mM NaOAc (pH 7)
- Elute 1 (Wash 2): 4 mL methanol — Elute 1 contains the 1-naphthylamine metabolite residue
- Elute 2: 4 mL methanol (2% formic acid) — Elute 2 contains the naptalam residue
- Evaporate and Reconstitute

Determination of naptalam and metabolite in drinking water

LC Conditions:
Instrument: Waters Alliance® Separations Module
Column: Waters XTerra® MS C_{18}, 2.1 x 100 mm
Injection Volume: 20 µL
Mobile Phase: 25% acetonitrile / 75% 10mM ammonium acetate (pH 5.5) to 90% acetonitrile in 6 minutes
Flow Rate: 200 µL / min

MS Conditions:
Instrument: Waters / Micromass ZMD™
Interface: Positive Electrospray (ESI+)
Multiple Selected-Ion Recording (SIR)





Results* (% recovery ± % RSD, n=4)

10 ppb	50 ppb
73 (6)	76 (8)

SIR Group	Time Mins.	Compound	Mass	Cone Voltage	Dwell Time
1	5 - 8	Naptalam	144,292,293	17V	0.8 secs.

* recovery measured against standards prepared in cucumber matrix

Optimized Oasis® MAX method for clopyralid and triclopyr

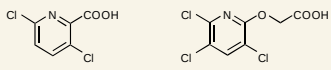
Conditions for Oasis® MAX cartridge, 6 cc, 500 mg
Part Number 186000865

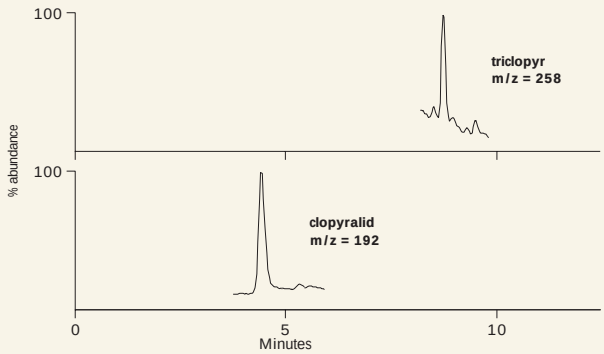
- Prepare Sample: pH 5 to 8
- Condition: 3 mL each: MTBE* / MeOH / H_2O — MTBE is employed as elution solvent to minimize humic interference from surface water. Therefore, precondition with this solvent.
- Load: 300 mL sample
- Wash 1: 3 mL 50 mM NaOAc (pH 7)
- Wash 2: 4 mL methanol
- Elute: 4 mL MeOH / MTBE / TFA (89:10:1) — Clopyralid is a stronger acid than formic acid. Therefore, formic acid cannot be utilized to elute this compound from Oasis® MAX sorbent. TFA was employed for elution of clopyralid.
- Evaporate and Reconstitute

* methyl *t*-butyl ether
diethyl ether can be used as an alternative to MTBE
TFA - trifluoroacetic acid

Determination of clopyralid and triclopyr

LC / MS Conditions:
Instrument: Waters Alliance® Separations Module with Waters / Micromass ZMD™
Interface: Positive Electrospray (ESI+)
Multiple Selected-Ion Recording (SIR)
Column: Waters XTerra® MS C_{18}, 2.1 x 100 mm
Injection Volume: 20 µL
Mobile Phase Gradient: 25% acetonitrile / 75% 10mM TFA (pH 2.1) to 90% acetonitrile in 6 minutes*
Flow Rate: 200 µL / min





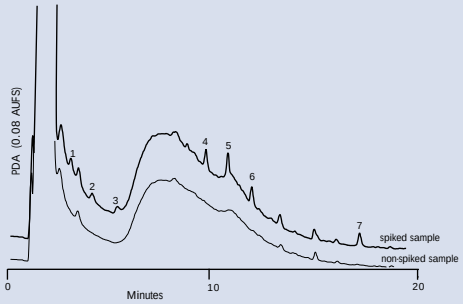
Oasis® MCX Methods for Basic Compounds

Generic Oasis® MCX method

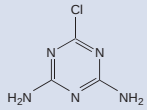
Conditions for Oasis® MCX Cartridge, 6 cc, 150 mg
Part Number 186000256

- Prepare Sample: adjust to pH 1.5 with HCl
- Condition: 2 mL methanol
  Rinse with 2 mL H_2O
- Load Sample
- Wash #1: 2 mL 0.1 N HCl
- Wash #2: 2 mL methanol
- Elute: 3 mL methanol (4% NH_4OH)
- Evaporate & Reconstitute

Determination of atrazine and metabolites



Column:	SymmetryShield™ RP_{18}, 3.9 x 150 mm
Mobile Phase:	A: phosphate buffer (20 mM, pH 6.8) B: acetonitrile
Gradient:	95% A for 2 min then linear to 25% A in 20 min
Flow Rate:	0.8 mL/min
Detection:	PDA (215nm)
Injection:	80 µL



Desethyldesisopropylatrazine

Drinking water samples (100 mL) were spiked with the herbicides and adjusted to pH 1.5. The samples were then analyzed using Oasis® MCX 6 cc, 150 mg cartridges using the protocol for basic compounds

	Compounds	(% Recovery - % RSD, n=5) 0.2 µg/L	1.0 µg/L
1.	Hydroxydesisopropylatrazine	94 (3)	85 (3)
2.	Desethyldesisopropylatrazine	75 (8)	76 (5)
3.	Hydroxydesethylatrazine	89 (6)	76 (7)
4.	Desisopropylatrazine	79 (4)	83 (2)
5.	Hydroxyatrazine	107 (7)	101 (2)
6.	Desethylatrazine	79 (5)	83 (3)
7.	Atrazine	89 (5)	77 (3)

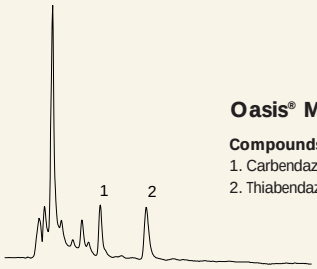
Optimized Oasis® MCX method for fungicides

Conditions for Oasis® MCX Cartridge, 6 cc, 150 mg
Part Number 186000256

- Prepare Sample: adjust to pH 10 with NaOH
- Condition: 2 mL methanol
  Rinse with 3 mL 2% NH_4OH
- Load Sample
- Wash 1: 2 mL 2% NH_4OH
- Wash 2: 2 mL 30% methanol/4% NH_4OH
- Wash 3: 2 mL 0.1 N HCl (Make Analytes Cationic)
- Wash 4: 2 mL methanol
- Elute: 3 mL methanol (4% NH_4OH)
- Evaporate and Reconstitute

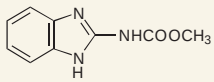
Determination of carbendazim and thiabendazole

Column:	XTerra® RP_{18}, 4.6 x 100 mm (3.5 µm DP)
Mobile Phase:	72.5% phosphate buffer, (20 mM pH 6.8) 27.5% acetonitrile
Flow Rate:	1.0 mL/min
Detection:	PDA (288nm)
Injection:	20 µL

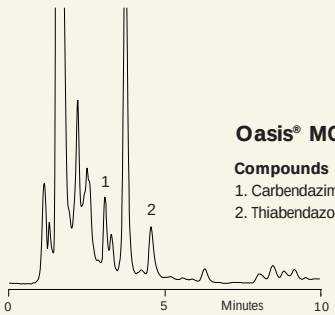


Oasis® MCX Optimized Method

Compounds	% Recovery, n=4
1. Carbendazim	82% (2%RSD)
2. Thiabendazole	96% (2% RSD)

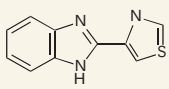


Carbendazim



Oasis® MCX Generic Method

Compounds	% Recovery, n=4
1. Carbendazim	81% (8%RSD)
2. Thiabendazole	94% (10% RSD)



Thiabendazole

Oasis® SPE Methods for Natural Products

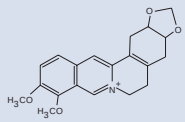
Oasis® HLB method for alkaloids in Goldenseal

Conditions for Oasis® HLB cartridge, 3 cc, 60 mg
Part Number WAT094226

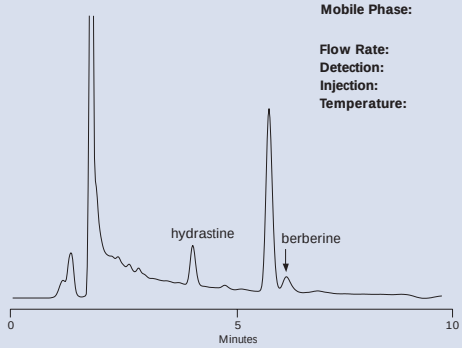
- Condition / Equilibrate: 1 mL methanol / 1 mL 150 mM K_2HPO_4
- Load: 3 mL of diluted sample
- Wash: 1 mL 30% methanol / 150 mM K_2HPO_4
- Elute: 3 mL methanol

Determination of alkaloids in Goldenseal, commercial products or whole leaf

Column:	Symmetry® C_{18}, 4.6 x 150 mm, 3.5 µm
Mobile Phase:	A: 100 mM KH_2PO_4, 73% B: Acetonitrile, 27%
Flow Rate:	1.2 mL/min
Detection:	UV @ 230nm
Injection:	10 µL
Temperature:	30° C



Berberine*



- Recovery, measured with certified standards at 100 ppm in reagent water, was > 80 % for hydrastine and berberine
- All analyses gave results within ± 20 % of the expected values with the exception of the liquid supplement

Sample pre-preparation
100 mg sample is extracted with 25 mL of ethanol:water (70:30)
0.3 mL of the ethanolic extract is diluted 1:10 with 150 mM K_2HPO_4*

*Berberine, a quaternary amine, requires a high salt concentration to enhance reversed-phase retention

Goldenseal extract before SPE cleanup

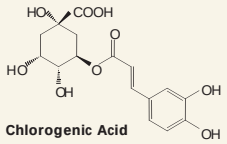
Goldenseal extract after SPE cleanup using Oasis® HLB

Oasis® MAX method for phenolics in echinacea

Conditions for Oasis® MAX cartridge, 3 cc, 60 mg
Part Number 186000367

- Condition / Equilibrate: 1 mL methanol / 1 mL water
- Load: 3 mL of diluted sample
- Wash 1: 2 mL of 50 mM sodium acetate
- Wash 2: 2 mL of methanol
- Dry Cartridge: air dry for 15 min with vacuum
- Elute: 3 mL MTBE / methanol / TFA (49:49:2)
- Evaporate and Reconstitute: 1 mL mobile phase

Determination of phenolics in Echinacea, commercial products or whole leaf



Chlorogenic Acid

Column:	Symmetry® C_{18}, 4.6 x 250 mm, 5 µm
Mobile Phase:	A: 0.1% phosphoric acid B: Acetonitrile
Gradient:	10% B initial, then linear gradient to 22% B in 13 min, to 40 % B in 40 min
Flow Rate:	1.5 mL/min
Detection:	UV @ 330 nm
Injection:	10 µL
Temperature:	35° C

- Recovery, measured with certified standards at 100 ppm in reagent water, was greater than 85 % for all compounds
- All analyses gave results within ± 35 % of the expected values with the exception of the liquid supplement (see goldenseal analysis)
- The selective SPE extraction and cleanup procedure provided a convenient analysis of echinacea phenolics in complex matrix such as in herbal tea

Sample pre-preparation
100 mg sample is extracted with 25 mL of ethanol:water (70:30)
1 mL of the ethanolic extract is diluted 1:3 with water (~ pH 7)

Herbal tea extract before SPE cleanup

Herbal tea extract after SPE cleanup using Oasis® MAX

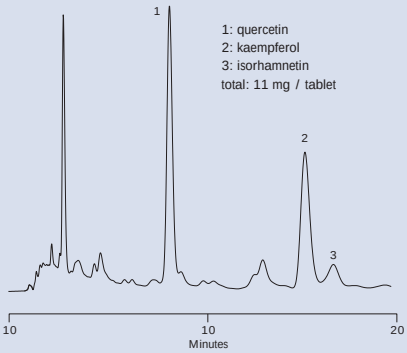
Oasis® SPE Methods for Natural Products

Oasis® HLB method for flavonoids in ginkgo

Conditions for Oasis® HLB cartridge, 3 cc, 60 mg
Part Number WAT094226

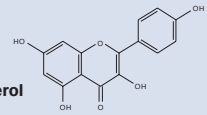
- Condition / Equilibrate: 1 mL methanol / 1 mL water
- Load: 3 mL of diluted sample
- Wash 1: 2 mL 5% methanol / water
- Wash 2: 2 ml methanol / 50mM NH_4OAc (30:70) pH 5
- Dry Cartridge: air dry for 15 min with vacuum
- Elute: 3 ml MTBE / methanol (80:20)
- Evaporate and Reconstitute: 1 mL mobile phase

Determination of flavonoids in Ginkgo, commercial products or whole leaf



1: quercetin
2: kaempferol
3: isorhamnetin
total: 11 mg / tablet

Column:	Symmetry® C_{18}, 4.6 x 250 mm, 5 µm
Mobile Phase:	A: 0.5% Phosphoric acid, 50% B: Methanol, 50%
Flow Rate:	1.5 mL/min
Detection:	UV @ 270nm 0.02 AUFS
Injection:	10 µL
Temperature:	25° C



Kaempferol

Sample pre-preparation
1 g sample is refluxed in 50 mL of ethanol:
3M HCl (70:30) for 2.5 hr
The cooled sample is adjusted to exactly 100 mL
0.3 mL of the ethanolic extract is diluted 1:10 with water

- Recovery, measured with certified standards at 100 ppm in reagent water, was 82 % for quercitin and > 90 % for the other compounds
- All analyses gave results within ± 40 % of the expected values with the exception of the capsule (+ 60%)
- The selective SPE extraction and cleanup procedure provided a convenient analysis of ginkgo flavonoids in a complex matrix (Herbal One with 16 herbal ingredients)

Ginkgo extract before SPE cleanup

Ginkgo extract after SPE cleanup using Oasis® HLB

Oasis® HLB method for fractionation of natural product crude extracts

Conditions for Oasis® HLB Cartridge, 6 cc, 200 mg
Part Number WAT106202

- Prepare Sample — Dilute sample to 95% aqueous **Low—adjust pH—High**
- Condition: 3 mL methanol / 3 mL water
- Load: up to 500 mL of sample
- Wash 1: 3 mL 5% methanol in water — Remove weakly retained polar compounds **Low—adjust pH—High**
- Wash 2: 3 mL methanol — Remove moderately-polar and weakly retained non-polar compounds **Low—adjust pH—High**
- Wash 3: 3 mL 70 / 30 acetonitrile / MeOH — Remove moderately retained non-polar compounds **Low—adjust pH—High**
- Wash 4: 3 mL 90 / 10 MeOH / DCM — Remove strongly retained non-polar compounds **Low—adjust pH—High**

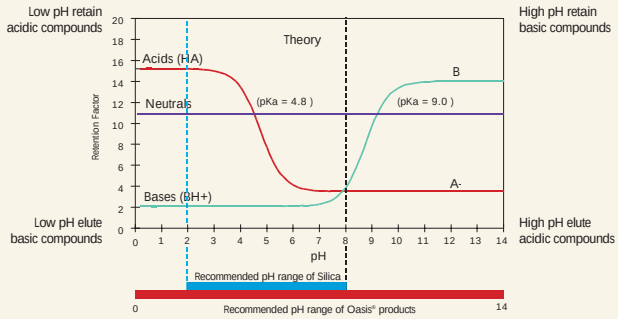
Selecting elution solvents by relative strength for the generic SPE method

Solvent*	Solvent Type	Relative Elution Strength**	Comments
Methanol	proton donor	1.0	disrupts H-bonding
Acetonitrile	dipole-dipole	3.1	medium polarity drugs
Tetrahydrofuran	dipole-dipole	3.7	medium polarity drugs
Acetone	dipole-dipole	8.8	medium polarity drugs
Ethyl Acetate	dipole-dipole	high	nonpolar drugs and GC compatible
Methylene Chloride	dipole-dipole	high	nonpolar drugs and GC compatible

* *When using solvents other than methanol, add 10-30% methanol to disrupt H-bonding with the Oasis® HLB sorbent.*

** *High-Purity Solvent Guide. Burdick & Jackson Laboratories, Inc. Solvent Properties of Common Liquids, L.R. Snyder, J. Chromatogr.*, 92, *223 (1974); J. Chromatogr. Sci.* 16, *223 (1978)*

Selecting pH for optimal retention and elution



Ordering Information

Description	Particle Size (µm)	Qty	Part Number
Oasis® HLB Extraction Cartridges			
1 cc/30 mg	30 µm	100	WAT094225
1 cc/30 mg with Gilson ASPC™ Adapter	30 µm	500	WAT058882
3 cc/60 mg	30 µm	100	WAT094226
6 cc/200 mg	30 µm	30	WAT106202
6 cc/500 mg LP*	60 µm	30	186000115
12cc/500 mg LP	60 µm	20	186000116
20 cc/1 gram LP	60 µm	20	186000117
35 cc/6 gram LP	60 µm	10	186000118
Plus/225 mg LP	60 µm	50	186000132
Vac RC/60 mg	30 µm	50	186000381
Vac RC/30 mg	30 µm	50	186000382
New! Glass 5 cc/200 mg	60 µm	30	186000683
A cleanliness test is performed on several cartridges from each lot to check for traces (ng levels) of two common phenols and six phthalates.			
Oasis® HLB Extraction Column			
New! 3.9 mm x 20 mm	25 µm	1	186002042
New! 2.1 mm x 20 mm	25 µm	1	186000706
Holder Kit for 2.1 mm x 20 mm Cartridge Column		1	186000262
Oasis® HLB Prospekt™ Cartridges**			
Prospekt™ 2 mm x 10 mm	30 µm	100	186000258
Prospekt™ 2, 2mm x 10 mm	30 µm	96	186001196

Description	Particle Size (µm)	Qty	Part Number
Oasis® MCX Extraction Cartridges			
1 cc/30 mg	30 µm	100	186000252
3 cc/60 mg	30 µm	100	186000254
3 cc/60 mg LP	60 µm	100	186000253
6 cc/150 mg	30 µm	30	186000256
6 cc/150 mg LP	60 µm	30	186000255
Vac RC/60 mg	30 µm	50	186000261
Vac Rc/60 mg LP	60 µm	50	186000380
Oasis® MAX Extraction Cartridges			
1 cc/30 mg	30 µm	100	186000366
3 cc/60 mg	30 µm	100	186000367
3 cc/60 mg LP	60 µm	100	186000368
6 cc/150 mg	30 µm	30	186000369
6 cc/150 mg LP	60 µm	30	186000370
Vac RC/30 mg	30 µm	50	186000372
Vac RC/60 mg	30 µm	50	186000371
Vac Rc/60 mg LP	60 µm	50	186000378

*LP=Large Particle **For use with the Spark Holland Prospekt™ system

Choice of Cartridge Based on Sample Size

Sample size	Cartridge size
1-10 mL	1 cc, 30 mg or 3 cc, 60 mg
10-100 mL	3 cc, 60 mg or 6 cc, 200 mg
100-500 mL	6 cc, 200 mg or 6 cc, 500 mg (LP)
500-1000 mL	6 cc, 500 mg (LP) or 12 cc, 1 g (LP)

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