



Thermo Scientific

SolEx Cartridges

Product Manual

P/N: 088175-02

November 2013

Product Manual

for

Dionex SolEx Cartridges and Columns

SolEx HRPHS, 48 Pack, 60 mg/3 mL, P/N 088124, 150 mg/3 mL, P/N 088125
SolEx HRPHS, 48Pack, 100 mg/ 6 mL, P/N088126, 200 mg/6 mL, P/N088127
SolEx HRPHS, 36 Pack, 500 mg/6 mL, P/N 088128
SolEx SAX, 48 Pack, 60 mg/3 mL, P/N 088105, 150 mg/3 mL, P/N 088106
SolEx SAX, 48 Pack, 100 mg/ 6 mL, P/N088107, 200 mg/6 mL, P/N088108
SolEx SAX, 36 Pack, 500 mg/6 mL, P/N 088109
SolEx SCX, 48 Pack, 60 mg/3 mL, P/N 088189, 150 mg/3 mL, P/N 088099
SolEx SCX, 48 Pack, 100 mg/ 6 mL, P/N088101, 200 mg/6 mL, P/N088102
SolEx SCX, 36 Pack, 500 mg/6 mL, P/N 088103
SolEx WAX, 48 Pack, 60 mg/3 mL, P/N 088111, 150 mg/3 mL, P/N 088112
SolEx WAX, 48 Pack, 100 mg/ 6 mL, P/N088113, 200 mg/6 mL, P/N088114
SolEx WAX, 35 Pack, 500 mg/6 mL, P/N 088115
SolEx WCX, 48 Pack, 60 mg/3 mL, P/N 088117, 150 mg/3 mL, P/N 088118
SolEx WCX, 48 Pack, 100 mg/ 6 mL, P/N088119, 200 mg/6 mL, P/N088121
SolEx WCX, 36 Pack, 500 mg/6 mL, P/N 088122

SolEx C8, 100 Pack, 100 mg/1 mL, P/N 074415, 50 Pack, 500 mg/3 mL, P/N 074413, 30 Pack, 1 g/6 mL, P/N 074411
SolEx C18, 100 Pack, 100 mg/ 1 mL, P/N 074623, 50 Pack, 500 mg/3 mL, P/N074412, 50 Pack, 500 mg/6 mL, P/N
074417, 30 Pack, 1 g/ 6 mL, P/N 074410
SolEx C18 (unendcapped), 30 Pack, 1 g/6 mL, P/N 074416
SolEx C18-Clean, 30 Pack, 1 g/6 mL, P/N 075895
SolEx C8-Clean, 50 Pack, 500 mg/ 6 mL, P/N 075897
SolEx C18-525, 30 Pack, 1.5 g/6 mL, P/N 075896
SolEx Silica, 50 Pack, 500 mg/6 mL, 074589
SolEx Carbon, 20 Pack, 2 g/6 mL, P/N 074590
SolEx GCB, 30 Pack, 500 mg/ 6 mL, P/N 075898

SolEx HRP, 2.1 x 20 mm, P/N 074400
SolEx HRP Cartridge Holder, P/N 074403
SolEx HRP RSLC, 2.1 x 20 mm, P/N 075590

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Revision History:

Revision 01, September, 2013, Original Publication.

Revision 02, November, 2013, Minor changes to Table 1 on Page 9.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:



SAFETY

Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.



WARNING

Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.



CAUTION

Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.



NOTE

Indicates information of general interest.

IMPORTANT

Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.

Tip

Highlights helpful information that can make a task easier.

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1. Introduction

The Dionex™ SolEx™ line of sample pretreatment products includes silica-based and polymeric-based stationary phase chemistries in barrel, disposable cartridge and RSLC formats. The barrel-format cartridges are designed for compatibility with the Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction (SPE) instrument for large sample volume applications. Large sample volumes are necessary when the required detection limits of the targets are below that achieved by direct injection or the preconcentration of less than about 20 mLs of sample. The barrel cartridges have luer-slip outlets that are compatible with the 3-mL and 6-mL volume pistons of the 280 instrument. The SolEx HRP is available in disposable cartridge and stainless steel RSLC formats for on-line applications.

The SolEx product line includes polymeric stationary phases built on a new, high capacity base particle, with a narrow size distribution yielding high efficiency with low backpressure. Four of the stationary phase chemistries are mixed mode ion exchange/reversed phase and the SolEx HRPHS phase is hydrophilic reversed phase resin. It also has selectivity for phenolic and azo functionalities. In addition to its general hydrophilic reversed applications, the HRPHS is useful for trapping humic acids. The online SolEx HRP resin is lower capacity than SolEx HRPHS, designed for compatibility with online elution into HPLC analytical columns.

The Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction (SPE) instrument is a high-throughput workstation dedicated specifically for automating SPE. The Dionex AutoTrace 280 instrument automates the cartridge or disk conditioning, sample loading, rinsing, and eluting steps for large volume aqueous solution extractions (20 mL to 20 L). The unit can process up to six samples in parallel, automatically conditioning, washing and eluting SPE cartridges or disks with a choice of up to five reagents. The unit also segregates aqueous and solvent waste using separate waste lines for efficient, cost effective waste handling. The Dionex AutoTrace 280 instrument is designed to automate the cartridge or disk requirements for U.S. EPA water analysis methods.

2. How to Choose an SPE Phase

SPE chemistries can be used in several types of methodologies. The phase can be selected so that the target(s) species are retained while the matrix passes thru to waste. Both the targets and the matrix component(s) can be retained by different mechanisms and the matrix can be selectively washed away, leaving later elution of the targets. Therefore it is important to identify the chemical properties of the targets and the matrix components so that the phase with the most appropriate properties can be chosen.

The order of the topics below reflects the decision tree with most general to most specific parameters considered in order.

2.1 Polymer vs. Silica

The decision to choose polymeric or silica-based stationary phase products for SPE is generally guided by pH requirements and relative retention of targets and matrix on the phase.

Bonded silica products have a safe operating range generally pH 3-8. The bonded phases are generally non-aromatic which means that highly aromatic targets can be more easily eluted with organic solvents. Highly polar targets are generally not well-retained and selectivity among species containing aromatic rings can be limited. The phases are generally not mixed-mode.

Polymeric stationary phases have a useful pH range of 0-14. The substrate particles in the polymeric SolEx products are based on divinylbenzene and therefore afford retention via adsorption and pi-pi interactions in addition to the surface graft functionality, as reflected in the product name. For example the SolEx SCX product has both reversed phase and strong cation exchange functionality, as discussed in Section 4.

2.2 Ion Exchange Functionality

There are four ion exchange functionalities available in the polymer group of SolEx sorbents, SAX, SCX, WAX and WCX. The strong anion exchange phase (SAX) bears quaternary ammonium functionality which means that the phase holds a positive charge (exchanging anions) regardless of pH. By contrast the weak anion exchange (WAX) phase bears amine functionality which means that it only holds a positive charge (capable of exchanging anions) at pH below about 7. The strong cation exchange (SCX) phase bears sulfonate (SO_3^-) sites which means it can exchange cations down to a pH <1. By contrast the weak cation exchange (WCX) phase bears carboxylate sites which have $\text{pK}_a \sim 4$. This means that the WCX phase is neutral and will not hold cations at pH <3. Of course a weak ion exchange site is 95% charged about 2 pH units from the pK_a , so there is some capacity in the proximity of the pK_a .

The ion exchange phases are characterized by their ion exchange capacities, which allow the calculation of how much sample can be held on the phase. For example, if a phase contains 1000 $\mu\text{Eq/g}$ of capacity and the cartridge contains 100 mg of resin, then the cartridge contains 100 μEq of capacity. If a sample contains 50 $\mu\text{g/mL}$ of sodium, for example, the question might be, how many milliliters of sample load on the cartridge before the resin bed is consumed. 50 $\mu\text{g/mL}$ sodium is about 2.2 $\mu\text{Eq/mL}$, so the 100 μEq cartridge capacity (See Table 2)/ 2.2 $\mu\text{Eq/mL}$ sample matrix concentration = 45 mL sample can be processed.

2.3 Reversed Phase Retention

The mixed mode ion exchange, HRPHS and bonded silica phases contain reversed phase capacity. Therefore elution of species containing hydrophobic moieties can be accomplished with organic solvent. These solvents include methanol, acetonitrile, methylene chloride or others as prescribed by existing methods. It is also convenient to elute targets using solvent if the method requires that the solvent be evaporated.

2.4 Barrel Size

Larger barrel sizes have larger diameters and, in general, higher bed weights than smaller barrels. The larger diameters are more rugged for samples containing particulates; colloids etc that can clog the inlet frits. If samples are fairly clean the smaller barrel sizes usually offer a cost advantage. As a rule of thumb for low particulate samples, a 1 mL barrel can handle about 50 mL, a 3 mL barrel can treat about 200 mL and a 6 mL barrels can treat about 500-1000 mL sample. See section 3.4.1 for an example.

2.5 Bed weight

Bed weight is usually chosen based on knowledge of sample composition. If a sample can be treated and an acceptable recovery of a target can be achieved then it is usually a cost savings to attempt a lower bed weight. A lower bed weight will also require less volume in the elution step. Sometimes the matrix will use more capacity, and sometimes the targets will use the majority of the capacity, so an understanding of the sample composition is very helpful. Example applications can also provide guidance. See section 3.4.1 for an example.

2.6 Target Analytes

Ionizable and permanently charged target analytes or matrix species can be retained and eluted from the ion exchange phases using fairly standard protocols in most cases. Permanently charged species including quaternary ammonium and sulfonates are retained on WCX and WAX respectively at a pH where the stationary phase is charged. The matrix species can be washed away and then these strong ion targets can be eluted using solvent at a pH that neutralizes the phase. Ionizable species including amines and carboxylic acids can be retained on SCX and SAX respectively at a pH where the analytes are charged. The matrix can be washed away at the same pH and a buffer in solvent is applied that neutralizes the analytes, thus eliminating their retention by ion exchange. Examples of these protocols can be found in the Applications section.

Neutral targets are best retained by reversed phase interactions, or in the case of Silica, normal phase. Neutral targets can be retained on the WCX and WAX phases at a buffer pH where the phase is neutral, pH <3 for WCX and pH >7 for WAX.

3. Product Usage

3.1 SolEx Product Line and Associated Chemistries

Table 1 Recommended Applications

Cartridge Type	Format	Functional Group	Example Applications
HRPHS	3-, 6- mL barrels	Divinylbenzene (DVB)/ polyvinylpyrrolidone (PVP, high)	General reversed phase, polyphenolics, azo compounds
SAX	3-, 6- mL barrels	DVB-quaternary ammonium	Ionizable (weak) anions, carboxylic acids
SCX	3-, 6- mL barrels	DVB-sulfonate	Ionizable (weak) cations, amines
WAX	3-, 6- mL barrels	DVB-amine	Permanent anions, inorganic and organic, sulfonates, sulfates
WCX	3-, 6- mL barrels	DVB-carboxylate	Permanent cations, inorganic and organic, quaternary ammonium
HRP	Online cartridge and RSLC column	DVB-PVP (low)	General neutral organics
C8, C18	1-, 3-, 6- mL barrels	Bonded silica	Pesticides, herbicides, hydrocarbons
Phthalate-free C8, C18	6-mL barrel	Bonded silica	Trace analysis
Silica	6-mL barrel	Acid-washed unbonded silica	Polar compounds
Activated Carbon	6-mL barrel	charcoal	Nitrosamines, 1,4-dioxane
Graphitized Carbon	6-mL barrel	Graphitized carbon particles	Herbicides

Table 2 Ion Exchange Capacities of the Ion Exchange Cartridges

Cartridge Type	Bed Weight/Barrel Volume (mg/mL)	Ion Exchange Capacity (μ Eq/cartridge)
SAX	60/3	54
SAX	150/3	135
SAX	100/6	90
SAX	200/6	180
SAX	500/6	450
SCX	60/3	42
SCX	150/3	105
SCX	100/6	70
SCX	200/6	140
SCX	500/6	350
WAX	60/3	72
WAX	150/3	180
WAX	100/6	120
WAX	200/6	240
WAX	500/6	600
WCX	60/3	72
WCX	150/3	180
WCX	100/6	120
WCX	200/6	240
WCX	500/6	600

3.1.1 HRPHS

The HRPHS stationary phase is a neutral resin comprised of a 22 µm, high surface area divinylbenzene base particle grafted with polyvinylpyrrolidone polymer. This material has properties of a hydrophilic reversed phase material and also has specific high capacity for polyphenolics including humic acids, and azo-containing substances such as azo-dyes. The hydrophilic reversed phase properties allow high recovery of a hydrophobic target, triclosan, as shown in the application below.

The barrel cartridges are compatible with the AutoTrace 280 high volume solid phase extraction instrument. The instrument can be programmed with method steps that exactly mimic a typical offline SPE protocol.

3.1.1.1 Method Determination of Bactericide from Tap Water

In this application, triclosan is extracted from a sample containing a hand-washing soap in tap water. The elution solvent is methanol.

Sample Prep: Autotrace 280, 100mL 1/3000 Softsoap in Sunnyvale tap water

Barrel: SolEx HRPHS, 200mg /6mL barrel

Flow rate: 10mL/min

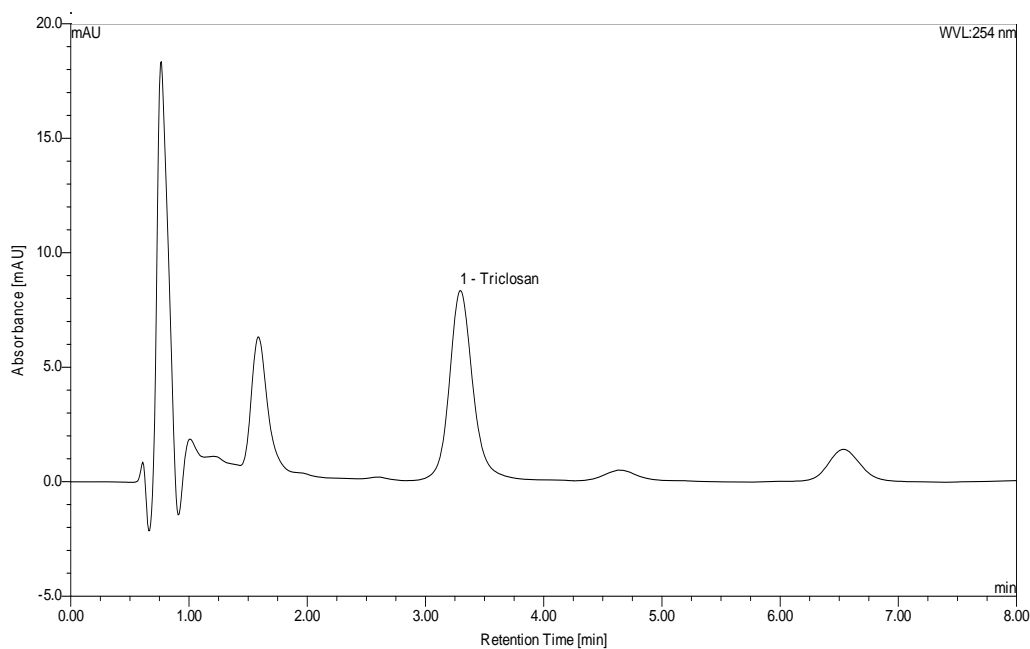
Elution solvent: 5 mL Methanol

AutoTrace 280 Method:

Step No.	Step Description	Step Function
1	Condition cartridge with 5.0mL of MeOH into solvent waste	Condition
2	Condition cartridge with 10.0mL of water into aqueous waste	Condition
3	Dry cartridge with gas for 5.0 minutes	Dry
4	Load 100.0mL of sample onto cartridge at 10mL/min	Load
5	Rinse cartridge with 5.0mL of water into aqueous waste	Wash
6	Collect 5.0mL fraction into sample tube using methanol	Elute

Column: Acclaim 120, 2X50mm
 Flow rate: 0.21mL/min
 Eluent: 65/35 v/v acetonitrile/water
 Detection: UV 254nm
 Peak: 1. Triclosan
 Result: Recovery, 96%

Figure 1 Determination of Triclosan in Sunnyvale Tap Water After Preconcentration using SolEx HRPHS in AutoTrace 280



3.1.1.2 Removal of Polyphenolic Species

The HRPHS stationary phase has high selectivity for polyphenolic compounds including tannic and humic acids. The retention mechanism is via H-bonding and charge transfer so these compounds can be retained even during elution of other organic species using organic solvent. Polyphenolic compounds are best retained under slightly acidic conditions, where the phenol groups are protonated.

Step No.	Step Description	Step Function
1	Condition cartridge with 5.0mL of MeOH into solvent waste	Condition
2	Condition cartridge with 10.0mL of water into aqueous waste	Condition
3	Dry cartridge with gas for 5.0 minutes	Dry
4	Load 100.0mL of sample onto cartridge under slightly acidic conditions at 10mL/min	Load
5	Rinse cartridge with 5.0mL of water into aqueous waste	Wash
6	Collect 5.0mL fraction into sample tube using methanol at a pH where the targets are ionized. Humic acids will be retained.	Elute

3.1.2 WAX

The WAX stationary phase is comprised of a 22 µm medium surface area divinylbenzene resin particle grafted with an amine functionality. The ion exchange capacity is about 1200 µEq/g. This phase is mixed mode reversed phase and anion exchange. Strong acids are retained at low pH where the amine sites on the resin are charged and therefore capable of ion exchange, and eluted raising the pH and therefore neutralizing the stationary phase. This is the common methodology when the target species are permanently (strongly) charged anions such as sulfonates.

3.1.2.1 Method Determination of LAS in ASTM Wastewater

In this application, the common surfactant linear alkylbenzene sulfonate (LAS), a mixture of sulfonates, is extracted from ASTM waste water matrix. The WAX phase is charged at low pH and neutral at high pH. The strongly acidic sulfonates are retained at low pH, matrix components are washed away, and the LAS is eluted by neutralizing the ion exchange sites on the stationary phase. Table 3 shows comparison of recoveries using several bed weights and barrel cartridge volumes. One advantage of using larger volume barrel cartridge is the improved ruggedness for “dirty” matrices afforded by the larger diameter and therefore larger surface area. Also shown in this data is the improved recovery at a minimum of 100 mg bed weight.

Sample Prep: AutoTrace 280, 20mL 0.005% LAS in wastewater

The wastewater was prepared according to ASTM Designation D5905-98 (Standard practice for the preparation of substitute wastewater).

Procedure:

1. Place 0.400g flour, 2.000g ocean salts, 0.080g Kaolin, 20.0mL Triton X-100 solution (1.200g Triton X-100 to water and dilute to 1L), and 120mL Beer (shake vigorously, loose cap. Allow to sit refrigerated for 24 h to dissipate carbonate and then allow to reach room temperature);
2. Blend on lowest setting for 30s. Allow most of the foam to subside before transferring. Transfer quantitatively to 2-L volumetric flask and dilute to 2L with water

Barrel: SolEx WAX, see Table 3
 Flow rate: 5 mL/min except as noted
 Elution solvent: 5 mL 5% NH₄OH in methanol

AutoTrace 280 Method:

Step No.	Step Description	Step Function
1	Condition cartridge with 5.0mL of MeOH into aqueous waste	Condition
2	Condition cartridge with 10.0mL of water into solvent waste	Condition
3	Dry cartridge with gas for 5.0 minutes	Dry
4	Condition cartridge with 5.0 mL of 1% v/v formic acid in water into aqueous waste	Equilibrate
5	Load 20.0mL of sample onto cartridge at 5mL/min	Load
6	Rinse cartridge with 5.0mL of 1% v/v formic acid in water into aqueous waste	Wash 1
7	Rinse cartridge with 5.0mL of 1% v/v formic acid in methanol into solvent waste	Wash 2
8	Collect 5.0mL fraction into sample tube using 5% NH ₄ OH in methanol	Elute

3 – Product Usage

Analytical Column: Acclaim Surfactant Plus, 3 μ m, 3 x 100 mm

Gradient:

Time (min)	MeCN	0.1M NH ₄ OAc, pH 5
-6	25	20
0	25	20
10	80	20

Temperature: 30°C

Flow rate: 0.6mL/min

Inj. volume: 5 μ L

Detection: UV at 225nm

Peaks: Linear alkylbenzene sulfonate (LAS), chain lengths as noted

Figure 2 Determination of LAS by Chain length after Preconcentration from Waste Water Using SolEx WAX in AutoTrace 280

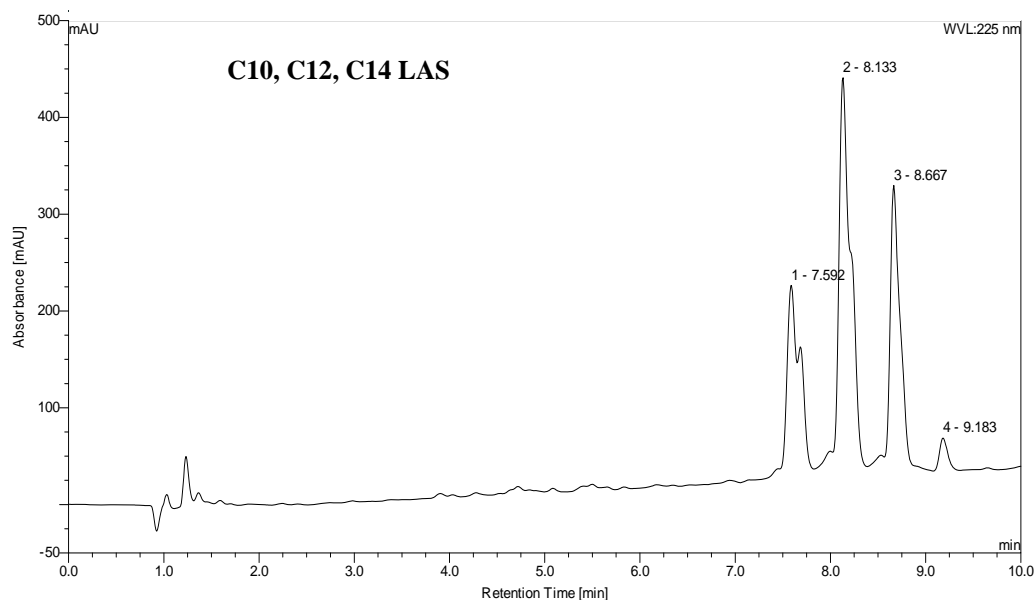


Table 3 Recovery of LAS as a Function of Bed Size

Sample	60 mg/3 mL	150 mg/3 mL	100 mg/6 mL	200 mg/6 mL
0.005% LAS, 20 mL (3 uEq)				106%
0.005% LAS, 30 mL (4.5 uEq)	81%	103% (10 mL/min loading)	90.5%	102%
0.005% LAS in wastewater, 20 mL (3 uEq)	91%	92.5%	100%	99%

3.1.3 WCX

The WCX stationary phase is comprised of a 22 µm medium surface area divinylbenzene resin particle grafted with carboxylate functional groups. The ion exchange capacity is 1000 µEq/g and the pKa of the weak acid sites is about 4. This phase is recommended for the solid phase extraction of strong (permanently charged) cations such as quaternary ammonium compounds. The phase is conditioned at pH 8 or higher. The phase is washed and the targets are eluted by protonating the stationary phase to carboxylic acid (neutral) thus eliminating the ion exchange retention mechanism. For some applications recoveries can be improved by adding a Soak-to-Elute step before the Elute step in the method. The resin is shipped in the ammonium acetate form.

3.1.3.1 Method Concentration of Quaternary Ammonium Compounds from Water

Sample Prep: AutoTrace 280
 Analytes: Quaternary ammonium compounds in tap water
 Barrel: SolEx WCX, 150 mg/3 mL
 Flow rate: 10 mL/min
 Elution solvent: 5 mL 10% formic acid in methanol

AutoTrace 280 Method:

Step No.	Step Description	Step Function
1	Condition cartridge with 5.0mL of MeOH into solvent waste	Condition
2	Condition cartridge with 10.0mL of water into aqueous waste	Condition
3	Condition cartridge with 5.0 mL of 1% v/v NH ₄ OH in water into aqueous waste	Equilibrate
4	Load 50.0mL of sample onto cartridge at 10 mL/min	Load
5	Rinse cartridge with 5.0mL of 1% v/v NH ₄ OH in water into aqueous waste	Wash 1
6	Collect 5.0mL fraction into sample tube using 10% formic acid in methanol/water	Elute

3.1.4 SAX

The SAX stationary phase is comprised of a 22 µm high surface area divinylbenzene resin particle grafted with an aromatic quaternary ammonium functionality. The ion exchange capacity is 900 µEq/g and the sites bear a permanent charge. This phase is recommended for the solid phase extraction of weak acid anions such as carboxylates. The phase is conditioned to a pH at which the target analytes are charged (about 2 pH units above the pKa). The sample is loaded, the phase washed, and the targets are eluted with solvent and at the pH about 2 pH units below the pKa, The target analytes are eluted as neutral species by reversed phase.

3.1.4.1 Method Large Volume Solid Phase Extraction of Naproxen in Surface Water

Naproxen is a substituted naphthalene bearing a carboxylic acid functional group. The pKa is 4.69. In the standard methodology, naproxen is retained when charged at high pH on a strong anion exchange phase, and eluted with solvent when neutralized at low pH.

Sample Prep:	AutoTrace 280
Analytes:	Weak acids, naproxen in surface water
Barrel:	SolEx SAX, 150 mg/3 mL
Flow rate:	5 mL/min
Elution solvent:	5 mL 5% formic acid in methanol
AutoTrace 280 Method:	

Step No.	Step Description	Step Function
1	Condition cartridge with 5.0mL of MeOH into aqueous waste	Condition
2	Condition cartridge with 10.0mL of water into solvent waste	Condition
3	Dry cartridge with gas for 5.0 minutes	Dry
4	Condition cartridge with 5.0 mL of 1% v/v NH ₄ OH in water into aqueous waste	Equilibrate
5	Load 50.0mL of sample onto cartridge at 10 mL/min	Load
6	Rinse cartridge with 5.0mL of 1% v/v NH ₄ OH in water into aqueous waste	Wash 1
7	Rinse cartridge with 5.0mL of 1% v/v NH ₄ OH in methanol into solvent waste	Wash 2
8	Collect 5.0mL fraction into sample tube using 5% formic acid in methanol	Elute

Analytical Column:	Acclaim mixed mode WCX-1, 4.6.x 150 mm
Mobile Phase:	40/60 v/v MeCN/NH ₄ OAc pH 5.2 (20 mM total)
Temperature:	30 °C
Injection volume:	5 µL
Flow Rate:	1 mL/min

3.1.5 SCX

The SCX stationary phase is comprised of a 22 µm high surface area divinylbenzene resin particle grafted with aromatic sulfonic acid groups. The ion exchange capacity is 700 µEq/g and the phase is mixed mode with both reversed phase and cation exchange functionality. This phase is recommended for the SPE of weak cations such as weak base amines.

3.1.5.1 Method Determination of Atenolol in Water

Atenolol contains a secondary amine and can be retained by cation exchange on an SCX phase.

In this application the cartridge is conditioned with elution mobile phase in order to minimize possible background interferences using UV detection for an early-eluting target such as atenolol.

The ion exchange capacity of the SCX phase is 700 µEq/g. As an example, a 150 mg bed weight cartridge will have $700 \mu\text{Eq/g} \times 0.15 = 105 \mu\text{Eq}$ of cation exchange capacity. A matrix containing 50 mg/L each of sodium and calcium contains about 4.8 mEq/L = 4.8 µEq/mL. This means that one 150 mg cartridge of SCX can hold $105 / 4.8 = 22$ mLs sample matrix. A 500 mg bed can hold about 75 mL sample matrix.

In order to develop the atenolol application then, one first determines the sample volume necessary to concentrate atenolol to a useful concentration based on the detection mode. Next, estimate the cation content of the matrix and calculate the bed size necessary to hold both the matrix and the target. In the application below, the 50 mL of 1 mg/L atenolol in a 50 ppm sodium/50 ppm calcium matrix can be concentrated using a 500 mg SCX bed, with capacity to spare.

Sample Prep: AutoTrace 280
 Barrel: SolEx SCX, 150 mg/3 mL
 Flow rate: 10 mL/min
 Elution solvent: 5 mL 5% NH₄OH in methanol
 AutoTrace 280 Method:

Step No.	Step Description	Step Function
1	Condition cartridge with 5.0mL of 5% NH ₄ OH in MeOH into aqueous waste	Condition
2	Condition cartridge with 10.0mL of water into solvent waste	Condition
3	Dry cartridge with gas for 5.0 minutes	Dry
4	Condition cartridge with 5.0 mL of 1% v/v formic acid in water into aqueous waste	Equilibrate
5	Load 50.0mL of sample onto cartridge at 10 mL/min	Load
6	Rinse cartridge with 5.0mL of 1% v/v formic in water into aqueous waste	Wash 1
7	Rinse cartridge with 5.0mL of 1% v/v formic acid in methanol into solvent waste	Wash 2
8	Collect 5.0mL fraction into sample tube using 5% NH ₄ OH in methanol	Elute

Analytical column: Acclaim PA2, 2 x 150 mm
 Mobile Phase: 60% methanol/ 40% 0.2% NH₄OH pH 10
 Flow rate: 0.25 mL/min
 Temperature: 30 °C
 Injection volume: 5 µL
 Detection: UV 254nm

3.1.6 Silica Bonded Phases and Silica

The C8 and C18 bonded phases are the two most commonly used sorbent types for off-line SPE and are used for the concentration of pesticides, herbicides, hydrocarbons and other organic contaminants from water as approved by the US EPA. The particle size is 40-63 μm with a surface area of 470-530 m^2/g and a pore volume of 0.70-0.85 cm^3/g .

Phthalate-free C8 and C18 products are used for the extraction of pesticides from water and excellent for trace analysis. The particle size is 40-63 μm with a surface area of 470-530 m^2/g and a pore volume of 0.70-0.85 cm^3/g . These products feature Teflon® frits and are packed in mylar bags to minimize phthalate contamination. The unbounded silica is an acid-washed sorbent and is used to remove polar impurities and purify polar compounds. The particle size is 40-63 μm , has a pore size of about 60 Å and a surface area of 500 m^2/g .

3.1.7 Carbon and GCB

The activated carbon (charcoal) SPE cartridges can be used for the extraction of nitrosamines in drinking water (US EPA Method 521) and for the extraction of 1,4-dioxane in drinking water (US EPA 522). The activated carbon is porous and has a surface area of about 972 m^2/g . Graphitized carbon is used for the extraction of herbicides from drinking water (US EPA Method 535). This carbon is a nonporous graphitized carbon with a surface area of about 90 m^2/g .

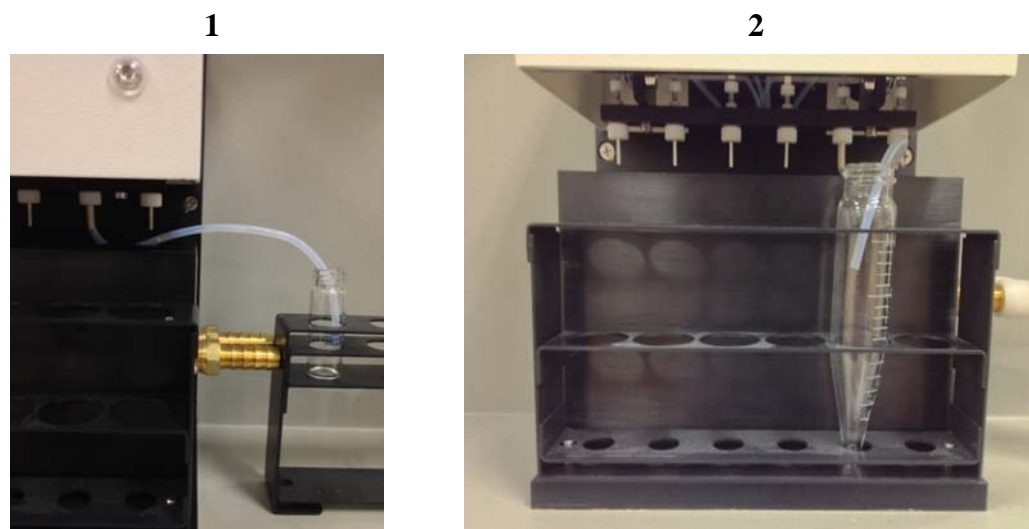
3.1.8 Online Products

SolEx HRP, 2.1 x 20 mm, and SolEx HRP RSLC, 2.1 x 20 mm contain a 12 μm medium surface area hydrophilic reversed phase resin. These products are in housings compatible with 10-32 fitting connections in online configurations. The SolEx HRP device is a disposable PEEK cartridge that is housed in a re-usable stainless steel (SST) housing and the SolEx HRP RSLC product contains 12 μm resin in a 2.1 x 20 mm SST column with high pressure (RSLC) end fittings. These products are covered in a separate product manual.

4. Troubleshooting

In general, each step in the method can be tested by collecting a fraction during each step and checking for the presence of the target analytes. Fig. 3 shows a setup for collecting fractions during method development. A piece of 1/8" O.D. x 1/16" I. D. Teflon (TFE) tubing is slipped onto the outlet line corresponding to the cartridge that is in use.

Figure 3 Setup for Testing Fractions During Method Development in AutoTrace 280 (1 or 2)



Premature breakthrough during loading or washing, or lack of elution will show which steps in the method should be modified. Fundamentally, either the targets are not concentrating during loading or they are not eluting.

4.1 Troubleshooting Table

Issue	Possible Cause	Action
Low Recovery on SAX or SCX	Sample pH not at full ionization of weak anion/cation target.	Adjust sample pH 2 pH units above or below pKa.
	Conditioning step before loading does not leave stationary phase at pH needed for ionization of targets.	Increase volume of conditioning step or increase base/acid in conditioning mobile phase.
	Insufficient elution by organic solvent in elution step.	If solvent is already at 100%, change solvents.
	Insufficient elution by acid or base in elution step.	Check pH of sample after elution; if necessary increase acid or base concentration, add a soak-to-elute step as necessary. Test by adding a second elution on the same cartridge.
	Ionic strength x volume of applied samples greater than capacity of the bed.	Change to a high bed weight cartridge or decrease amount of sample applied.
Low Recovery on WCX or WAX	Conditioning step before loading does not leave stationary phase at pH where the phase is charged.	WAX should be conditioned at about pH 3 and WCX should be conditioned at about pH 7 (or higher).
	Insufficient elution by organic solvent in elution step.	If solvent is already at 100%, change solvents.
	Insufficient elution by acid or base in elution step.	Check pH of sample after elution; if necessary increase acid or base concentration, add a soak-to-elute step as necessary. Test by adding a second elution on the same cartridge.
	pH of elution mobile phase is not where the stationary phase is neutralized.	Adjust elution mobile phase to pH 2 pH units above or below pKa.
	Ionic strength x volume of applied samples greater than capacity of the bed.	Change to a high bed weight cartridge or decrease amount of sample applied.
Low Recovery on HRPHS	Low retention during loading due to targets being ionized at sample pH.	Adjust pH as necessary.
	Insufficient elution by organic solvent in elution step.	Increase elution volume or % organic solvent in elution reagent.