

## A Unified Solid Phase Extraction Platform for Disk and Cartridge Based Extractions

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### Keywords

EPA Method 525, EPA Method 522,  
Pesticides, Phthalates, Adipates, PAHs,  
1,4-Dioxane, SPE Disk, SPE Cartridge,  
SPE Automation



### ABSTRACT

Solid phase extraction of environmental samples typically involve either SPE cartridges or disks, depending on cost, availability, efficiency and sample matrix requirements. Many labs utilize both options in their routine extractions. Current market research has shown a lack of automated systems that can perform both cartridge and disk-based extractions with high efficiency. Compounded with the range of disk and cartridge formats, labs often purchase different types of extractors based on the application.

### INTRODUCTION

PromoChrom's 8-Channel SPE-03 system was designed for using SPE cartridges. It offers high sample throughput and full automation including sample bottle rinsing. With simple modifications, the instrument is capable of running not only larger cartridges, but commercially available 47-mm and 50-mm SPE disks and disk assemblies, with the benefits of positive pressure and unattended operation. This offers more flexibility and a compact footprint when working with disk-based extractions. The design approach and extraction performance will be presented and discussed for a number of common applications.

## DESIGN APPROACH

The SPE-03 has a small 34 cm x 34 cm footprint, excluding sample bottles and racks. Up to 8 SPE cartridges can be mounted within the system. Its default cartridge adapters are compatible with 1/3/6 mL SPE cartridges. 12 mL and 20 mL SPE cartridges can also be mounted on the same cartridge rack using 6/12/20 mL SPE cartridge adapters.

Liquid handling is built around our patented 8-Channel valve and 2 sets of positive pressure syringe pumps. This keeps the design compact and simple.

For the first time in SPE automation, we applied the same positive pressure approach to SPE disks and disk assemblies. This is made possible through an extended platform (disk rack) to create sufficient space.



Figure 1 - SPE-03 disk rack mounted with various disk and cartridge formats

Different disks and cartridges can be adapted using our disk holders or adapter caps as shown below.



Figure 2 - Disk holders for 47-mm/50-mm disks



Figure 3 - Enviro-Clean Universal 525 cartridges with adapter caps



Figure 4 - Pre-assembled BAKERBOND® Speedisk™ with adapter caps

## APPLICATIONS

### EPA Method 525.2 – Using UCT Enviro-clean Universal 525 Cartridge



Figure 5 - UCT Enviro-Clean Universal 525 Cartridge



Figure 6 - SPE-03 System with Enviro-Clean Cartridge

A US lab performs EPA Method 525.2 manually using UCT Enviro-clean Universal Cartridges for 92 analytes. These large cartridges are similar to pre-assembled disks and extraction is done by mounting them on a stainless steel vacuum manifold. The operator is required to manually condition cartridges, direct liquid waste, control sample flow rate and perform bottle rinsing. Each extraction takes about 90 minutes.

Using the SPE-03, up to 8 samples can be extracted in the same amount of time, without human attendance. The system automates all extraction steps with controlled flow rates. The SPE-03 method below was translated from their manual procedure.

**Solvent 1** = MeOH, **Solvent 2** = Water, **Solvent 3** = 1:1 EtAc/DCM, **Solvent 4** = EtAc, **Waste 1** = Aqueous, **Waste 2** = Organic

Table 1 - EPA Method 525.2 steps programmed on the SPE-03

Action	Inlet 1	Flow	Volume
Elute W2	Solvent 3	50 mL/min	10 mL
Wait	Time Based		1 min
Elute W2	Solvent 3	50 mL/min	10 mL
Air-Purge W2	Air	50 mL/min	20 mL
Elute W2	Solvent 1	30 mL/min	5 mL
Wait	Time based		1 min
Elute W2	Solvent 1	30 mL/min	5 mL
Elute W1	Solvent 2	30 mL/min	20 mL
Add Samp W1	Sample	50 mL/min	1020 mL
Air-Purge W1	Air	50 mL/min	20 mL
N2 Cartridge	Time based		5 min
Rinse	Solvent 4	70 mL/min	10 mL
Air-Purge R	Air	70 mL/min	2 mL
Collect 2	Sample	30 mL/min	10 mL
Wait	Time based		1 min
Collect 2	Sample	30 mL/min	5 mL
Rinse	Solvent 3	70 mL/min	10 mL
Collect 2	Sample	30 mL/min	10 mL
Wait	Time based		1 min
Rinse	Solvent 3	70 mL/min	10 mL
Air-Purge R	Air	70 mL/min	2 mL
Collect 2	Sample	30 mL/min	10 mL
Wait	Time based		1 min
Collect 2	Sample	30 mL/min	15 mL

#### Pre-conditioning

Clean sorbent with 10 mL 1:1 EtAc/DCM  
1 min soak in between each 5 mL stroke

Purge liquid from cartridges

Condition sorbent with 10 mL MeOH with 1 min soak  
in between each 5 mL stroke

Wash sorbent with 20 mL water

#### Sample Loading

#### Sorbent Drying

Purge large water droplets followed by 5 min N<sub>2</sub> dry

#### EtAc Rinse and Elute

Rinse sample bottles with 10 mL of 1:1 EtAc  
Purge remaining EtAc in rinse lines  
Deliver rinsate through sorbent and collect in  
fraction row 2, with 1 min soak in between

#### 1:1 EtAc/DCM Rinse and Elute

Rinse sample bottles with 10 mL of 1:1 EtAc/DCM  
Deliver rinsate through sorbent, and collect in  
fraction row 2, with 1 min soak in between

Rinse sample bottles with another 10 mL of 1:1  
EtAc/DCM, purge solvent in rinse lines  
Deliver rinsate through sorbent, and collect in  
fraction row 2, with 1 min soak in between

15 mL air to purge residual solvent from cartridge

Below are the average recoveries for 8 x 1L samples extracted by the SPE-03 and 4 x 1L samples extracted on the manual manifold.

## Pesticides

Table 2 – Pesticide recoveries on the SPE-03 and Manual Manifold at 1 ug/L

Compound	SPE-03	Manual	Compound	SPE-03	Manual
Hexachlorocyclopentadiene	83%	91%	Butylate	116%	106%
Pentachlorophenol [4 ug/L]	122%	117%	Molinate	129%	117%
Etridiazole	119%	109%	Terbutryn	109%	110%
Chlorneb	120%	108%	Propachlor	118%	107%
Hexachlorocyclohexane, alpha	117%	110%	Ethoprop	127%	116%
Hexachlorocyclohexane, beta	112%	107%	Trifluralin	100%	98%
Hexachlorocyclohexane, delta	114%	106%	Chlorpropham	122%	112%
Simazine	120%	117%	Propazine	137%	129%
Atrazine [2 ug/L]	126%	117%	Pronamide	123%	117%
Lindane	117%	110%	Terbacil	106%	105%
Chlorothalonil	116%	107%	Ametryn	106%	106%
Alachlor [2 ug/L]	129%	116%	Prometon	110%	108%
Heptachlor	106%	108%	Bromacil	117%	114%
Dacthal (DCPA)	114%	106%	Metolachlor	115%	108%
<b>Aldrin</b>	<b>83%</b>	63%	Chlorpyrifos	129%	122%
Heptachlor Epoxide	114%	110%	Diphenamid	115%	109%
gamma-Chlordane	94%	95%	Butachlor	118%	110%
Endosulfan I	105%	97%	Norflurazon	117%	112%
Endosulfan II	120%	112%	Hexazinone	120%	112%
alpha-Chlordane	108%	101%	Acetochlor	115%	108%
4,4'-DDD	103%	102%	Thiobencarb	118%	115%
4,4'-DDE	95%	97%	Ethion	109%	106%
4,4'-DDT	105%	103%	Trithion	106%	108%
Dieldrin	111%	106%	Dimethoate	125%	129%
Endrin	132%	113%	Terbufos Sulfone	125%	116%
Chlorobenzilate	116%	109%	Captan	120%	100%
Endrin Aldehyde	91%	83%	<b>Diazinon</b>	<b>99%</b>	52%
Endosulfan Sulfate	128%	115%	Malathion	121%	115%
Methoxychlor	115%	115%	methyl-Parathion	119%	116%
Permethrin <sup>1</sup> [2 ug/L]	111%	112%	Parathion	130%	120%
Dichlorvos	125%	118%	Metribuzin	120%	117%
EPTC	121%	109%			

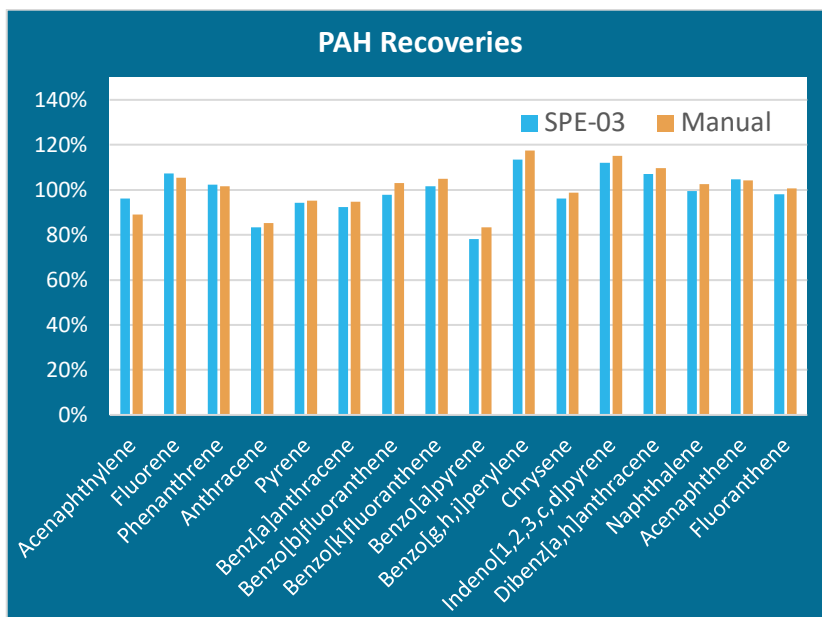
1. Permethrin recovery is based on the combined recovery of cis-Permethrin and trans-Permethrin.

The SPE-03 yielded comparable pesticide recoveries to the manual extraction. Most notably, the compounds Aldrin and Diazinon which have been problematic for manual extraction recovered much higher using the SPE-03. This is speculated to be due in part to the controlled flow rate of the system.

## PAHs

Table 3 - PAH recoveries on the SPE-03 and Manual Manifold at 1ug/L

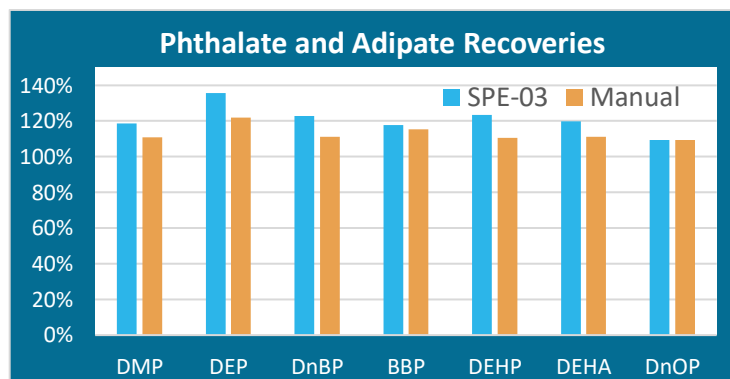
Compound	SPE-03	Manual
Acenaphthylene	96%	89%
Fluorene	107%	106%
Phenanthrene	102%	102%
Anthracene	83%	85%
Pyrene	94%	95%
Benz[a]anthracene	92%	95%
Benzo[b]fluoranthene	98%	103%
Benzo[k]fluoranthene	102%	105%
Benzo[a]pyrene	78%	83%
Benzo[g,h,i]perylene	113%	118%
Chrysene	96%	99%
Indeno[1,2,3,c,d]pyrene	112%	115%
Dibenz[a,h]anthracene	107%	110%
Naphthalene	100%	103%
Acenaphthene	105%	104%
Fluoranthene	98%	101%



## Phthalates and Adipates

Table 4 - Phthalate and Adipate recoveries on the SPE-03 and Manual Manifold at 1ug/L

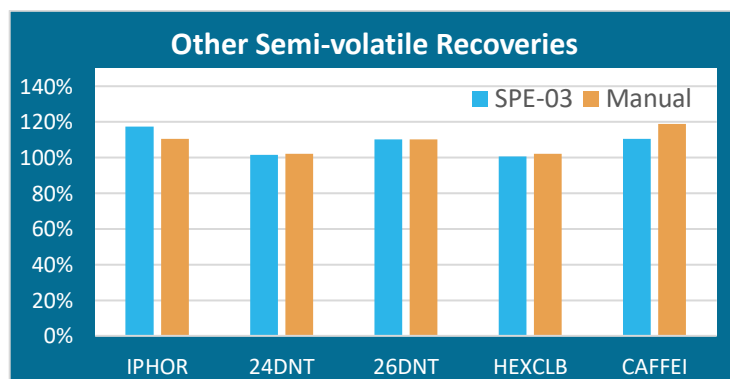
Compound	SPE-03	Manual
Dimethylphthalate (DMP)	119%	111%
Diethylphthalate (DEP)	136%	122%
Di-n-Butylphthalate (DnBP)	123%	111%
Di(2-ethylhexyl)adipate (BBP)	118%	116%
Di(2-ethylhexyl)phthalate (DEHP)	124%	111%
Butylbenzylphthalate (DEHA)	120%	111%
Di-n-octyl phthalate (DnOP)	109%	110%



## Other Semi-volatiles

Table 5 - Other Semi-volatile recoveries on the SPE-03 and Manual Manifold at 1ug/L

Compound	SPE-03	Manual
Isophorone (IPHOR)	118%	111%
2,4-Dinitrotoluene (24DNT)	102%	102%
2,6-Dinitrotoluene (26DNT)	110%	110%
Hexachlorobenzene (HEXCLB)	101%	102%
Caffeine (CAFFEI)	110%	119%



The remaining classes of target analytes saw similar recoveries between both setups.

## EPA Method 525.2 – Using Biotage Atlantic® SPE Disk



Figure 7 - Biotage Atlantic® C18 Disks

Another lab uses the Biotage Atlantic® C18 SPE disk to perform EPA Method 525.2 for a smaller list of 22 compounds. Their existing method was developed on a large automated setup with 8 single-channel modules. The procedure was translated on the SPE-03 and the disks mounted using PromoChrom's disk holders. Up to 8 samples can be extracted simultaneously on a single unit. The following results were obtained from 4 x 1L samples, spiked at 2 ug/L.

Table 6 - EPA 525.2 results on the SPE-03 at 2 ug/L

Target Compounds	Recovery	RSD
Hexachlorocyclopentadiene	85%	6.1%
Propachlor	105%	2.7%
Hexachlorobenzene	93%	0.9%
Atrazine	101%	2.5%
Simazine	97%	2.1%
Lindane	107%	0.8%
Alachlor	106%	0.7%
Heptachlor	101%	2.5%
Metribuzin	84%	6.3%
Metolachlor	108%	0.0%
Aldrin	96%	4.9%
Heptachlor Epoxide	106%	2.4%
Butachlor	108%	2.0%
Trans-Chlordane	109%	2.8%
Cis-Chlordane	103%	3.5%
Trans-Nonachlor	112%	6.1%
Dieldrin	104%	3.8%
Endrin	103%	2.8%
Bis(2-ethylhexyl) adipate	107%	1.4%
Methoxychlor	98%	5.3%
Bis(2-ethylhexyl) phthalate	108%	3.5%
Benzo(a)pyrene	91%	3.4%



Figure 8 - Biotage Atlantic® C18 Disks mounted on the SPE-03

All compounds recovered between 84% to 112%, demonstrating excellent performance of the SPE-03 using Atlantic SPE disks.

## 1,4-Dioxane – Using CDM Empore™ Carbon SPE Disk



Figure 9 - CDM Empore™ carbon disks



Figure 10 - SPE-03 using CDM Empore™ carbon disk

The Regional Municipality of Waterloo uses Empore™ Carbon SPE disks to determine 1,4-Dioxane in water samples. Their method was also translated from an older automated extractor onto the SPE-03 system. The disks are mounted in PromoChrom's disk holders.

Compared to 6mL SPE cartridge extraction following EPA Method 522 for 1,4-Dioxane, the SPE-03 uses 5 to 6 times the solvent and sample flow rate for these 47mm disks. It takes just 80 minutes to fully-automate the extraction of all 8 x 1L samples.

Below are the Initial Demonstration of Capability (IDC) and Minimum Detection Limit (MDL) results.

Table 7 - IDC Results at 2.5 ug/L

	1	2	3	4	5	6	7	8	9	10	Average	RSD
<b>Recovery</b>	104%	107%	100%	93%	103%	103%	104%	100%	106%	98%	101%	4.1%

Table 8 - MDL Results at 0.25 ug/L

	1	2	3	4	5	6	7	8	9	10	Average	RSD
<b>Recovery</b>	108%	112%	116%	116%	104%	108%	116%	108%	104%	116%	111%	4.5%

## 1,4-Dioxane – Using 6mL 2g Coconut Charcoal SPE Cartridge

The same system can be used with 6mL SPE cartridges for 1,4-Dioxane following EPA Method 522. Using a slower flow rate for the SPE cartridge, 8 x 500mL samples are extracted in 132 minutes, yielding the following results from the Erie County Water Authority:

Table 9 - IDC Recoveries at 5.0 µg/L

	1	2	3	4	Average	RSD
<b>Recovery</b>	114%	111%	106%	85%	104%	12.6%

Table 10 - MRL recoveries at 1.0 µg/L

	1	2	3	4	5	6	7	Average	RSD
<b>Recovery</b>	111%	114%	105%	106%	112%	113%	106%	110%	3.4%

## CONCLUSIONS

The SPE-03 is a versatile system that works for a wide range of solid phase extraction applications. Using an extended platform with necessary holders and adapters, it applies the benefits of positive pressure and controlled flow rates to SPE disks and cartridges of many sizes and form factors. Up to 8 samples can be extracted simultaneously within a small foot print which frees up lab space for other equipment.

Instead of supervising hours of extraction process each day, operators can simply load the samples and run the system with the touch of a button. The SPE-03 automates all extraction steps including bottle rinsing, so that attention can be focused on other lab activities.

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