

# A Novel Solid Phase Extraction Disk that Combines the Advantages of SPE Cartridges and Conventional 47mm disks while Overcoming Their Challenges

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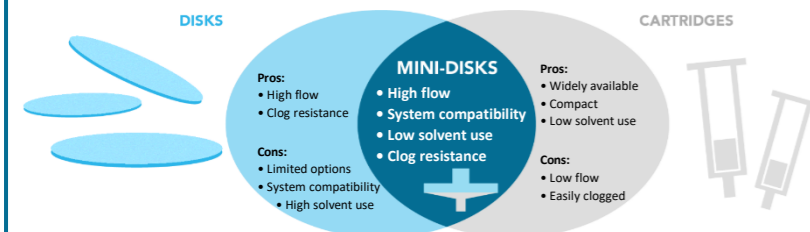
## ABSTRACT

To capture the advantages of:

- **SPE disks** in extraction speed and clogging resistance
- **SPE cartridges** in system compatibility and low solvent use



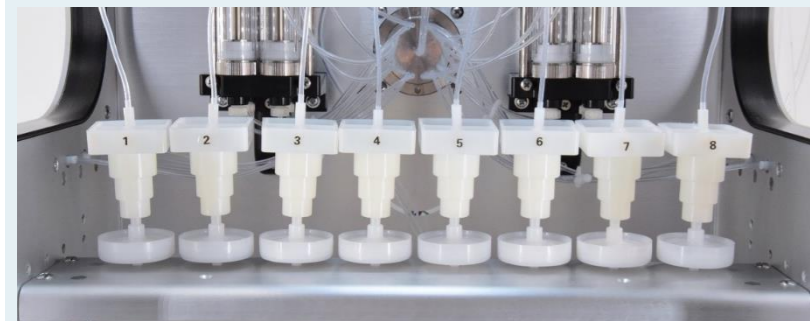
While eliminating their short-comings.



## INTRODUCTION

Solid phase extraction (**SPE**) is often the most challenging and time-consuming process in the analysis of large volume water samples. Majority of SPE are performed using SPE cartridges, even though SPE disks offer much higher flow rate and clogging resistance. This is due in part to the higher cost and larger volume of solvent usage. In addition, most automated SPE systems are only compatible with SPE cartridges, thereby further limiting the use of disks.

To capture the advantages of SPE disks in extraction speed and overcome the shortcomings in solvent consumption and automation, PromoChrom developed a suite of **Mini-disks**. The Mini-disks come in a format similar to a 30-mm syringe filter and a cross section area 5 times of a 6-mL SPE cartridge. The increased cross-section area and optimized sorbent properties enables the Mini-disks to work with much higher flow rates than SPE cartridges, whereas a smaller size than conventional disks makes the Mini-disks easily adaptable to cartridge type SPE instruments and consume less solvents.



This poster discusses the extraction of **EPA Method 525.2** and **TCLP Semi-volatile Organic Compounds (SVOCs)** using Mini-disks MD-525-30 and MD-BNA-30 with the SPE-03 automated solid phase extraction system.

## EPA METHOD 525.2 COMPOUNDS

EPA Method 525.2 is used for the determination of organic compounds spanning pesticides, PAHs, phthalates and other pollutants in water using SPE and GCMS.

## MATERIALS

- **Mini-disk:** PromoChrom Mini-disk with mixed-mode polymers for extracting both hydrophilic and hydrophobic compounds (Cat. No.: MD-525-30).
- **Instrument for Extraction:** SPE-03 8-channel SPE system with MOD-00P configuration for automatic rinsing of up to 1 L sample bottles.
- **Instrument for analysis:** Agilent GCMS with Agilent J&W HP-5MS 30m x 0.25mm x 0.25 um column.

## METHOD

Four samples consisting of 950 mL of reagent water spiked at 2 ug/L and acidified to pH <2 were extracted on the SPE-03. 5 mL of DCM, Ethyl Acetate and Methanol was used for pre-conditioning. Final bottle rinse and cartridge elution consists of 7.5 mL of Ethyl Acetate and 5 mL of DCM. Sample loading was performed at 55 mL/min. The extraction took 100 minutes for a batch of 8 samples. Collected fractions were concentrated to 1 mL prior to GCMS analysis.

## RESULTS

Below are the results based on the absolute recoveries of each compound.

Pesticides		
Analytes	%Recovery	%RSD
Aldrin	86.2	1.5
Hexazinone	137.4	3.3
4,4-DDD	126.8	5.5
4,4-DDE	94.9	1.6
4,4-DDT	88	10.4
Alachlor	100.6	0.6
Ametryn	98.7	2.9
Atrason	77.6	2
Atrazine	102.3	1
Bromacil	97.1	2.2
Butachlor	109.7	0.3
Butylate	94.9	1.1
Carboxin	98.9	7
gamma-Chlordane	98.9	12.7
trans-Chlordane	90.7	10.4
alpha-Chlordane	62.2	8.5
Chlormeb	104.2	0.6
Chlorobenzilate	124.4	10.6
Chlorothalonil	106.4	3.3
Chlorpropham	105.3	4.4
Cycloate	98.9	2
Dacthal	101.2	3.2
Diethrin	97.3	0.8
Diphenamid	105.1	0.9
Endosulfan I	95.3	2.8
Endosulfan II	106.8	9.3
Endosulfan Sulfate	112.5	9.2
Endrin	128.3	9.4
Endrin Aldehyde	100.9	15.6
EPTC	96.4	1.9
Etridiazole	109.3	0.6
Fenarimol	122.3	3
Fluridone	115.6	6.4
Heptachlor	91.6	22.7
Heptachlor Epoxide	85.9	12.1
Hexachlorocyclopentadiene	75.4	21.9
alpha-BHC	101.8	0.5
beta-BHC	104.2	2.6
delta-BHC	101.9	2.2
gamma-BHC	104.1	1.3
HCCPD	75.4	21.9
Lindane	106	2
Methoxychlor	112.6	9.5
Metolachlor	105.5	1.9
Metribuzin	99.3	2
MCG-264 B	102.1	5.6
MCG-264A	101.5	12.5
Molinate	100.1	1.5
Napropamide	108.7	1.3
Norflurazon	113.2	9.3

Pesticides (continued)		
Analytes	%Recovery	%RSD
Pebulate	102.1	0.7
Pentachlorophenol	76.7	5.5
Permethrin, cis	109.5	4.2
Permethrin, trans	106.2	4
Prometon	94.3	3.3
Prometryn	100.3	2.6
Pronamide	100	1.9
Propachlor	98.7	1.9
Propazine	106.3	3.5
Simazine	93.3	2.1
Simetryn	86.1	2.2
Strofos	109.9	17.8
Tebuthiuron	92.2	4.2
Teacale	89.5	3.6
Terbutryn	103.6	0.7
Triademefon	108.3	4.2
Tricyclazole	124.5	5.6
Trifluralin	88.8	1.7
Vernolate	95.6	1.4

PAHs		
Analytes	%Recovery	%RSD
Acenaphthylene	101.3	1
Anthracene	102.4	2.5
Benzo(a) anthracene	102.5	1.9
Benzo(a) pyrene	117.9	2.2
Benzo(b) fluoranthene	117.6	2.1
Benzo(e,h,i) perylene	112.5	2.1
Benzo(k) fluoranthene	103.7	0.6
Chrysene	102.5	1.9
Dibenzo(a,h) anthracene	86.1	2.9
Fluorene	102.1	1
Indenopyrene	89.9	4.7
Perylene-d12	112.4	12.1
Phenanthrene	94.8	1.9
Pyrene	101.7	1.3

Phthalates, Adipates, others		
Analytes	%Recovery	%RSD
Butylbenzylphthalate	124.1	7
Di 2-ethylhexyl adipate	109	9.2
Di 2-ethylhexyl phthalate	124.3	5.7
Diethylphthalate	91.4	3.6
Dimethylphthalate	105.7	0.9
Di-n-butylphthalate	114.7	4
1,3-Dimethyl-2-Nitrobenzene	96.8	2.2
2,4-Dinitrotoluene	96.9	4.4
2,6-Dinitrotoluene	101.9	1.6
Hexachlorobenzene	85.7	14.5
Isophorone	91.9	4.6
Triphenyl phosphate	86.3	22.9

Most of the 95 compounds recovered within the range of 70-130% and %RSD <20%. Alpha-Chlordane is the only compound that recovered lower than 70%, which can be further optimized by tweaking the extraction and analysis parameters.

It was also found that mixing the stock standard solution in 5 mL of MeOH before spiking into the samples helped with the recovery of more volatile compounds such as hexachlorocyclopentadiene, hexachlorobenzene, and aldrin.

Overall, the MD-525-30 Mini-disk offers a quick and effective approach to extracting a long list of EPA Method 525.2 compounds including pesticides, PAHs, phthalates and other organic compounds. Using a flow rate of 55 mL/min, the extraction speed is more than 5 times as fast as SPE cartridge extraction while only requiring minimal solvents for conditioning and elution.

## TCLP SEMI-VOLATILE COMPOUNDS



Merit Laboratories had been extracting TCLP SVOCs using 47-mm disks and a vacuum-based extractor, hereon referred to as the "benchmark". To overcome the limitations in sample throughput and variations in extraction time, PromoChrom's MD-BNA-30 Mini-disk and SPE-03 8-Channel extractor were introduced. While 47-mm disks require disk holders, the Mini-disks come pre-packaged for direct connection to the SPE-03 or any standard luer connector.

## MATERIALS

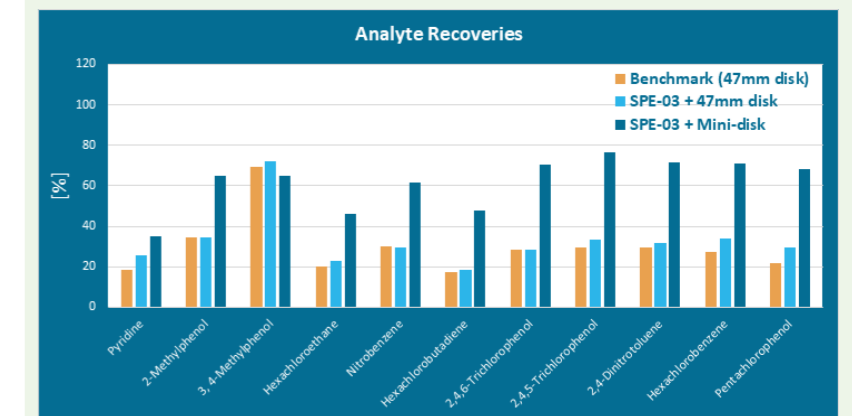
- **Mini-disk:** PromoChrom Mini-disk with mixed-mode polymers for extracting acidic, basic and neutral compounds (Cat. No.: MD-BNA-30).
- **Instrument for Extraction:** SPE-03 8-channel SPE system with MOD-00P configuration for automatic rinsing of up to 1L sample bottles.
- **Instrument for analysis:** Agilent GCMS

## METHOD

The SPE-03 was first validated using 47-mm disks to achieve similar results to the benchmark. The method was developed based on the benchmark using similar amounts of conditioning and elution solvents. Sample loading was performed at 50 mL/min. Some method development was required, including the use of nitrogen drying to effectively purge liquids during conditioning and elution. This was not needed for the Mini-disks. PromoChrom's mixed-mode Mini-disks were validated with similar steps and then introduced into routine extractions. The Mini-disk results were taken from LCS and LCS duplicates across 8 different extraction batches on different days.

## RESULTS

As shown in the plot below, recoveries comparable to the benchmark (orange) was achieved by the SPE-03 using 47-mm disks (light blue). Compared to using 47-mm disks on the benchmark and SPE-03, the Mini-disk (dark blue) demonstrated a significant improvement in overall analyte recoveries. The basic compound, Pyridine, has also been a challenging analyte in the past due to its low recoveries, which saw more than 30% improvement on the Mini-disk.



Compared to the 47-mm disk, the improved performance of the Mini-disk is attributed in part to the optimized sorbent proportion, packing distribution and flow design within its housing. The smaller disk area also allows for easier conditioning, drying and elution.

## CONCLUSIONS

PromoChrom's Mini-disks demonstrated good recoveries of a wide range of organic compounds under EPA Method 525.2 and acidic, neutral and basic compounds in the TCLP semi-volatiles list. The compact and pre-packaged format requires minimal solvent and makes them easily integrable with the SPE-03 8-Channel automated system or even vacuum manifolds. While 47-mm disks would require inserting into disk holders and cleaning the holders after extraction, the Mini-disk comes with luer fittings for direct connection.

Regardless of using cartridges, disks or Mini-disks, PromoChrom's SPE-03 system provides an efficient and versatile platform that extracts 8 samples simultaneously. The fixed extraction time using positive-pressure syringe pumps provides laboratories with predictable turnaround times.

## ACKNOWLEDGEMENTS

We would like to thank Merit Laboratories for using our MD-BNA-30 mini-disks and SPE-03 system and sharing results for this poster. We would also like to thank Rick Chen from PromoChrom Technologies for validating our MD-525-30 Mini-disks for the EPA Method 525.2 compound list.