

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

Using **Mini-disks** 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 shortcomings.



INTRODUCTION

Solid phase extraction (SPE) is the most time-consuming process in the analysis of large volume water samples. Majority of SPE are performed using SPE cartridges, even though SPE disks offer higher flow rate and clogging resistance. This is due in part to the higher cost and solvent usage. In addition, SPE disks require more complicated mounting with holders and screens which limit their ease-of-use.

To capture the advantages of SPE disks in speed and overcome the shortcomings in solvent consumption, PromoChrom developed a suite of **Mini-disks**. The Mini-disks come in a format similar to a 30-mm syringe filter for easy attachment and a cross section area 5 times of a 6-mL SPE cartridge. The increased cross-section area and optimized sorbent properties enable the Mini-disks to work with much higher flow rates than SPE cartridges. The small format keeps solvent use similar to cartridges.

To further enhance extraction efficiency and automation, PromoChrom introduced the new **Presto 8-Channel accelerated SPE system** which features continuous pumps designed for high flow applications. This poster discusses the extraction of **EPA Method 525.2** and **Brevetoxins in seawater** using Mini-disks with the Presto.



Figure 1 - Presto Accelerated SPE System



Figure 2 - Mini-disks within the Presto

EPA METHOD 525.2

EPA Method 525.2 is used for the determination of Persistent Organic Pollutants (POPs) spanning pesticides, PAHs, phthalates, etc. in water using SPE and GCMS.

MATERIALS

- **Mini-disk:** PromoChrom Mixed-Mode Mini-disk (Cat. No.: MD-525-30)
- **Equipment for Extraction:** Presto 8-channel Accelerated SPE system
- **Instrument for analysis:** Agilent GCMS, 30m x 0.25mm x 0.25 um column

METHOD

8 x 1L samples spiked at 2 ug/L and adjusted to pH of 2 were extracted using PromoChrom's Presto system and mixed-mode Mini-disk. 5 mL of EtAc, MeOH and water were used to condition the Mini-disks at 50 mL/min followed by sample loading at 40 mL/min. After 10 mins of disk drying, 5 mL of EtAc and 8 mL of DCM were used to rinse the bottles and elute the Mini-disks at 60 mL/min.

RESULTS

Pesticides			Pesticides (continued)		
Analytes	Recovery	RSD	Analytes	Recovery	RSD
Dichlorvos	87.7	5.9	Endosulfan I	91.9	10.9
Hexachlorocyclopentadiene	87.6	2.4	Endosulfan II	95.0	10.8
EPTC	93.0	8.2	Tetrachlorvinphos	115.5	8.4
Mevinphos	94.2	11.1	Butachlor	104.7	10.7
Butylate	92.1	7.7	Trans-nonchlor Chlordane	90.4	4.3
Vernolate	91.0	7.6	Napropamide	102.9	11.0
Etridiazole	97.0	9.4	Tricyclazole	103.2	11.1
Pebulate	90.6	7.7	4,4-DDD	89.7	7.6
Terbufos	99.9	9.7	4,4-DDE	89.1	3.8
Chlorobeb	91.7	7.6	4,4-DDT	89.4	5.8
Tebuthiuron	104.4	12.7	Dieldrin	94.0	9.5
Molinate	91.9	6.9	Carboxin	88.6	5.5
Propachlor	93.3	11.8	Endrin	103.0	9.4
Cycloate	91.9	9.7	Chlorobenzilate	102.3	12.1
Ethoprophos	97.1	12.7	Endrin Aldehyde	102.4	11.9
Chlorpropham	99.9	12.0	Endosulfan Sulfate	106.8	8.5
Trifluralin	98.0	12.5	Norflurazon	114.1	9.2
Disulfoton Sulfone	92.4	10.7	Hexazinone	103.6	10.8
Hexachlorobenzene	88.2	1.6	Endrin Ketone	103.0	9.4
Atraton	88.2	4.2	Methoxychlor	112.2	7.9
Simazine	105.6	10.0	Fenarimol	122.3	5.8
Prometon	89.6	6.1	cis-Permethrin	98.5	13.0
Atrazine	93.3	11.5	trans-Permethrin	91.6	10.0
alpha-HCH	113.5	9.2	Fluridone	125.9	2.3
beta-HCH	92.4	9.1			
delta-HCH	93.2	9.2			
gamma-HCH	93.2	7.8			
Pentachlorophenol	113.5	8.9			
Chlorothalonil	114.2	9.3			
Propazine	92.3	10.8			
Merphos	95.8	8.4			
Pronamide	94.9	12.6			
Diazinon	91.5	11.1			
Methyl paraoxon	101.1	9.6			
Disulfoton	88.8	2.0			
Terbacil	98.4	12.3			
Metribuzin	97.4	14.2			
Simetryn	91.9	9.5			
Heptachlor	95.0	8.5			
Alachlor	97.9	11.4			
Ametryn	92.6	8.8			
Prometryn	90.8	10.4			
Terbutryn	94.4	13.0			
Bromacil	104.3	10.3			
Aldrin	88.8	3.2			
Metolachlor	101.3	11.0			
Chlorpyrifos	90.5	9.7			
Triademefon	107.0	7.9			
Dacthal	92.3	9.6			
Fenamiphos	89.9	7.5			
MGK-264 B	100.8	12.3			
MGK-264 E	103.1	11.2			
Diphenamid	95.8	12.8			
alpha-Chlorodane	91.7	3.8			
Heptachlor Epoxide	98.8	8.1			
gamma-Chlorodane	89.5	6.3			

Figure 3 - EPA Method 525.2 compounds extracted on the Presto using Mini-disks

The Presto with Mini-disks achieved excellent 85% to 130% recovery for all 107 compounds, with %RSD <15%.

*Di 2-ethylhexyl adipate results were based on 4 samples due to contamination in the other 4 positions during extraction. The contamination source was determined to be dust in the lab which was later removed by wiping down all surfaces with IPA.

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 higher flow rates, 8 x 1L samples can be extracted within an hour compared to more than 3 hours using SPE cartridges. Using the Presto further allows unattended operation and greater efficiency compared to manual or other automated systems.

BREVETOXINS IN SEAWATER

Prior to its release, PromoChrom's Presto system was Beta-Tested in a variety of conditions at the Mote Marine Lab in Florida. Additionally, PromoChrom's C18 and mixed-mode Mini-disks were compared to their original 3 mL C18 SPE cartridges for the extraction of brevetoxins in salt water.



MATERIALS

- **SPE Media:** PromoChrom C18 Mini-disk (Cat. No.: MD-C18-30) and Mixed-Mode Mini-disk (Cat. No.: MD-525-30), C18 3 mL 200 mg SPE cartridge
- **Equipment for Extraction:** Presto 8-channel Accelerated SPE System
- **Instrument for analysis:** Agilent LCMSMS

METHOD

The extraction of brevetoxins involve conditioning the SPE media with 5 mL of MeOH followed by 5 mL of water. 500 mL of aqueous samples are then loaded before the SPE media is dried with air or nitrogen for 9 minutes. Finally, 12 mL of MeOH is used to elute the SPE media and collect the fractions for analysis.

The lab was originally using the SPE-03 system with 3 mL SPE cartridges at a slow flow rate of 5 mL/min. Using the Presto with C18 Mini-disks, the flow rates were increased to 50 mL/min for conditioning and sample loading, while elution was performed at 60 mL/min.

RESULTS

The C18 Mini-disk performed similarly to the SPE cartridges, whereas the Mixed Mode Mini-disk had lower recoveries overall. There was substantial time savings achieved by utilizing the Mini-disks in conjunction with the Presto system, with extraction times for a 500 mL sample dropping from >2 hours to just 45 minutes.

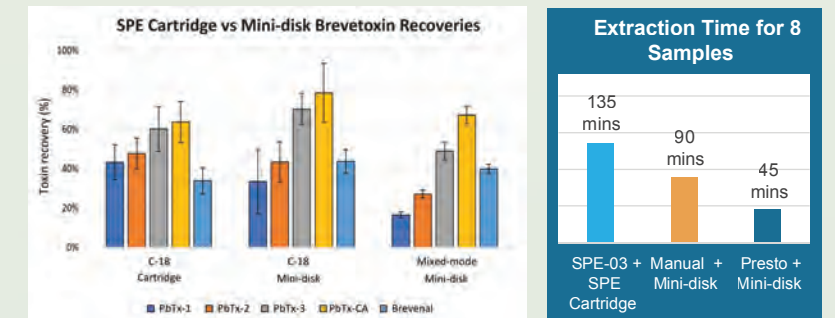


Figure 4 - Brevetoxin extraction recoveries and time using cartridge vs Mini-disk

HANDLING CHALLENGING SAMPLES

PromoChrom's Anti-clogging Tips attached to the sample lines of the Presto system allowed smooth extraction of seawater samples filled with substantial quantities of sediment and algae. The extractor also includes pressure and flow regulation to maintain flow rate within an acceptable pressure range. According to Mote, processing speed was consistent at 50 mL/min, and did not affect recovery.



Figure 5 - Using Anti-clogging Tips for seawater samples with sediment and algae

CONCLUSIONS

PromoChrom's Mini-disks can achieve excellent recoveries at much higher extraction speeds than SPE cartridges, while providing lower solvent use and easier handling than 47mm SPE disks. The wider surface area also increases its capacity for sample particulates which alleviates clogging when running challenging samples.

When paired with the Presto 8-Channel extractor with continuous sample loading pumps, users can achieve maximum time savings and unsupervised operation. The use of anti-clogging tips further enhances the tolerance of sample particulates.

ACKNOWLEDGEMENTS

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