

SPE-03 Multi Channel Cleanup Station

For Automatic Sample Preparation in Trace Analysis

About PromoChrom

PromoChrom Technologies focus on development of sample preparation solutions for trace analysis. Since year 2005, PromoChrom have developed SPE-01 cleanup station, SPE-03 cleanup station, SPE-04 online/offline SPE, LC-04SP valve system and SPE-06 mini SPE. Each of the instruments are targeting specific applications. SPE-01 has been used for cleanup in analysis of pesticide residues and extractable petroleum pollutants in soil. SPE-03 has been used for water quality monitoring. LC-04SP has been used to build multi dimensional HPLC.

In 2011, PromoChrom developed flow-path-integration technique for liquid handling. The technique is based on ideas from integrated circuit and lab on a chip manufacturing. It combines various switching valves into one liquid handling module. The technique simplifies the structure of our instruments considerably, making the instruments more affordable and more reliable.

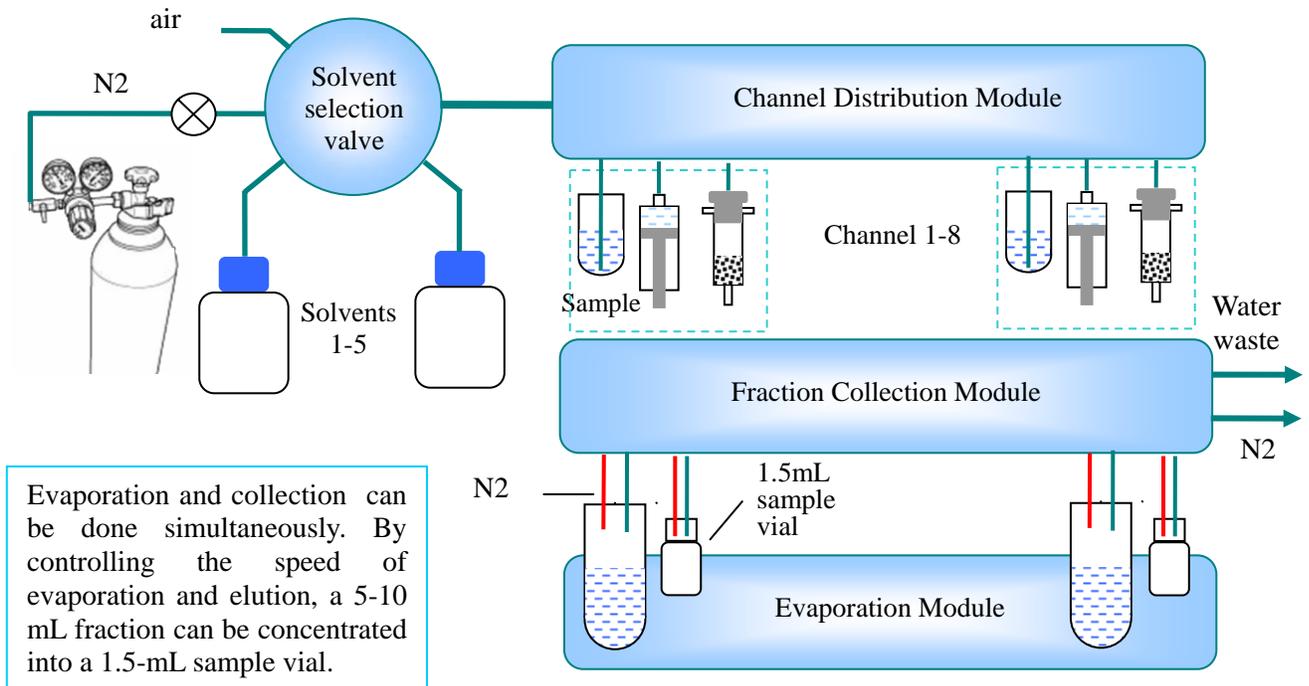
SPE-03 multi-channel cleanup station is designed for simultaneous extraction of multiple samples. It can automatically fulfill all the necessary actions for solid phase extraction, such as conditioning of SPE columns, sample loading, washing, drying sorbent using nitrogen, and fraction collection. By providing constant flow rate and well controlled elution procedures, SPE-03 helps to improve quality and efficiency of trace analysis and release chemists from tedious sample preparation routines. It can be used for extraction of large volume water samples, as well as small volume samples (such as soil extracts, forensic samples, and food samples).

Thanks to the flow-path-integration technique, the weight and dimension of SPE-03 are much smaller than other multi channel SPE instruments.



1. Working principle of SPE-03

The following diagram illustrates the working principle of SPE-03:



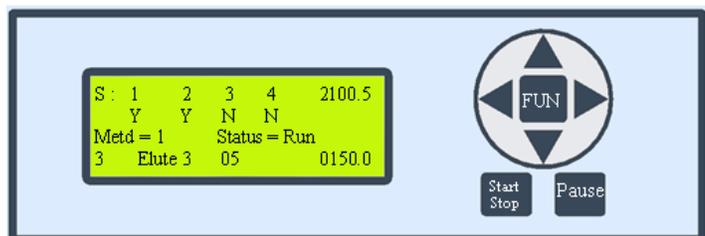
As shown in the above diagram, the major components of SPE-03 are a stream selection valve, a channel distribution module, and an optional evaporation module. It does not need other stream selection valves or 3-way solenoid valves. The tubing layout is also simple. The instrument can be configured in 4, 6 or 8 channels according to buyers budget and required sample throughput.

2. Features

2.1 Easy operation

SPE-03 uses built-in methods. They can be easily edited. The operation of instrument involves only 7 buttons. Below is a typical routine operation procedure:

- Place sample probe in samples,
- Install columns and collection tray
- Select method,
- Press the start/stop button.



The screen indicates samples 1 and 2 are being processed using method 1. The total volume per sample is 2100.5 mL and 150 mL has been processed. Currently the instrument is running the 3rd step of the method, which is using solvent 3 to elute SPE column at a flow rate of 5 mL/min.

The instrument will process the samples automatically according to the selected method.

2.2 Column blockage detection and smart handling

The system can detect the blockage of SPE column and reduce the flow rate accordingly. If blockage still occur, the instrument will pause to wait for human attendance.

2.3 Small footprint and simple structure due to flow path integration technique

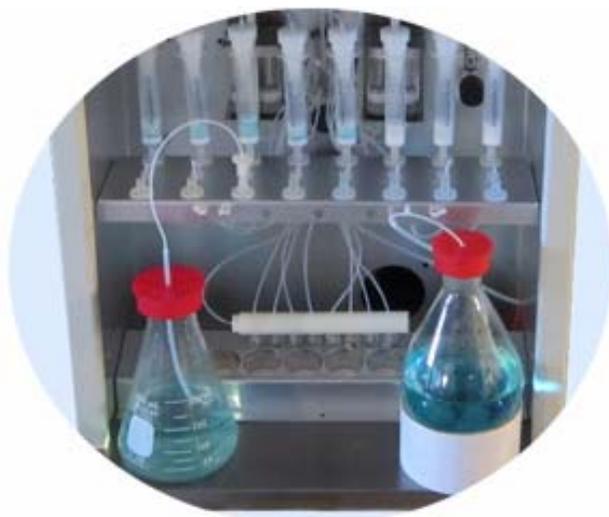
Normal automated SPE instruments involves many switching valves and complex tubing connections. A multi channel SPE is normally heavy and of large size. The tubing connection is also complex, making maintenance difficult.

Thanks to the flow-path-integration technique, the weight and dimension of SPE-03 are around half of other multi channel SPE instruments. For a 8-channel SPE-03, the weight is only 12 kg.

2.4 Use various SPE columns and containers for fractionsw

SPE-03 adopts an innovative adapter to deal with variations in diameter of SPE columns. The adapters can be easily adjusted when different type columns are used. Its wide flow rate range makes it also suitable for disk extractions.

The instrument can also use containers of different shap for sample and fraction collection. These containers can be capped during while the instrument is processing sample. It minimizes solvent evaporation. Thus the work can be carried out without using a fume hood.



2.5 Versatile applications

SPE-03 uses two sets of tubing for sample loading. Long and wider tubing is used for large volume water samples, and narrower and shorter tubing is used for small samples. In addition to large volume water samples,

SPE-03 is also suitable for small volume extracts from soil or food samples.

2.6 Automatic wash of sample tubing and container

To prevent cross contamination, function for cleaning of sample tubing is built into the methods. Two solvents may be used to wash the tubing and container. The rinsate may be left inside the container or added back to the columns.

2.7 Online evaporation (optional)

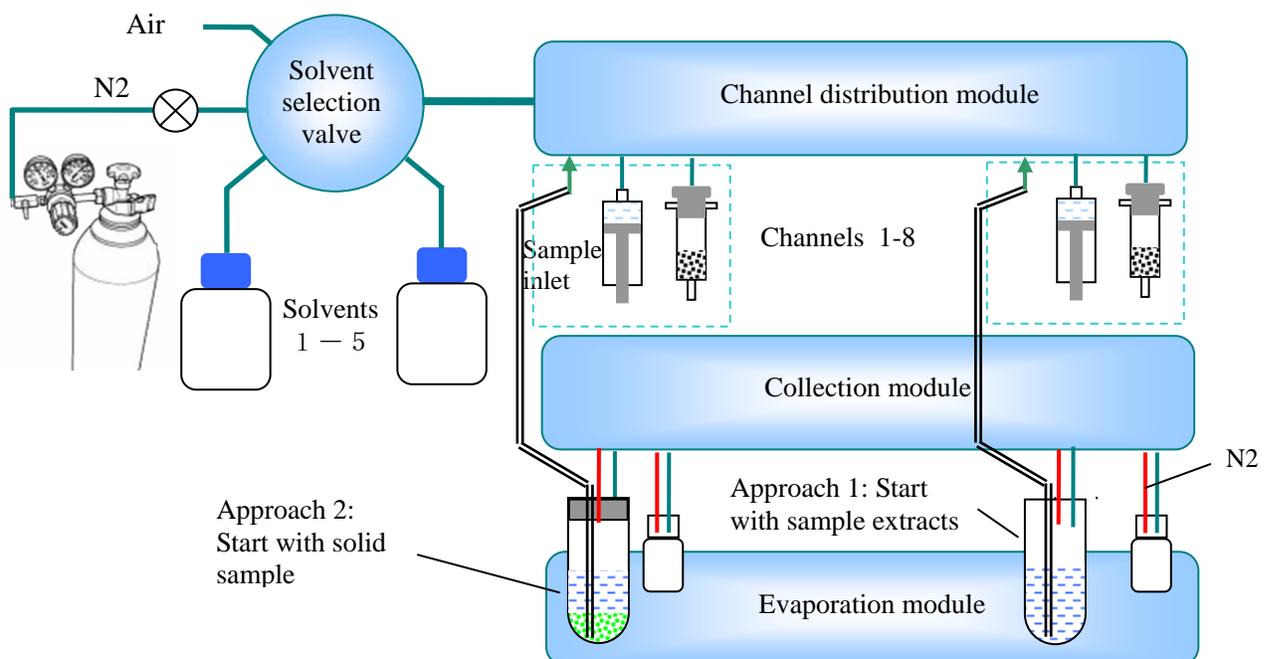
Evaporation and collection can be done simultaneously. By controlling the speed of evaporation and elution, a 5-10 mL fraction can be concentrated into a 1.5-mL sample vial. The liquid level in the sample vial can be maintained below 0.5 mL. It can be adjusted manually using the marker on the vials after the completion of the evaporation.

In addition to evaporation of fraction, the evaporation module can also evaporate sample extraction before loading it to column and can even carry out heat assisted sample extraction.

3. Applications

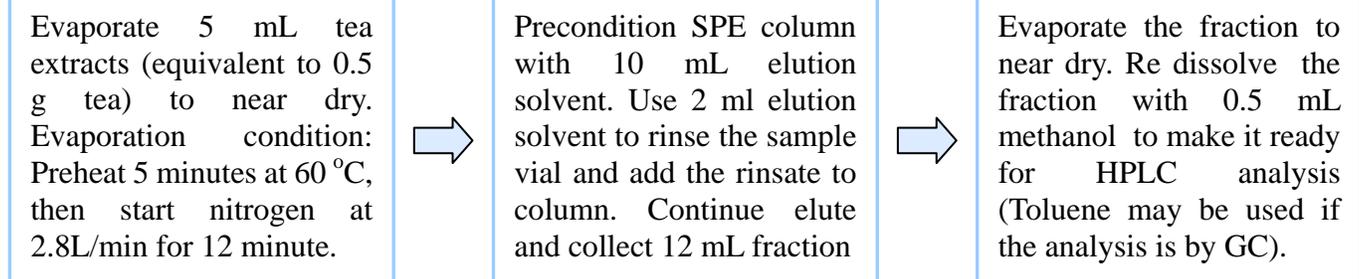
3.1 Full automation of sample preparation for pesticide residue analysis in solid samples

Sample preparation for pesticide residue analysis normally involves four step: 1) Sample extraction, 2) evaporation and solvent change of the extracts, 3) column cleanup, 4) fraction evaporation and solvent change prior to GC or HPLC analysis. This application uses analysis of methyl parathion in tea as an example to demonstrate how SPE-03 can automate all the sample preparation procedures. The following diagram shows the set up of the instrument for this application:



This application uses two approaches. Approach 1 starts from evaporation of sample extracts, whereas approach 2 starts from the extraction of the sample. The column used for cleanup is graphitized carbon black (500 mg/3 mL), the elution solvent is acetone + dichloromethane (1:1), the solvent for extraction is acetone.

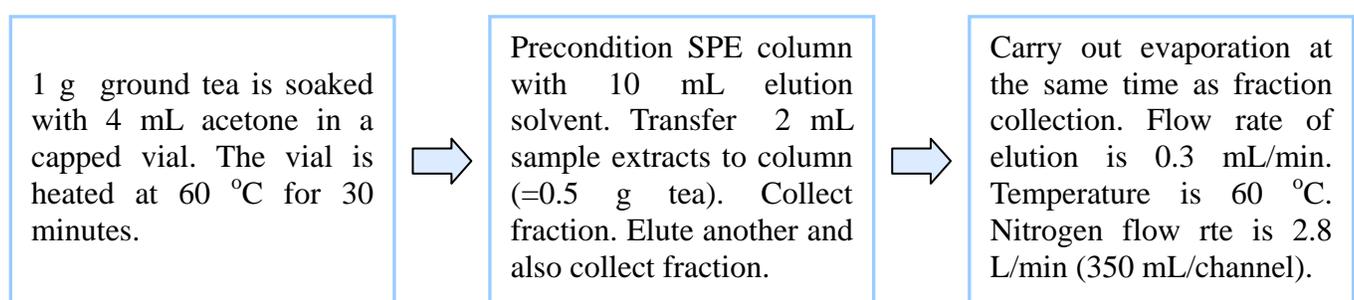
Procedures for approach 1:



SPE-03 method parameters for approach 1:

No.	Act	Flow rate	Volume	Remarks
1	Heat	60 (°C)	5.0 (min)	Pre heat at 60 °C for 5 minutes
2	Elute 1	8	10	Pre condition SPE column using 10 mL elution solvent
3	Evap F1	10(no use)	12 (min)	Evaporate sample extracts for 12 minutes
4	Clean 1	8	2.0	Add 2 mL elution solvent to sample vial
5	Add samp	8	0.1	Transfer the liquid in sample vial to column and collect the fraction.
6	Collect 1	8	2.5	
7	Elute 1	8	0.0	Connect to elution solvent and collect 10 mL fraction.
8	Collect 1	8	10.0	
9	Evap F1	10 (no use)	25	Evaporate the fraction for 25 minutes

Procedures for approach 2:



SPE-03 method parameters for approach 2:

No.	Act	Flow rate	Volume	Remarks
1	Heat	60 (°C)	30.0 (min)	Extract 30 minute with heat at 60 °C
2	Elute 1	8	10	Pre condition SPE column using 10 mL elution solvent
3	Add samp	8	0.2	Transfer 2 mL sample extracts to column and collect fraction/evaporation. The flow rate is 0.3 mL/min.
4	Con F2	8(no use)	2.0	
5	Elute 1	8	0.0	Elute with the elution solvent and continue collection and evaporation.
6	Con F2	8(no use)	10.0	

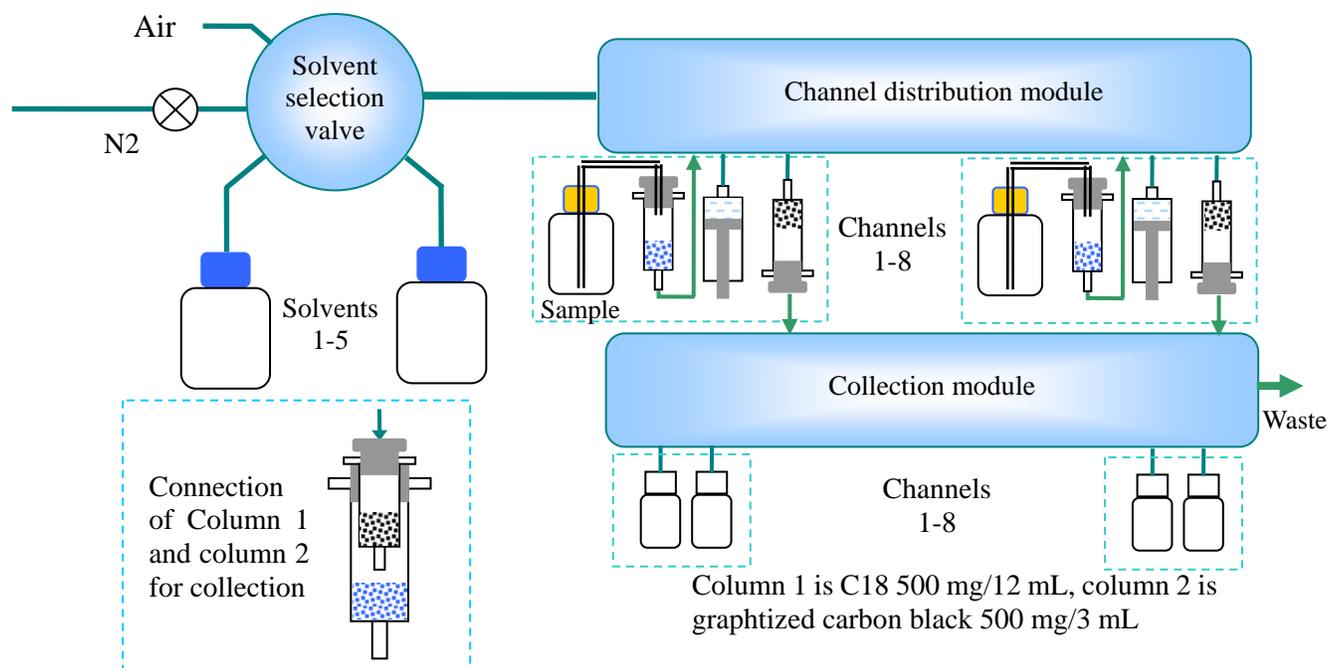
In a recovery test for approach 1, 5 ug methyl parathion was added to 5 mL tea extracts (equivalent to 0.5 g tea). In recovery for approach 2, 2 ug methyl parathion was added to 1 g ground tea before starting the SPE processing. The processed sample was analysed by HPLC. For both case, the recovery is above 95%.

The time for processing 1 batch samples (8 samples) is 50 minutes for approach 1 and 80 minutes for approach 2. In case of manual approach, the time used on column cleanup is similar to automated approach. The major difference is with the evaporation procedures. It normally needs 30 minutes for 1 sample.

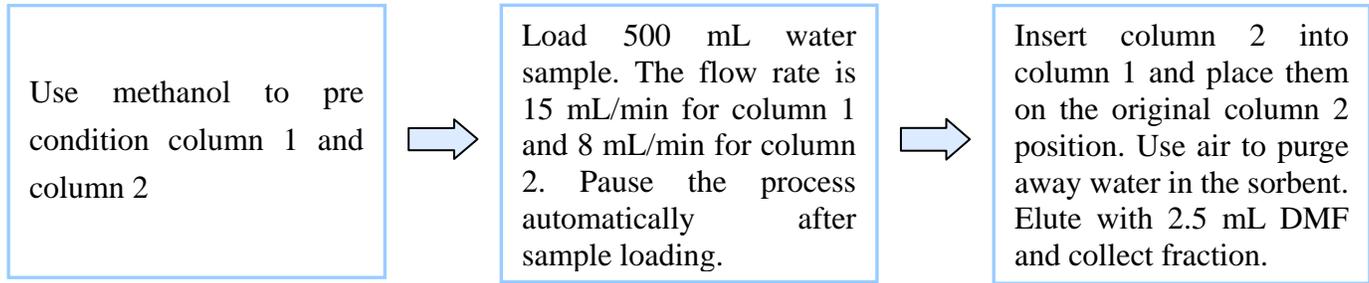
3.2 Simultaneous extraction of pollutants of various properties in large volume water samples

In analysis of large volume water samples, the pollutants to be monitored can have vary different properties. One sample may need to be processed several times to have all these pollutants extracted. This application shows a new method for simultaneous extraction of pollutants with very different properties. The targeted compounds used are phenathrene, methyl parathion, caffeine, and benzidine.

It uses a C18 column (column 1) to trap compounds of low polarity and a graphitized carbon black column (column 2) to trap compounds of mid and high polarity. The difference of this approach with other two-column-in-series methods is that the column 1 is placed in the inlet of loading pump and column 2 is placed in the outlet of the pump. Water sample is first drawn into column 1, and then pushed into column 2. Among the advantages of this arrangement over the conventional two column methods are: 1) much lower resistance in liquid delivery and more tolerance to blockage on columns; 2) Particles and sticky compounds (such as benzo pyrene and PCBs) are retained by column 1. They will not cause damage to the syringe pump and valves. It also avoid cross contamination. The following diagram shows the set up of the instrument:



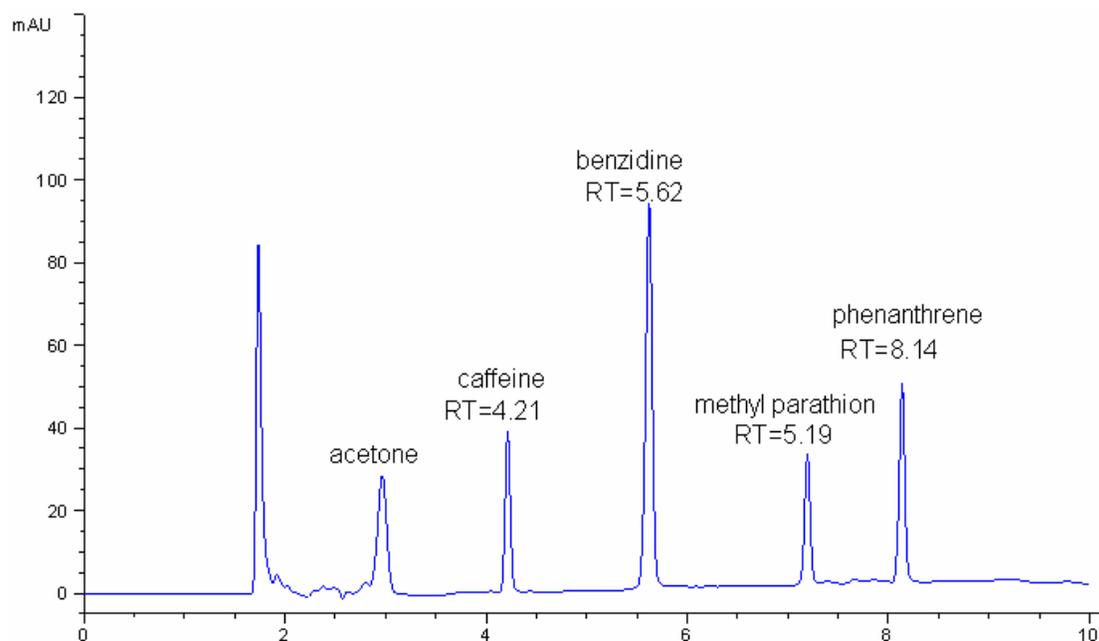
Below is the procedures for the extraction:



SPE-03 method parameters:

No.	Action	Flow rate	Volume	Remarks
1	Rinse 1	10	10.0 (min)	Use solvent 1 (methanol) to rinse columns 1 and 2
2	Add sam	8	500.0	Load 500 mL sample
3	Pause	8(no use)	0.0(no use)	Pause automatically. After replacing the two columns, press the "Pause" button to resume processing.
4	Elute 2	8	10.0	Use water to wash columns 1 and 2
5	Blow air	10	10.0	Use air to purge away water in columns
6	Elute 3	6	0.1	Change elution solvent to solvent 3 (DMF) and collect 2.5 mL fraction
7	Collect 1	6	2.5	

The collected fractions are analysed by HPLC. HPLC conditions: Instrument, Agilent 1100 with a quaternary pump and a VWD detector; mobile phase A, 15 mM ammonium acetate solution; mobile phase B, acetonitrile; gradient program, increase B from 10% to 95% over a 6 minute duration, hold for 2.5 minutes, then reduce B to 10% over a 1.5 minute duration; flow rate, 1.5 mL/minute; column, PCT-C18 4.6x250 mm with 5 um particle; detection wavelength, 276 nm; injection volume 20 uL.



Graphitized carbon black is a very unique sorbent. In addition to the capability of a C18 sorbent for compounds of low polarity, it can also trap compounds of mid and high polarity. As the adsorption of graphitized carbon black is too strong, a larger volume solvent is needed to elute out all the trapped analytes. To avoid this problem, elution from a reversed direction is often used. A limit of graphitized carbon black is with molecules of large conjugated system, such as PAHs. These compounds will be very difficult to come out once they are trapped by graphitized carbon black, even with elution from a reversed direction.

In the present application, analytes of low polarity (include PAHs) are trapped by a C18 column. Only analytes with mid and high polarity will enter the graphitized carbon black column.

For a recovery test, four compounds were added to tap water: caffeine, 10 ppb; benzidine, 20 ppb; methyl parathion 10 ppb; phenanthrene, 10 ppb. To protect benzidine from decomposition by the bleaching reagent, 20 mg sodium thiosulfate was added to tap water before addition of benzidine.

Among the four compounds, caffeine, methyl parathion, and phenanthrene all give recovery above 95%. The recovery of benzidine is only 18-20%. Further analysis indicates that methyl parathion and phenanthrene are all trapped by C18 column, caffeine is mainly trapped by graphitized carbon black with 5-7% trapped by C18 column, benzidine is trapped around 10% by each column. The low recovery of benzidine is due to its strong ionization. It may be improved by using ion pair reagents or add an ion exchange sorbent to the C18 column (or a mixed mode column).

Using two columns in this arrangement does not increase the liquid transfer resistance. Making the operation more tolerant to blockage by the particles from the samples. When it is necessary, a third column may be added after column 2. By using elution in reversed direction, a 2.5 mL solvent can recover all the trapped analytes. A further concentration is thus avoided.

4. Specifications

Sample capacity	4, 6, or 8 per batch
Volume of sample	1 to 4000 mL
Material of wetted parts	Teflon, 316 stainless steel, Pyrex glass
System control	Microcontroller with keypad data entry and/or computer
Method	Permanent storage of three methods with instrument
Method functions	Pre condition, load sample, elution with 5 solvents, wash of sample line, drying with nitrogen, fraction collection.
Waste handle	Collect organic waste and aqueous waste separately
Pump flow rate	0.5 to 60 mL/min
Pressure limit of pump	6 bar
Pump reproducibility (C.V.%)	<1.5
Power consumption	< 3.0 A at 24 VDC
Evaporation temperature	Ambient to 90 °C
Minimum evaporation volume	0.5 mL
Weight	12.5 Kg
Dimension (cm)	34 x 34 x 45 (width x depth x height)

4. Order information

Part No.	Description	Price (US\$)
SPE-03-01	Includes 4-channel SPE-03 mainframe, 24V power supply, and user manual.	
SPE-03-02	Includes 8-channel SPE-03 mainframe, 24V power supply, and user manual.	
SPE-03-03	Includes 6-channel SPE-03 mainframe, 24V power supply, and user manual.	
MOD-001	Evaporation module	



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