PromoChrom Technologies

SPE-03 Multi-Channel Cleanup Station

For Automatic Sample Preparation in Trace Analysis

About PromoChrom

PromoChrom Technologies focuses on the development of sample preparation solutions for trace analysis. Since year 2005, PromoChrom has developed SPE-01 cleanup station, SPE-03 cleanup station, SPE-04 online/offline SPE's, LC-04SP valve system and SPE-06 mini SPE. Each of the instruments target specific applications.
In 2011, PromoChrom developed the flow-path-integration technique for liquid handling. It combines various switching valves into one liquid handling module. The technique simplified 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 relieve 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). SPE-03 is available in several configurations to meet the needs in budget and sample throughput. If the evaporation option is added, SPE-03 will come with a front cover as shown below.

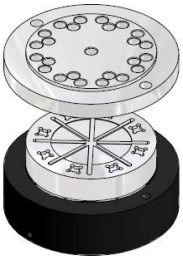
SPE-03
with evaporation function

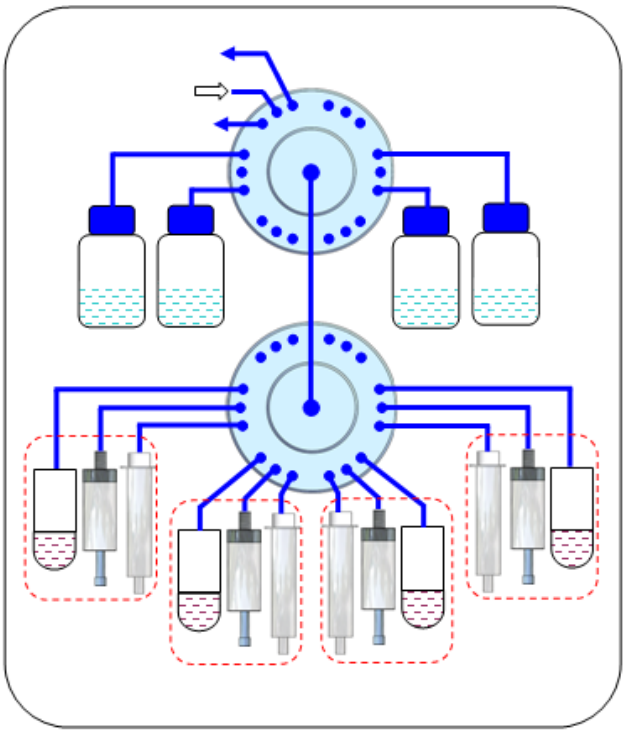
1. Working principle of SPE-03

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

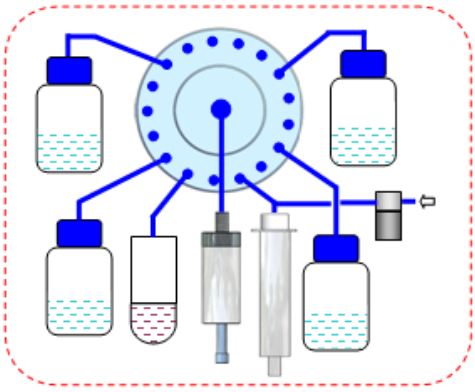
SPE-3 uses two multi-functional valves, regardless of number of channels.

PromoChrom's multi-functional valve is based on the flow-path-integration technique. The function of one such valve is equivalent to several normal stream selection valves and isolation valves. Our valves are made of ceramic to resist damage from particles. Below shows one of the multi-functional valves.





Multi-channel SPE systems from other brands normally use several valve modules as shown below. Each module consists of one stream selection valve and one isolation valve. Every channel has one of these modules. Because of the module complexity, a multi-channel SPE system is heavy (around 40 KG) and limited in channel count. Very few can have more than 6 channels.



Module for one channel in a typical multi- channel SPE

Valve components used in a four channel SPE.

PromoChroms's SPE-03 SPE systems have 8 channels and only weigh 12 KG. Using only two special valves, SPE-03 can not only perform normal SPE cleanup, but also do evaporation of samples and collected fractions.

2. Features

2.1 Small footprint and simple structure due to flow-path-integration technique

Normal automated SPE instruments involve many switching vales and complex tubing connections. A multi-channel SPE is normally heavy and of large size. The tubing connection is also complex, making maintenance difficult. The weight and size of SPE-03 are less than half of other multi-channel SPE instruments. For an 8-channel SPE-03, the weight is only 12 kg. A simplified structure also reduces the chance or error and simplifies routine maintenance.

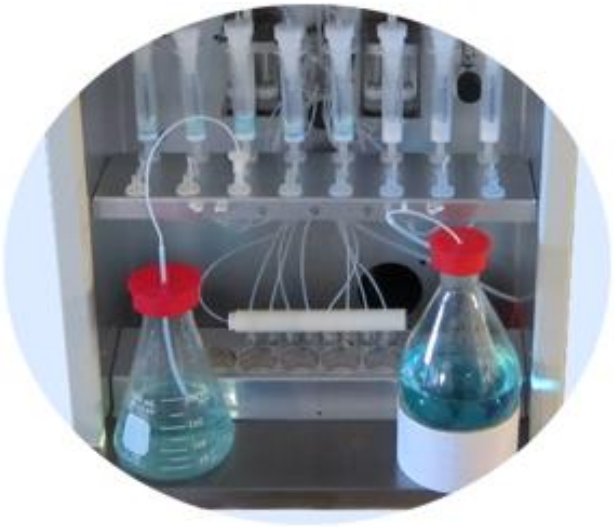
2.2 Column blockage detection and smart handling

The system can detect the blockage of SPE column and reduce the flow rate accordingly. If blockage continues, the instrument will pause to wait for the user to step in.

2.3 Use various SPE columns and containers for fractions

SPE-03 uses 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 suitable for disk extractions as well.

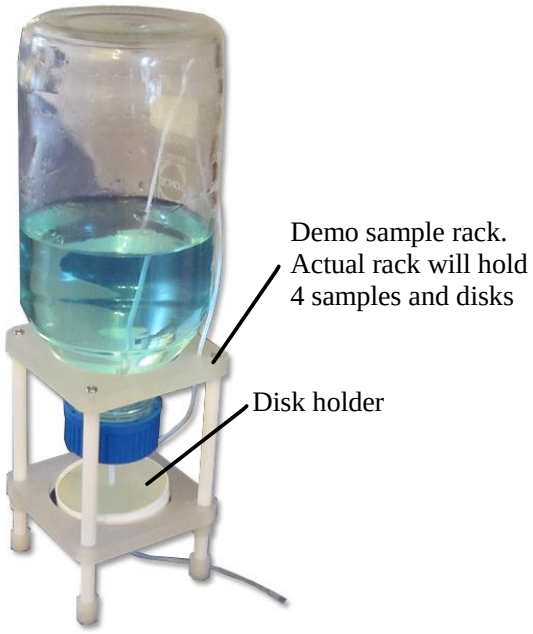
The instrument can also use containers of different shapes for sample and fraction collection. These containers can be capped while the instrument is processing samples. It minimizes solvent evaporation. Thus the work can be carried out without using a fume hood.



2.4 Versatile applications for both SPE cartridges and SPE disks

SPE-03 comes with two sets of tubing for sample loading. The longer and wider tubing is used for large volume water samples, while the 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.

SPE-03 can use both cartridges and disks for large volume water samples. The default configuration is for cartridges only. A disk kit (MOD-003) is required for each channel that is using SPE disks. The kit includes a 47mm disk holder, a cap adapter for connecting sample bottle with the disk holder, and necessary tubing for connecting the disk with SPE-03 instrument. Sample racks (MOD-004) are for mounting disk holders and sample bottles. Each rack is capable of mounting 4 sample bottles and disk holders. An 8-channel system would need 2 racks and 8 disk kits.



For disk operation, SPE-03 can perform activation of the membrane, sample loading, sample bottle cleaning, washing and drying of the membrane, and fraction collection. Adding the disk function does not affect use of SPE cartridges.

2.5 Automatic washing of sample tubing and container

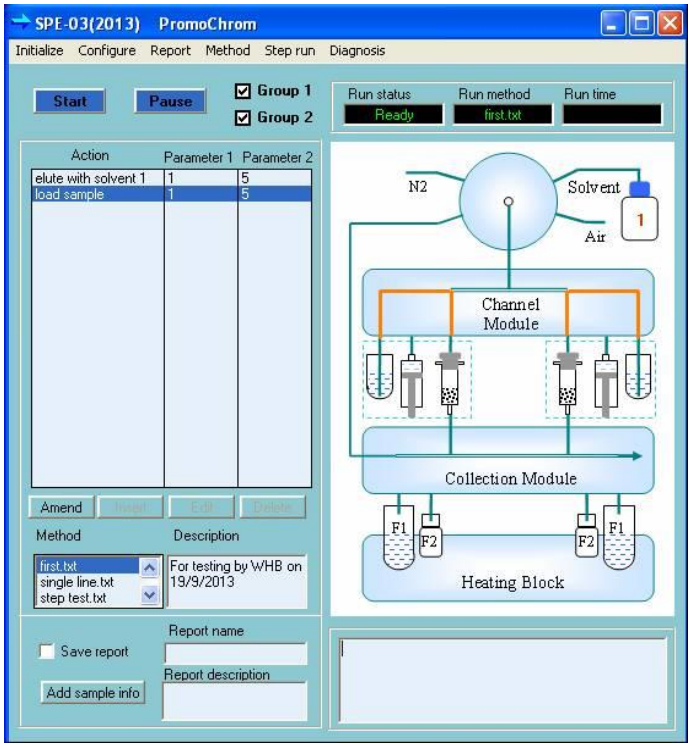
To prevent cross-contamination, function for cleaning of sample tubing is built into the methods. Two solvents may be used to wash the tubing and container. The rinsate may be left inside the container or added back to the columns.

2.6 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 to below 0.5 mL. After evaporation, more solvent can then be manually added using the vial markers as reference. In addition to evaporation of fraction, the evaporation module can also evaporate sample extract before loading it to the column and can even carry out heat assisted sample extraction.

2.7 User-friendly computer software (optional)

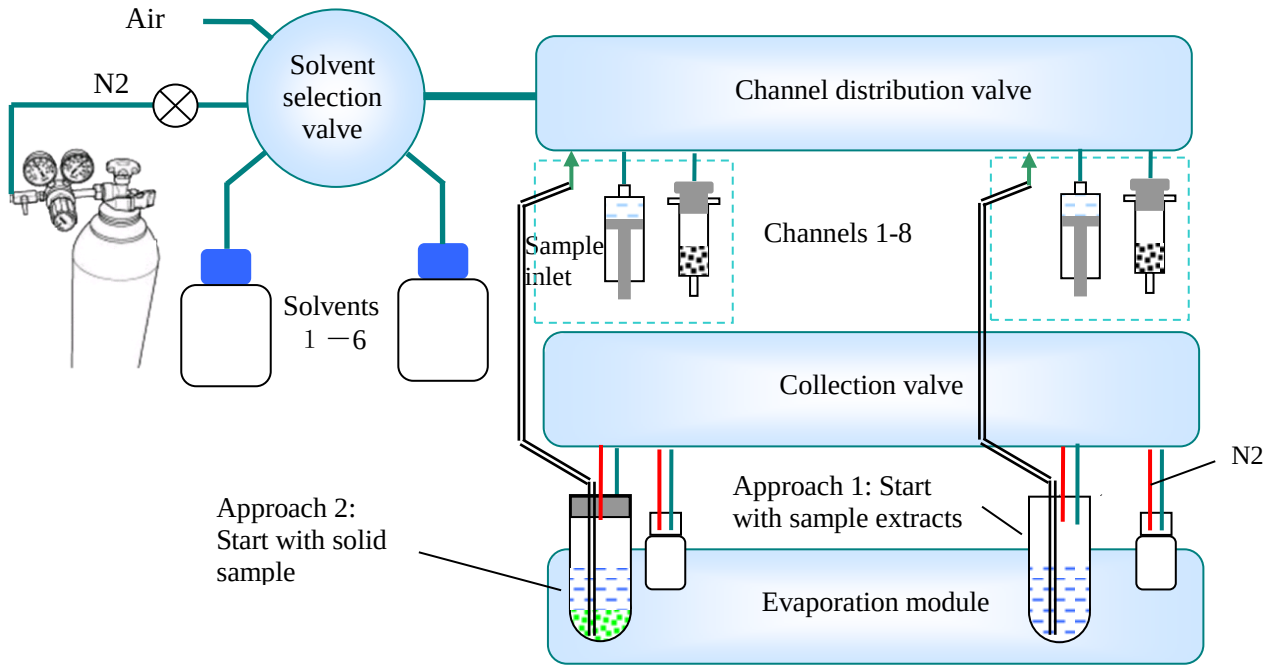
SPE-03 can be controlled using a computer when many methods are used for routine work. The control software has a graphical interface to indicate real time status of the instrument. Among the helpful features are step run function for step by step method development and diagnosis tool for trouble shooting.


SPE-03(2013) PromoChrom
Initialize Configure Report Method Step run Diagnosis
Start
Pause
Group 1
Group 2
Run status
Ready
Run method
first.txt
Run time
Action
Parameter 1
Parameter 2
elute with solvent 1
1
5
load sample
1
5
Amend
Insert
Edit
Delete
Method
Description
first.txt
single line.txt
step test.txt
For testing by WHB on 19/9/2013
Save report
Report name
Report description
Add sample info
N2
Solvent
Air
1
Channel Module
Collection Module
F1
F2
F2
F1
Heating Block

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 steps: 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.



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

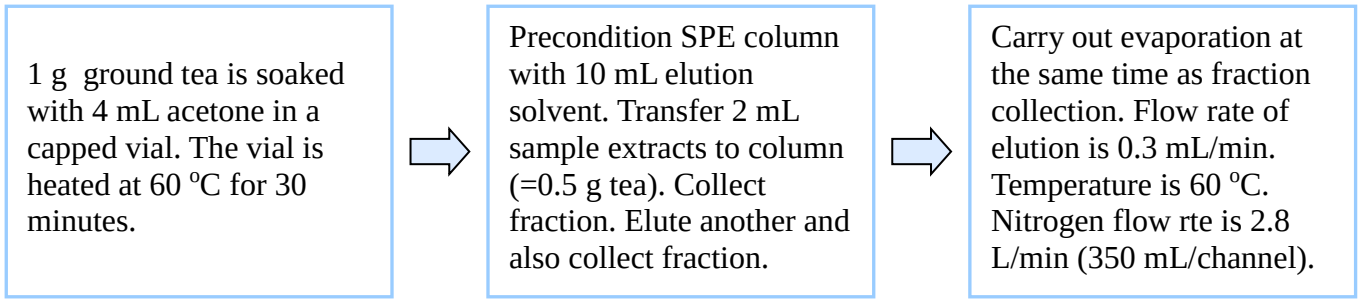
Procedures for approach 1:

Evaporate 5 mL tea extracts (equivalent to 0.5 g tea) to near dry. Evaporation condition: Preheat 5 minutes at 60 °C, then start nitrogen at 2.8 L/min for 12 minute.	Precondition SPE column with 10 mL elution solvent. Use 2 ml elution solvent to rinse the sample vial and add the rinsate to column. Continue elute and collect 12 mL fraction	Evaporate the fraction to near dry. Re-dissolve the fraction with 0.5 mL methanol to make it ready for HPLC analysis (Toluene may be used if the analysis is by GC).

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	

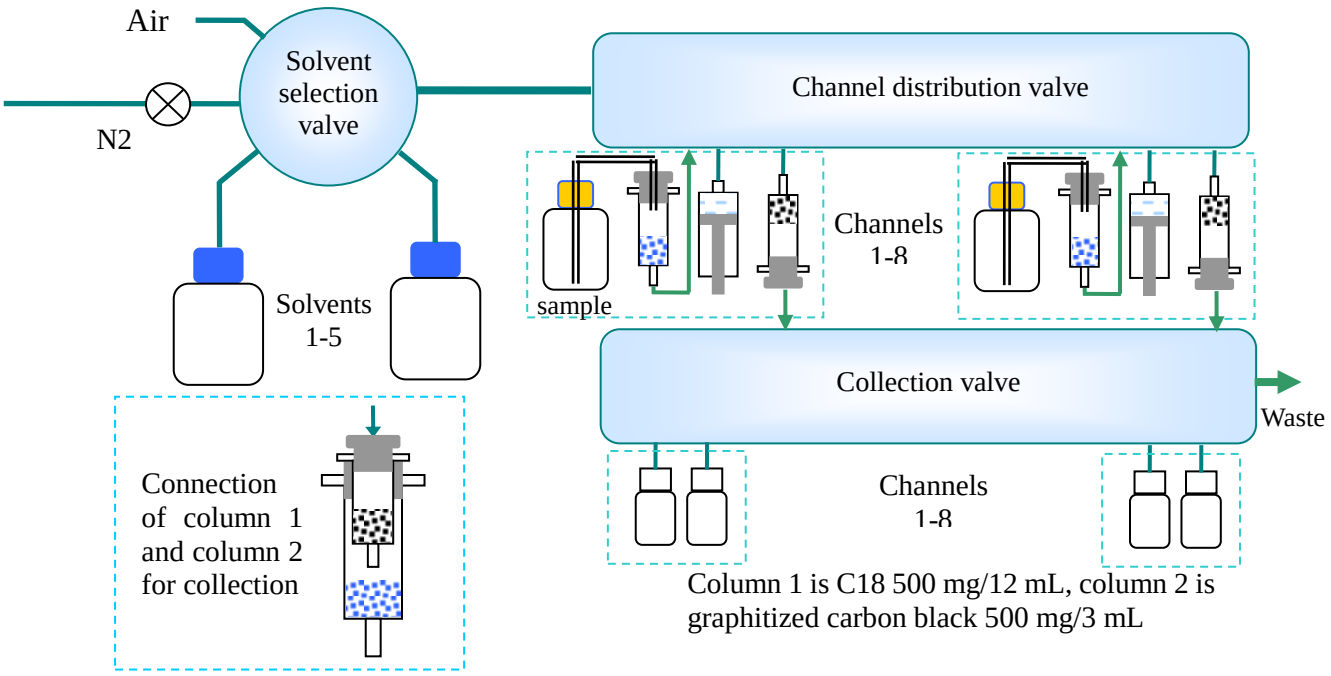
In a recovery test for approach 1, 5 ug methyl parathion was added to 5 mL tea extracts (equivalent to 0.5 g tea). For approach 2, 2 ug methyl parathion was added to 1 g ground tea before starting the SPE processing. The processed sample was analyzed by HPLC. For both cases, the recovery is above 95%.

The time for processing 1 batch of samples (8 samples) is 50 minutes for approach 1 and 80 minutes for approach 2. In the 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 takes 30 minutes for 1 sample.

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

In the analysis of large volume water samples, the pollutants to be monitored can have very 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 avoids cross contamination. The following diagram shows the set up of the instrument:



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 sample	8	500.0	Load 500 mL sample
3	Pause	8(not used)	0.0(not used)	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	

Below is the procedure for the extraction:

Use methanol to pre-condition column 1 and column 2

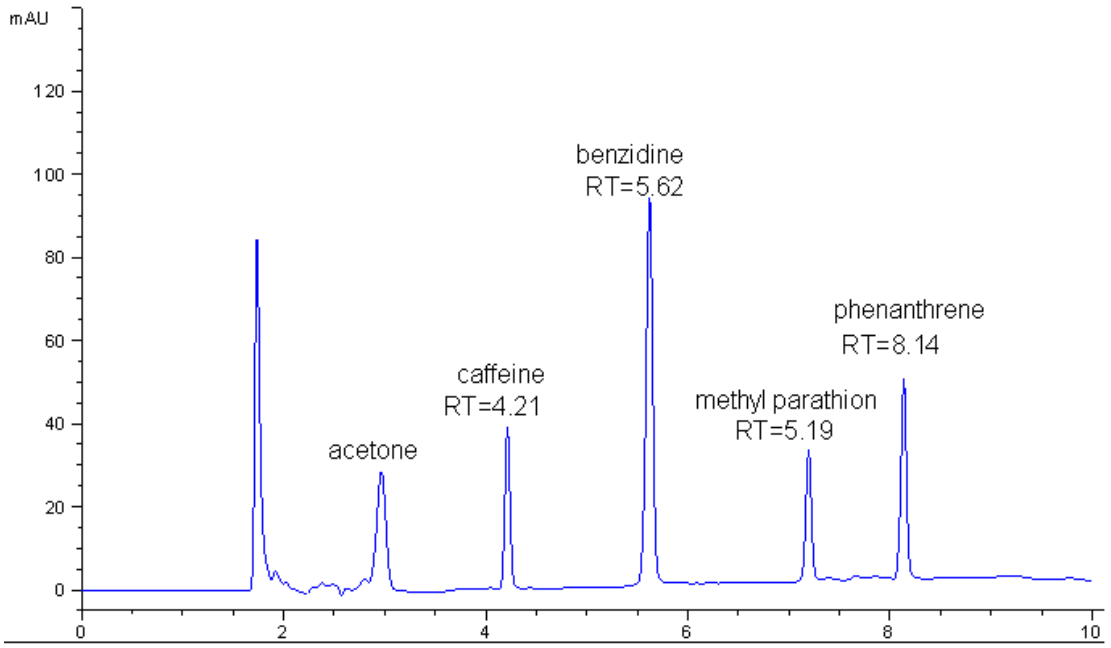
→

Load 500 mL water sample. The flow rate is 15 mL/min for column 1 and 8 mL/min for column 2. Pause the process automatically after sample loading.

→

Insert column 2 into column 1 and place them on the original column 2 position. Use air to purge away water in the sorbent. Elute with 2.5 mL DMF and collect fraction.

The collected fractions are analyzed 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 collect 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

Parameters	SPE-03
Sample capacity	4, 6, or 8 per batch depending on configuration
Volume of sample	0.5 to 4000 mL
Material of wetted parts	Teflon, 316 stainless steel, Pyrex glass
System control	Microcontroller with keypad data or computer
Types of solvent	6
Method functions	Pre-condition, load sample, elution with 6 solvents, wash of sample line, dry column with nitrogen, fraction collection.
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 80 ^{o}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
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
MOD-002	Software for computer control
MOD-003	Disk kit for using 47mm SPE disks, includes disk holder, sample bottle adapter and tubing. One disk kit is required for each channel.
MOD-004	Sample and disk rack, holds 4 sample bottles and disks per rack

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