

Application

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Extraction/Concentration Apparatus Saves Time and Ensures High Recovery of Semivolatiles from Water, for Capillary GC

An apparatus designed and made by Supelco performs both the semivolatiles extraction and concentration steps. This saves equipment set-up and clean-up time, eliminates a transfer step, and allows recovery of the extraction solvent for proper disposal. Detector responses for extracted and concentrated analytes and analytes directly injected onto the capillary GC column, at the same concentrations, were nearly identical. The apparatus also provided nearly 90% recovery of the extraction solvent.

Key Words:

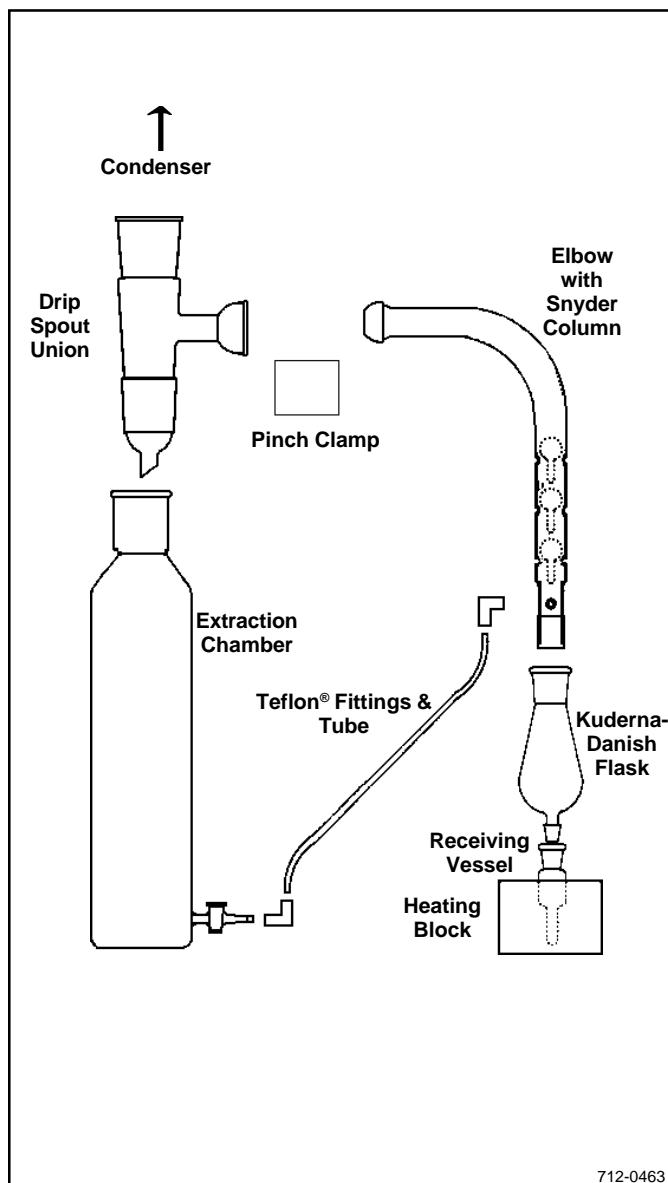
- semivolatiles
- water analyses
- sample extraction
- sample concentration

US Environmental Protection Agency (EPA) methods for semivolatiles analysis (e.g., Method 625 for wastewater, SW-846 Method 8270 for hazardous waste, Contract Laboratory Program Statement of Work) involve extracting 1 liter samples of water with methylene chloride, using a separatory funnel or continuous extraction, followed by concentration of the recovered compounds using a Kuderna-Danish apparatus. According to SW-846 Method 3520, continuous liquid-liquid extraction may be used as an alternative procedure when separatory funnel extractions might lead to emulsion formation and poor analyte recovery. To maximize analyte recovery, EPA CLP methodology now requires that continuous extraction be used for semivolatiles analysis, in place of separatory funnel extractions.

An apparatus designed and made by Supelco (Figure A) combines the extraction and concentration steps in one device. This saves set-up and clean-up time, eliminates a transfer step, and allows recovery of the extraction solvent for proper disposal. A heating block has been designed to be the heating source for the extractor/concentrator. The heating block is especially advantageous when long heating times (e.g., overnight) could allow a water bath to dry.

Supelco's R&D chemists evaluated the extraction/concentration apparatus for use in US EPA methods. They added 0.5mL each of our Acids Surrogate Spike Mix (Cat. No. 4-8875) and our Base-Neutrals Surrogate Spike Mix (Cat. No. 4-8925) to 1 liter water samples, then extracted the samples overnight (18-24 hours) with 500mL of methylene chloride. A single extraction procedure, with no sample pH adjustment, was used. The following day, the stopcock in the extractor/concentrator was closed and the extract was concentrated to 10mL. The heating block concentrates a sample in about the same time as by water bath, 15-40 minutes. Recovery rates for the spiked analytes were

Figure A. Supelco™ Extractor/Concentrator



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Table 1. Recovery of Surrogate Standards, Using Various Extraction/Concentration Apparatus

Surrogate Compound (Conc.)	Retention Time (min.)	Apparatus [#]				CLP Limits
		1	2	3	4	
2-Fluorophenol (100µg/mL)	3.2	95.0	122*	94.9	112*	21-110
Phenol-d ₆ (100µg/mL)	6.1	97.5	133*	96.0	113*	10-110
Nitrobenzene-d ₅ (50µg/mL)	7.3	94.8	123*	96.0	108	35-114
2-Fluorobiphenyl (100µg/mL)	10.6	92.3	122*	96.2	111	43-116
2,4,6-Tribromophenol (50µg/mL)	13.0	95.6	169*	93.7	107	10-123
4-Terphenyl-d ₄ (50µg/mL)	16.8	92.8	130	103	119	33-141

1 – Supelco extractor/concentrator with Supelco heating block
 2 – Other extractor/concentrator with Supelco heating block
 3 – Kuderna-Danish concentrator with Supelco heating block
 4 – Kuderna-Danish concentrator with water bath

Column: PTE™-5 QTM, 15m x 0.53mm ID, 0.5µm film
 Oven: 40°C (4 min) to 280°C at 15°C/min
 Carrier: helium, 46cm/sec
 Det.: FID (350°C)
 Inj.: 1µL, splitless (0.75 min purge, 250°C).

*Value exceeds EPA CLP limits

Extracted/concentrated samples – 1L water plus 0.5mL Acids Surrogate Spike Mix (Cat. No. 4-8875) and 0.5mL Base-Neutrals Surrogate Spike Mix (Cat. No. 4-8925), extracted in 500mL methylene chloride and concentrated to 10mL.
 Directly injected samples – 10mL methylene chloride plus 0.5mL Cat. No. 4-8875 and 0.5mL Cat. No. 4-8925.

determined by comparing peak areas on a capillary gas chromatogram to those for directly injected analytes. These values also were compared to recovery rates for alternative extraction/concentration apparatus. Values for directly injected analytes and for extracted and concentrated analytes, at the same concentrations, were virtually identical (Table 1).

In addition to analyte recovery, we monitored the level of the extraction solvent in both the extraction chamber and the concentration apparatus before extraction, after extraction, and after concentration. Table 2 summarizes the data for the after-extraction and after-concentration steps, based on an initial volume of 500mL of methylene chloride. The Supelco unit provided good average recovery of the extraction solvent, 86%. Solvent loss occurs during both extraction and concentration, but is more likely during extraction because of the longer time involved.

We concluded that by combining the extraction and concentration steps the device saves time, provides analyte recovery rates acceptable for EPA methodology, and ensures good recovery of the extraction solvent. Multiple-part construction makes cleaning easy and minimizes the chance of breakage. Relative to a

water bath, the heating block appears to be a superior means of heating the receiving vessel, especially when overnight extraction could allow a water bath to dry.

If you are following US EPA methodology for extracting semivolatiles from water samples, we highly recommend the Supelco Extractor/Concentrator. The apparatus is complete as shown in Figure A, including both the condenser and the heating block. The only additional equipment required is a hot plate for the heating block. Alternatively, you can use our Stead/Therm™ Powered Heating Block with the extractor/concentrator. Replacement parts are available; parts of the extractor/concentrator are compatible with our continuous liquid/liquid extractor (Cat. No. 6-4769).

Table 2. Recovery of Extraction Solvent After Using Extraction/Concentration Apparatus

Apparatus	After Extraction (mL)	After Concentration (mL)	Recovery (%)
Supelco	470	430	86
	—	430	86
	—	450	90
	400	410	82
	—	420	84
			86 (mean)
Other	—	380	76
	380	380	76
	410	390	78
	430	400	80

Ordering Information:

Supelco Extractor/Concentrator Complete as shown in Figure A	64807-U
Heating Block	64811-U
Stead/Therm Powered Heating Block	
120 VAC	64814
220 VAC	64830
PTE-5 QTM Fused Silica Capillary Column 15m x 0.53mm ID, 0.5µm film	25355

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, FAX 814-359-5468) for expert answers to your questions.

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