



High Throughput Microwave Assisted Solvent Extraction of Semi-Volatile Organic Compounds Using US EPA Method 3546

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Abstract

Conventional extraction techniques with shakers, Soxhlet, or sonication for the preparation of environmental organic samples are time consuming, generate large volumes of solvent waste, and expose workers to toxic organic solvents for extended periods of time. Pressurized solvent extraction uses less solvent, but can only process six samples per hour and is limited by sample size. Microwave assisted solvent extraction (MASE) is a proven technique that is fast, uses significantly less solvent than conventional techniques, and is cost-effective. For these reasons, MASE is an approved procedure for the extraction of semi-volatile organic compounds from soil, sediment, clay, sludge, and solid waste as listed in US EPA Method 3546.

Most environmental laboratories that follow US EPA Method 3546 will prepare and analyze several hundred samples per week. These laboratories require instrumentation that is more sophisticated and user-friendly for higher throughput. The recent introduction of MARSXpress Plus vessels with disposable glass liners meets their needs for user-friendly and high throughput instrumentation as the system can process up to 24 samples simultaneously.

The MARSXpress Plus vessels with disposable glass liners were utilized in the extraction of semivolatile organic compounds from spiked soils following US EPA Method 3546. The solution containing the extracted compounds was analyzed with a GC-MS. The spike recovery results are discussed.

Instrumentation

The MARS 6 with MARSXpress Plus vessels and disposable glass liners processes up to 24 samples simultaneously. The MARSXpress Plus vessel is an easy-to-assemble, high throughput

vessel with a disposable glass liner. The glass liner is easily removed and replaced after each extraction run. The temperature of the vessels is monitored and controlled with Contactless All-Vessel IR Temperature Sensors.



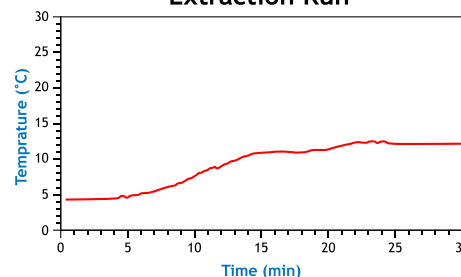
Analytical Procedure

A 20 gram sample of lake soil and a 20 gram sample of clay soil were added to the glass liner of a MARSXpress Plus vessel with a 300 ppb spike solution containing aldrin; alpha-chlordane; hepta-chlor epoxide; 4,4'-DDE; and benzene 1,2,3,5-tetrachloro-4,6-dimethyl. The glass liner was inserted in the MARSXpress Plus vessel, the plug was inserted, the cap was tightened, and the samples stood for 60 minutes. After 60 minutes, the vessels were uncapped, 30.00 mL of acetone/hexane (1:1) was added, the vessels were capped and tightened, and the vessels were inserted in the turntable. Each of the samples and blanks was prepared in triplicate. A One Touch extraction method was created in the MARS 6. Samples were cooled to room temperature and the supernatant was transferred to vials with a pipet. Samples were refrigerated until injected on an Agilent 7890A Series Gas Chromatograph with an Agilent 5975 Series Mass Selective Detector.

Extraction Conditions			
	Ramp Time (minutes)	Hold Time (minutes)	Extraction Temperature (°C)
MARS 6 with MARSXpress Plus Vessel and Disposable Glass Liner	20	10	115

Results

Extraction Run



MARS 6 with MARSXpress Plus Vessel and Glass Liner Spike Recovery Results of 300.0 ppb Semivolatile Organic Compounds

	Benzene, 1,2,3,5-tetrachloro-4,6-dimethyl	Aldrin	Heptachlor Epoxide	Alpha Chlordane	4,4'-DDE
Lake Soil					
Average	271.50	265.41	312.94	285.78	300.96
% Recovery	90.50	88.47	104.31	95.26	100.32
RSD	11.48	10.00	13.49	9.96	9.40
Clay Soil					
Average	281.80	271.91	278.60	290.33	295.27
% Recovery	93.93	90.64	92.87	96.78	98.42
RSD	5.52	3.54	8.70	5.50	6.67

Discussion

The extraction run had a controlled ramp to 115 °C and the temperature was stable during the 10 minute hold at 115 °C in accordance with EPA guidelines.

All solvent remained in the glass liner - there was no solvent between the glass liner and the Teflon® vessel, therefore no sample was lost during extraction.

The precision and accuracy of the recovery data of semivolatile organic compounds with the MARS 6 and MARSXpress Plus vessel and disposable glass liners are comparable to or better than techniques such as Soxhlet, pressurized solvent extraction, sonication, and shakers.

A 20 gram sample was used for the extraction. However, the disposable glass liners can be used to extract up to a 30 gram sample for most soils.

Conclusions

Microwave assisted solvent extraction with the MARS 6 and MARSXpress Plus vessels:

- Use much less solvent as compared to conventional techniques

- Extract semivolatile organic compounds from 24 different soil samples in less than 45 minutes

- Is a very simple system to use with the easiest to assemble vessels on the market

- Do not need cleaning as the glass liners are disposed after each run

- Achieve accuracy and precision values better than conventional techniques and pressurized solvent extraction

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