Investigation of Carryover or Cross-Contamination in the Thermo Scientific Dionex ASE 200 Accelerated Solvent Extractor System

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Key Words
Accelerated Solvent Extraction, ASE, Polyaromatic Hydrocarbons, Polychlorinated Biphenyls, U.S. EPA Method 3545A

Goal
To demonstrate that the accelerated solvent extraction technique is exhaustive and does not result in carry over or cross contamination in-between samples.

Executive Summary
Accelerated solvent extraction is a sample preparation technique that uses elevated temperature and pressure to increase extraction efficiency in solid and semi-solid samples. This technique significantly reduces the amount of time and solvent required for extraction when compared to traditional techniques such as Soxhlet. The accelerated solvent extraction technique ensures a high degree of reproducibility by running each sample individually under the preset method conditions. This sequential mode of operation uses a common pathway to collect the extracts and carry-over or cross contamination was investigated to ascertain the viability of this technique for multiple samples processed in a single batch. Two sets of extractions were run on soil and sediment contaminated with PAHs (up to 1,500 mg/kg) and PCBs (up to 3,700 µg/kg) and carryover was not observed. These results demonstrate that the accelerated solvent extraction technique is exhaustive and all compounds will be removed from the common extract pathway when using an optimized extraction method.

Introduction
Accelerated solvent extraction (ASE) is an innovative sample preparation technique that combines elevated temperatures and pressures with liquid solvents to achieve fast and efficient removal of analytes of interest from various matrices. With accelerated solvent extraction technique, extractions can be done in very short periods of time and with minimal amounts of solvent as compared to conventional sample extraction techniques like Soxhlet or sonication. For example, 10 g samples can be completely extracted in less than 15 min with less than 15 mL of solvent. Accelerated solvent extraction technique has been demonstrated to be equivalent to existing extraction methodologies such as Soxhlet and automated Soxhlet for most RCRA (Resource Conservation and Recovery Act) analytes from solid and semisolid samples. It meets the requirements of U.S. EPA Method 3545, Pressurized Fluid Extraction.

With the small amount of solvent used relative to the sample size, carryover or cross-contamination could be potential concerns with the accelerated solvent extraction technique and the Thermo Scientific™ Dionex™ ASE™ 200 Accelerated Solvent Extractor. Two sets of experiments were conducted to investigate these concerns. The experiments performed included the extraction of heavily loaded soil and sediment samples followed by extracting blank samples and the determination of target analytes in both extracts.

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Instrumentation

- Dionex ASE 200 system*
- Thermo Scientific™ Dionex™ DX 500 HPLC system with AD20 (UV detector) and fluorescence detector
- Gas chromatograph (GC) with electron capture detector (ECD)

*Equivalent or improved results can be achieved using the Dionex ASE 150 or 350 systems

Experimental

In the first experiment, 7 g samples of a highly contaminated soil (SRS100-103, Fisher Scientific, 11 wt% solvent extractable) were extracted at 17.2 MPa (2500 psi), 100 °C with toluene/methanol (1:1), with 5 min heat-up and 5 min static times. Analyses of the extracts were done by HPLC using UV at 254 nm and fluorescence at 325 nm excitation and 410 nm emission. In this case, the target analytes were polycyclic aromatic hydrocarbons (PAH). After the extraction, the cell was removed and replaced with a cell containing clean sand. A second extraction was performed using the same conditions. These steps were repeated with three separate new samples to determine reproducibility. No rinsing of the instrument was performed between the extractions.

The second experiment was conducted using Standard Reference Material (SRM) 1939 (river sediment from the National Institute of Standards and Technology (NIST), Gaithersburg, MD). This sample has a high level of coextractable components (about 30 wt%) in addition to the PCBs for which this material is certified. In this experiment, 8 g of the sediment were extracted with hexane/acetone (1:1) at 100 °C and 17.2 MPa (2500 psi) with 5 min heat-up and 5 min static periods.

After the extraction, a second extraction was performed on the same sample and collected in a separate vial. This process was repeated with three samples. No rinsing of the instrument was performed between the extractions. The analysis of the extracts was performed by GC with ECD. These experiments were performed to determine not only if material remained in the tubing of the instrument, but to determine the completeness of the extraction of the sample in the cell itself.

Results and Discussion

The experiments on the contaminated soil were performed to determine if any material remained and was not completely flushed from the tubing and valves that are in line with the extraction cell. Carryover was determined by comparing the total area counts from the samples to those obtained from the blanks for the UV and fluorescence detectors on the HPLC analysis method for PAH.

The results are shown in Table 1. These are the percentages of the total area seen in the first extraction of the sample that were obtained from the extract of the empty cell. These results show that an insignificant level of carryover exists. It should be noted that it is unlikely that this carryover was due to analytes because the recovery of the PAH was quantitative compared to the certified values. No PAH were detected in second extracts or blanks (minimum detectable quantity (MDQ) was 0.2 mg/kg, and the certified concentration of the PAHs ranged from 31 to 1500 mg/kg).

In the experiments with the river sediment sample, the total area counts (minus the solvent blank) of the first extract were compared to those of the second. The results are summarized in Table 2. This small amount of material in the second extract was actually residual coextractable material because the recovery of the target compounds, PCBs, was quantitative compared to the certified values. No PCBs were detected in the second extract (MDQ = 0.1 µg/kg, and the concentration of the analytes ranged from 180 to 3700 µg/kg). This indicates that the lines are cleaned out well, and complete extraction is achieved with these small volumes of solvent.

The Dionex ASE 200 system has low dead-volume, which translates into no cross-contamination between samples. It can be programmed to perform automatic rinses between samples to allay concerns of cross contamination between samples. Extensive testing for this was also conducted as part of the EPA equivalency studies. Accelerated solvent extraction is an exhaustive extraction technique, and when quantitative extraction occurs, no analytes are left in the sample for subsequent extractions.

Table 1. Carryover of PAH in the Dionex ASE 200 system after extraction from contaminated soil.

	HPLC with UV Detection	HPLC With Fluorescence Detection	Acclerated Solent Extraction Recovery vs Certified Values
PAH Carryover n=3	0.024%	0.13%	106% Recovery 5.5% RSD

Table 2. Carryover of PCBs in the Dionex ASE 200 system after extraction from contaminated river sediment.

	GC Analysis with ECD	Acclerated Solent Extraction Recovery vs Certified Values
PCB Carryover n=3	2.6%	102% Recovery 4% RSD

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