PSE fast

APPLICATIONS

#619

Pressurized Solvent Extraction (PSE) of Arsenic Compounds from Carrots

Introduction

Dietary sources and drinking water provide the major pathways to human arsenic exposure. Since drinking water mainly contains inorganic forms of arsenic, which are toxic, it is an environmental and regulatory concern to determine the chemical forms of arsenic in food of terrestrial origin where toxic arsenic compounds have been found in the water supply. Because of the variable toxicity levels of arsenic compounds found in foods, total arsenic determinations alone do not provide an adequate assessment of risk. In order to accurately determine dietary risk, it is necessary to achieve a quantitative extraction of individual arsenic species. Unfortunately, conventional methods of extraction are labor intensive and consume large volumes of organic solvent.

Pressurized solvent extraction is a new technique that reduces solvent consumption and sample preparation time. Solvent is pumped into an extraction vessel containing the sample and is heated and pressurized. The pressurized solvent at high temperature accelerates the extraction process by increasing the solubility of the analyte in the solvent and also increasing the kinetic rate of desorption of the analyte from the sample matrix.

The *fast* PSE is an automated system which processes six samples simultaneously. The parallel processing technology of the *fast* PSE dramatically increases sample throughput compared to Soxhlet and pressurized solvent extraction systems that employ serial processing. In addition to rapid extraction times, significant reduction in solvent consumption is achieved.

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Pressurized solvent extraction can be used to replace Soxhlet and sonication techniques traditionally used to extract arsenic from solid foods, and is approved for use as EPA Method 3545A. This application describes the rapid pressurized extraction of arsenic compounds from freeze dried carrots for further speciation studies.

Equipment

- ✓ Applied Separations' *fast* PSE Pressurized Solvent Extractor
- ✓ 11 mL Extraction Vessels-Cat.#10625
  - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Analytical balance
- ✓ Blender or food processor

Solvents and Materials

- ✓ Water- ultra-pure 18 mΩ
- ✓ *Spe-ed*™ Matrix- Cat. #7950
- ✓ Collection Vials (60mL for extract collection)- Cat. #10650
- ✓ Disposable Syringe Filter, 0.45µm, nylon
- ✓ Ottawa Sand- Cat. #10548 (Acid washed for 4 HR with hot 50 % nitric acid and rinsed with water until neutral pH.)
- ✓ S/S Vessel Frits- Cat. #10710
- ✓ Cellulose Filter Disks – Cat. #10711

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Summary of Method

1. Prepare Sample

2. Load Sample

3. Run Sample

4. Collect Extract

Procedure

Prepare Sample

Prepare carrots by washing off dirt or debris from outer surface under running water. Remove carrot ends and any dark or rotten spots. Rinse carrots with water and then air-dry. Homogenize carrots in a blender or an equivalent product. Ensure that the sample is homogeneous before proceeding. Freeze dry sample and grind so that the sample passes through a 40 mesh screen. Failure to prepare a totally uniform sample may result in inconsistent results.

Load Sample

Prepare the extraction vessels for analysis by placing a cellulose filter disk in the bottom opening followed by a 10µm s/s frit, and secure them in place with a retaining nut. Mix 1 gram of dried ground carrots with 4 grams of acid washed Ottawa sand and load prepared sample into the vessel. Add clean Ottawa sand to within 1 cm of the top of the vessel's interior flange as directed by User's Manual.

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Place the extraction vessel into the instrument as described in the *fast* PSE operator's manual. Ensure that the pump is primed and that the extraction solvent is H_2O. Place a pre-cleaned collection vial in the instrument for each sample, and program the instrument using the following parameters:

Extraction Conditions

Program the following extraction parameters on the fast PSE

Program A Mode – 11 mL vessels

Solvent:	H_2O
Temperature:	100 ° C
Pressure:	100 Bar
Static:	1 minute
Solvent Module:	1*
Cycles:	3
Pause:	N=0
Flushing Program:	Solvent/gas/repeat flush: 1 min/2 min/0

***Note:** If automatic solvent selection module is used, enter the appropriate position number (i.e. 2, 3, or 4).*

Optimize the conditions as needed. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction vessel is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the samples. Any pressure in the range of 100 BAR should suffice. Once established, the same parameters should be used for all samples extracted for the same analysis type.

Collect Extract

Collect each extract in a clean 60mL vial. Allow the extract to cool after the extraction is complete. Before total arsenic determination can be achieved, the carrot extracts should be passed through 0.45µm nylon syringe filters.

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References

US EPA Method 3545A – Pressurized Fluid Extraction
Vela et al. *Analyst*, 2001,**126**,1011-1017.

Safety

The use of organic solvents, elevated temperatures, and high pressures present potential safety concerns in the laboratory. Common sense laboratory practices can be employed to minimize these concerns. However, the following sections describe additional steps that should be taken.

Extraction vessels in the *fast* PSE oven are hot enough to burn unprotected skin. Allow the vessels to cool before removing them from the oven, or use appropriate protective equipment (e.g. insulated gloves or tongs) as recommended by the manufacturer.

During the gas purge step, some solvent vapors may exit through a vent port in the instrument. Connect this port to a fume hood or other means to prevent release of solvent vapors to the laboratory atmosphere. This precaution also applies to the removal of post extraction solvent from the collected extract.

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