

The Pressurized Solvent Extraction (PSE) of Arsenic Compounds from Seaweed

Introduction

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.

Seafood is a major source of dietary arsenic exposure. One environmental and regulatory concern is the presence of arsenic compounds found in seaweed products, which are mainly consumed by the Asian Pacific subpopulations. The majority of arsenic found in seaweed is generally in the form of non-toxic organo-arsenic compounds, while toxic inorganic arsenic typically constitutes only a small component of the total arsenicals. Because of the variable toxicity levels of arsenic compounds found in foods, total arsenic determinations alone do not provide an adequate risk assessment. In order to accurately determine dietary risk, it is necessary to achieve a quantitative extraction of individual arsenic species. Without it, the extraction process may selectively remove the non-toxic species while leaving the toxic species undetected within the solid matrix.

Traditionally, arsenic compounds are determined by applying various solvent extraction methods including Soxhlet and sonication techniques. Pressurized solvent extraction can be used to replace the labor intensive Soxhlet and sonication procedures and is approved for use as EPA Method 3545A. In addition to significant time savings and solvent reduction, pressurized solvent extraction provides a more quantitative extraction of arsenic compounds from seaweed than conventional methods.

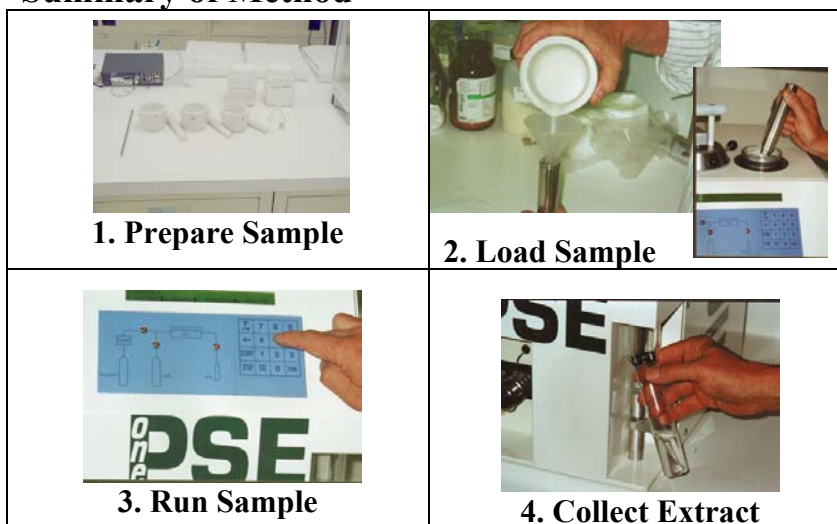
Equipment

- ✓ Applied Separations' Pressurized Solvent Extractor
 - *one* PSE or *fast* PSE
- ✓ 11mL Extraction Vessel- Cat. #10625
 - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Osterizer blender (for sample homogenization)

Solvents and Materials

- ✓ Methanol (HPLC grade)
- ✓ *Spe-ed*TM Matrix- Cat. #7950
- ✓ Cellulose Disk- Cat. #10711
- ✓ Collection Vials (60mL for extract collection)- Cat. #10650
- ✓ Ottawa Sand- Cat. #10548
- ✓ Vessel Frits- Cat. #10710
- ✓ Empore Filter Aid Glass Beads (Varian)

Summary of Method



Procedure

Prepare Sample

Prepare the extraction vessel(s) for analysis by placing a cellulose filter disk in the bottom opening followed by a 10µ s/s frit, and secure them in place with the retaining nut. Lyophilize seaweed samples in a freeze dryer. Homogenize the dried seaweed in an Osterizer blender or an equivalent product. Ensure that the sample is homogeneous before proceeding. Failure to prepare a totally uniform sample may result in inconsistent results. After homogenizing the samples, mix the seaweed with the glass beads as a dispersion media. Failure to do so could result in clogging the PSE vessel.

Load Sample

Load prepared sample into an 11mL vessel. Place the extraction vessel into the instrument as described in the *onePSE* operator’s manual. Place a pre-cleaned collection vial in the instrument for each sample. Ensure that the collection vial is sufficiently large to hold the extract.

Run Sample

Select the appropriate stored extraction program and start.

Extraction Conditions

Program the following extraction parameters on the one PSE

Program A Mode – 11mL vessel

Oven temperature:	22°C
Pressure:	100 BAR
Static time:	1 minute
Cycles	3
Solvent:	MeOH/H2O 30/70
Purge:	N=2
Flush program:	Solvent/gas/repeat flush: 20 sec/2min/0

Optimize the conditions as needed. 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. At the end of the extraction sequence, remove collection vial from the needle port and place vial in an evaporation apparatus. Remove solvent using low heat under a gentle stream of nitrogen. Analyze as required.

References

Gallagher et al. *Fresenius J Anal Chem* (2001) 369:71-80
US EPA Method 3545 – Pressurized Fluid Extraction

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 cells in the oven are hot enough to burn unprotected skin. Allow the cells 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.