

Extraction of Fat from Fresh Meat Products by Pressurized Solvent Extraction (PSE)

Introduction

Fat is traditionally removed from canned meat products using tested methods such as the Association of Official Analytical Chemists (AOAC) Method 960.39 (16th edition). This is a solvent-based method that employs a Soxhlet apparatus to extract fat from meat products. Depending on the reflux rate used, the method takes 4 – 6 hours, not including the 1 – 1.5 hour pre-extraction heating period.

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. Pressurized solvent extraction can be used to replace Soxhlet and sonication techniques and is approved for use as EPA Method 3545A.

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.

This application describes a procedure for the extraction of fat from fresh meat products using pressurized solvent extraction.

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#610





Equipment

- ✓ Applied Separations' *fast* PSE Pressurized Solvent Extractor
- ✓ 11 mL Extraction Vessels-Cat.#10625
 - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Solvent Concentration System
- ✓ Microwave Oven-for sample drying
- ✓ Analytical balance
- ✓ Mortars (90mm o.d. Coors #60316) and pestles (Coors #60317)

Solvents and Materials

- ✓ Petroleum ether (b.p. 35 - 60°C) or Hexane (ACS grade)
- ✓ Nitrogen-high purity grade to purge the extraction vessel.
- ✓ S/S Frits (10 micron)- Cat. #10710
- ✓ Collection Vials (60mL for extract collection)-Cat.#10650
- ✓ *Spe-ed*TM Matrix-Cat.#7950
- ✓ Cellulose Filter Disk-Cat. #10711
- ✓ Ottawa Sand – Cat. #10548

Summary of Method

 <p>1. Prepare Sample</p>	 <p>2. Load Sample</p>
 <p>3. Run Sample</p>	 <p>4. Collect Extract</p>

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#610

Procedure

Prepare Sample

Ensure that the meat product sample is homogeneous before proceeding with this method. Failure to prepare a totally uniform sample may result in inconsistent results. Place 2g of *Spe-ed* Matrix in a weighing dish. Accurately weigh a 1 – 1.5 (to 0.1mg) sample onto the *Spe-ed* Matrix already in the weighing dish. If the sample has a very low fat content (less than 2%), it may be advisable to adjust the ratio as follows: 4g *Spe-ed* Matrix and 2-3g meat product sample using a 22mL instead of an 11mL extraction vessel. Pour the weighed material carefully into the mortar and grind with the pestle until uniform. Scrape all particles from pestle into the mortar and set the pestle aside. Place only the mortar in a microwave oven and heat for four minutes at a high setting. When mortar cools, regrind mixture with pestle until smooth

Load Sample

Prepare the extraction vessels for analysis by placing a cellulose filter disk in the bottom opening followed by a 10 μ m s/s frit, and secure them in place with a retaining nut. Place adaptor and funnel in top opening. Pour the sample mixture through a funnel into the extraction vessel. Weigh out and add an additional gram of *Spe-ed* Matrix to the mortar. Swirl the material around in the mortar with the pestle to sweep up residual sample. Transfer this additional material to the extraction vessel and tap the vessel on the bench top to settle and compact the contents. Add clean Ottawa sand to within 1 cm of the top of the vessel's interior flange as directed in the User's Manual.

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 petroleum ether or hexane. Place a pre-cleaned collection vial in the instrument for each sample, and program the instrument using the following parameters:

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#610

Extraction Conditions

*Program the following extraction parameters on the fast PSE
Program B Mode – 11 mL vessels*

Solvent:	Petroleum Ether
Temperature:	125 ° C
Pressure:	100 Bar
Static:	1 minute
Solvent Module:	1*
Refills/Volume:	7/2
Pause:	N=0
Flushing Program:	Solvent/gas/repeat flush: 1min/ 2min/ 0

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

Collect Extract

Collect each extract in a clean 60mL vial. Allow the extract to cool after the extraction is complete. When collection vials are cool, remove from the *fast* PSE and place vials in an evaporation apparatus. Remove solvent using low heat under a gentle stream of nitrogen. Dry vial in oven set at 100°C for 1 hour. Weigh dried vial, calculate the percent recovery, and report result.

Results

Example:

Extraction of Fat from Fresh Meat Products

Several types of fresh meat products were selected for analysis by the *fast* PSE method and for comparison with an official method. The fat content of these samples ranged from 0.85 - 29%. The 11mL vessels were used for most of the samples and the sample sizes ranged from

1.0 – 3.0g. (In one set of samples the 22mL vessels were used, and in this case the sample size was increased to 3g.) The samples extracted in the Soxhlet apparatus ranged from 3.0 – 3.5g. The data obtained by the *fast* PSE method were compared directly with AOAC Method 960.39. This comparison of the two methods is shown in the following table:

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#610

Percent Fat Recovery from Fresh Meat Products *fast* PSE vs. Soxhlet

Mean \pm s.d.

Sample	<i>fast</i> PSE	n	AOAC 960.39	n
Frankfurter	29.59 \pm 0.15 (day 1)	3	29.46 \pm 0.26	3
	29.68 \pm 0.21 (day 2)	3		
Sausage	26.18 \pm 0.48	5	26.12 \pm 0.48	3
Ground Beef	24.63 \pm 0.32 (11mL)	3	23.42 \pm 0.42	3
	24.87 \pm 0.12 (22mL)	3		
#1 Turkey Breast	1.54 \pm 0.09	3	1.49 \pm 0.39	3
#2 Turkey Breast	1.44 \pm 0.06	5	1.07 \pm 0.10	3
#3 Turkey Breast	0.85 \pm 0.05	6	0.65 \pm 0.03	3

In the above table, note that 3 – 6 determinations were made for each set of samples extracted on the *fast* PSE while only sets of 3 samples were extracted on the Soxhlet apparatus, yet for every type of meat sample analyzed, the means for the *fast* PSE sets were higher and the standard deviations (s.d.) lower than those obtained by the official method, indicating the repeatability possible with the *fast* PSE technique. The amount of solvent used for each *fast* PSE extraction was about 20mL in 11mL vessels compared with 200 – 220mL for the Soxhlet technique.

To determine day-to-day method variability, a frankfurter sample was analyzed on subsequent days on the *fast* PSE. Note the agreement obtained between these two sets. A test also was performed to determine the agreement between sets of ground beef samples analyzed on the same day in both 11mL and 22mL extraction vessels. The results for both sets were in close agreement with each other, although the sample sizes and total solvent volumes varied considerably.

The above *fast* PSE method for fresh meats employs the same solvent, petroleum ether or hexane, as the Soxhlet method; however it differs in notable ways, such as faster sample preparation, shorter extraction times, and significantly lower solvent consumption.

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#610

References

US EPA Method 3545A – 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 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.