

## **Extraction of Fat from Canned 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 (16<sup>th</sup> 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 may take 4 – 6 hours.

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 canned meat products using pressurized solvent extraction.

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#609





## Equipment

- ✓ Applied Separations' *fast* PSE Pressurized Solvent Extractor
- ✓ 11 mL Extraction Vessels-Cat.#10625
  - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Microwave Oven-for sample drying
- ✓ Evaporation Apparatus
- ✓ 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*<sup>TM</sup> Matrix-Cat.#7950
- ✓ Cellulose disk-Cat. #10711
- ✓ Ottawa Sand-Cat. #10548

## Summary of Method

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

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#609

## Procedure

### *Prepare Sample*

Place 2 g of *Spe-ed* Matrix in a weighing dish. Accurately weigh a 1 – 1.5 (to 0.1 mg) 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: 4 g *Spe-ed* Matrix and 2-3 g meat product sample using a 22 mL (Applied Separations Cat.#10626) instead of an 11 mL 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. Ensure that the meat product sample is homogeneous before proceeding with this method. Failure to use a totally uniform sample may result in inconsistent results. 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 $\mu$ 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. Place a pre-cleaned collection vial in the instrument for each sample. 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:



#609

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### *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 vials from the fast PSE and place in an evaporation apparatus. Remove solvent using low heat under a gentle stream of nitrogen. Dry vials in oven set at 100°C for 1 hour. Weigh dried vials, calculate the percent recovery, and report result.

## Results

### *Example:*

#### **Extraction of Fat from Canned Meat Products**

Three sample types of canned meat products were selected for analysis by the fast PSE method and for comparison with an official method. The three types were canned fresh pork and canned dog and cat foods. The sample sizes used for these determinations by the fast PSE method ranged from 1 – 1.5 g while those used with the Soxhlet ranged from 3 – 3.5 g. No difficulties were encountered in obtaining good repeatability using such small samples in the fast PSE 11mL vessels. The data obtained by the fast PSE method were compared with those using AOAC Soxhlet method 960.39.

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#609

A comparison of the *fast* PSE and the Soxhlet methods for the canned meat products is shown in the following table:

## Percent Fat Recovery from Canned Meat Products PSE vs. Soxhlet

Mean  $\pm$  s.d. (RSD)

Sample	<i>fast</i> PSE	n	AOAC 960.39	n
Fresh Pork	17.73 $\pm$ 0.17 (0.90)	5	17.80 $\pm$ 0.16 (0.90)	3
Cat food (turkey & giblet)	7.14 $\pm$ 0.08 (1.1)	6	7.13 $\pm$ 0.11 (2.50)	3
Dog food (gravy with chicken)	3.11 $\pm$ 0.04 (1.29)	6	2.80 $\pm$ 0.04 (1.29)	3

The amount of solvent used for each *fast* PSE extraction was about 20 mL compared with 200 – 220 mL for the Soxhlet technique. For the above table, 5 – 6 determinations were made for each sample by the *fast* PSE method whereas only 3 determinations were performed on the same sample by the Soxhlet method. In spite of the larger number of meat samples in each set, the *fast* PSE technique gave consistently the same or lower RSDs than those obtained by the official Soxhlet apparatus. The means for each sample also compared favorably with the official method indicating the accuracy and repeatability possible with the *fast* PSE.

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

### References

US EPA Method 3545A – Pressurized Fluid Extraction

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#609

## 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.