

Total Extractable Fat Using Pressurized Liquid Extraction (PLE)



Introduction

Regulating fat content is essential to maintaining the quality of food and feed products. The food industry extracts total fat from food samples to analyze for Priority Organic Pollutants (POPs). For several compounds of concern like PCB and PBDE congeners, results are often expressed in amount for gram of lipid.

Traditionally performed by Soxhlet extraction, fat determination is a time consuming process requiring large volumes of solvent and lengthy extraction times. By using pressurized liquid extraction, the entire extraction process can be performed in under an hour resulting in higher throughput.

The following application details the use of the FMS, Inc PLE system for the extraction of total fat from a variety of sample matrices.

Instrumentation and Consumables

- FMS, Inc. PLE system
- FMS, Inc. SuperVap™ Concentrator
- FMS, Inc. 200 mL direct-to-vial concentrator tubes
- Mettler Toledo analytical balance
- FMS, Inc PLE extraction cells (10, 20 40 mL)
 - *Alloy 20 cells required for Hydrolysis

Consumables

- Fisher Pesticide Optima* n-Hexane
- Agilent Hydromatrix
- FMS PLE re-usable end caps

Procedure

Weigh extract collection vials
 Sample aliquots are weighed
 Samples dried with Hydromatrix
 Dried samples transferred are into PLE extraction cells

PLE System

1. Cells filled with n-Hexane
2. Cells pressurized to 1500 PSI
3. Cells heated to 140 °C for 20 minutes
4. Cells cooled and flushed with n-Hexane (equal to cell volume)
5. Cells purged with N₂ into SuperVap Concentrator

SuperVap Concentrator

1. Preheat temp: 20 minutes at 60 °C
2. Evaporation mode: 60 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts to total dryness

*Evaporator tubes rinsed with N-Hexane to ensure no collected material sticks to evaporator tube walls.

Post evaporation, weigh collection vials and subtract the pre-weight values.





Results

Table 1: Fat calculations for PLE extracts

Sample	Mean Percent Lipid	RPD	Expected Lipid %	Percent Difference
Animal Feed 1	6.22%	1.0%	6.27%	-0.7%
Animal Feed 2	3.94%	1.3%	4.01%	-1.9%
Peanut Butter	50.04%	4.8%	50%	0.1%
Almonds				
Non-Dairy Creamer	2.31%	2.7%	2%	14.2%
Parmasean Cheese	24.37%	1.3%	25%	-2.6%
Rolled Oats	5.69%	2.4%	6.25%	-9.4%
Lard	97.7%	0.5%	100%	-2.1%
	Percent Lipid		Expected Lipid %	
NIST 1946	10.05%		10.17%	-1.2%
Fish Tissue				

Conclusions

Recovered fat calculations yielded values that are close to expected values. RPDs between duplicates demonstrate good reproducibility between the performed extractions. The addition of the NIST 1946 reference material provided a certified fat value for QC of the extraction batch.

When comparing the PLE extraction (< 1 hour total extraction time) to a Soxhlet extraction lasting 16-24 hours, the value of adopting PLE extractions becomes quickly evident. With the demonstrated diversity of matrices capable of being extracted, the PLE is a clearly superior option for high throughput facilities performing fat extractions.

For more information contact FMS at:
 FMS, Inc.
 580 Pleasant Street
 Watertown, MA 02472
 Phone: (617) 393-2396
 Fax: (617) 393-0194
 Email: onlineinfo@fms-inc.com
 Web site: fmsenvironmental.com