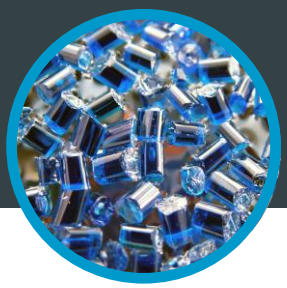
Application Note

Extraction of Antioxidants from High Density Polyethylene (HDPE) By Pressurized Liquid Extraction (PLE®)



Introduction

Plastics and other polymers are used in a wide spectrum of applications from industrial usage to packaging, including the packaging of food and pharmaceutical products. To enhance functionality, chemical additives are often added to polymers. These additives enable polymers to be more durable, pliable and make them less subject to degradation. To ensure that the specified amounts of an additive or combination of additives are incorporated into a polymer, after the extrusion process, a rapid and accurate analytical method is required to measure additives and other process by-products.

This application note demonstrates the use of Pressurized Liquid Extraction using the FMS PLE® system to extract polymer additives for quatitative analysis by HPLC. Four specific additives of known concentrations were extracted from high density polyethylene (HDPE) and the recoveries measured.

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Instrumentation and consumables

- FMS, Inc. PLE® System
- Thermo Certified GC/MS auto sampler vials
- FMS 5 mL Stainless Steel PLE Cartridge
- FMS Universal Stainless Steel PLE End Caps

Reagents

- Fisher Optima* 2-Propanol
- Fisher Optima* Cyclohexane

Sample preparation and PLE procedure

Sample Prep

1. A sample of high density polyethylene was milled to a fine particle size to increase surface area.
2. Duplicate samples of 2 grams each were weighed out and transferred to FMS 5 mL extraction cells and sealed with stainless steel re-usable end caps.
3. Samples were loaded on FMS PLE® system.

Pressurized Liquid Extraction

Solvents Mix: 2-Propanol/Cyclohexane (95:5)

Solvent pump rate: 35 mL/min

Pre-heat temperature: 100 °C

Extraction temperature: 120 °C

Extraction time: 20 minutes

Cycles: 3

Total time: 1.5 hours

FMS, Inc. PLE® and SuperVap®

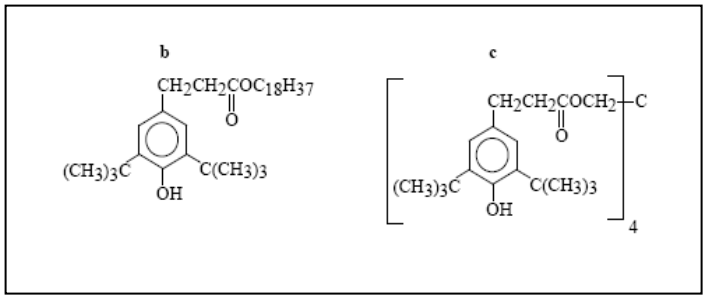
FMS
Fluid Management Systems

Results

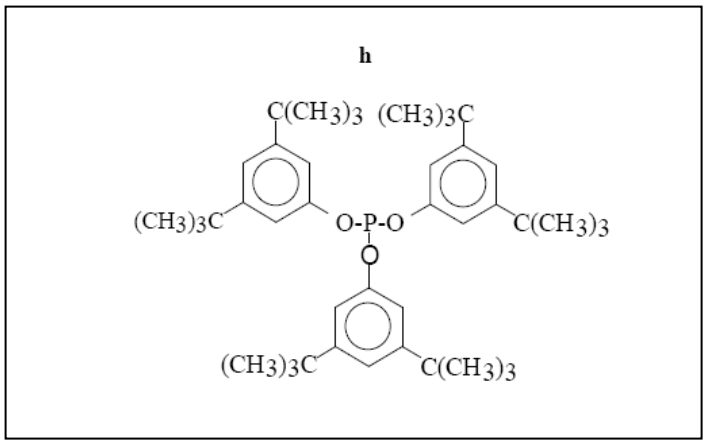
Table 1: Dual extractions of HDPE

	Cell #1	Cell #2	RSD
Erucamide®	70.80%	71.60%	0.712
Irganox® 1010	81.30%	81.00%	0.8115
Irganox® 1076	81.20%	80%	0.806
Irgafos® 168	116.60%	115.60%	1.161

Molecular formulas of two hindered phenols extracted. b) Irganox® 1076 & c) Irganox® 1010



Molecular formula for aromatic phosphite extracted, h) Irgafos® 168



Conclusions

Analysis of the four antioxidant additives in the HDPE samples demonstrates the FMS PLE system's ability to generate excellent recoveries for all four compounds (70-130%). The low relative RPDs also demonstrate the ability to generate consistent, reproducible extraction efficiencies between runs.

The advantages of Pressurized Liquid Extraction over traditional Soxhlet extractions are faster analyses (1.5 hours versus 15 hours) and reduced solvent volume (up to 200 mL). This Pressurized Liquid Extraction technique using the FMS PLE extraction system is an ideal method for extracting antioxidant additives from HDPE.

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