

Extraction of Additives from Plastics



Summary

The EDGE® automated extraction system is the most advanced extraction system available for the extraction of additives from plastics. The EDGE combines pressurized fluid extraction and dispersive solid phase extraction to dramatically reduce the sample preparation time and the potential of human error. The extraction is fast, simple, and repeatable in under 20 minutes, including sample cooling and system washing.

Extraction of polypropylene and polyethylene were performed with the EDGE, using sonication per ASTM methods D6043-09 and D6953-11. Recovery results are published and discussed.

Introduction

It is important to know the composition of plastic materials from both quality and safety perspectives. Plastic materials are a staple in our world and we have contact with these materials on a daily basis. From packaging to cell phone cases, to medical devices, plastic materials are everywhere. Plastics performance can be affected based on the amount of antioxidants and erucamide slip additives present. As manufacturers and consumers, it is important that we have confidence in the quality of our plastic products. Furthermore, from a safety perspective, it is important to ensure that no contaminants are leaching from these materials to which we are continuously exposed. The ability to extract additives from plastics with a quick and simple process is crucial to the plastics industry.

The extraction of additives from plastics is difficult for a number of reasons. The notably low melting point of the plastic makes it difficult to extract in a heated system. The plastic must be transformed to extract, but not melted. Reaching that balance can be challenging. Furthermore, some traditional extraction techniques, such as soxhlet and sonication, can be time-consuming and require large volumes of solvent.

The EDGE is capable of producing a cooled extract that is ready for analysis in less than 20 minutes, using no more than 40 mL of solvent. The EDGE is also capable of a single or dual-solvent wash that cleans the system and mitigates risk of carryover.

Materials and Methods

Reagents

Polypropylene (P/N 427888) and polyethylene (P/N 427799) were sourced from Sigma Aldrich. ASTM D6042-96 Calibration Mix was sourced from Restek and used as the standard to calibrate and spike the samples. Tinuvin P (P/N 533203) was sourced from Sigma Aldrich and used as the internal standard. Isopropanol was used as the sonication, extraction, rinse and wash solvent.

Sonication Method

Either polypropylene or polyethylene (1 g) spiked with 0.7 mL of 50 ppm standard was sonicated in 20 mL of isopropanol for 1 hour. The extract was then brought to dryness and reconstituted in 900 μ L of isopropanol with 100 μ L of the internal standard. The sample was filtered through a 0.45 μ m syringe prior to analysis.

Sample Preparation

Either polypropylene or polyethylene (1 g) was weighed into an assembled Q-Cup® containing a M1 Q-Disc® on the bottom with a C9 Q-Disc on top. The samples were spiked with 0.7 mL of 50 ppm standard. The Q-Cups were placed in the EDGE removable rack (each with a 40 mL clear glass collection vial) and the rack was slid into position on the EDGE. The CEM approved EDGE method for additives in the respective plastic was run. The extract was then brought to dryness and reconstituted in 900 μ L of isopropanol with 100 μ L of the internal standard. If analysis is performed within ~30 minutes, additional filtration is not necessary. However, if additives begin to precipitate out, the sample can be filtered through a 0.45 μ m syringe prior to analysis.

EDGE Methods

Additives in Polypropylene

Q-Disc: M1 + C9
Solvent: Isopropanol
Top Add: 30 mL
Bottom Add: 10 mL
Rinse: 0 mL
Temperature: 80 °C or 120 °C
Hold Time: 15 min
Wash 1: 15 mL Isopropanol
Wash 2: 15 mL Isopropanol

Additives in Polyethylene

Q-Disc: M1 + C9
Solvent: Isopropanol
Top Add: 30 mL
Bottom Add: 10 mL
Rinse: 0 mL
Temperature: 90 °C
Hold Time: 15 min
Wash 1: 15 mL Isopropanol
Wash 2: 15 mL Isopropanol

Analysis

A portion of 10 μ L of each sample was injected into a Waters Acquity UPLC with a PDA detector for analysis. A Restek Ultra C8, 5 μ m, 150 x 4.6 mm column with a flow of 1 mL/min and a 16 min ramp from 25% A (water) and 75% B (acetonitrile) to 100% B. The absorbance at 230 nm and the standard addition method was used for quantification.

Results and Discussion

The EDGE efficiently extracted the additives from both polypropylene and polyethylene in under 20 minutes, including sample cooling and system washing. **Figure 1** is a representative HPLC chromatogram showing clean separation of the additives of interest. **Table 1** contains the retention times and purpose of the standard compounds, which are common plastic additives. **Table 2** shows the recovery data for the extraction of additives from polypropylene on the EDGE at two temperatures.

The data for the extraction of polypropylene at both 80 °C and 120 °C is present in **Table 2**. It can be seen that no data was available for the extraction of BHT at 120 °C. BHT is known to be labile at higher temperatures and a lower temperature must be used to extract BHT. Conversely, the higher temperature was needed to extract the erucamide. The extraction recovery for the remaining compounds was comparable at both temperatures. Depending on the analyte of interest, different temperatures may be optimal.

Table 3 shows the recovery data for the extraction of additives from polypropylene on both the EDGE and via sonication. **Table 4** shows the recovery data for the extraction of additives from polyethylene on both the EDGE and sonication. It is clear that the EDGE performed better for polypropylene and polyethylene for all compounds. The EDGE not only offered the benefits of automation, but also yielded better results.

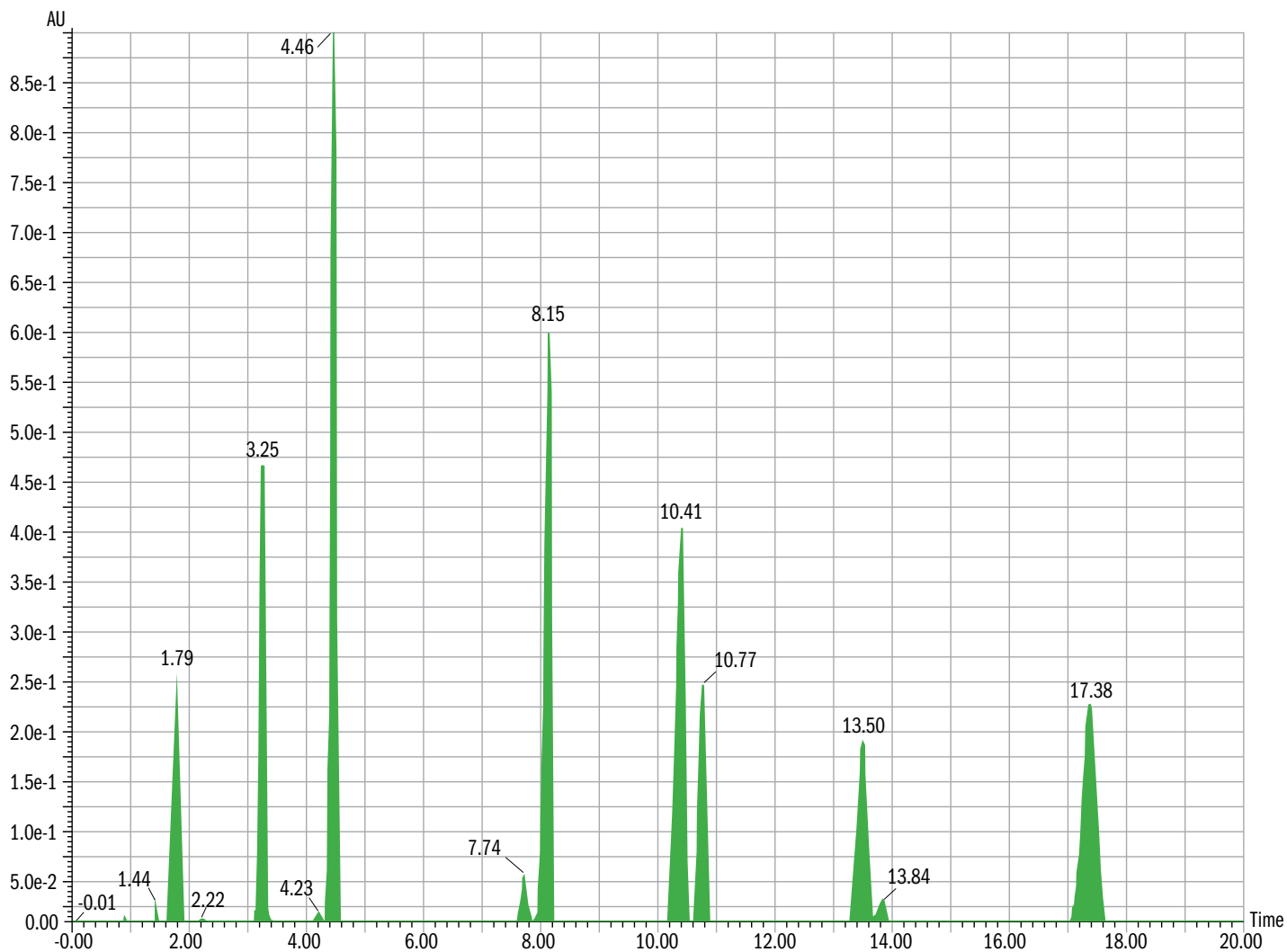


Figure 1: HPLC Chromatogram of Additives of Interest

Table 1: Standard Compound Information

Standard Compound	Retention Time (min)	Purpose
Tinuvin	3.3	Internal Standard
BHT	4.5	1° Antioxidant
Erucamide	7.8	Slip Agent
Irganox 3114	8.2	Antioxidant
Vitamin E	10.5	Antioxidant
Irganox 1010	10.8	1° Antioxidant
Irganox 1076	13.5	1° Antioxidant
Irgafos 168 OX Phosphite	13.9	
Irgafos 168	17.4	2° Antioxidant

Table 2: Recovery Data for the Extraction of Additives from Polypropylene

Standard Compound	80 °C (n=4)	RSD	120 °C (n=4)	RSD
BHT	90	34	N/A	N/A
Erucamide	N/A	N/A	90	4.2
Irganox 3114	86	5.6	81	3.1
Vitamin E	93	6.7	98	2.9
Irganox 1010	75	11	78	2.0
Irganox 1076	88	3.1	83	2.8
Irgafos 168 OX Phosphite	99	13	107	19
Irgafos 168	92	6.3	98	4.1
Irgafos Total	93	6.3	98	4.1

Table 3: Recovery Data for the Extraction of Additives from Polypropylene via the EDGE and Sonication

Standard Compound	ASTM (%)	EDGE (%)
BHT	79	91
Erucamide	65	90
Irganox 3114	58	86
Vitamin E	63	98
Irganox 1010	54	78
Irganox 1076	59	88
Irgafos 168 OX Phosphite	67	107
Irgafos 168	54	98
Irgafos Total	56	98

Table 4: Recovery Data for the Extraction of Additives from Polyethylene via the EDGE and Sonication

Standard Compound	ASTM (%)	EDGE (%)
BHT	62	111
Erucamide	48	86
Irganox 3114	58	91
Vitamin E	63	67
Irganox 1010	45	63
Irganox 1076	52	75
Irgafos 168 OX Phosphite	40	75
Irgafos 168	51	93
Irgafos Total	48	87

Conclusion

The extraction process used on the EDGE automated extraction system allowed for efficient extraction of additives from plastics. One simple extraction method with adjusted temperatures was utilized for all plastics, which greatly simplified the process. Additives were extracted more efficiently with the EDGE than with the traditional sonication method, due to the rapid extraction and filtration that is only possible with Q-Cup technology. No additional steps were required prior to analysis.

In this study, polypropylene and polyethylene were the focus. However, a similar method would be applicable for other plastic samples. The EDGE, with its efficient plastic extraction method, is ideal for manufacturers that want repeatable and reliable results for all samples.

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