

Extraction of Phthalates from Polyethylene and Polyvinyl Chloride



Abstract

Phthalates are used as plasticizers in a wide range of common products and over recent years have garnered a lot of attention due to their potential negative health impacts. These impacts are so concerning that the use of some phthalates have been banned in children's toys by the Consumer Product Safety Commission (CPSC). The ability to quickly and accurately extract phthalates from plastics such as polyvinyl chloride is advantageous and can help aid in ensuring the safety of the products made of these materials. With its patent pending Q-Cup Technology™ the EDGE™ can extract plastic samples in less than 10 minutes. The proposed method has been found to be faster and more effective than other methods for this application.

Introduction

Phthalates have been used since the 1950's to soften plastics and can be found in products that most people encounter on a daily basis, such as personal care products, vinyl flooring, children's lunch boxes, backpacks and toys. Even more alarming is that phthalates can be found in many items that young children, who are more prone to put things in their mouths, come in contact with. Phthalates have been described as dangerous chemical toxins and that can damage the liver, kidneys, lungs, and reproductive systems and have been linked to alteration of DNA integrity. Since they are not chemically

bound to the plastic, phthalates are continuously being released into the environment and people are exposed through repeated contact. The extraction of phthalates from plastics needs to be a quick and simple process so that manufacturers can confidentially release products that meet the safety guidelines of the CPSC.

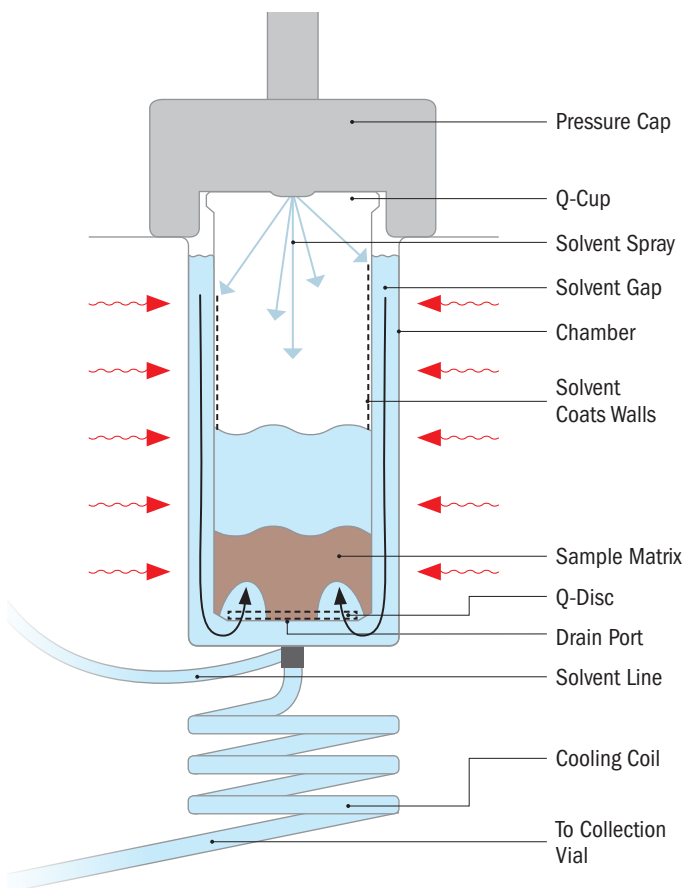
The extraction of phthalates from plastics is difficult for a number of reasons. First, the complexity of the samples and the low melting point of the plastic make it difficult to extract just the analyte of interest. Next, the traditional methods for extraction of phthalates are time consuming and often yield extracts that are cloudy and contain multiple co-extracts, making analysis difficult. Lastly, is the potential for unacceptable levels of carryover from sample to sample. The EDGE is capable of producing a clean, filtered, and cooled extract that is ready for analysis in less than 10 minutes. Each 10 minute extraction cycle also includes an efficient dual solvent wash which cleans the system and eliminates risk of carryover.

Instrumentation

The EDGE uses Q-Cup Technology that combines the process of Pressurized Fluid Extraction and Dispersive Solid Phase Extraction in one instrument that yields rapid and efficient extraction. The easy-to-assemble Q-Cup™ sample holder offers a unique open cell concept that creates a dispersive effect and promotes rapid extraction and filtration. The result is fast, simple and efficient extractions.

Sample preparation in the EDGE could not be easier. Simply place a Q-Disc™ into the Q-Cup™ base and screw the two parts together. The EDGE can accommodate a broad range of sample sizes, from less than a gram to multi-gram samples. The EDGE will use only 40 mL of solvent per extraction which includes solvent for diffusive extraction and sample rinse. Cleaning of the system can use up to an additional 30 mL of solvent. The rapid heating of the extraction chamber in combination with diffusive action allows a temperature of up to 180 °C to be achieved in less than 2 minutes.

Figure 1: The EDGE Process



Sample is Loaded

The Q-Cup is automatically loaded into the chamber by the auto sampler. The pressure cap then creates a pressurized seal on the top of the Q-Cup.

Solvent is Extracted

Solvent is first added through the bottom to fill the gap between the chamber and Q-Cup, this aids in heat transfer. Then, solvent is added through the top of the Q-Cup to wet the sample.

As the chamber walls are heated, the pressure in the gap increases. This overcomes the pressure inside the Q-Cup, forcing the solvent to disperse into the sample.

Extract is Collected

Once the sample reaches temperature, the solvent is dispensed through the Q-Disc, the cooling coil, and into a collection vial.

Procedure and Method

0.5 g of polyethylene or polyvinyl chloride was weighed into an assembled Q-Cup containing a Q-Disc. The Q-Cups were placed in the EDGE removable rack each with a collection vial and the rack was slid into place on the EDGE. The One Touch Method™ for polyethylene and One Touch Method for polyvinyl chloride were used respectively. The extracts were injected into the Agilent 7890A with a 5975C MSD for analysis adhering to EPA 8270. A Phenomenex ZB-5MSplus 30 m, 0.25 mm column was used.

Samples

A polyethylene CRM-PE001 and polyvinyl chloride CRM-PVC001 were purchased from SPEX CertiPrep. CRM's were extracted via the EDGE and soxhlet. A 70/30 mixture of acetone/cyclohexane was used as the extraction and rinse solvent for polyethylene. A 50/50 mixture of isopropanol/cyclohexane was used as the extraction and rinse solvent for polyvinyl chloride. The system was washed with acetone and isopropanol.

Results and Discussion

The EDGE, using Q-Cup Technology for the extraction of phthalates from plastics such as, polyethylene and polyvinyl chloride, yielded comparable recoveries than the standard soxhlet method. Furthermore; the EDGE method used less solvent than the alternative method. Table 1 shows the % recovery of the extraction of phthalates from polyethylene versus soxhlet. Table 2 shows the % recovery of the extraction of phthalates from polyvinyl chloride versus soxhlet. EDGE is a good option to economically and accurately extract phthalates from plastics. All methods for sample prep, extraction and analysis were based on CPSC-CH-C1001-09.1.

Table 1: % recovery data as compared to soxhlet for polyethylene

Phthalate	Average Recovery (% Soxhlet)
Bis (2-ethylhexyl) Phthalate	101
Di-n-octyl Phthalate	94

Table 2: % recovery data as compared to soxhlet for polyvinyl chloride

Phthalate	Average Recovery (% Soxhlet)
Butylbenzyl Phthlate	76
Diethyl Phthlate	74
Di-n-octyl Phthate	76

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