Extraction of Pesticides from Difficult Food Matrices

Introduction

The QuEChERS method has been shown to be practical for pesticide analysis on a number of different sample types and is increasingly being employed on more difficult matrices. Unfortunately, this process is a manual multi-step process that can be time consuming. With so many different types of food matrices and pesticide residues to be analyzed there is a need for a faster simplified extraction method. Furthermore, some matrices either by their nature or their economic value, can be difficult to analyze with just the QuEChERS method alone. In this application note we propose the EDGE™ as an alternative to the QuEChERS method for the extraction of pesticides from food matrices including known difficult matrices. With its patent pending Q-Cup Technology™ the EDGE can extract a difficult food sample, including the dispersive solid phase cleanup, in less than 5 minutes in one automated step.

Abstract

More and more consumers want to know what is in their food, particularly anything that could be harmful, such as pesticides. There is a driving need for pesticide analysis and the list of pesticides regulated throughout the world continues to increase. The QuEChERS method has become a widely accepted method to extract pesticides from food matrices. Due to the large number of pesticides to monitor and the low method detection limits, pesticide analysis can be a big challenge. Alternative methods can help aid in this challenge giving improved recoveries for difficult matrices with a faster and simplified method.

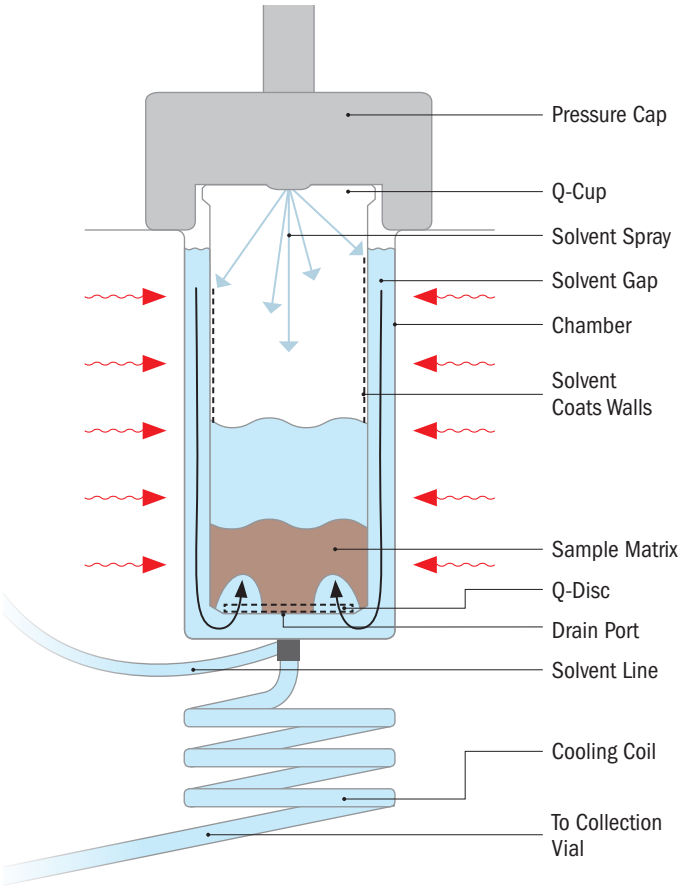
The manual multi-step process of the QuEChERS method requires multiple sample transfers and generates a lot of consumable waste. With the EDGE the sample and sorbents are together in one sample cell leading to extraction and cleanup in one step. In under 5 min the food sample is extracted using Q-Cup Technology which performs both extraction and cleanup of the sample. The collected extract is filtered, cooled and ready for analysis. Included in the run time is both the rinsing of the sample and washing of the system ensuring no carryover. EDGE offers the fastest pesticide extraction possible in one simple method.

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. Add any sorbents or salts needed for cleanup or water removal and then add the sample. Food samples can be wet or dry and as large as 15 g. The EDGE will use only 40 mL of solvent per extraction, which includes solvent for diffusive extraction, sample rinse, and cleaning of the system. 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 Added

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.

Chamber is Heated

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

1 g of primary-secondary amine (PSA) was added to an assembled Q-Cup containing a Q-Disc. For strawberries, 5 g of $Na2SO_4$ was added to the Q-Cup prior to the PSA. 5 g of rice, strawberries, or avocado, spiked with 250 µl of a 500 ppm spiking solution, were then added to the Q-Cup. The salt, sorbent and sample created layers and were not mixed. 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 Pesticide Residues was used. The extracts were filtered through a 0.45 µm syringe filter and injected into a Waters Acquity UPLC with a Xevo TQD triple quad mass spectrometer for analysis. An Acquity UPLC BEH C18 1.7µm 2.1x50 mm column with a flow of 0.45 mL/min and a 6 min ramp from 95% A (water w/ 10 mM $C2H7NO_2$) and 5% B (methanol w/ 10 mM $C2H7NO_2$) to 5% A and 95% B. A 10 uL sample was injected into the UPLC. Two MSD transitions were used for quantification for each pesticide.

Samples

Rice, strawberries and avocado were obtained from a local grocery store and homogenized with a grinder. All samples were spiked with organophosphorus mix A from Sigma Aldrich. PSA and sodium sulfate were purchased from Sigma Aldrich. Spiked samples were extracted via the EDGE. Acetonitrile was used as the extraction and rinse solvent. The system was washed with water and acetonitrile.

Results and Discussion

The EDGE efficiently extracted the pesticides from rice, strawberries, and avocado in under 5 minutes, including sample cleanup, filtration, cooling and system washing. Table 1 shows the recovery data of multiple pesticides from rice, strawberries, and avocado. These food matrices show that EDGE is effective for both dry and wet samples. Furthermore, avocado is a known difficult sample due to its high fat content and the EDGE was able to yield an extract with sufficient cleanup in just one automated step resulting in good recoveries. No matter the food sample EDGE offers a fast and simple extraction method that includes the cleanup process and is a good alterative to the QuEChERS method.

Table 1: % Recovery of Pesticides from Spiked Rice, Strawberry and Avocado

Pesticide	Rice	Strawberry	Avocado
Tokuthion	87	93	86
Guthion	90	90	85
Dichlorvos	88	120	116
Methyl parathion	95	107	107
Dursban	89	100	93
Ronnel	90	102	97
Disulfoton	92	92	89
Mocap	94	103	93

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