Extraction of Pesticides from Difficult Matrices

Summary

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. The EDGE® provides a valuable alternative to the QuEChERS method for the extraction of pesticides from food matrices, including matrices known to be difficult. With its patent pending Q-Cup Technology™, the EDGE can extract a difficult food sample, including the dispersive solid phase cleanup, in less than 7 minutes in one automated step.

Introduction

More and more consumers want to know what is in their food, particularly anything that could be harmful, such as pesticides. This creates a driving need for pesticide analysis; this need for testing is further bolstered with an ever-increasing list of regulated pesticides. The QuEChERS method has become a widely accepted method to meet some of these challenges; however, due to the large number of pesticides to monitor and the low method detection limits, 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 a single sample cell, meaning extraction and cleanup is performed in one step. The collected extract is automatically 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 automated pesticide extraction possible in one simple method.

Materials and Methods

Reagents

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. Primary Secondary Amine (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.

Sample Preparation

1 g of PSA was added to an assembled Q-Cup containing a C9 and M2 Q-Disc. The C9 Q-Disc was first placed in the bottom cap of the Q-Cup, then the M2 Q-Disc was placed in with the textured side up. The Q-Cup bottom cap and body were then firmly secured. For strawberries, 5 g of Na_2SO_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 position on the EDGE. The CEM approved EDGE method for Pesticide Residues was used.

EDGE Method

Q-Disc: C9 with M2

Extraction Solvent: Acetonitrile

Top Add: 20 mL

Bottom Add: 10 mL

Rinse: 0 mL

Temperature: 100 °C

Hold Time: 1 min

Analysis

The extracts were diluted to the 30 mL certified graduated mark on the vial 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 ammonium acetate) and 5% B (methanol w/ 10 mM ammonium acetate) to 5% A and 95% B. A 10 µL sample was injected into the UPLC. Two MSD transitions were used for quantification for each pesticide.

Results

The EDGE efficiently extracted the pesticides from rice, strawberries, and avocado in under 7 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 demonstrate that EDGE is effective for both dry and wet samples. Furthermore, avocado is known to be a difficult sample due to its high fat content and the EDGE was able to yield an extract with sufficient cleanup in a single automated step resulting in good recoveries.

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

Conclusion

The extraction process used on the EDGE automated extraction system allowed for a broad range of food samples to be extracted efficiently. One CEM approved extraction method was utilized for all samples, both dry and wet, greatly simplifying the sample preparation process. With an automated method using Q-Cup Technology, pesticides were more efficiently extracted than with the traditional QuEChERS process. In this study, three matrices and a few pesticides were examined, however; this same method would be applicable for all food samples and a wide range of pesticides. Some pesticides are known to be heat labile. For samples where temperature is a concern, a room temperature extraction can be used on the EDGE. The EDGE, with its efficient pesticide extraction method, is ideal for testing labs that want repeatable results for all samples with just one method.

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