Extraction of Pesticides from a Strawberry CRM

Abstract

The QuEChERS method has become the industry standard for pesticide clean-up and extraction of a wide variety of food samples. Unfortunately, this process is a manual, multi-step process that can be time consuming and riddled with error. Furthermore, there are so many different QuEChERS kits for various food types that it can be overwhelming to determine which kit is best for any given sample.

In this application note, we discuss the use of the EDGE® automated extraction system with Q-Matrix® Hydra. The EDGE had the same recovery as traditional QuEChERS methodology but in an automated platform that included sample rinsing, filtration, and system washing.

Introduction

As consumers, we want to know what is in our food that could be harmful, including pesticides, fillers, and anything that could be leached from packaging. As a manufacturer, we need to ensure that our products are safe and fall within allowable limits of contaminants. Pesticide analysis is of particular interest, as the long-term effects of pesticide exposure are well-documented. While pesticide testing is already very routine with QuEChERS methodology, there is room for improvement. Achieving a more complete extraction in less time in an automated fashion has been a request of food manufacturers for years. As limits of detection continue to drop and turnaround time for batch release becomes more urgent, a faster, more efficient extraction method is necessary.

In this experiment, the techniques of QuEChERS and EDGE automated solvent extraction will be compared. QuEChERS utilizes salts and sorbents in a multi-step manual clean-up and extraction process. With QuEChERS, the total time to extract one sample is between 20 and 60 minutes. The EDGE automated solvent extraction system performs sample extraction and clean-up in a single 7-9 minute cycle that includes sample rinsing, filtration, and system washing. The use of Q-Matrix Hydra further simplifies the extraction process by removing water from wet samples in situ, so that no additional handling of the sample is required.

Materials and Methods

Reagents

A Strawberry CRM (T19253QCSale) was purchased from FAPAS. Q-Matrix Hydra is a product of CEM Corporation. Sodium acetate, magnesium sulfate, and primary secondary amine were purchased in bulk from Silicycle. Samples were extracted via the EDGE or QuEChERS method. Acetonitrile with 1% acetic acid was used as the extraction, rinse, and wash solvent.

QuEChERS Method

A portion of 10 g of strawberry CRM was weighed into a 50 mL centrifuge tube. A volume of 10 mL of acetonitrile with 1% acetic acid was added to the tube and vortexed for 1 minute on a VWR Analog Vortex Mixer. A portion of 1.5 g of sodium acetate and 6 g of magnesium sulfate were added to the tube, which was capped, shaken for 1 minute, and then centrifuged at 6000 rpm for 5 minutes in a Thermo CL2 Centrifuge.

A volume of 1 mL of the acetonitrile layer was added to a 20 mL centrifuge tube containing 150 mg of magnesium sulfate and 50 mg of primary secondary amine. The tubes were shaken for 1 minute and centrifuged at 6000 rpm for 5 minutes. The supernatant was transferred to a vial for analysis. All samples and blanks were prepared in triplicate.

Sample Preparation

A portion of 2.5 g of Q-Matrix Hydra and 5 or 10 g of strawberry CRM were weighed into an assembled Q-Cup® containing a S1 Q-Disc® stack (C9+G1+C9 sandwich). A Q-Screen® was placed on top of each sample using a Q-Screen tool. The Q-Cups were placed in a rack, along with 50 mL polypropylene conical tubes, and the rack was slid into position on the EDGE. The EDGE was programmed with either the standard Pesticide Residues method for the 5 g sample or the modified Pesticide Residues method, which includes agitation and multiple rinses to increase analyte recovery, for the 10 g sample. The extract was transferred to a Q-Dry™ solvent evaporator for evaporation to <5 mL. Extracts were then diluted to 5 mL with acetonitrile with 1% acetic acid. All samples and blanks were prepared in triplicate. The extracts were transferred to vials for analysis.

EDGE Methods for Pesticides from a 5 g Sample

Q-Disc: S1 Q-Disc stack (C9+G1+C9 sandwich)
Sample Size: 5 g

Cycle 1
Extraction Solvent: Acetonitrile with 1.0% Acetic Acid (v/v)
Top Add: 25 mL
Bottom Add: 0 mL
Rinse: 5 mL
Temperature: 40 °C
Hold Time: 03:00 (mm:ss)

Wash 1
Wash Solvent: Acetonitrile with 1.0% Acetic Acid (v/v)
Wash Volume: 10 mL
Temperature: 40 °C
Hold Time: 00:03 (mm:ss)

EDGE Methods for Pesticides from a 10 g Sample

Q-Disc: S1 Q-Disc stack (C9+G1+C9 sandwich)
Sample Size: 10 g

Cycle 1
Extraction Solvent: Acetonitrile with 1.0% Acetic Acid (v/v)
Agitation: 02:00 (mm:ss)
Top Add: 15 mL
Bottom Add: 0 mL
Rinse: 5 mL
Temperature: 40 °C
Hold Time: 04:00 (mm:ss)

Cycle 2 (Rinse Only)
Extraction Solvent: Acetonitrile with 1.0% Acetic Acid (v/v)
Top Add: 0 mL
Bottom Add: 0 mL
Rinse: 10 mL
Temperature: - - -
Hold Time: - -:- -

Wash 1
Wash Solvent: Acetonitrile with 1.0% Acetic Acid (v/v)
Wash Volume: 10 mL
Temperature: 40 °C
Hold Time: 00:03 (mm:ss)

Analysis

A volume of 2 µL of each extract was injected into a Waters Acquity UPLC with a Xevo TQD triple quad mass spectrometer. Separation was done with a Restek ARC-18, 2.7 µm, 100 x 2.1 mm column with a flow of 0.4 mL/min. An elution program with a 7-minute ramp from 95% A (water with 10 mM ammonium acetate and 0.2% formic acid) and 5% B (methanol with 10 mM ammonium acetate and 0.2% formic acid) to 100% B was programmed. MRM transitions were used for quantification for each pesticide. Each sample was analyzed in triplicate.

Results and Discussion

The EDGE efficiently extracted the pesticides from the strawberry CRM in 7–9 minutes, including filtration, cooling, and system washing. **Table 1** (page 3) shows the EDGE and QuEChERS recovery data of multiple pesticides from the strawberry CRM. Comparable recoveries were obtained between the EDGE and QuEChERS extractions. It is important to note that the QuEChERS method was developed for matrices such as strawberries, so good recovery values should be expected. The ability of the EDGE to match the efficiency of the QuEChERS method for a standard matrix, such as strawberries, validates that it can be a suitable alternative method. Since the QuEChERS method is a tedious manual process, the EDGE offers the benefit of a simplified automated extraction with comparable recoveries.

Conclusion

The extraction process used on the EDGE automated extraction system allowed for a strawberry CRM to be extracted efficiently in a single step. With one automated method, pesticides were efficiently extracted with recoveries comparable to the traditional QuEChERS process. The addition of agitation on the EDGE allowed for extraction of a larger sample and use of less solvent than when agitation is not used. The increase in sample size gives greater confidence into sample homogeneity with respect to the total batch sampled. Furthermore, a minimum of 60% time savings was realized when compared to the QuEChERS method. This time savings leads to increased sample throughput and overall laboratory productivity.

In this study, a wet food sample of strawberry CRM was the focus; however, the same method would be applicable for all food samples, wet and dry, 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 performed on the EDGE. The EDGE, with its efficient pesticide extraction method, is ideal for testing labs that want repeatable results for all food samples using one simple automated method.

Table 1. Recovery Data from a Strawberry CRM Extracted via the EDGE and QuEChERS

Pesticide	EDGE 5 g Sample (% Recovery)	EDGE 10 g Sample (% Recovery)	QuEChERS (% Recovery)
Aldicarb Sulfoxide	92	100	111
Dimethoate	105	93	92
Etoxazole	69	65	60
Monocrotophos	82	98	96
Pirimicarb	87	102	62
Triticonazole	81	95	102

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