# The Extraction of Pesticides from Black Tea

## Abstract

As consumers grow more aware of the pesticides in their food, the need for efficient and automated extraction methods for pesticides analysis continues to grow. The typical method used for the extraction of pesticides from food is the QuEChERS method, but the use of this method has many drawbacks, including the weighing of salts, multiple sample transfers, and waste generation. The EDGE®, an automated extraction system, improves upon the QuEChERS method for extraction by eliminating the need for sample transfers and minimizing the use of salts or sorbents. In this application note, the EDGE was used to extract pesticides from black tea, resulting in acceptable recoveries of 80-120% and acceptable reproducibility with RSD values less than 20%. The EDGE is an ideal choice for food laboratories looking to automate their workflow.

## Introduction

Consumers are increasingly concerned about the pesticides found in their food. With the emergence of pesticide resistance and the continued development of new pesticides, the list of formally regulated pesticides continues to grow. As pesticides can be toxic depending on their concentration level, it is critical to test for these compounds. Efficient methods to extract these pesticides are needed. Typically, the industry standard for the extraction of pesticides from food is the QuEChERS method. This method includes the tedious addition of salts and sorbents to a sample, manual shaking, and multiple sample transfers, ultimately making it a time-consuming method that generates substantial waste. One extraction can take between 20 to 60 minutes. Thus, innovative improvements to this method are needed.

The EDGE is an automated extraction system that improves upon the traditional QuEChERS method. The EDGE does not require the use of salts/sorbents, which often leave behind residual material that can cause problems on LC-MS and GC-MS systems. The EDGE's Q-Cup® technology relieves the need for multiple sample transfers, thus generating less waste. In this application note, the EDGE was used to extract a large panel of pesticides from 2 g of black tea. The EDGE rapidly and efficiently extracted over 140 pesticides with high recoveries (>80%) and acceptable RSDs (<20%). The EDGE extracted, filtered, and cooled the extracts quickly. The EDGE is an ideal alternative method for food testing laboratories seeking to improve their workflow through automation.

## Materials and Methods

### Reagents

UHPLC-grade acetonitrile was used as the extraction and wash solvent. Loose black tea was purchased from a local grocery store. A custom pesticide mixture was made for spiking. Methanol, water, ammonium formate, and formic acid were used for analysis.

### Sample Preparation

Q-Cups were assembled with the S1 Q-Disc® stack (C9+G1+C9 sandwich), and 2 g of black tea were directly weighed into each Q-Cup. The black tea was spiked with 20 µg/kg of a pesticide mix. The black tea was then covered with a Q-Screen® to prevent the matrix from floating upon the addition of solvent.

The Q-Cup containing the spiked sample was then extracted on the EDGE using the parameters below. Each extraction was collected in a 50 mL centrifuge tube, volume was confirmed at 15 mL within centrifuge tube, and then transferred to a vial for analysis.

## EDGE Method for the Extraction of Pesticides from Black Tea

Q-Disc: S1 Q-Disc Stack (C9+G1+C9 sandwich)

**Cycle 1**
Extraction Solvent: Acetonitrile
Top Add: 10 mL
Bottom Add: 0 mL
Rinse: 0 mL
Temperature: 40 °C
Hold Time: 1:30 (mm:ss)

**Cycle 2 (Rinse Only)**
Extraction Solvent: Acetonitrile
Top Add: 0 mL
Bottom Add: 0 mL
Rinse: 5 mL
Temperature: - - -
Hold Time: - -:- -

**Wash**
Wash Solvent: Acetonitrile
Wash Volume: 10 mL
Temperature: 40 °C
Hold: 0:03 (mm:ss)

# Analysis

A volume of 5 µL of each sample was injected into an Agilent UHPLC with a 6490A Mass Spectrometer for analysis. A Eclipse Plus C8, 1.8 µm, 2.1 x 100 mm column with a flow of 0.3 mL/min and a multi-stage elution program with a 17 minute ramp from 100% B (water with 2% methanol, 5mM ammonium formate, 0.1% formic acid) to 100% A (methanol with 2% water, 5mM ammonium formate, and 0.1% formic acid) was programmed. MRM transitions were used for quantification.

# Results

**Table 1** lists the recoveries and RSDs of the pesticides extracted for n=3 samples, or in triplicate. The EDGE was able to efficiently extract 144 pesticides from spiked black tea, filter the extract, and cool the sample to room temperature in under 10 minutes. The recoveries for these 144 pesticides were greater than 80%, indicating excellent recovery of each compound. The RSD values were less than 20%, indicating good reproducibility. For this sample type, the EDGE did not require the use of salts, sorbents, or any cleanup materials, which is advantageous because these materials can interfere and affect the recovery of certain pesticides.

# Conclusion

The EDGE utilizes automation to improve upon the typical extraction approach, QuEChERS, which is widely used for pesticide extraction. The Q-Cup technology used by the EDGE does not require multiple sample transfers and decreases waste generation. In this application note, the EDGE efficiently extracted the pesticides from 2 g of black tea in under 10 minutes without the use of the salts or sorbents required for the QuEChERS method. The EDGE also filtered and cooled the extract and recovered more than 80% of each pesticide with favorable RSDs. The EDGE provides a rapid, efficient, automated alternative to the manual QuEChERS method and is a great solution for food laboratories working to streamline their extraction process with automation.

**Table 1**. The Recovery of A Panel of Pesticides from Spiked Black Tea

| Compound | Recovery (%)n=3 | RSD (%) n=3 |
|---|---|---|
| 2,4-D | 84% | 3% |
| Acetamiprid | 87% | 5% |
| Acrinathrin | 83% | 10% |
| Ametoctradin | 92% | 3% |
| Anilofos | 88% | 0% |
| Azinphos-ethyl | 85% | 4% |
| Azinphos-methyl | 93% | 10% |
| Azoxystrobin | 87% | 12% |
| Benalaxyl | 85% | 0% |
| Bifenthrin | 91% | 13% |
| Bitertanol | 114% | 15% |
| Boscalid | 93% | 1% |
| Bromacil | 86% | 6% |
| Bromuconazole | 86% | 12% |
| Bupirimate | 92% | 4% |
| Buprofezin | 93% | 2% |
| Carbaryl | 92% | 9% |
| Carbendazim | 84% | 1% |
| Carbendazim d3 | 84% | 2% |
| Chlorantraniliprole | 100% | 3% |
| Chlorbromuron | 105% | 3% |
| Chlorfenvinphos | 97% | 2% |
| Chlorfluazuron | 87% | 7% |
| Chloridazon | 109% | 5% |
| Chlorotoluron | 87% | 2% |
| Chloroxuron | 109% | 0% |
| Chromafenozide | 95% | 6% |
| Clomazone | 91% | 8% |
| Coumaphos | 96% | 8% |

| Compound | Recovery (%)n=3 | RSD (%) n=3 |
|---|---|---|
| Cyazofamid | 80% | 5% |
| Cyflufenamid | 81% | 2% |
| Cyhalofop-butyl | 91% | 5% |
| Cyproconazole | 120% | 4% |
| Deltamethrin | 86% | 5% |
| Diazinon | 83% | 14% |
| Dichlorvos $D_6$ | 81% | 2% |
| Diethofencarb | 94% | 4% |
| Difenoconazole | 95% | 14% |
| Difenoxuron | 94% | 0% |
| Diflubenzuron | 111% | 4% |
| Dimethomorph | 94% | 14% |
| Diuron | 98% | 4% |
| Edifenphos | 82% | 9% |
| EPN | 95% | 17% |
| Epoxiconazole | 81% | 11% |
| Ethion | 82% | 5% |
| Ethiprole | 94% | 5% |
| Ethoprophos | 82% | 1% |
| Etofenprox | 94% | 6% |
| Famoxadone | 93% | 5% |
| Fenamidone | 103% | 3% |
| Fenamiphos-sulfoxide | 84% | 13% |
| Fenarimol | 107% | 2% |
| Fenazaquin | 95% | 4% |
| Fenbuconazole | 111% | 4% |
| Fenhexamid | 99% | 1% |
| Fenoxycarb | 95% | 12% |
| Fenpropathrin | 98% | 9% |
| Fenpyroximate | 97% | 11% |
| Fenthion-sulfone | 81% | 10% |
| Fenthion-sulfoxide | 89% | 11% |
| Fenuron | 84% | 2% |
| Flazasulfuron | 85% | 1% |
| Flonicamid | 99% | 0% |
| Fludioxonil | 84% | 1% |
| Fluometuron | 100% | 4% |
| Fluopicolide | 85% | 9% |
| Fluopyram | 82% | 4% |
| Fluquinconazole | 100% | 0% |
| Fluxapyroxad | 90% | 12% |
| Fosthiazate | 83% | 1% |
| Hexythiazox | 88% | 18% |
| Imidacloprid | 100% | 1% |

| Compound | Recovery (%)n=3 | RSD (%) n=3 |
|---|---|---|
| Indoxacarb | 108% | 7% |
| Ioxynil | 80% | 1% |
| Iprodione | 81% | 8% |
| Isoprocarb | 81% | 8% |
| Isoprothiolane | 83% | 3% |
| Isoproturon | 92% | 2% |
| Isoxaflutole | 89% | 7% |
| Lenacil | 93% | 4% |
| Malathion | 81% | 4% |
| Mandipropamid | 109% | 9% |
| Metconazole | 89% | 14% |
| Methiocarb-sulfone | 115% | 1% |
| Methiocarb-sulfoxide | 100% | 0% |
| Methoxyfenozide | 95% | 2% |
| Metobromuron | 85% | 1% |
| Metolachlor | 89% | 5% |
| Metolcarb | 85% | 7% |
| Metrafenone | 81% | 10% |
| Monolinuron | 89% | 15% |
| Monuron | 89% | 1% |
| Neburon | 91% | 9% |
| Novaluron | 92% | 4% |
| Oxadiargyl | 85% | 13% |
| Oxasulfuron | 91% | 1% |
| Paraoxon-methyl | 93% | 1% |
| Penconazole | 84% | 2% |
| Pencycuron | 87% | 0% |
| Permethrin | 101% | 3% |
| Phenthoate | 89% | 9% |
| Phosalone | 90% | 5% |
| Phosmet | 94% | 15% |
| Profenofos | 92% | 10% |
| Promecarb | 88% | 3% |
| Prometryn | 83% | 5% |
| Propaquizafop | 91% | 9% |
| Propargite | 85% | 3% |
| Propazine | 80% | 1% |
| Propiconazole | 89% | 1% |
| Propyzamide | 96% | 8% |
| Proquinazid | 85% | 9% |
| Prosulfocarb | 99% | 6% |
| Pyraclostrobin | 104% | 0% |
| Pyridaben | 85% | 8% |
| Pyridaphenthion | 96% | 4% |

| Compound | Recovery (%)n=3 | RSD (%) n=3 |
|---|---|---|
| Pyridate | 97% | 3% |
| Pyriproxyfen | 90% | 10% |
| Quinalphos | 86% | 7% |
| Quizalofop (free acid) | 102% | 5% |
| Quizalofop-ethyl | 92% | 13% |
| Rotenone | 114% | 5% |
| Spirodiclofen | 93% | 6% |
| Spiromesifen | 114% | 18% |
| Spirotetramat | 91% | 10% |
| Tebuconazole | 111% | 6% |
| Tebufenozide | 103% | 0% |
| Tebufenpyrad | 96% | 11% |
| Teflubenzuron | 84% | 4% |
| Teflubenzuron | 103% | 3% |
| Terbuthylazine | 85% | 5% |
| Tetraconazole | 93% | 7% |
| Tetramethrin | 91% | 3% |
| Thiacloprid | 103% | 4% |
| Thiamethoxam | 99% | 1% |
| Thiobencarb | 86% | 16% |
| Triadimenol | 111% | 7% |
| Triazophos | 87% | 5% |
| Trifloxystrobin | 91% | 8% |
| Triflumuron | 86% | 6% |
| Triticonazole | 115% | 1% |
| XMC (3,5-xylyl methylcarbamate) | 111% | 6% |
| Zoxamide | 99% | 4% |

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