

# 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 <sub>6</sub>	81%	2%
Diethofencarb	94%	4%
Difenoconazole	95%	14%
Difenoxyuron	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%
Fonicamid	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%
Oxadiazyl	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-xyllyl methylcarbamate)	111%	6%
Zoxamide	99%	4%

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