Extraction of Pesticides from Cannabis

Summary

The need for accurate testing of cannabis and cannabis based products is critical as more states approve recreational and medical marijuana use each year. Samples from flower to edibles, to consumer products and more, present challenges in both sample preparation and analysis, with pesticide analysis among the most challenging. In this application note, we propose the EDGE® as a method for the extraction of pesticides from cannabis. With its patent pending Q-Cup Technology™, the EDGE can extract pesticides from cannabis, including the dispersive solid phase cleanup, in less than one 7-minute automated step.

Introduction

Every year, more and more consumers want to know what is in the products they are purchasing, particularly anything that could be harmful, such as pesticides. Pesticides are potentially toxic to humans and can lead to acute and chronic health effects due to bioaccumulation. There is a driving need for pesticide analysis, and the list of pesticides regulated throughout the world continues to increase, particularly in the rapidly growing world of cannabis. The QuEChERS method is a widely accepted method to extract pesticides from food matrices; however, it has not proven to be suitable for pesticides extraction from cannabis. Pesticide analysis of cannabis is a challenge due to the large number of pesticides to monitor, the low method detection limits, and difficult cannabis matrix. There is a need for a simple and efficient method for the extraction of pesticides from cannabis that yields high recoveries and repeatable results.

The EDGE offers that simplicity and efficiency by containing the sample and sorbents together in one sample cell, leading to extraction and cleanup in one step. In under 7 minutes, the sample is extracted and the collected extract is filtered, cooled and ready for analysis. Each EDGE method includes rinsing of the sample to increase recovery efficiency and washing of the system to eliminate carryover risk. EDGE offers the fastest automated pesticide extraction possible in one simple method.

Materials and Methods

Reagents

Cannabis samples at the Washington State Department of Agriculture Chemical and Hop Laboratory were first knifemilled and then cyromilled. GCMS spike mix 1 and spike mix 2 and LCMC spike mix 1 and spike mix 2 were prepared by the Washington State Department of Agriculture Chemical and Hop Laboratory. QuEChERS Dispersive SPE 15 mL part number 5982-5058 was purchased from Agilent. A solution of 1% acetic acid in acetonitrile was used as the extraction, rinse, and wash solvent.

Sample Preparation

The Q-Cup® was assembled with a C9 Q-Disc® placed beneath an M2 Q-Disc with textured side facing up. The contents of an Agilent QuEChERS dispersive SPE 15 mL tube were added to the assembled Q-Cup. A sample of 1.5 g of cannabis was weighed directly into the Q-Cup containing the sorbent. GCMS samples were spiked with 200 µL of spike mix 1 and 200 µL of spike mix 2. LCMS samples were spiked with 61 µL spike mix 1 and 61 µL of spike mix 2. The Q-Cups were placed in the EDGE removable rack, each with a collection vial, and the rack was positioned in the EDGE. The CEM approved EDGE method for Pesticide Residues in Cannabis was used.

EDGE Method

Q-Disc: M2 & C9

Extraction Solvent: 1% Acetic Acid in Acetonitrile

Top Add: 20 mL

Bottom Add: 5 mL

Rinse: 5 mL

Temperature: 40 °C

Hold Time: 2 minutes

Analysis

The extracts were diluted to a known volume and GCMS samples injected into a Agilent 7010, and LCMS samples injected into a Sciex 6500QTRAP equipped with an LC. GCMS and LCMS methods were run according to the Washington State Department of Agriculture Chemical and Hop Laboratory protocols.

Results

The EDGE efficiently extracted over 400 pesticides from cannabis in under 7 minutes, including sample cleanup, filtration, cooling, and system washing. **Table 1** shows the recovery data of multiple pesticides from cannabis via GCMS analysis. **Table 2** shows the recovery data of multiple pesticides from cannabis via LCMS analysis. For a complete list of all pesticides evaluated, please contact CEM. EDGE was able to yield an extract with sufficient cleanup in just one automated step, resulting in good recoveries.

Table 1: % Recovery of Pesticides from Spiked Cannabis via GCMS

Pesticide	Recovery
Acetochlor	87%
Aldrin	86%
Allethrin	90%
Benfluralin (Benefin)	86%
BHC-alpha (benzene hexachloride)	98%
BHC-beta	89%
Bifenazate	82%
Captan	84%
Chlordane-cis (alpha)	84%
Chlordane-trans (gamma)	85%
Chlorfenapyr	88%
Chlorpropham	95%
Chlorpyrifos	87%
Cyhalothrin (lambda)	109%
DCPA (Dacthal, Chlorthal-dimethyl)	87%
DDD-p,p'	78%
DDE-o,p'	87%
DDE-p,p'	86%
DDT-p,p'	80%
Dichlorobenzonitrile, 2,6- (Dichlobenil)	90%
Diclofop-methyl	93%
Dicofol pp	86%
Dieldrin	87%
Endosulfan I (alpha isomer)	106%
Endosulfan II (beta isomer)	80%
Ethalfluralin	92%
Ethion	87%
Ethoprophos (Ethoprop)	83%
Fenamiphos (Phenamiphos)	90%
Fipronil sulfone	92%

Pesticide	Recovery
Fipronil	91%
Fludioxonil	93%
Heptachlor exo-epoxide (isomer B)	91%
Heptachlor	100%
Methidathion	80%
MGK-264 (zengxiaoan)	86%
Naphthalenol-1 (1-naphthol)	101%
Oxadiazon	89%
Oxyfluorfen	89%
Parathion (Parathion Ethyl)	87%
Parathion-methyl	82%
PCNB	88%
Pentachloroaniline (PCA)	89%
Pentachlorobenzene(PCB)	95%
Phenylphenol, 2- (OPP)	98%
Phorate	80%
Piperonyl butoxide	104%
Procymidone	90%
Profenofos	73%
Pronamide (Propyzamide)	83%
Quinoxyfen	103%
Resmethrin-cis (Cismethrin)	110%
Tefluthrin, cis-	88%
Terbacil	86%
Terbufos	82%
Tetradifon	115%
Thiobencarb (Benthiocarb)	89%
THPI (Tetrahydrophthalimide, cis-1,2,3,6-)	86%
Trifluralin	87%
Vinclozolin	84%

Table 2: % Recovery of Pesticides from Spiked Cannabis via LCMS

Pesticide	Recovery
3-Hydroxycarbofuran	89%
Abamectin	99%
Acephate	81%
Acetamiprid	88%
Acibenzolar-S-methyl	112%
Alachlor	93%
Aldicarb	89%
Ametryne	108%
Atrazine	105%
Azinphos-methyl	104%
Azoxystrobin	100%
Bendiocarb	96%
Benoxacor	102%
Bensulide	103%
Bensulide Oxon	106%
Bromacil	96%
Buprofezin	85%
Carbaryl	84%
Carbendazim	98%
Carbofuran	94%
Carboxin	91%
Carfentrazone-ethyl	105%
Chlorantraniliprole	107%
Chlorpyrifos Oxon	99%
Clofentezine	106%
Clomazone	103%
Clothianidin	88%
Coumaphos	97%
Coumaphos Oxon	99%
Cymoxanil	99%
Diazinon	93%
Dichlorvos	101%
Difenoconazole	104%
Diflubenzuron	105%
Dimethenamid	102%
Dimethoate	98%
Dimethomorph	100%
Dinotefuran	86%
Disulfoton Oxon	103%
Disulfoton SN	100%
Disulfoton SX	100%

Pesticide	Recovery
Diuron	89%
Etoxazole	97%
Famoxadone	100%
Fenazaquin	94%
Fenbuconazole	101%
Fenhexamid	115%
Fenoxycarb	106%
Flonicamid	91%
Flubendiamide	109%
Flufenoxuron	111%
Flumioxazin	96%
Fluopyram	105%
Fluquinconazole	102%
Fluridone	89%
Flusilazole	104%
Flutriafol	111%
Hexythiazox 2	85%
Imazalil	103%
Imazethapyr	98%
Imidacloprid	91%
Indoxacarb	105%
Isoxaben	98%
Kresoxim-methyl	108%
Malathion	117%
Malathion Oxon	97%
Mandipropamid	94%
Methamidophos	90%
Methiocarb	100%
Methomyl	88%
Methoxyfenozide	94%
Metolachlor	103%
Metribuzin	90%
Norflurazon	102%
Norflurazon Desmethyl	102%
Novaluron	107%
Omethoate	84%
Oxamyl	85%
Oxamyl Oxime	84%
Oxydemeton-methyl	87%
Oxydemeton-methyl SN	94%
Parathion-methyl Oxon	99%

Pesticide	Recovery
Pendimethalin	95%
Phorate SN	104%
Phorate SX	101%
Phosalone	113%
Pirimiphos-methyl	90%
Prometon	100%
Prometryne	114%
Propamocarb HCl	84%
Propazine	108%
Propetamphos	100%
Propoxur	97%
Pyraclostrobin	97%
Pyraflufen-ethyl	107%
Pyridaben	106%
Pyrimethanil	104%
Pyroxsylam	103%
Saflufenacil	102%
Sethoxydim	96%
Simazine	103%
Spinetoram A	103%
Spinetoram B	93%
Spinosad A	106%
Spirodiclofen	86%
Spiromesifen	110%
Sulfoxaflor	89%
Tebufenozide	101%
Tebufenozide	81%
Tebuthiuron	97%
Tetraconazole	85%
Thiabendazole	88%
Thiacloprid	91%
Thiamethoxam	97%
Thiazopyr	100%
Thiodicarb	97%
Thiophanate-Methyl	95%
Triallate	105%
Trichlorfon	92%
Trifloxystrobin	97%
Trifloxysulfuron-Na	102%
Triflumizole	113%
Triforine	100%

Conclusion

The extraction process used on the EDGE automated extraction system allowed for efficient extraction of pesticides from cannabis. One CEM approved extraction method was utilized for all samples, both GCMS and LCMS amenable, that greatly simplified the sample preparation process. With an automated method, pesticides can be extracted more efficiently than with the traditional QuEChERS process. Furthermore, only a 1.5 g sample size was required. In this study, the focus was the extraction of pesticides; however, the EDGE can be used for the extraction of cannabinoids, terpenes, and mycotoxins as well.

The EDGE, with its efficient pesticide extraction method, is ideal for cannabis testing labs that want repeatable results with one automated method and smaller sample sizes. We would like to thank the Washington State Department of Agriculture Chemical and Hop Laboratory for extracting the cannabis on the EDGE and running the analysis.

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