Maximization of Analytical Cannabis Extractions and Sample Clean-Up Through the Use of a Single Process Combined Pressurized Fluid and Dispersive Solid Extraction (EDGE)

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INTRODUCTION

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 some matrices, either by their nature or their economic value, like cannabis, can be difficult to analyze with just the QuEChERS method alone. These cannabis samples can show lower recoveries of pesticides and other target analytes than are often observed with more traditional agricultural products. An improved combined extraction and clean-up method is proposed in which both the extraction and dispersive solid phase extraction (DSE) steps are combined and heated using a pressurized fluid extraction and adding heat and pressure to the process increases the efficiency leading to a better sample clean-up and DSE improved analyte recoveries. In this study, a new combined extraction system was optimized to increase sample processing throughput, efficiency and recovery in a one-step process. Different analytes including pesticides, cannabinoids and terpenes were examined to determine improvement of recovery and method efficiency of the combined extraction apparatus. The new method showed marked improvement in sample clean-up, throughput and sample extraction recovery for cannabis testing.

METHOD & MATERIALS

Materials

- HPLC Grade Acetonitrile
- LCMS Grade Water
- Formic Acid
- Organic Hemp Flower Tea composed of 100% Cannabis Hemp Flowers
- QuEChERS AOAC 2 mL PSA Clean-Up Kit

SPEX CertiPrep Standards

- LC-ESCA-141 (mix of cannabis pesticides)

Sample Preparation

Initial Sample Preparation:

Whole hemp flowers were ground using SPEX SamplePrep Freezer Mill.

- Grinding Conditions
  - 2 g of Hemp Flowers

METHOD & MATERIALS (cont'd)

- Program
  - Precool for 20 minutes
  - Grind for 5 cycles (2 minutes per cycle)
  - Each cycle = 2 minutes cooling
  - Impact rate = 16 impacts per second

Sample Spiking

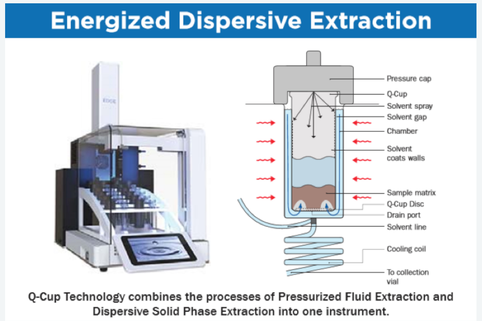
Ten grams of ground Cannabis hemp flowers were spiked with 1 mL of SPEX CertiPrep Cannabis Pesticide Standard LC-ESCA-141 (@ 1,000 µg/mL). The material was rolled for several hours and dried overnight.

Sample Extraction & Clean-Up

CEM Edge Q-cups were loaded with a C1 Q-Cup disk with one gram of ground Cannabis hemp flowers. The extraction system applies heat and cycles solvent through the Q-Cup to create an extremely efficient and quick digestion (see Figure 1).

In half of the samples, the contents of a SPEX QuEChERS AOAC-PSA (2 mL) clean-up tube was added prior to the addition of the one gram of ground Cannabis hemp flowers. One set of samples spiked and unspiked were extracted using 25 mL Acetonitrile with 0.05% Glacial Acetic Acid at 50 °C for 5 minutes with a one minute hold. The second set was extracted using the same solvent at 100 °C for 5 minutes with no hold.

Figure 1. CEM Energy Dispersive Extraction Process



Q-Cup Technology combines the processes of Pressurized Fluid Extraction and Dispersive Solid Phase Extraction into one instrument.

Instrument Conditions

- LC Conditions:
  - C18 2.6 µm 100 x 3.00 mm
    - 0.5 mL per minute
    - 10 µL injection
    - Gradient: Water (0.1% Formic) & Acetonitrile (0.1% Formic)
      - 98% Water to 98% Acetonitrile over 30 minutes
- MS Conditions:
  - ESI
    - Pos & Neg modes
    - TIC & EIC

RESULTS & DISCUSSION

The target spiking of the nine pesticides examined was 4 µg/mL in the extract. The recovery of the pesticides ranged from 77 to 155% (see Table 1).

Table 1. Pesticide Recovery Efficiencies from Cannabis using Energized Dispersive Extraction

% Recovery	No Clean-Up 50 °C	No Clean-Up 100 °C	AOAC PSA Clean-Up 50 °C	AOAC PSA Clean-Up 100 °C
Acephate	77	147	155	144
Methomyl	99	98	110	102
Dimethoate	91	94	101	99
Imidacloprid	81	85	96	98
Systhane	90	105	94	92
Abamectin	113	148	162	161
Carbaryl	95	95	98	98
Azoxystrobin	109	116	111	99

The lowest recoveries were found in samples that did not use the dSPE clean-up materials. Overall recoveries increased with the application of heat and the use of dispersive clean-up materials. Slightly better recoveries were found at the 50 °C samples in conjunctions with dSPE. Large amounts of the pesticides acephate and abamectin were found above the spiked concentration suggesting that the material already contains a measurable amount of those pesticides.

The new energized dispersive extraction method for the extraction of pesticides from difficult matrices yielded comparable or better recoveries compared to previous QuEChERS studies. Furthermore; the new energized dispersive method offered faster run times, and an automated simplified approach compared to alternative methods. Energized dispersive extraction offers a good and economical option for the extraction of pesticides from all types of food and nutraceutical matrices.

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