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QuEChERS Sample Preparation for GC-MS Determination of Organophosphorous Pesticides in Beef

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Abstract

This application note demonstrates that QuEChERS methodology is well-suited as a sample preparation step prior to GC-MS determination of pesticides in beef tissue. The QuEChERS extraction and subsequent dispersive SPE cleanup steps provide a faster analysis with less toxic reagents, and similar detection limits compared with other methods.

Benefits

- Rapid sample preparation of beef tissue using QuEChERS methodology
- Prepare 30 samples in one hour
- Straightforward SPE cleanup using dispersive SPE

Introduction

QuEChERS was developed to be quicker, easier, cheaper, safer than, and as rugged as alternative methods of sample preparation. The methodology was developed for analysis of pesticide residues in fruits and vegetables, prior to GC-MS or LC-MS. There are also maximum residue limits (MRLs) for many pesticides in animal products, requiring the determination of pesticide residues in these matrices. Here, we demonstrate the suitability of QuEChERS methodology for determining organophosphorous pesticides in beef muscle tissue. The pesticides chosen have MRLs ranging from 50 to 4000 ppb in beef muscle for US markets.

For a successful analysis using QuEChERS methodology, the sample matrix must be easily homogenized and suitable for extraction with acetonitrile. The sample should contain sufficient water for the liquid partition cleanup, following the addition of the QuEChERS salts. Also, the compound(s) of interest must have a suitable partition co-efficient when partitioned between acetonitrile and salt-saturated water. In this study, a small amount of water was added to the samples to achieve 80% water content. The results of this study indicate that QuEChERS methodology is well-suited as a sample preparation step prior to GC-MS determination of pesticides in beef tissue. The QuEChERS extraction and subsequent dispersive SPE cleanup steps provide a faster analysis with less toxic reagents, and similar detection limits compared with other methods.

Experimental

Sample Description

Initial extraction (QuEChERS):

Place 10 g of homogenized ground beef into a 50-mL centrifuge tube. Add 2 mL water and 10 mL acetonitrile (ACN), then shake the tube vigorously for 1 min. Add contents of DisQuE pouch salts for CEN QuEChERS (p/n 186006813), and shake vigorously for 1 min. Centrifuge for 3 min at 4000 rpm, and take a 1-mL aliquot of the supernatant (top layer) for dSPE cleanup.

dSPE cleanup:

Transfer the 1-mL aliquot of supernatant to a 2-mL dSPE cleanup tube that contains 150 mg of magnesium sulfate, 50 mg PSA sorbent, and 50 mg C_{18} sorbent (p/n 186004830). Shake vigorously for 1 min. Transfer a portion of the supernatant to the LCMS Certified Vial for GC-MS analysis.

GC conditions

System:	Agilent 6890
Column:	Rxi-5Sil MS, 30 meter x 0.25 mm (I.D.), 0.25 µm df
Injection volume:	1 µL
Carrier gas:	Helium
Flow rate:	1.0 mL/min (constant flow)
Temp. program:	80 °C initial (hold for 1 min), 10 °C/min to 280 °C, and hold for 10 min
Sample vials:	LC-MS Certified (p/n 600000751CV)

MS conditions

Mass spectrometer: Waters Quattro micro GC

The mass spectrometer was operated in positive electron impact (EI+) mode. Data was collected at 70 electron energy (eV) using selected ion monitoring (SIR). The ions monitored consisted of the following, with the principal quantification ion listed first.

Compound	SIR (*m/z*)	Retention time (min)
Dimethoate	87.0, 93.0,125.0	11.8
Chlorpyrifos methyl	286.1, 288.1,125.0	13.1
Malathion	173.2, 127.1, 125.0	13.7
Tribufos	169.1, 202.2, 113.0	15.6
Coumaphos	362.3, 226.2, 210.1	19.6

Results and Discussion

Figure 1 shows GC-MS (SIR) chromatograms obtained from analysis of a beef sample spiked at 200 µg/kg (ppb) of each pesticide. Table 1 shows the recovery data. Recovery was calculated by comparing the SIR peak area for samples spiked prior to QuEChERS extraction (pre-spiked samples) with the SIR peak area for samples spiked after QuEChERS extraction (post-spiked samples).

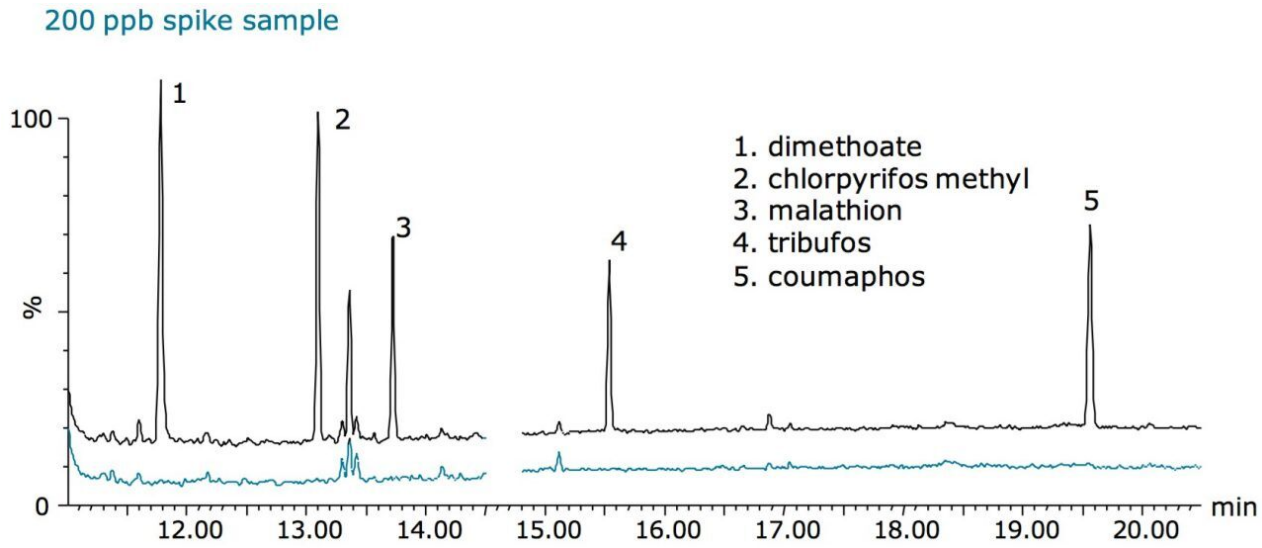

Figure 1. GC-MS chromatograms of unspiked ground beef samples (blue trace), and ground beef samples spiked with 200 ppb organophosphorus pesticides (black trace).

Compound *OP Pesticides*	Spike level (ppb)	% Recovery	% RSD
Dimethoate	20	89.4	7.2
Chlorpyrifos methyl	20	74.6	6.8
Malathion	20	93.9	13.8
Tribufos	20	71.8	3.7
Coumaphos	20	88.0	6.5
Dimethoate	200	98.9	6.1
Chlorpyrifos methyl	200	88.0	7.5
Malathion	200	107.0	9.6
Tribufos	200	74.8	6.9
Coumaphos	200	95.1	7.3

Table 1. Recovery for organophosphorus pesticides in ground beef using the DisQuE pouch for QuEChERS (n=5).

Conclusion

- The DisQuE pouch (QuEChERS) method is effective for extraction of organophosphorus pesticides from ground beef with consistent recoveries ranging from 75% to 100%.
- A straightforward dispersive SPE procedure provides excellent sample cleanup, prior to GC-MS analysis.
- A lower limit of quantification (LOQ) of 20 ppb was easily achieved using splitless injection. Using a large volume injector, sub-ppb detection limits are possible.
- The QuEChERS-based method allows the analyst to prepare more samples faster, with performance equivalent to alternative methods of analysis.

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