ChloroFiltr™

A NEW SORBENT FOR THE EFFICIENT REMOVAL OF CHLOROPHYLL IN QuEChERS APPLICATIONS

ABSTRACT

QuEChERS is a multiclass, multiresidue method for the extraction, clean-up and analysis of pesticides and other regulated compounds in food products and other biological matrices. Due to the wide variety and the complexity of natural product matrices, abundant quantities of chlorophyll, lipids, sterols or other components are co-extracted by this procedure. Since the development of the QuEChERS approach, numerous modifications and method improvements have evolved. These improvements are designed to enhance sample cleanup and quantification of analytes by removing interfering compounds to yield cleaner chromatograms during PTV/LVI GC/MS and LC/MS/MS. The QuEChERS method uses graphitized carbon black (GCB) to remove chlorophyll. While GCB is effective in removing chlorophyll from samples, it also removes planar pesticides such as chlorothalonil, coumaphos, hexachlorobenzene, thiabendazole, terbufos, quintozene and others, resulting in low recoveries for these analytes. UCT has developed a new sorbent that effectively removes chlorophyll without the loss of planar analytes. The ChloroFiltr sorbent is used in QuEChERS clean-up products in place of GCB. No method modifications are necessary. This paper discusses the use of ChloroFiltr in place of GCB and shows its effectiveness in removing chlorophyll.

INTRODUCTION

Proper sample preparation in QuEChERS analyses is important in order to achieve the highest accuracy and recovery values for pesticide residues. Natural products are complex matrices and contain large pigments molecules such as chlorophyll that are of no analytical interest. Most often chlorophyll interferes with identification of the compounds of interest by contamination of analytical instrumentation causing downtime reducing overall throughput. An ideal extraction method would selectively reduce or remove chlorophyll without affecting the actual analytical compounds of interest. Results would be cleaner chromatograms easing peak identification and interpretation.

During extraction procedures used in QuEChERS methods, chlorophyll, present in green agricultural products, is a significant interfering co-extractant. Graphitized carbon black (GCB) is added to dispersive SPE tubes for the removal of chlorophyll in plant samples to improve the performance of the QuEChERS method. However, significant problems are encountered using GCB. While GCB readily removes chlorophyll it also has strong affinity for many planar pesticides such as coumapahos, thiabendazole, quintozene and many others reducing the recovery of many of these pesticides of interest.

UCT has developed a new sorbent to selectively remove chlorophyll from green plant residues while leaving polar pesticides behind in the acetonitrile extract. This new sorbent is known as the ChloroFiltr. The ChloroFiltr sorbent is a white, cross-linked polymeric powder compatible with extraction solvents used with the QuEChERS method. It is a strong yet selective sorbent for chlorophyll and used in place of graphitized carbon black (GCB) in UCT dispersive extraction centrifuge tubes. Substitution of ChloroFiltr for GCB does not affect established laboratory procedures. The use of the ChloroFiltr solid-phase sorbent has been shown to strongly adsorb chlorophyll from plant material reducing unwanted background noise and interfering chromatographic peaks. Its use over GCB offers significant reduction in the sorption of polar pesticides resulting in significantly high recoveries of compounds of analytical interest over GCB.

This poster shows the results of a study using spinach as the test matrix. Over 400 pesticides compounds were spiked at concentrations ranging between 25-100 ng/g of sample. Spiked samples were divided into LC or GC amenable groups for analysis by GC/MS or LC/MSMS. These spinach samples were split then extracted using standard QuEChERS protocol, one sample using GCB and the other using ChloroFiltr. Results are plotted in bar graph format. Results for ChloroFiltr are shown in with green bars and show the % improvements of pesticide recovery over that obtained using only GCB.

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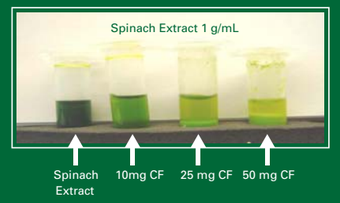
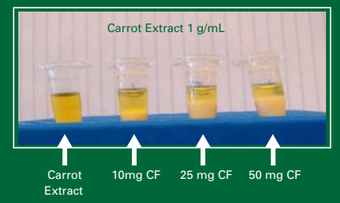
QuEChERS Spinach Extract (ACN) Showing Effectiveness of ChloroFiltr (CF) Used in Place of GCB



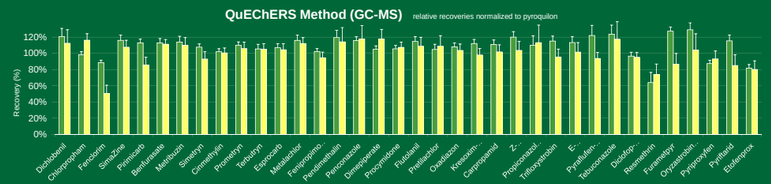
ChloroFiltr Does Not Remove Carotenoids Showing the Specificity of the Sorbent

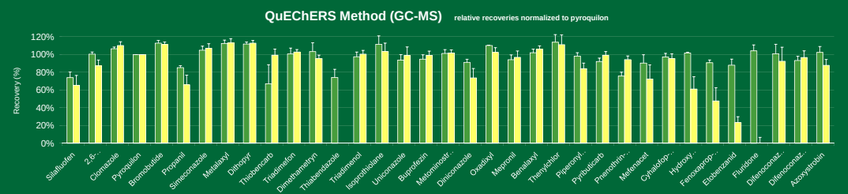


PRODCEDURES:

Determination of Acid Herbicides Residues by QuEChERS Using LC-MS/MS

1) Weigh 10 ± 0.1 g of sample into a 50 mL centrifuge tube
2) For spike sample mix working standard at 0.05 ppm level
3) Add 10 mL of 5% formic acid in acetonitrile
4) Shake 1 minute
5) Add 4 ± 0.2 g of magnesium sulfate anhydrous, (MgSO4), 1 ± 0.05 g NaCl, 1 ± 0.05 g tri-sodium citrate dehydrate and 0.5±0.03 g di-sodium hydrogen citrate sesquihydrate
6) Shake 1 minute
7) Centrifuge at 3,400 rpm for 5 minutes
8) Transfer all solution to another 50 mL centrifuge tube
9) Add 0.25 g of UCT C18, 1.5 g MgSO4 & 0.1 g alumina-N to solution
10) Add 0.35 g ChloroFiltr or graphitic carbon black
11) Shake
12) Centrifuge @ 3,400 rpm for 5 minutes
13) Transfer 5 mL aliquot to 10 mL tube
14) Evaporate to dryness @ 40 ± 2°C
15) Dissolve residue using 1 mL of mobile phase
16) Sonicate to dissolve if needed
17) Filter extract through a 0.2µm filter into LC vial

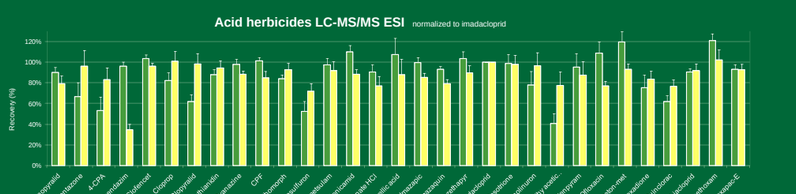




PRODCEDURES:

Determination of Multiresidues Using QuEChERS Extraction and UPLC-MS/MS

1) Weigh 10 ± 0.1 g of sample into a 50 mL centrifuge tube
2) Add 10 mL acetonitrile
3) For spike sample mix working standard at 0.05 ppm level
4) Cover the tube and vortex 1 minute
5) Add 4 ± 0.2 g of MgSO4 anhydrous, 1 ± 0.05 g NaCl, 1±0.05 g tri-sodium citrate dehydrate, 0.5 ± 0.03 g di-sodium hydrogen citrate sesquihydrate
6) Vortex 1 min and centrifuge at 3,400 rpm for 5 minutes
7) Transfer 1 mL of extract to a microcentrifuge tube containing 0.025 g PSA, anhy MgSO4, and 0.05 g ChloroFiltr or GCB
8) Vortex and centrifuge
9) Filter extract through 0.2µm filter into LC vial

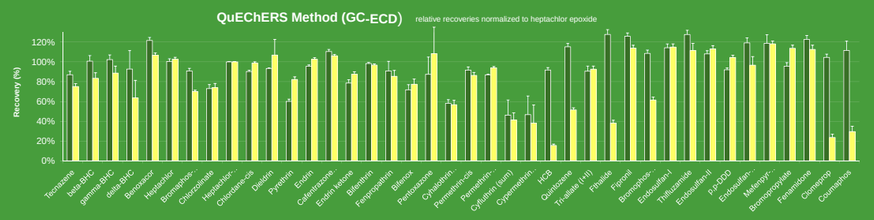


PRODCEDURES:

Determination of Multiresidues Using QuEChERS Extraction by GC-FPD

1) Weigh 10 ± 0.1 g of sample into a 50 mL centrifuge tube
2) Add 10 mL of 0.1% acetic acid in acetonitrile
3) For spike sample with the mixed working standard at 0.10 ppm level
4) Close the tube and vortex 1 minutes
5) Add 4 ± 0.2 g of magnesium sulfate anhydrous (MgSO4), 1 ± 0.05 g NaCl, 1±0.05 g tri-sodium citrate dehydrate and 0.5 ± 0.03 g di-sodium hydrogen citrate sesquihydrate.
6) Vortex for 1 minute
7) Centrifuge @ 3,400 rpm for 5 minutes
8) Transfer 5 mL of extract to centrifuge tube containing 0.25 g PSA, 0.75 g $MgSO_4$
9) Add 0.25 g ChloroFiltr or GCB
10) Shake then centrifuge @ 3,400 rpm for 5 minutes

LEGEND	= Chloro Filtr = Graphic Carbon



THE USE OF CHLOROFILTR CONTAINED IN DISPERSIVE QUECHERS TUBE REQUIRES NO CHANGE FROM CURRENT PROCEDURES!

GCB removes chlorophyll but also removes planar pesticides reducing recovery of these analytes

ENVIRO