Increasing Efficiency and Pesticide Recovery from the QuEChERS Approach for Fruit and Vegetable Products Using Ceramic Homogenizers

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Agilent Technologies

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Introduction

QuEChERS

- **Q**uick, **E**asy, **Ch**eap, **E**ffective, **R**obust and **S**afe
- Developed by the US FDA and EU Food Regulatory Agencies
- Procedure was validated in 2003, "toddler stage"
- Extraction and analysis of pesticides in food product
- Advancements in the analysis of other components
  - PCB, PAH, Antibiotics, Pharmaceuticals, Toxins
  - Other food-stuffs; meat fish, grains, nuts, juices, oils
  - Other matrices; soil, wine, biologicals

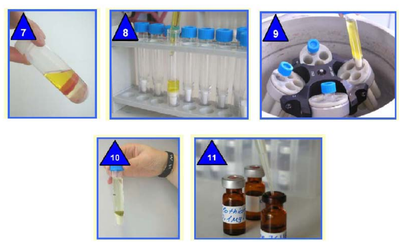
- QuEChERS
  - Basic procedure
  - Concerns and variance with procedure
- Homogenizers in QuEChERS extraction
  - Description and characterization
  - Implementation
  - Data and results

Experimental

QuEChERS Procedure

- 3 step process: extraction, dispersive-SPE, analysis
- 1st Extraction

- 2nd Dispersive-SPE



Experimental

Concerns and Variance with Procedure

- 1st Step Extraction
  - After addition of organic to sample, add extraction salts
    - Requires vigorous shaking, 1 min or greater
    - Variations is time and force shaking affect recovery
    - Variation from beginning to end of extraction, 100+ samples
    - Variations between technicians between labs
    - Affect recovery of pesticides

Results and Discussion

Homogenizers in QuEChERS Procedure

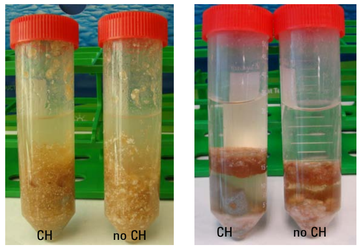
- Homogenizers are inert ceramic pieces
- Aggressively force the partitioning between the water and organic layers in QuEChERS
- Break up salt agglomerates
- Increase grinding/pulverizing of homogenized matrix

Ceramic Homogenizers (CH)

- Available in 3 sizes; 50 mL, 15 mL, and 2 mL
- Place 2 CH per tube

- Process samples the same
- Weight and angled sides of the ceramic homogenizers increase shaking capabilities

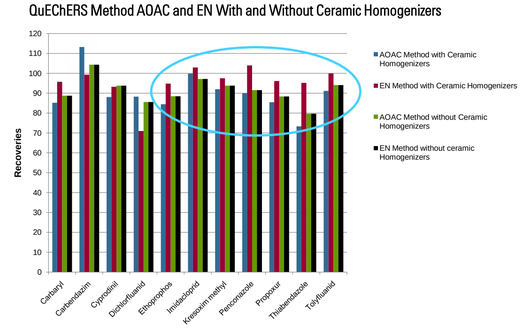
QuEChERS Extraction: With and Without Ceramic Homogenizers



- Determination of inertness of CH in the presence of matrix and pesticides
- Study to extract samples with and without CH by manual shaking
  - AOAC method 2007.01
    - 6 g $MgSO_4$, 1.5 g NaAcetate
  - EN method 15662
    - 4 g $MgSO_4$, 1 g NaCl, 1 g NaCitrate, 0.5 g disodium citrate sesquihydrate

Results and Discussion

QuEChERS Method AOAC and EN With and Without Ceramic Homogenizers



Graph 1: Pesticides used in QuEChERS study with and without ceramic homogenizers

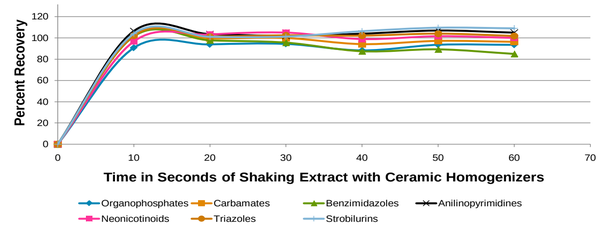
Procedure after Addition of Ceramic Homogenizers

- 1st Step Extraction
- With Ceramic Homogenizers
  - After addition of organic to sample, add extraction salts
    - ✓ Requires vigorous shaking, 1 min or greater
    - ✓ Variations is time and force shaking affect recovery
    - ✓ Variation from beginning to end of extraction, 100+ samples
    - ✓ Variations between technicians between labs
    - ✓ Affect recovery of pesticides
    - ➢ *"SHAKING"*

QuEChERS Extraction: Require Shaking for 1 min

- Investigate the ceramic homogenizers
  - Could they reduce the time required for shaking
    - Maintain acceptable recovery and RSD for all pesticides, classes
  - Setup a timed extraction study
    - Add the extraction salts
    - Shake for 10 seconds, adding 10 sec for each additional set
    - 10, 20, 30, 40, 50, 60 sec

Average Recovery of Several Classes of Pesticides within 20 Second Shaking Extraction and Ceramic Homogenizers



Graph 2:Pesticides used in study: Acephate, Carbaryl, Carbendazim, Cyprodinil, Imidacloprid, Imidazalil, Methamidophos, Penconazole, Propoxur, Pymetrozine, Thiabendazole, Thiophanate-methyl, Ethoprophos, Kresoxim-methyl ; Apple matrix

Conclusions

- QuEChERS procedure is very efficient "just enough" sample preparation
- Ceramic homogenizers addresses many of the "shaking" issues
- Inert, improves recoveries and RSDs for the pesticides and other compounds
- Disposable, can be used manually or with mechanical shakers
- Drastically decreases "shaking" time by 70%
- Improve sample throughput