

# Increased Pesticide Recovery in Fruit and Vegetable Products using the Geno/Grinder® with the QuEChERS Method



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## Abstract

Samples prepared for pesticide analysis according to the QuEChERS method are typically combined with solvent and buffering salts, then mixed by shaking for 1 min. by hand. In this study, GC-MS results of samples prepared using the standard, manual QuEChERS methods were compared with results for samples mechanically mixed using the Geno/Grinder.



## Experiment 1

### Sample Preparation

- ▶ Fresh strawberries, apples, and celery cut into small chunks (¼ - ½ in.)
- ▶ Spiked with 5 ppm of a solution of CAL-CARB 13 (a mix of 13 common pesticides, Spex CertiPrep) and refrigerated overnight.

Samples prepared as follows:

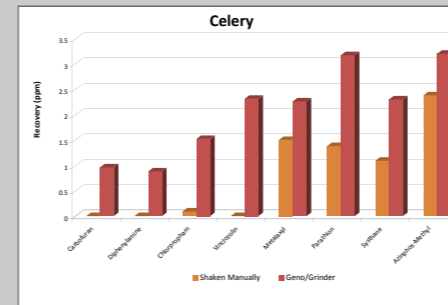
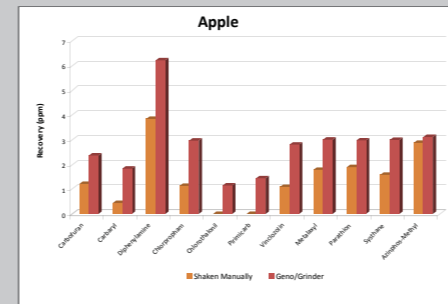
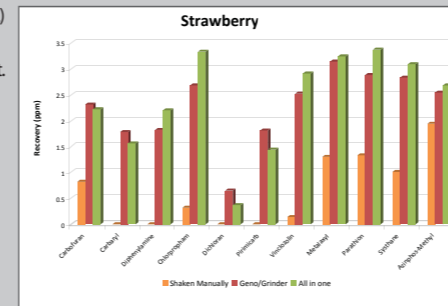
	Sample Set A	Sample Set B
Homogenization	Single-serve blender	Geno/Grinder @ 1500 strokes/min. in 50 ml tubes, using 3 ceramic grinding cylinders
Add Solvent	15 ml Acetonitrile (1% Acetic Acid)	15 ml Acetonitrile (1% Acetic Acid)
Add Salts	6 g MgSO <sub>4</sub> 1.5 g NaOAC	6 g MgSO <sub>4</sub> 1.5 g NaOAC
Shake	1 min. by hand	1 min. on Geno/Grinder at 1500 spm
Centrifuge	3 min. @ 3500 rpm	3 min. @ 3500 rpm

- ▶ For strawberry, a third set of samples was prepared: spiked strawberry chunks were ground in the presence of the solvent and salts (All in one).

### Clean Up and Analysis

- ▶ Removed supernatant liquid from each sample.
- ▶ Treated with PSA (25mg/ml extract), and GCB (5mg/ml extract).
- ▶ Mix:
  - Set A: shaken 30 sec. by hand.
  - Set B: shaken 30 sec. at 1500 spm on Geno/Grinder.
- ▶ Centrifuged all samples at 3200 rpm for 1 min.
- ▶ Removed supernatant liquid.
- ▶ Evaporated samples to near dryness.
- ▶ Brought total volume to 1 ml in toluene.
- ▶ Analyzed by GC-MS using a SCAN method.

### Results



## Experiment 2

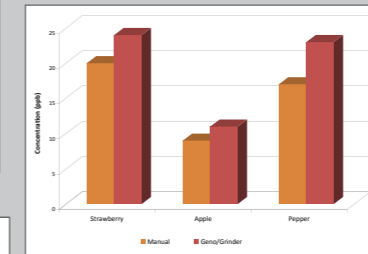
### Sample Preparation

- ▶ Produce samples obtained in homogenized form from UC Davis (IR-4 Project).
- ▶ Samples contained known pesticides in unknown concentrations.
- ▶ Samples prepared in 50 ml centrifuge tubes:

Matrix	Pesticide	Sample Size	ACN (1% Acetic Acid)	MgSO <sub>4</sub>	Salt 2
Strawberry	Flutianil	15 g	15 ml	6 g	1.5 g NaOAC
Apple	Flutianil	15 g	15 ml	6 g	1.5 g NaOAC
Green Pepper	Etoxazole	15 g	15 ml	6 g	1 g NaCl

- ▶ Shaking method:
  - Sample set A: 1 min. by hand
  - Sample set B: 1 min. on Geno/Grinder at 1500 spm using 2 ceramic grinding cylinders per 50 ml tube.
- ▶ Centrifuged samples: 3 min. @ 3500 rpm.
- ▶ Clean up as in Experiment 1 using PSA, GCB, and MgSO<sub>4</sub>.
- ▶ Analyzed by GC-MS using a SIM method.

### Results



Matrix	Manual			Geno/Grinder			Increase using Geno/Grinder (%)
	Conc. (ppb)	Std. Dev.	RSD (%)	Conc. (ppb)	Std. Dev.	RSD (%)	
Strawberry	20	1.0	5	24	1.7	7	20
Apple	9	0.8	9	11	1.4	12	18
Green Pepper	17	2.5	15	23	2.4	10	35

### Conclusions

- ▶ Pesticide recovery increased in all cases when the Geno/Grinder was used to shake samples (≥ 18%).
- ▶ Use of the Geno/Grinding gives improved sample mixing and pesticide extraction.
- ▶ Soft fruit can be homogenized in the presence of solvent and salts.
  - In Experiment 1, recovery was as good or better for strawberries when homogenized in the presence of solvents or salts.

### GC-MS Conditions

- ▶ HP 5890-GC
  - HP-5 capillary column (30 m x 0.25 µm)
  - Injection volume: 2.0 µL
  - Program: 70°C, 1 min; 20°C/min up to 230°C
  - Inlet: Splitless
    - Initial temp: 250°C
    - Purge: 50.0 ml/min for 2.00 min
    - Total flow: 54.0 ml/min
- ▶ 5972-MSD
  - SCAN & SIM acquisition methods
  - EM voltage: 2600
  - Scan range: 35-450 m/z

### Acknowledgement

Special thanks to Dr. Matt Hengel, UC Davis for providing homogenized material used in Experiment 2.