



eXtremelFV[®] vs SPE for the Analysis of Pesticides in Orange Juice by GC/MS

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Abstract

Pesticides act as toxins when found in sufficient quantities as residues in food. This is of particular importance for orange juice because it is consumed in high quantities by children. Sensitive, rapid, and cost effective analytical methods are required in order to reduce the risk to consumers.

Solid Phase Extraction (SPE) is a common sample preparation technique used prior to GC or LC analysis of pesticides in food. Typically, SPE is used to concentrate analytes, reduce interference from co-eluting molecules or to clean up/"filter" sample particulates. Drawbacks to the use of SPE include cost, sample preparation time, large sample volumes, use and disposal of organic solvents, and potentially poor recoveries. The continuing development of higher sensitivity instrumentation and improved filtration devices has led many labs to investigate whether methods can be adapted to eliminate the SPE step.

Thomson eXtreme[®] Filter Vials offer multi-layer filtration for viscous samples and samples containing up to 30% solid particulates. Filtration time from unfiltered sample transfer to filtered sample in an autosampler ready vial is only 15 seconds. The filter vial consists of two parts: a filter vial shell and a plunger which includes the multi-layer filter on one end and a vial cap on the other end. Samples are filtered by pipetting the sample into the filter vial shell, inserting the plunger into the shell, and then pushing the plunger into the shell.

Prior to the introduction of the eXtremelFV®s, many samples containing high levels of particulates were only "filtered" by using an SPE step in the method. These methods are readily amendable to the replacement of the SPE step with a much faster and lower cost eXtremelFV® step.

Experiment

Samples were prepared and analyzed at Micro Quality Labs, Burbank, CA.

Sample Preparation

- Spike 10mL of commercially available High Pulp Orange Juice with 1mL of 1 ppm pesticide standard mix in a 40mL vial.
- 2. Add one pack (approximately 6g) of Restek Extraction Salts (Restek catalog #26236) to the spiked orange juice.
- 3. Extract the spiked orange juice with 4 x 25mL portions of methylene chloride.
- 4. Concentrate to dryness using a Turbovap II concentrator.
- 5. Dissolve the residue in approximately 10mL of acetonitrile.
- 6. Vortex and sonicate the re-suspended residue with frequent swirling.
- 7. Split the re-suspended residue into two 5mL portions.
- 8. Dilute each 5mL portion with acetonitrile to 10mL using a

volumetric flask.

9. Label one flask "for SPE" and the other "for Thomson eXtreme|FV[®]".

eXtreme|FV® Cleanup Prior to Analysis

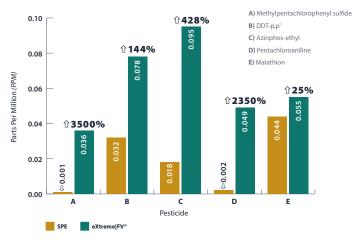
- 1. Add 400 μ L of the re-suspended residue from the flask labeled "for Thomson eXtremelFV®" to the shell of one Thomson eXtremelFV® 0.45 μ m, PTFE (P/N 85540-500).
- 2. Insert plunger completely.

Analysis

Samples were analyzed utilizing an Agilent Technologies[®] GC/MS, 7000 Triple Quad system equipped with a 7890A GC system and 7693 auto sampler.

SPE -vs- eXtremelFV®

Comparison of spiked pesticide recoveries



Conclusions

The Thomson eXtremelFV[®], 0.45µm PTFE, (P/N 85540) yielded 26% higher recoveries on average when tested with 87 common pesticides. In the cases highlighted in the results table, greater than 428% recovery increases were seen. In the case of Hexachlorobenzene, no pesticide was detected in the sample prepared by SPE and 0.019 ppm was detected in the sample prepared with the eXtremelFV[®]. The use of Thomson eXtremelFV[®], 0.45µm PTFE, as a substitute for SPE conforms to USP Method 561.

The results show Thomson eXtremelFV®s offer a viable alternative with higher recovery and less preparation time compared to SPE for the preparation of juices prior to pesticide analysis. \frown

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