series	cap color	membrane	pore size	part #
eXtreme|FV®	●	PTFE	0.45µm	85540

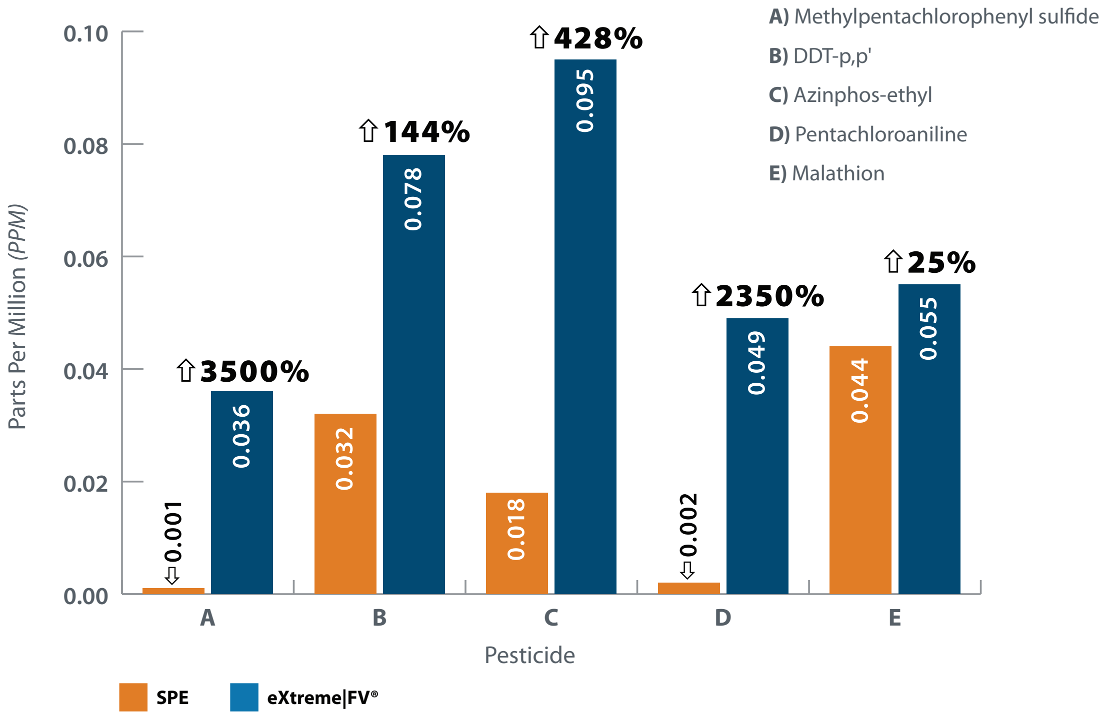
eXtreme|FV® vs SPE for the Analysis of Pesticides in Orange Juice by GC/MS

Authors: Uday Sathe1, Karine Aylozyan1, Lisa Wanders2, Joe Machamer2, & Sam Ellis2

Micro Quality Labs1 | Thomson Instrument Company2

SPE -vs- eXtreme|FV®

Comparison of spiked pesticide recoveries



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 eXtreme|FV®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 eXtreme|FV® step.

Experiment

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

Sample Preparation

1. 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®".

SPE Cleanup Prior to Analysis - Restek 6mL Combo SPE Cartridge

1. Wash one Restek 6mL Combo SPE Cartridge (packed with 200mg CarboPrep 200 and 400mg PSA Restek catalog #26127) with acetonitrile.
2. Add the 10mL portion of the re-suspended residue from the flask labeled "for SPE" to the SPE cartridge.
3. Elute the sample from the cartridge with 50mL of acetonitrile.
4. Concentrate the eluted sample to 10mL using a Turbovap II concentrator.

eXtreme|FV® Cleanup Prior to Analysis

1. Add 400µL of the re-suspended residue from the flask labeled "for Thomson eXtreme|FV®" to the shell of one Thomson eXtreme|FV® 0.45µm, PTFE (Thomson Part Number 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.

Compound/Sample Name	Avg. PPM SPE+ Routine Syringe Filter	Avg. PPM eXtreme|FV® PTFE (W/O SPE)
Alachlor	0.043	0.053
Aldrin	0.025	0.032
Azinphos-ethyl	0.018	0.095
Azinphos-methyl	0.023	0.115
BHC-alpha (benzene hexachloride)	0.026	0.033
BHC-beta	0.054	0.073
BHC-delta	0.062	0.081
BHC-gamma (Lindane, gamma HCH)	0.032	0.043
Bromophos-ethyl	0.025	0.057
Bromopropylate	0.063	0.076
Carbophenothion	0.051	0.071
Chlordane-cis (alpha)	0.04	0.052
Chlordane-oxy	0.034	0.042
Chlordane-trans (gamma)	0.039	0.049
Chlorfenvinphos	0.061	0.071
Chlorpyrifos	0.035	0.047
Chlorpyrifos-methyl	0.035	0.046
Cyfluthrin I	0.082	0.113
Cyhalothrin (lambda)	0.076	0.091
Cypermethrin I (Zeta)	0.082	0.117
Cypermethrin II {CAS # 52315-07-8}	0.08	0.113

Compound/Sample Name	Avg. PPM SPE+ Routine Syringe Filter	Avg. PPM eXtreme|FV® PTFE (W/O SPE)
Cypermethrin III (Beta)	0.058	0.104
Cypermethrin IV {CAS # 52315-07-8}	0.07	0.097
DCPA (Dacthal, Chlorthal-dimethyl)	0.04	0.048
DDD-o,p'	0.052	0.06
DDD-p,p'	0.056	0.066
DDE-o,p'	0.043	0.039
DDE-p,p'	0.045	0.057
DDT-o,p'	0.035	0.065
DDT-p,p'	0.032	0.078
Deltamethrin	0.053	0.102
Diazinon	0.028	0.035
Dicofol	0.033	0.028
Dieldrin	0.041	0.052
Dimethoate	0.061	0.077
Endosulfan I (alpha isomer)	0.041	0.076
Endosulfan II (beta isomer)	0.053	0.065
Endosulfan sulfate	0.061	0.074
Endrin	0.045	0.058
Ethion	0.057	0.069
Etrimfos	0.03	0.038
Fenchlorphos oxon	0.047	0.061
Fenitrothion	0.041	0.053
Fenpropathrin	0.068	0.078
Fensulfothion	0.1	0.117
Fenthion	0.041	0.05
Fenthion sulfone	0.081	0.107
Fenthion sulfoxide	0.106	0.134
Fenvalerate I	0.076	0.106
Fenvalerate II {CAS # 51630-58-1}	0.055	0.073
Fluvalinate-tau I	0.078	0.082
Fluvalinate-tau II {CAS # 102851-06-9}	0.058	0.084
Fonofos	0.023	0.028
Heptachlor	0.022	0.029
Heptachlor endo-epoxide (isomer A)	0.039	0.048
Heptachlor exo-epoxide (isomer B)	0.037	0.045
Hexachlorobenzene	0	0.019
Malaoxon (metabolite of Malathion)	0.07	0.086
Malathion	0.044	0.055
Mecarbam	0.052	0.062

Compound/Sample Name	Avg. PPM SPE+ Routine Syringe Filter	Avg. PPM eXtreme|FV® PTFE (W/O SPE)
Methidathion	0.063	0.08
Methylpentachlorophenyl sulfide	0.001	0.036
Mirex	0.042	0.056
Octachlorodipropyl ether (S421)	0.021	0.047
Omethoate	0.052	0.061
Paraoxon	0.071	0.08
Parathion	0.039	0.049
Parathion-methyl	0.035	0.045
Pendimethalin	0.038	0.048
Pentachloroaniline	0.002	0.049
Pentachloroanisole	0.017	0.021
Permethrin I	0.068	0.097
Permethrin II (trans)	0.071	0.115
Phosalone	0.005	0.089
Phosmet	0.031	0.104
Piperonyl butoxide	0.117	0.105
Pirimiphos-ethyl	0.044	0.053
Pirimiphos-methyl	0.04	0.05
Procymidone	0.064	0.082
Profenofos	0.058	0.071
Prothiofos	0.033	0.06
Quinalphos	0.042	0.061
Quintozene	0.02	0.028
Ronnel (Fenchlorphos)	0.031	0.04
Tecnazene (TCNB)	0.011	0.014
Tetradifon	0.062	0.077
Vinclozolin	0.043	0.052

THOMSON INSTRUMENT COMPANY Solutions ™ At Work

GCMS Data (links to PDF)

Without Internal Spike
SPE w/ Filtration | http://bit.ly/spe-spike
eXtreme|FV® 85540 | http://bit.ly/extreme-no-spike

With Internal Spike
USP 36 <561> with 0.1 PPM | http://bit.ly/usp-spike
eXtreme|FV® with 0.1 PPM | http://bit.ly/extreme-with-spike

Conclusions
The Thomson eXtreme 0.45µm, PTFE Filter Vials patented (Part#: 85540-500) 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 eXtreme|FV®. The use of Thomson eXtreme 0.45µm, PTFE Filter Vials as a substitute for SPE conforms to USP Method 561.

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

Time = Money

To process 6 samples	Traditional SPE or GPC	QuEChERS with SPE clean-up	QuECHERS with Thomson Filter Vial clean-up*	Thomson Filter Vial Benefits
Estimated (min.)	120	20	10	1
Solvent used (mL)	90	10-15	5	0.5
Chlorinated waste (mL)	30	none	none	none
Specialized equipment	Separatory funnels, water bath, evaporator, etc.	Vacuum pump, vacuum manifold	none	none

*Significant time & money savings because lengthy wash steps are eliminated!