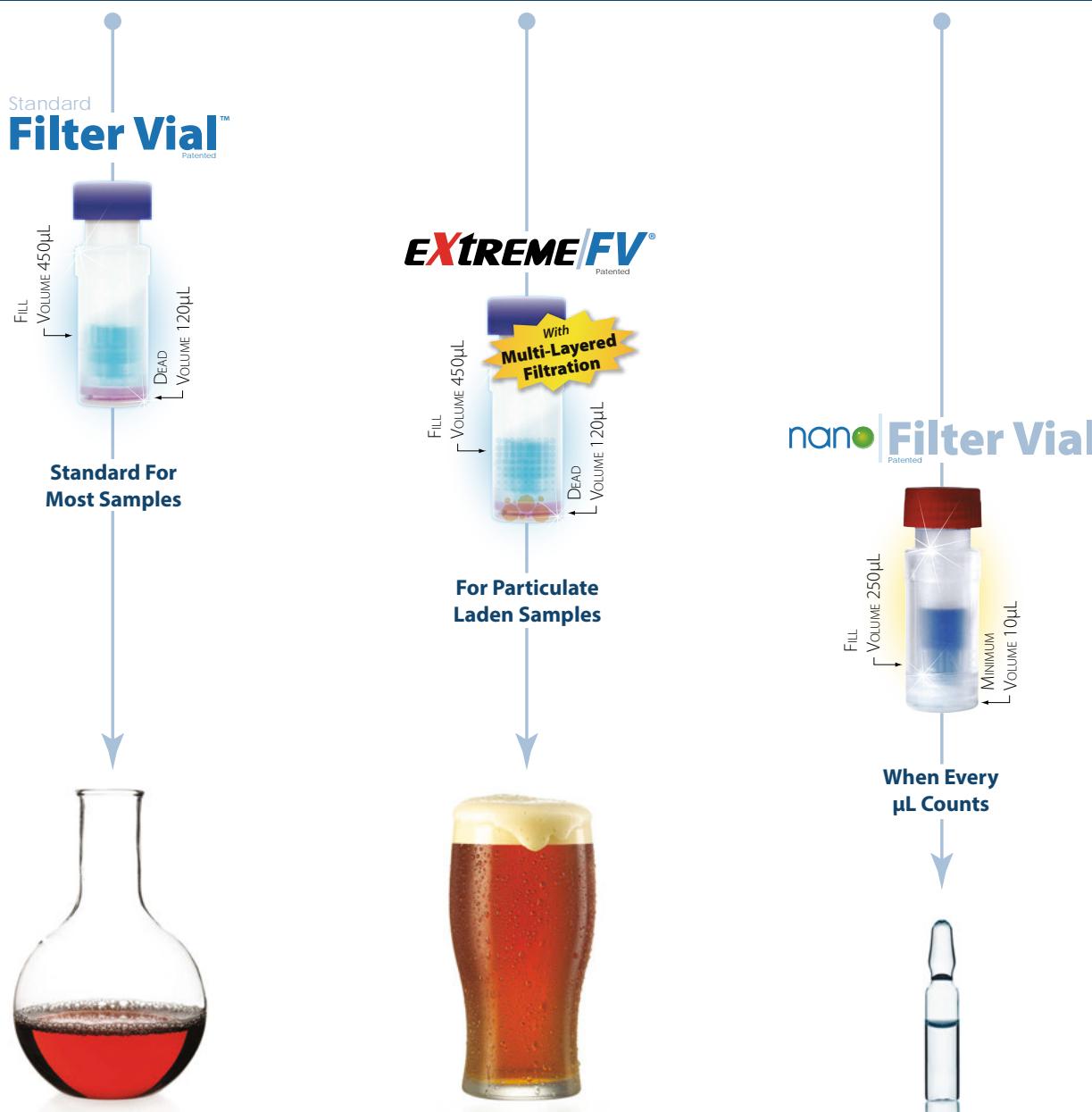


Filter Vial CATALOG

APPLICATION NOTES



BGB GC|LC
MS|OF

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Filter Vials have the following patents:
US 7,790,117, 8,211,384, 8,383,006, 8,322,539, EU patent 2268252,
EP2268252B1, Singapore Patent 164909, Worldwide Patents Pending

For up to date patent and trademark information please see htslabs.com.



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Table of Contents

Overview	4
Application Section	5
Membrane Material	6
Membrane Pore Size	6
Leachables Study	7
Standard Filter Vials	10
Open Access LCMS	11
Protein Precipitation	12
How to Mass Spec Your TLC Spots with Filter Vials	13
Corn Syrup Analysis Saccharides (E-61 CRA Test)	14
eXtreme FV®	15
Improved Sample Preparation Methods for Athlete	
Doping Analysis of Common Compounds in Urine by LCMS	16
Pesticide Applications - Soil & Vegetation	21
eXtreme FV® vs SPE for the Analysis of Pesticides in Orange Juice	23
Supplement Analysis of Huperzine A by HPLC	28
Antibody Analysis with eXtreme FV®	29
Analysis of Nitrosamines in Tobacco	30
nano Filter Vials™	31
10µL Filtration with 2µL Injection	32
Appendix	35
Chemical Compatibility	35
Compound Compatibility	40

Filter Vial Overview

Thomson Filter Vials (patented) are a single system which replaces HPLC Vials, HPLC Caps, Syringes, & Syringe Filters for the filtration of samples. In 15 seconds, Filter Vials allow for Sample Preparation of unfiltered samples to filtered samples in an autosampler ready vial. The filter vial consists of two parts: a filter vial shell and a plunger which includes a 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.

Thomson Filter Vials simplify general filtration by replacing syringes & syringe filters, centrifuging spin columns and/or liquid-liquid extractions.

Applications for Thomson Filter Vials include all sample types to be analyzed by HPLC, UHPLC, LC-MS and GC-MS.

Filter Vial



Max Fill Vol. 450 μ L
Dead Vol. 120 μ L

Standard Filter Vials (120 μ L Dead Volume)

Thomson Standard Filter (patented) can be used for samples containing less than 10% solid particulates. The filter vial consists of two parts: a filter vial shell and a plunger which includes a single layer filter on one end and a vial cap on the other end.

Applications for Thomson Standard Filter Vials include filtration of catalysts from organic and medicinal chemistry synthesis reactions, saccharide analysis in corn syrup, and in-vial protein precipitation.

eXtreme|FV®



Max Fill Vol. 450 μ L
Dead Vol. 120 μ L

eXtreme|FV® (Multi-Layered Filtration)

Thomson eXtreme|FV® (patented) offer multi-layer filtration for viscous samples and samples containing up to 30% solid particulates. The filter vial consists of two parts: a filter vial shell and a plunger which includes a multi-layer filter on one end and a vial cap on the other end.

eXtreme|FV® allow for compounds to be separated from the matrix which, results in both a higher signal to noise ratio and peaks that are more differentiated.

Prior to the introduction of the eXtreme|FV®, many samples containing high levels of particulates were “filtered” by using an SPE step in the method. These methods are readily amendable to the replacement of the SPE step using a rapid and lower cost eXtreme|FV® step.

Applications for Thomson eXtreme|FV® include filtration of cell and cell debris from cell culture; pesticide analysis in food, tissue, soil, and water; and toxicology analysis in blood and urine.

nano|Filter Vial™



Max Fill Vol. 250 μ L
Min Fill Vol. 10 μ L (for 2 μ L injection)

nano|Filter Vials™ (10 μ L Minimum Volume)

Thomson nano|Filter Vials™ offer a very low dead volume allowing one to filter as little as 10 μ L of sample with enough remaining filtrate to make a 2 μ L injection. The filter vial consists of two parts: a filter vial shell with mating bottom surface and a plunger which includes a filter on one end and a screw cap vial on the other end.

Applications include the analysis of enzymes, peptides, DNA, RNA, synthesis reaction intermediates, finished products, saliva, samples available in low volumes, in-vial evaporation and re-suspension for sample concentration and buffer/solvent change.

Application Selection



**See our Technical Library
for more Applications**
htslabs.com

nano|FV™ nano|FV™ Pre-Slit Cap Standard Filter Vial Low Evaporation extreme|FV®

10µL-100µL	●	●			
120µL-450µL			●	●	●
UPLC Compatible	●	●	●	●	●
GCMS Compatible	●			●	
≤ 30% Solids					●
Viscous		●			●
Replacement for SPE					●
General Liquids < 10% solids	●	●	●	●	●
Cell Fermentation	●	●	●	●	●
Particulate Removal	●	●	●	●	●
Automation Compatible	●	●	●	●	●
Small Molecules	●	●	●	●	●
Food & Supplements			●	●	●
Toxicology	●	●	●	●	●
Pesticides	●	●	●	●	●
Environmental	●	●	●	●	●
Sterile Testing	●				

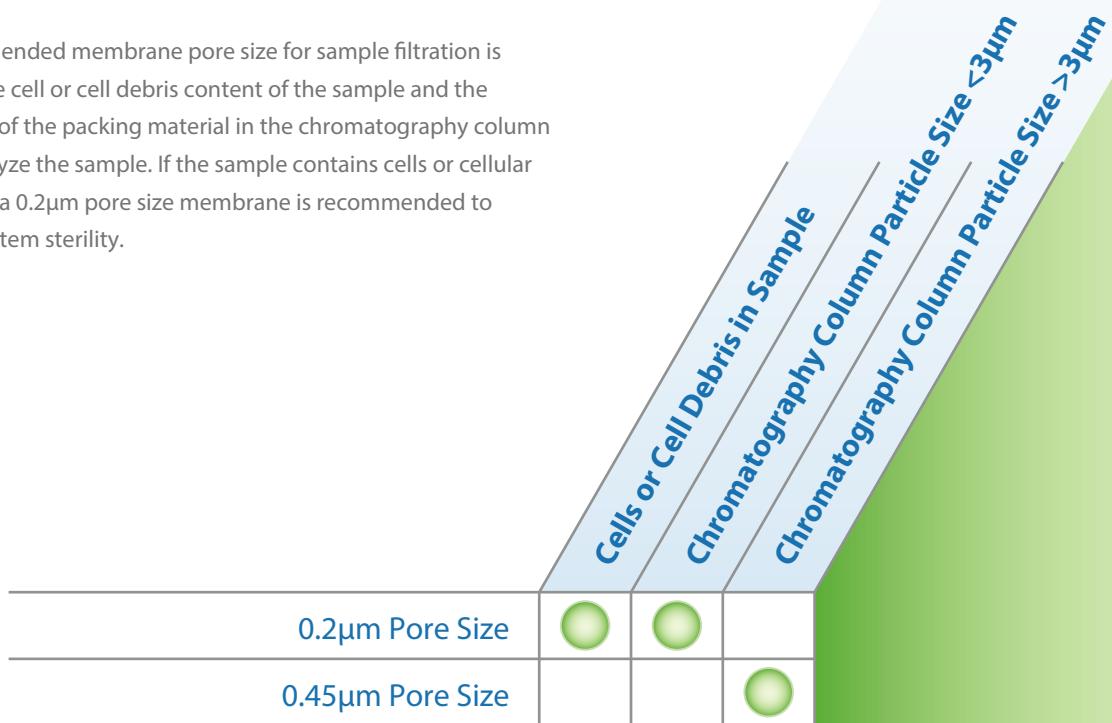
Filter Vial Membrane Material

The recommended membrane for sample filtration is based on the percentage of organic solvent in the sample and the amount of protein binding.

	AQUEOUS	ORGANIC	LOW PROTEIN BINDING
PTFE			
PVDF			
Nylon			
PES			

Filter Vial Membrane Pore Size

The recommended membrane pore size for sample filtration is based on the cell or cell debris content of the sample and the particle size of the packing material in the chromatography column used to analyze the sample. If the sample contains cells or cellular debris, then a 0.2µm pore size membrane is recommended to maintain system sterility.



Filter Vial Leachables

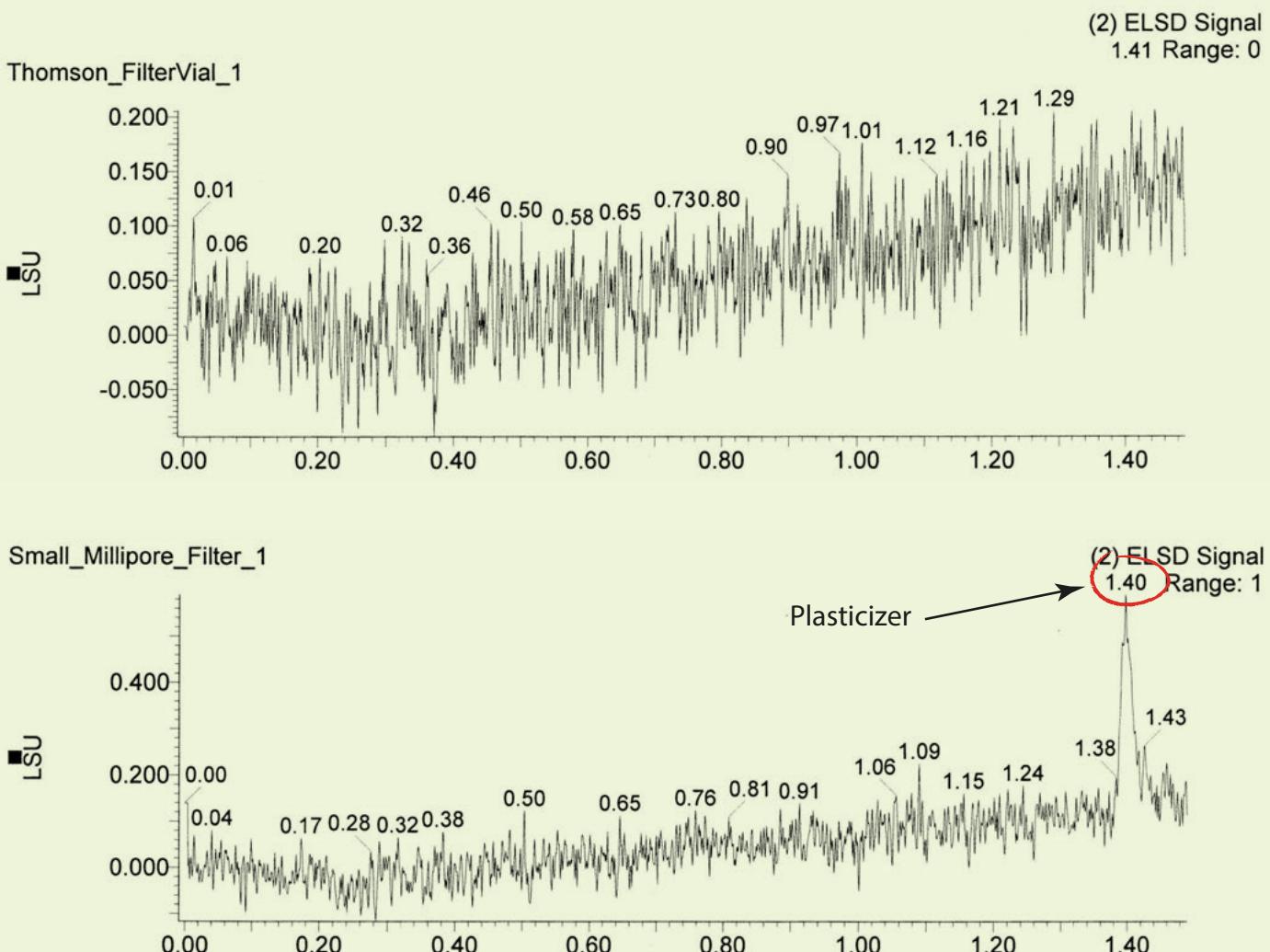
Thomson Filter Vials are manufactured without the use of plastizers or mold release agents making them LC/MS clean.

Testing with ELSD, PDA, and MS detection by Takeda Pharmaceutical showed no leaching from Thomson Standard Filter Vial with a 0.45um, PTFE membrane compared to significant leaching from Millipore Millex-FH® Filter, 0.45µM, hydrophobic PTFE, 4mm.

Method: A. Water B. ACN 45-90% with .05% TFA Ballistic Gradient over 1.4 minutes using Waters® Acquity® UPLC Thomson Filter Vial (patented) Part # 35540-500 Filter Vial 0.45µM hydrophobic PTFE, w/ Pre-Slit Cap Millipore Syringe Filter Part #:SLFHR04NL Millex-FH® Filter, 0.45µM, hydrophobic PTFE, 4mm, non-sterile

Plasticizer Leachable

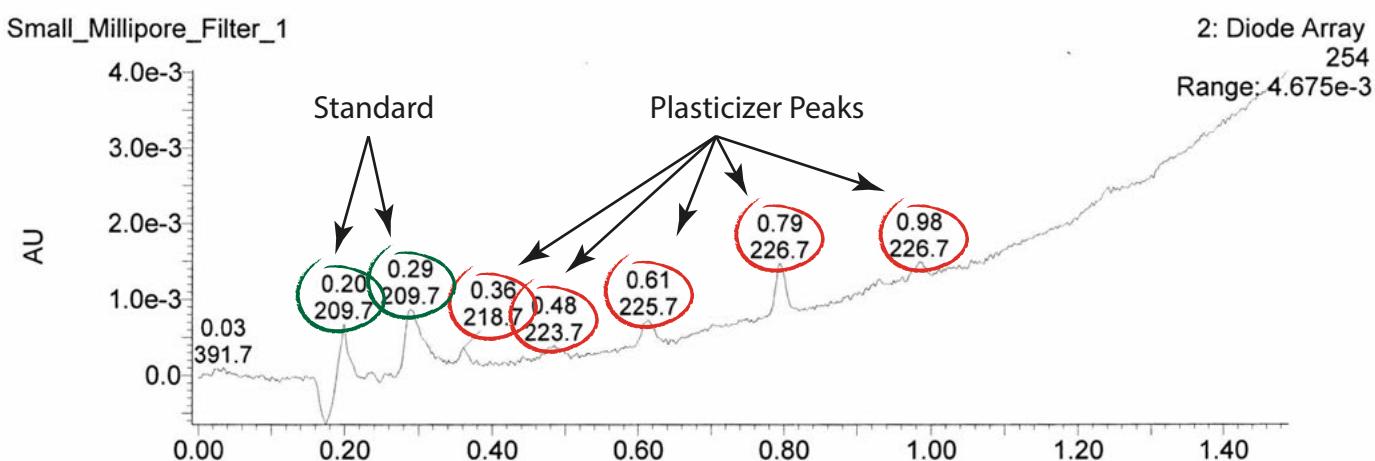
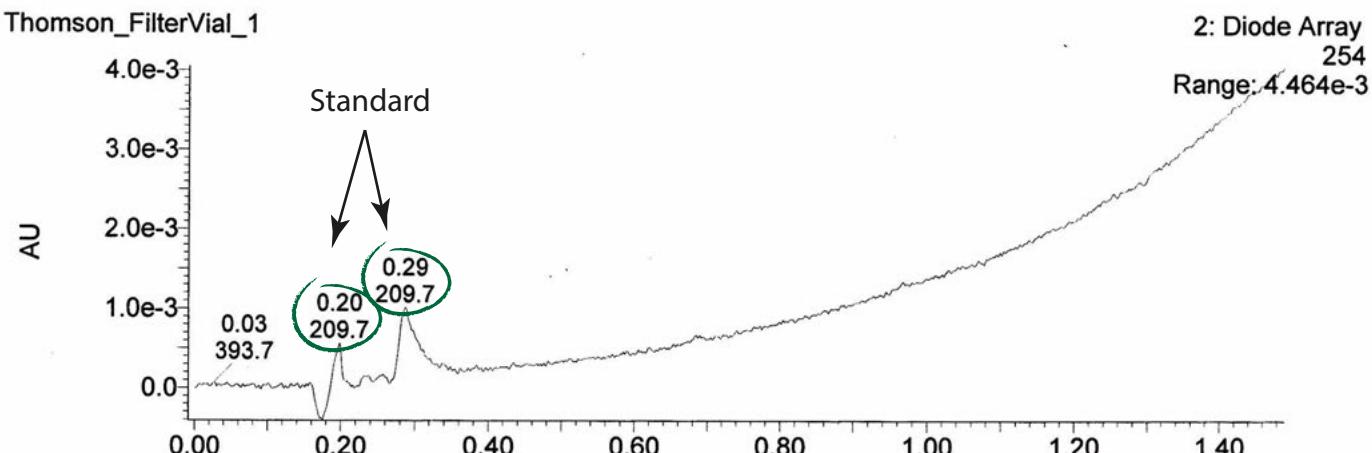
Testing by Takeda® Mass Spec. ES+ Data



Method: A. Water B. ACN 45-90% with .05% TFA Ballistic Gradient over 1.4 minutes using Waters® Acquity® UPLC Thomson Filter Vial (patented) Part # 35540-500 Filter Vial 0.45µM hydrophobic PTFE, w/ Pre-Slit Cap Millipore Syringe Filter Part #:SLFHR04NL Millex-FH® Filter, 0.45 µm, hydrophobic PTFE, 4 mm, non-sterile.

Plasticizer Leachable

Testing by Takeda® UV Data

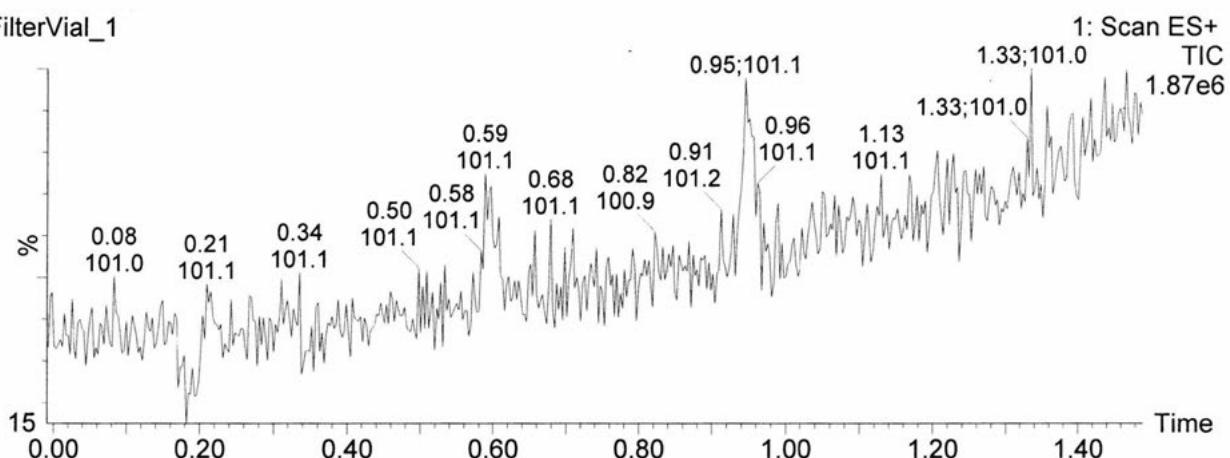


Method: A. Water B. ACN 45-90% with .05% TFA Ballistic Gradient over 1.4 minutes using Waters® Acquity® UPLC Thomson Filter Vial (patented) Part # 35540-500 Filter Vial 0.45µM hydrophobic PTFE, w/ Pre-Slit Cap Millipore Syringe Filter Part #:SLFHR04NL Millex-FH® Filter, 0.45 µm, hydrophobic PTFE, 4 mm, non-sterile.

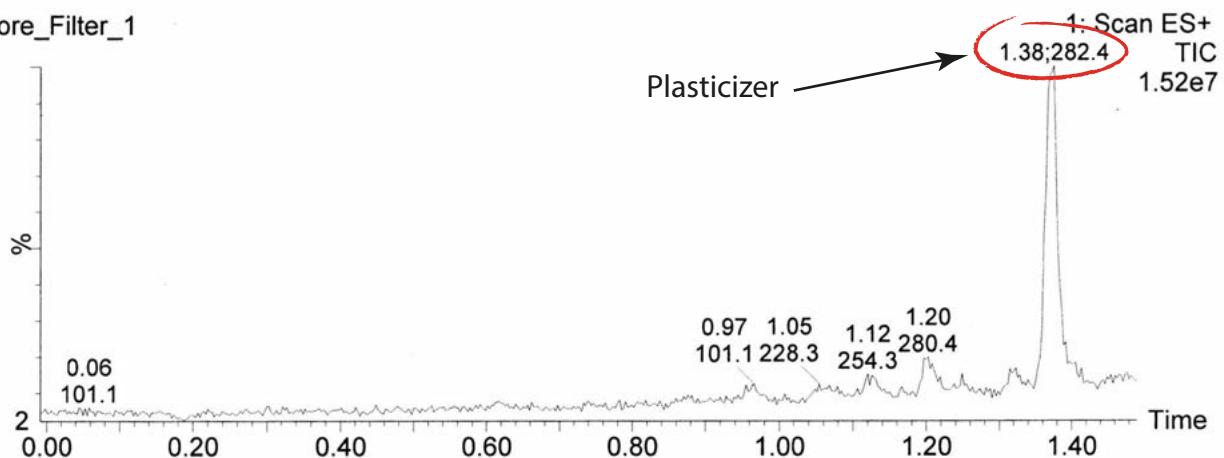
Plasticizer Leachable

Testing by Takeda® Mass Spec ES+ Data Part 2

Thomson_FilterVial_1



Small_Millipore_Filter_1



Method: A. Water B. ACN 45-90% with .05% TFA Ballistic Gradient over 1.4 minutes using Waters® Acquity® UPLC Thomson Filter Vial (patented) Part # 35540-500 Filter Vial 0.45uM hydrophobic PTFE, w/ Pre-Slit Cap Millipore Syringe Filter Part #:SLFHR04NL Millex-FH® Filter, 0.45 µm, hydrophobic PTFE, 4 mm, non-sterile.

Standard Filter VialTM

Patented

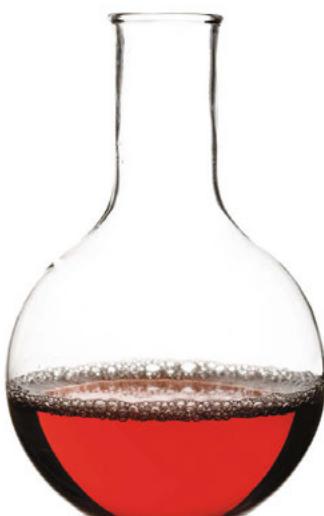


**Standard For
Most Samples**

Standard Filter Vials (120µL Dead Volume)

Thomson Standard Filter (*patented*) can be used for samples containing less than 10% solid particulates. The filter vial consists of two parts: a filter vial shell and a plunger which includes a single layer filter on one end and a vial cap on the other end.

Applications for Thomson Standard Filter Vials include filtration of catalysts from organic and medicinal chemistry synthesis reactions, saccharide analysis in corn syrup, and in-vial protein precipitation.



Filter Vial



(Pre-Slit Cap)

.2µM PTFE

Part No. 35530

.45µM PTFE

Part No. 35540

.2µM PVDF

Part No. 35531

.45µM PVDF

Part No. 35541

.2µM NYLON

Part No. 35538

.45µM NYLON

Part No. 35539

.2µM PES

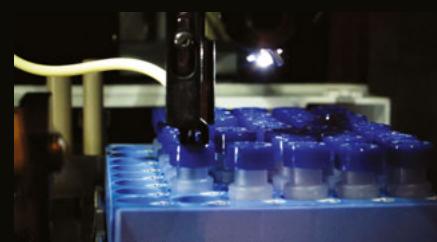
Part No. 35535

.45µM PES

Part No. 35545

SINGLE STEP® Filter Vials Patented

Open Access LCMS



→ "We've been pounding our walk up systems for **over a year** without a single clog."

-Justin
University of Arizona

Thomson Instrument Company is not affiliated with Waters® its product the UPLC® Aquity® or The University of Arizona.

Protein Precipitation with SINGLE StEP Filter Vials™ Patented



Vortex Sample

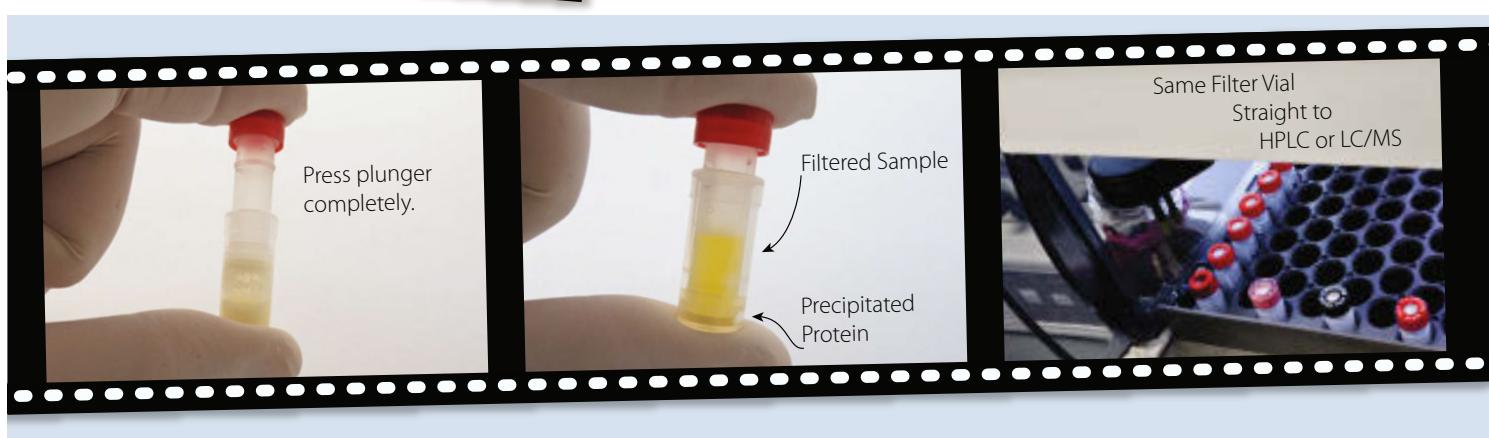
20-40 seconds



Protein Crash

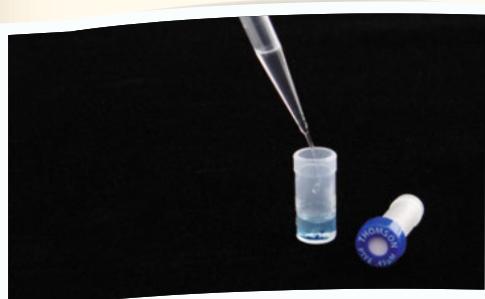
Recommended PVDF .2µM Filter Vial

Use #35531



How to Mass Spec Your TLC Spots Using Thomson Filter Vials

1. After eluting analytical TLC plates, scrape-off desired spots into Thomson Shell Vial



2. Add 0.4 mL EtOAc to Shell Vial



3. Next, insert Plunger, press half way down and shake or swirl to extract compound from silica gel.



4. Press down plunger completely to filter sample

5. Same Filter Vial straight to Mass Spec



CORN SYRUP ANALYSIS SACCHARIDES (LIQUID CHROMATOGRAPHY) with Filter Vials

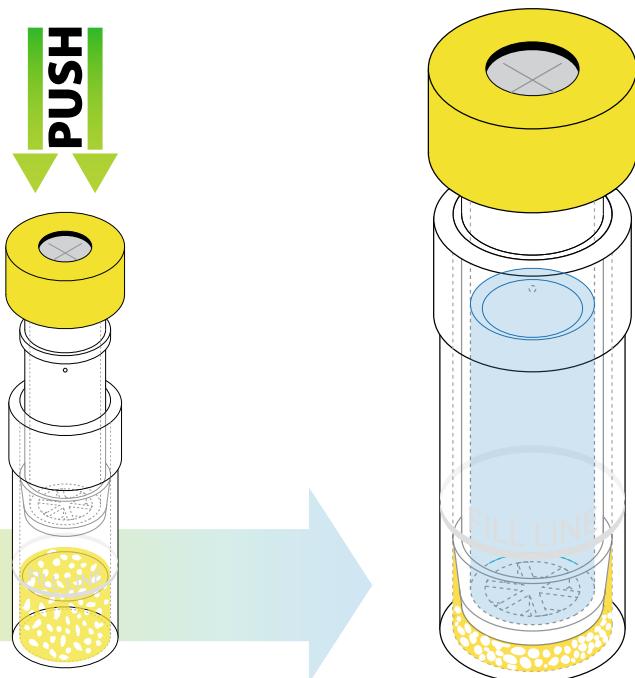
Patented

E-61
CRA Test*



Customers conducting the *CRA E-61* test prefer **Thomson PVDF .45µm Filter Vials** to the standard *syringe filter method*, for **Cost Savings & Speed of use.**

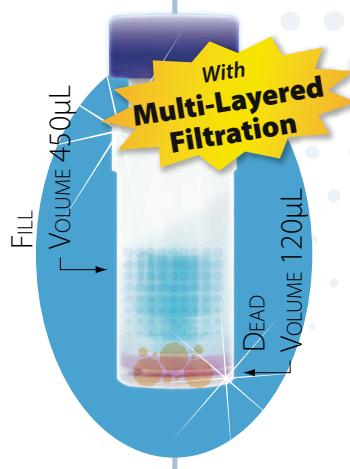
Thomson PVDF Filter Vial 0.45µm
Part Number 35541



*Thomson Instrument Company is not affiliated with Corn Refiners Association, Inc. and this system is not sanctioned by Corn Refiners Association, Inc., although some analysts are using Filter Vials in the Field for this purpose.

eXtreme|FV®

Patented



For Particulate Laden Samples

eXtreme|FV (Multi-Layered Filtration)

Thomson eXtreme|FV® (patented) offer multi-layer filtration for viscous samples and samples containing up to 30% solid particulates. The filter vial consists of two parts: a filter vial shell and a plunger which includes a multi-layer filter on one end and a vial cap on the other end.

eXtreme|FV® allow for compounds to be separated from the matrix which, results in both a higher signal to noise ratio and peaks that are more differentiated.

Prior to the introduction of the eXtreme|FV®, many samples containing high levels of particulates were “filtered” by using an SPE step in the method. These methods are readily amendable to the replacement of the SPE step using a rapid and lower cost eXtreme|FV® step.

Applications for Thomson eXtreme|FV® include filtration of cell and cell debris from cell culture; pesticide analysis in food, tissue, soil, and water; and toxicology analysis in blood and urine.



eXtreme|FV®

(Pre-Slit Cap)

.2µM PTFE

Part No. 85530

.45µM PTFE

Part No. 85540

.2µM PVDF

Part No. 85531

.45µM PVDF

Part No. 85541

.2µM NYLON

Part No. 85538

.45µM NYLON

Part No. 85539

.2µM PES

Part No. 85535

.45µM PES

Part No. 85545

Improved Sample Preparation Methods for Athlete Doping Analysis of Common Compounds in Urine by LCMS



Australian Government
Department of Industry



WORLD
ANTI-DOPING
AGENCY

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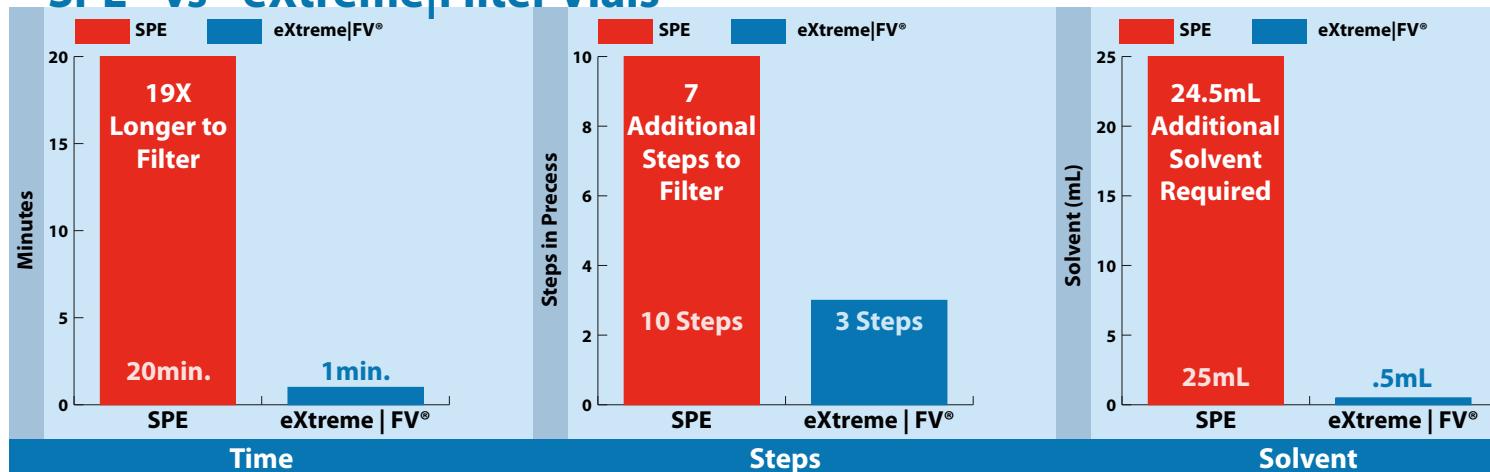
Authors : Dr. Catrin Goebel², Lisa Wanders¹, Sam Ellis¹

Thomson Instrument Company¹

Australian Sports Drug Testing Laboratory in the National Measurement Institute Department of Industry²

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SPE -vs- eXtreme|Filter Vials®



Abstract

Anti-doping testing by urine analysis requires fast and robust screening methods with repeatable sample preparation. Since, every sample has to be screened, methods are designed to be sufficiently sensitive and specific to identify all suspect samples. One must be careful to minimize false suspects. Ensuring samples are spiked with internal standards accordingly will help verify that samples are being extracted and tested correctly and with accurate uniformity.

The Australian Sports Drug Testing Laboratory, our collaborators, have invested time in determining a limited number of comprehensive screening methods. These methods, using Thomson's eXtreme Filter Vials (patented), comply with the World Anti-Doping Agency's (WADA) Prohibited List.

In exploring new methods labs have looked at both detection and sample prep as routes to quicker and more accurate analysis. Liquid chromatography coupled with mass spectrometry detection is prevalent, superseding many of the gas chromatographic coupled with mass spectrometry methods because of the simpler sample preparation. Specifically, the anti-doping testing

shown below consisted of sample preparation without the initial use of cumbersome traditional SPE methods, and instead consisted of the comparison of filtration techniques. Filter plates versus Thomson eXtreme Filter Vials (patented) were tested to determine which product allowed for a method of simple and quick urine analysis while complying with the WADA's guidelines.

Experiment

The experiments were performed at the National Measurement Institute (Australia) in the Sports Drug Testing Laboratory.

The 11.8 minute run time for the instrumental analysis meets the requirements of the WADA Technical Document- Minimum Required Performance Level (TD2013MRPL). This document details the analysis of a large number of analytes from the classes on the WADA Prohibited List, while meeting sensitivity requirements. The analytes included compounds in the following classes anabolic agents, B2-agonists, hormone antagonists and modulators, diuretics, stimulants, narcotics, glucocorticoids, B-blockers, etc.

Full Method:

A comparison between sample preparation using filter plates sourced from several different manufacturers, and Thomson eXtreme Filter Vials (patented) PVDF 0.2µm (85531-500) was conducted. The preparation with the Thomson eXtreme Filter Vials were automated using a Tecan robotics platform for liquid dispensing in the Thomson 48 position rack (#35010-RACK), and 48 position press (#35010).

Direct Urine Preparation:

1. Label each eXtreme Filter Vial with sample/quality control sample information.
2. Pipette 200 µL of each sample into labeled eXtreme Filter Vial.
3. Add 200 µL of the Mefruside Internal Standard (300 ng/mL in 0.5% formic acid) to each filter vial cup.
4. Place the eXtreme Filter Vial tops onto each vial and press shut.

LCHRMS System:

UPLC coupled to High Resolution Mass Spectrometry with an electrospray source in full scan mode. Data acquisition in both positive and negative polarity modes within a single 11.8 min chromatographic run.

Column: C18, 2.1mm x 50mm, 1.7µm

Column Temperature: 30 °C

Flow rate: 300µL/min

Mobile Phase:

A: 0.3% aqueous Formic Acid in Water

B: 0.3% Formic Acid in Acetonitrile

Gradient:

Time	A%	B%
0.00	95	5
0.50	95	5
3.50	80	20
5.50	75	25
7.00	43	57
8.00	10	90
8.60	10	90
8.80	95	5

Injection volume: 10µL

Sample tray temperature: 18°C

Column Temperature: 30°C

Method run time: 11.8 minutes

Gas: UHP Nitrogen

Conclusions

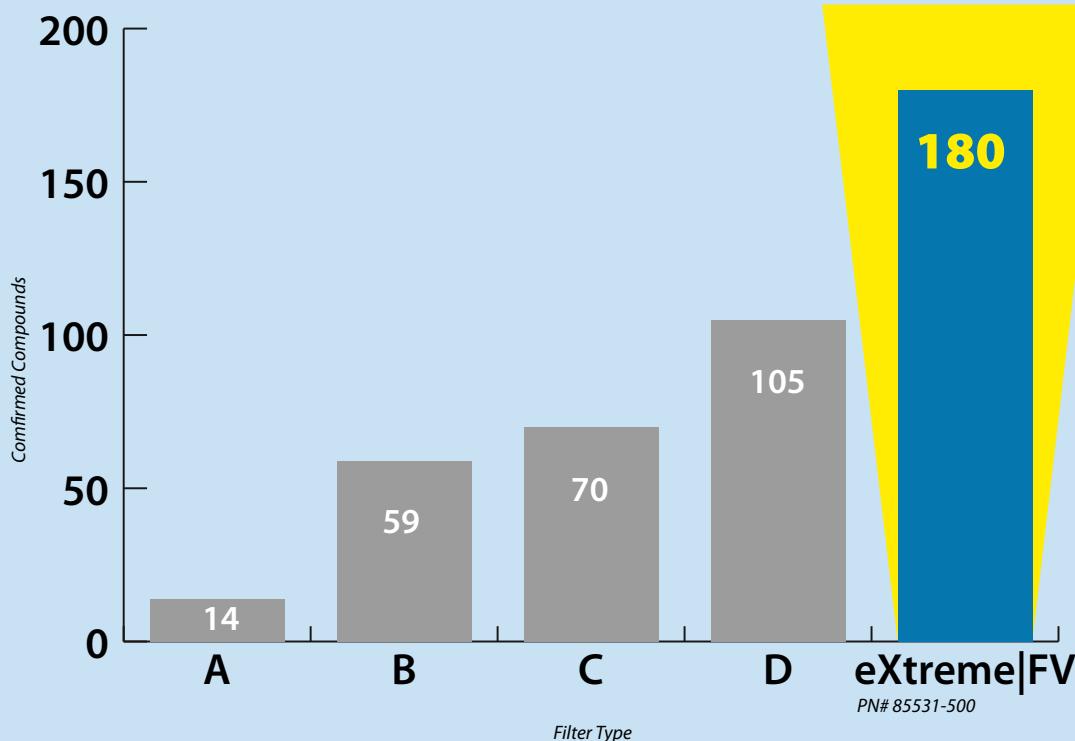
The Thomson eXtreme Filter Vials (patented) PVDF 0.2µm (85531-500) performed the best in compound extraction and identification while allowing the end user to follow the WADA validated method. The elimination of SPE steps from laboratory methods is a large time saver, and enables urine-direct-injection solely using Thomson eXtreme Filter Vials for filtration. Together the Thomson 48 position Filter Vial Press and automation enabled 48 position rack equaled timing of filter plate methodology but provided the best extraction and identification of all filter types. A total of 180 compounds can be identified through the screening analysis with the Thomson eXtreme Filter Vials (patented) PVDF 0.2µm (85531-500).

The method presented is being used for the analysis of athlete's urine samples for banned substances at the Australian Sports Drug Testing Laboratory.

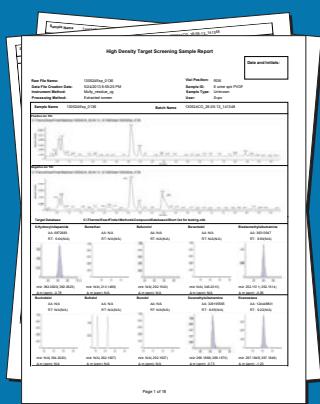
Acknowledgments

We would like to thank Dr. Catrin Goebel, Director, of Australian Sports Drug Testing Laboratory in the National Measurement Institute, Department of Industry (a WADA accredited laboratory in Australia) for her extensive testing. Dr. Goebel is also an Executive member of World Association of Anti-Doping Scientist.

Comparison of Filter Types

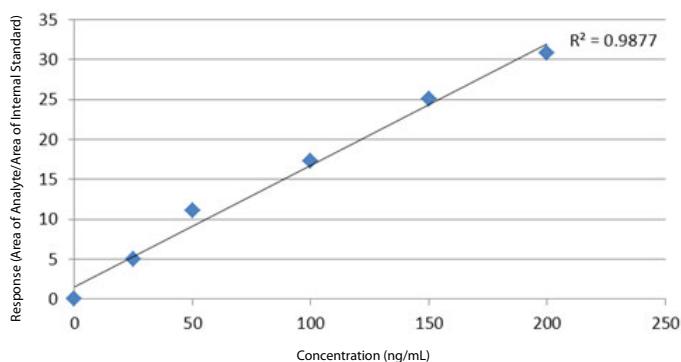


To View All Chromatograms
Visit <http://bit.ly/wada-data>

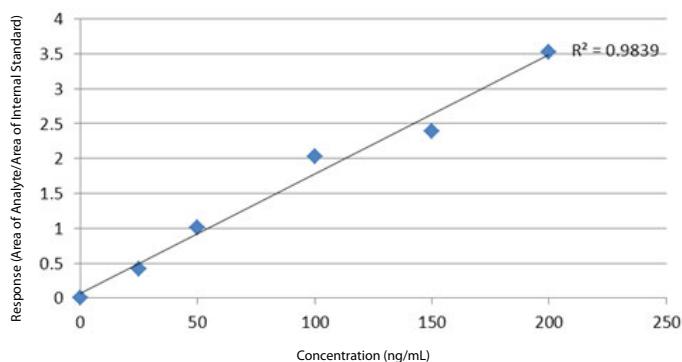


Linearity of The Analysis Method Was Assessed Over a Range From 25% To 200% Of MRPL With R2 Generally Being Greater Than 0.98

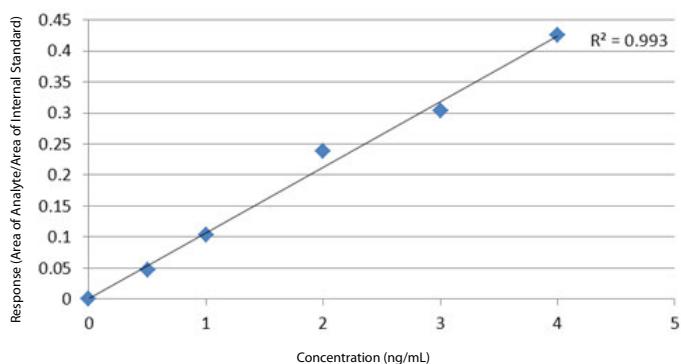
Acebutolol (P2)



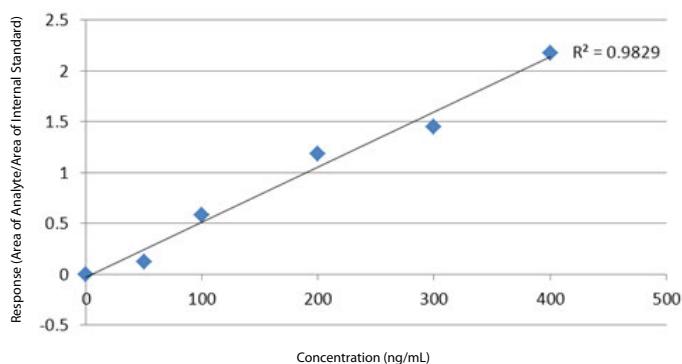
Pemoline (S6)



Norfentanyl (S7)



Quinethazone (S5)



Time is Equal



With automation our customers are utilizing Filter Vials at the same speed filter plates were used in the past.

Confirmed Compounds (180)

Sample Name 130524Exp_0136

Raw File 130524Exp_0136

Batch Name 130524CG_26-05-13_141348

Compound Name	Found		Confirmed
State			
5-Hydroxyindapamide	Confirmed	S5.16 Furosemide	Confirmed
Bisdesmethylsibutramine	Confirmed	S5.17 Hydrochlorothiazide	Confirmed
Desmethylsibutramine	Confirmed	S5.20 Mefruside metabolite 2	Confirmed
Exemestane	Confirmed	S5.21 Indapamide	Confirmed
ISD.01 Mefruside (+)	Confirmed	S5.22 Metolazone	Confirmed
ISD.02 Mefruside (-)	Confirmed	S5.23 Polythiazide	Confirmed
ISD.03 D3-epitestosterone glucuronide	Confirmed	S5.24 Torasemide	Confirmed
ISD.04 D3-epitestosteronea	Confirmed	S5.25 Triamterene	Confirmed
M1.03 AICAR	Confirmed	S5.26 Xipamide	Confirmed
M1.04 GW1516	Confirmed	S6.00 Caffeine	Confirmed
P2.03 Atenolol	Confirmed	S6.00 Cis-4-Methylaminorex	Confirmed
P2.05 Bisoprolol	Confirmed	S6.00 Cotinine (Nicotine metab)	Confirmed
P2.12 Esmolol	Confirmed	S6.00 MBDB	Confirmed
P2.14 Metipranolol	Confirmed	S6.00 Methoxyamphetamine	Confirmed
P2.16 Nadolol	Confirmed	S6.00 Methylenedioxymethamphetamine	Confirmed
P2.17 Nadoxolol	Confirmed	S6.01 Adrafinil	Confirmed
P2.18 Oxprenolol	Confirmed	S6.03 Amiphenazole	Confirmed
S1.00 Clenbuterol	Confirmed	S6.04 Amphetamine	Confirmed
S1.00 Gestrinone	Confirmed	S6.07 Benzoylecgonine	Confirmed
S1.00 Methylidienolone	Confirmed	S6.09 Benzylpiperazine	Confirmed
S1.00 Methyltriienolone	Confirmed	S6.10 Carphedon	Confirmed
S1.00 Metribolone	Confirmed	S6.11 Cathine	Confirmed
S1.00 Tetrahydrogestrinone	Confirmed	S6.14 Crotethamide	Confirmed
S1.00 Tibolone	Confirmed	S6.15 Cyclazodone	Confirmed
S1.00 Zilpaterol	Confirmed	S6.17 Ephedrine	Confirmed
S1.01 3'-Hydroxystanolozolol	Confirmed	S6.17 Phenylpropanolamine	Confirmed
S1.02 4'-Hydroxystanolozolol	Confirmed	S6.17 Pseudoephedrine	Confirmed
S3.01 Bambuterol	Confirmed	S6.18 Etamivan	Confirmed
S3.03 Formoterol	Confirmed	S6.20 Etilefrine	Confirmed
S3.04 Salbutamol	Confirmed	S6.25 Fenetylline	Confirmed
S3.05 Salmeterol	Confirmed	S6.30 Hydroxy mesocarb	Confirmed
S3.06 Terbutaline	Confirmed	S6.32 Isometheptene	Confirmed
S4.00 Andarine	Confirmed	S6.33 Methylenedioxymethamphetamine (MDA)	Confirmed
S4.1.00 Exemestane metabolite	Confirmed	S6.34 Methylenedioxymethamphetamine(MDMA)	Confirmed
S4.1.01 Aminoglutethimide	Confirmed	S6.43 Methylphenidate	Confirmed
S4.2.00 Raloxifene	Confirmed	S6.44 Modafinil	Confirmed
S4.3.00 Fulvestrant	Confirmed	S6.45 Modafinil Acid (metabolite)	Confirmed
S4.5.00 GW1516 (501516)	Confirmed	S6.46 Nikethamide	Confirmed
S5.00 Methazolamide	Confirmed	S6.49 Oxilofrine	Confirmed
S5.00 Piretanide	Confirmed	S6.50 Pemoline	Confirmed
S5.00 Quinethazone	Confirmed	S6.51 Pentetrazol	Confirmed
S5.00 Spironolactone	Confirmed	S6.53 Phenmetrazine	Confirmed
S5.00 Trichlormethiazide	Confirmed	S6.56 Pholedrine	Confirmed
S5.01 Acetazolamide	Confirmed	S6.57 p-Hydroxy amphetamine	Confirmed
S5.02 Althiazide	Confirmed	S6.62 Ritalinic Acid	Confirmed
S5.02 Amiloride	Confirmed	S6.64 nor-Selegiline	Confirmed
S5.03 Bendroflumethiazide	Confirmed	S7.00 Methylecgonine	Confirmed
S5.03 Benzthiazide	Confirmed	S7.03 Codeine	Confirmed
S5.04 Bumetanide	Confirmed	S7.06 Hydromorphone	Confirmed
S5.05 Canrenone	Confirmed	S7.08 Morphine	Confirmed
S5.06 Chlorexolone	Confirmed	S8.04 JWH018 N-(5-hydroxypentyl) metabolite	Confirmed
S5.07 Chlorothiazide	Confirmed	S8.05 JWH073 N-butanoic acid metabolite	Confirmed
S5.08 Chlorthalidone	Confirmed	S9.03 Budesonide	Confirmed
S5.09 Clopamide	Confirmed	S9.05 Cortisol	Confirmed
S5.1.01 Probencid	Confirmed	S9.06 Cortisone	Confirmed
S5.10 Cyclopenthiazide	Confirmed	S9.12 Flumethasone	Confirmed
S5.11 Cyclothiazide	Confirmed	S9.16 Fluticasone propionate metabolite	Confirmed
S5.12 Dichlorphenamide	Confirmed	S9.17 Methylprednisolone	Confirmed
S5.13 Epitizide	Confirmed	S9.18 16a-OH-Prednisolone	Confirmed
S5.14 Eplenerone	Confirmed	S9.18 Prednisolone	Confirmed
S5.15 Etacrylic acid (frag?)	Confirmed	Sildenafil	Confirmed
		Tadalafil	Confirmed
		Vardenafil	Confirmed

PESTICIDE APPLICATIONS

SOIL | VEGETATION

Vegetation & Soil Application

1. Samples are extracted using 20g of homogeneous, ground sample
2. Sample clean-up was achieved using Thomson eXtreme Filter Vials (PTFE .2µm & PVDF .2µm)

The following compounds were seen in both soil and vegetation:

MCPP	Quinclorac
Clopyralid	Fluroxypyr
Aminopyralid	MCPA
Picloram	Diflufenzopyr
Dicamba	

System: UPLC ®/MS/MS®

HPLC Column: Zorbax Rx C8, 150 x 2.1 mm id

HPLC Guard Column: Agilent Eclipse XDB-C8, 2.1 x 12.5mm, 5 micron

Column Temperature: 35°C

Autosampler Temperature: 15°C

Injection Volume: 10µl

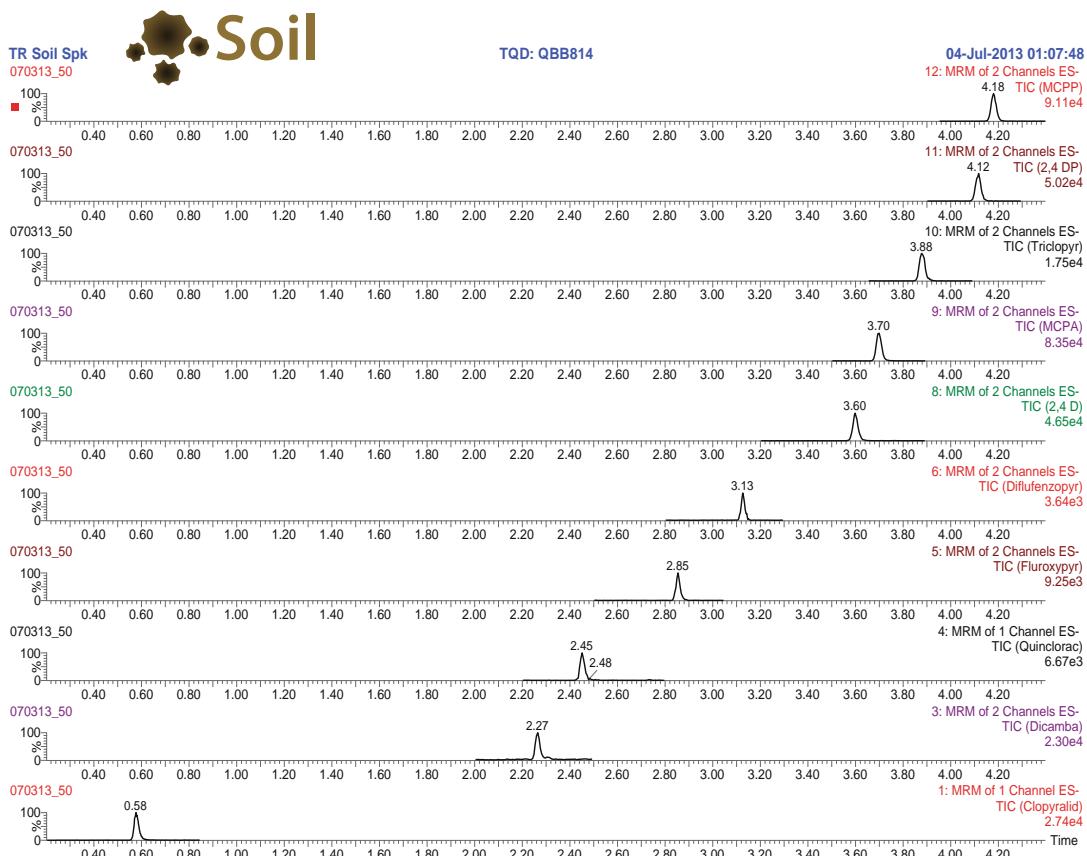
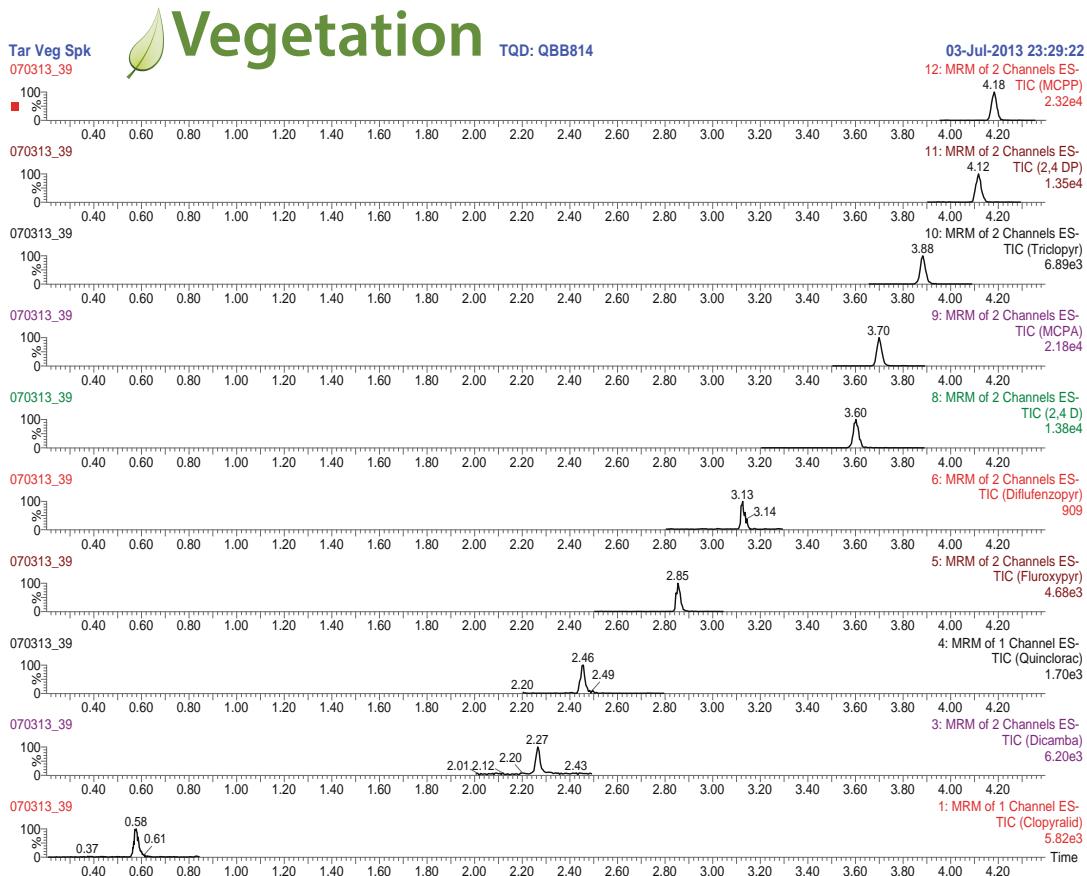
Run Time: 8 min

Solvent A : 0.15% Glacial Acid in Water

Solvent B: 0.15% Glacial Acid in ACN

Gradient:

Time (min)	Flow Rate (ml/min)	%A	%B
Initial	0.8	95	5
1	0.8	95	5
2	0.8	80	20
3	0.8	70	30
4	0.8	60	40
5	0.8	50	50
5.5	0.8	5	95
6.5	0.8	5	95
7	0.8	95	5



eXtreme Filter Vials® vs SPE for the analysis of Pesticides in Orange Juice



MICRO
QUALITY LABS INC.

Thomson Instrument Company is not affiliated with Micro Quality Labs Inc..

Micro Quality Labs Inc. is not affiliated with Thomson Instrument Company or endorse Thomson's products.

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

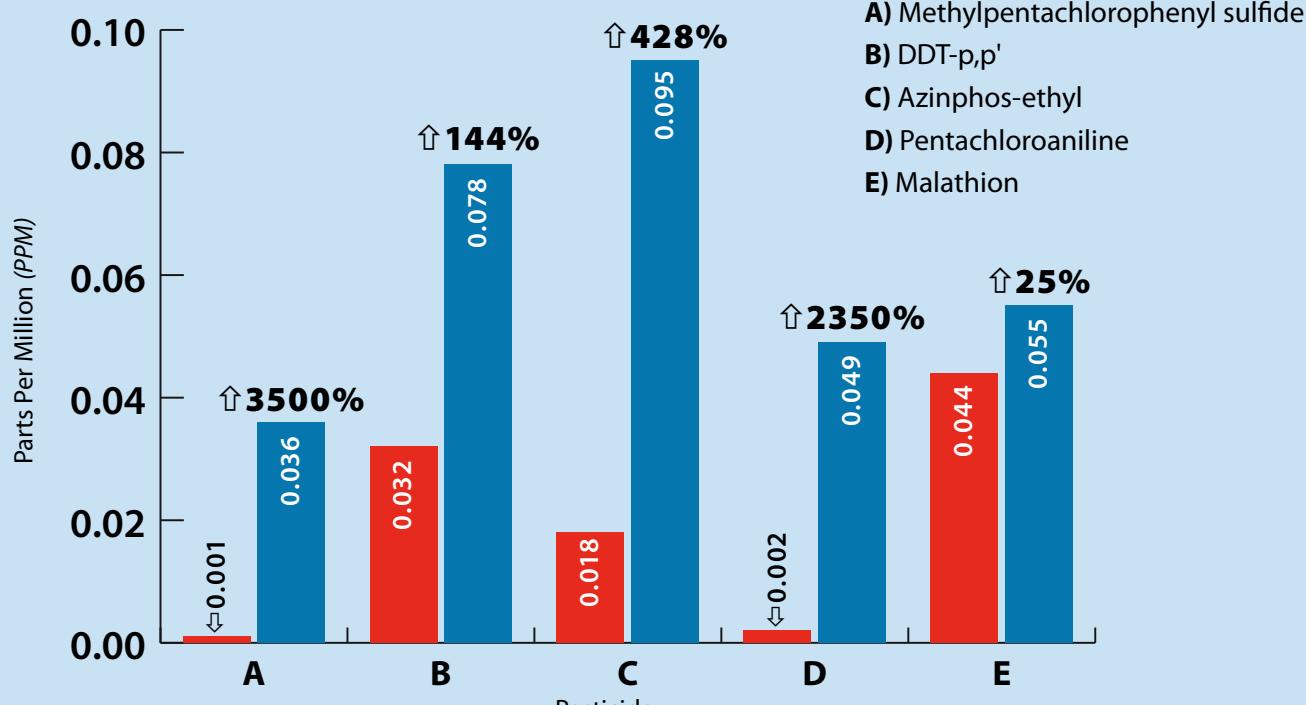
Micro Quality Labs¹

Thomson Instrument Company²

For reprints contact folks@htslabs.com



Comparison of Pesticide Recoveries



SPE

eXtreme|FV®

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 Filter Vials, 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 Filter Vial 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 Filter Vial".

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.

Thomson eXtreme Filter Vial Cleanup Prior to Analysis

- 1.) Add 400µL of the re-suspended residue from the flask labeled "for Thomson eXtreme Filter Vial" to the shell of one Thomson eXtreme Filter Vial 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	SPE+ ROUTINE Syringe FILTER	ONLY EXTREME FV 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
Chlорfenvinphos	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
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
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

GCMS Data (links to PDF)

With Out 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 (*Thomson #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 Filter Vial. 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 Filter Vials offer a viable alternative with higher recovery and less preparation time compared to SPE for the preparation of juices prior to pesticide analysis.

Restek or its products are not affiliated with Thomson Instrument Company

SUPPLEMENT ANALYSIS OF HUPERZINE A BY HPLC

.45µm eXtreme|FV Nylon

Huperzine A Summary

1. Samples are extracted with 10mM HCl (aqueous)
2. Non-soluble plant parts or excipients are filtered out using a 0.45µm Nylon filter
3. Samples are injected onto the HPLC System

Figure I: Chromatogram of Huperzine A extracted from the Chinese Club Moss, *Huperzia serrata*

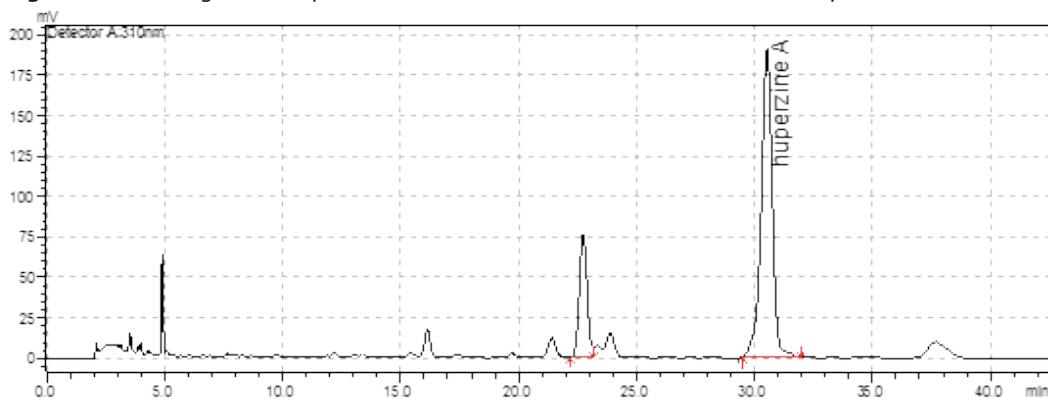
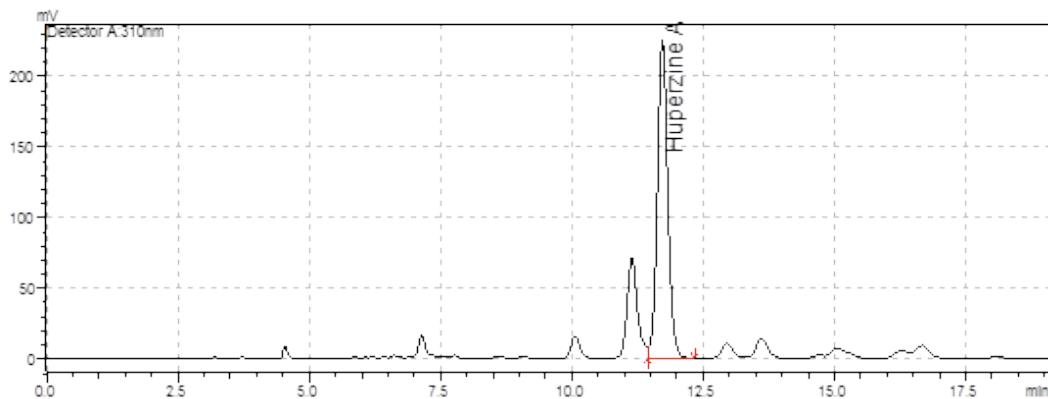


Figure II: Chromatogram of Huperzine A extracted from a Club Moss Powdered Extract



Antibody Analysis with eXtreme|FV®



HPLC Column and Method

Column

Poros® Protein A by Applied Biosystem® 2-1001-00 Column

Method

A Solvent: PBS pH 7.4

B Solvent: 150 millimolar Sodium Chloride pH 2.2

Isocratic 6 minute run on an Agilent® 1200

Filter Vials Allow

- Real Time Monitoring
- Quantify Antibodies
- Ideal For Timepoints
- Accurate On The Fly Adjustments
- Fits In Standard Autosampler



Thomson Instrument Company is not affiliated with Agilent Technologies®, Corning Life Sciences®, Applied Biosystem® a part of Life Technologies® or any of their products.

ANALYSIS OF NITROSAMINES IN TOBACCO

Prep:

1. 0.25g of unburned/smokeless tobacco sample
2. Extracted with 100mM ammonium acetate solution, filtered with eXtreme|FV® PVDF 0.45 µm

HPLC:

Injection Volume:

5µL

Column:

Waters Xterra MS C18, 50x4.6mm, 5µm

Aqueous phase:

5mM ammonium acetate in HPLC water

Organic Phase:

5mM ammonium acetate in 95/5 acetonitrile/water blend.

Gradient:

Time [min] Organic %

0	5
1	5
2	35
5	35
6	5
8	5

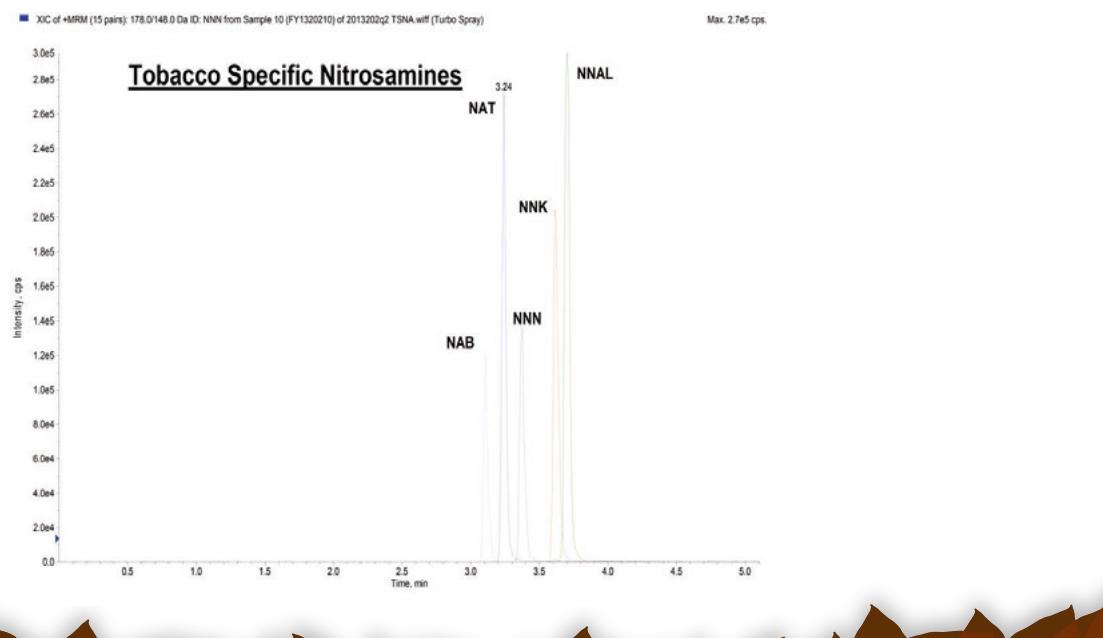
Flow rate: 1mL/min

Temperature: 60°C

Detection: MS/MS

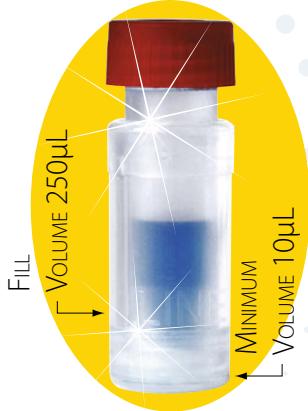
Analyte Ion pair Q1/Q3 (amu)

NAB	192/162	N-Nitrosoanabasine
NAT	190/160	N-Nitrosoanatabine
NNK	208/122	N-Nitrosonornicotine
NNN	178/148	4-(Methylnitrosamino)-1-(3-pyridyl)-1-butanone
NNAL	210/180	4-(Methylnitrosamino)-1-(3-pyridyl)-1-butanol



nano|Filter Vial™

Patented



**When Every
µL Counts**

nano|Filter Vials (10µL Dead Volume)

Thomson nano|Filter Vials™ offer a very low dead volume allowing one to filter as little as 10µL of sample with enough remaining filtrate to make a 2µL injection. The filter vial consists of two parts: a filter vial shell with mating bottom surface and a plunger which includes a filter on one end and a screw cap vial on the other end.

Applications include the analysis of enzymes, peptides, DNA, RNA, synthesis reaction intermediates, finished products, saliva, samples available in low volumes, in-vial evaporation and re-suspension for sample concentration and buffer/solvent change.



nano|Filter Vial™



.2µM PTFE

Part No. 15530

Part No. 25530 (Pre-Slit Cap)

.45µM PTFE

Part No. 15540

Part No. 25540 (Pre-Slit Cap)

.2µM PVDF

Part No. 15531

Part No. 25531 (Pre-Slit Cap)

.45µM PVDF

Part No. 15541

Part No. 25541 (Pre-Slit Cap)

.2µM NYLON

Part No. 15538

Part No. 25538 (Pre-Slit Cap)

.2µM PES

Part No. 15535

Part No. 25535 (Pre-Slit Cap)

Date: 05-05-05
Instrument: ZQ12

10 μ L FILTRATION

WITH

2 μ L

INJECTION

**Open Access LCMS - ZQ12 - FRENICA1**

File:FRENICA1_ZQ12-1087-1

Notes:DEPTH 2 -10uL

Time:16:18:04

Method:C:\MassLynx\09_LC-C8-BroadRange-ApH.olp

Page 1

Submitter:FRENICA1

TA-Project:AS

Date:03-Oct-2011

Instrument:ZQ12

ID:MICRO VIAL

Vial:1:44

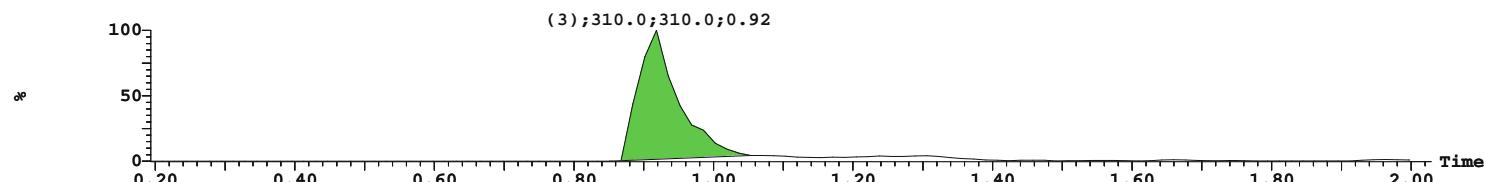
Sample: 1

Printed: Mon Oct 03 16:21:39 2011

Sample Report:

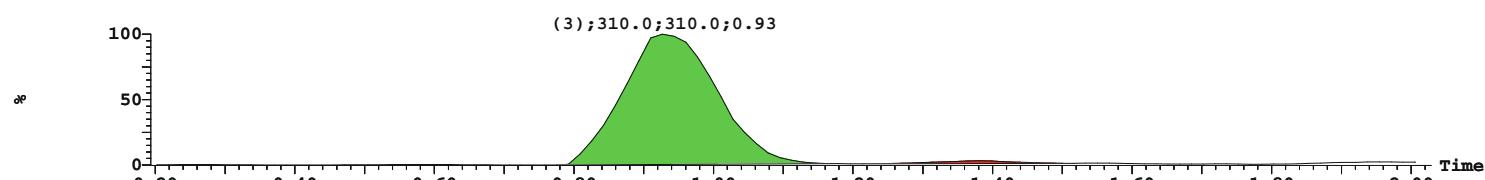
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1.2e+007



2: MS ES- :308.992+369.013

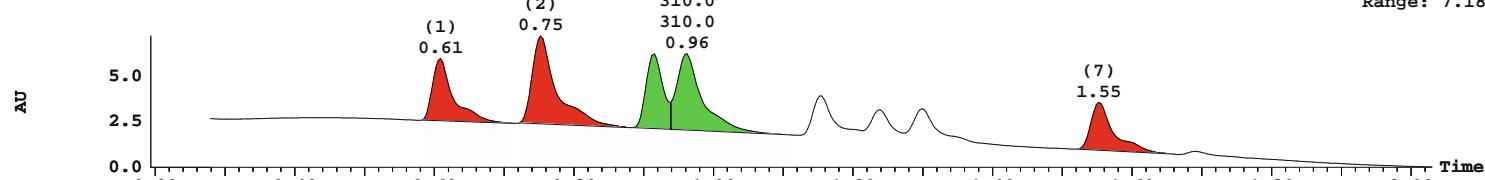
9.8e+004



3: UV Detector: TIC

7.182

Range: 7.182



Peak Number	Time	Area %
1	0.61	16.61
2	0.75	27.61
3	0.92	16.36
4	0.96	25.32
7	1.55	14.11

**Open Access LCMS - ZQ12 - FRENICA1**

File:FRENICA1_ZQ12-1087-1

Notes:DEPTH 2 -10uL

Time:16:18:04

Method:C:\MassLynx\09_LC-C8-BroadRange-ApH.olp

Submitter:FRENICA1

TA-Project:AS

Date:03-Oct-2011

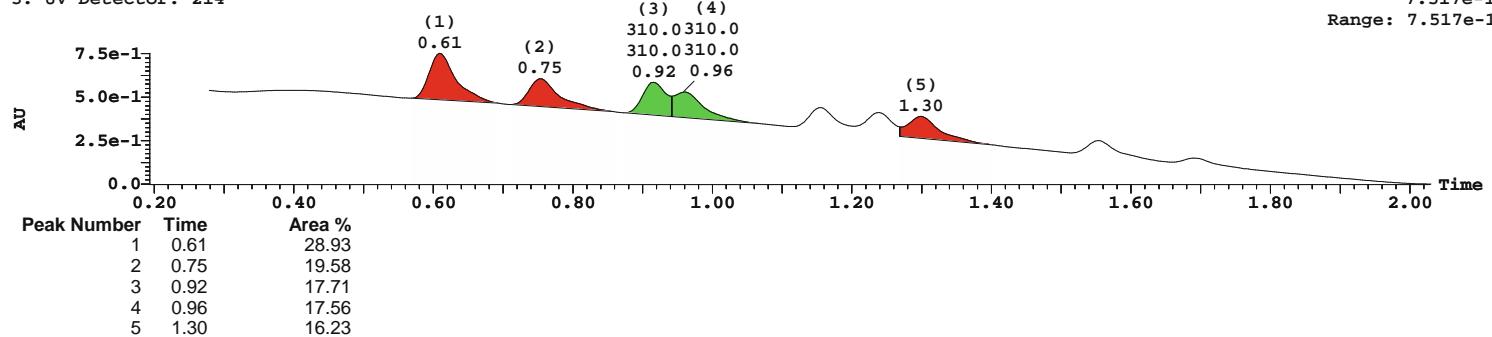
Instrument:ZQ12

Page 2

Printed: Mon Oct 03 16:21:39 2011

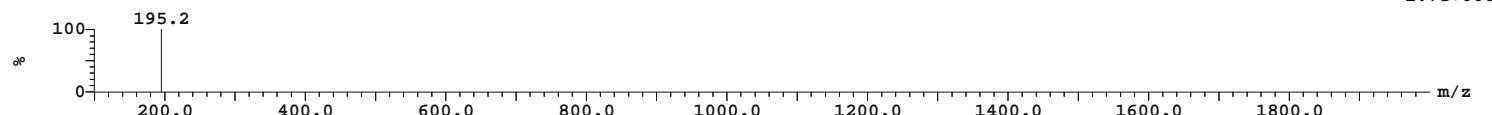
Sample Report (continued):

3: UV Detector: 214



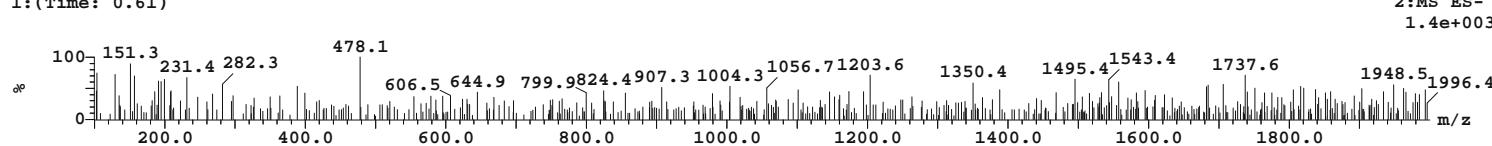
Peak ID	Time	Mass Found	Base Peak	State
1	0.61	195.2	195.2	Diversity fail

1:(Time: 0.61)



Peak ID	Time	Mass Found	Base Peak	State
1	0.61	478.1	478.1	Diversity fail

1:(Time: 0.61)



Appendix

Chemical Compatibility

	Housing Materials		Filter Materials		
	PP	PTFE	PVDF	PES	nylon
Acetic Acid (glacial) <i>acid, organic</i>	TST	R	R	R	NR
Acetone <i>ketone</i>	R	R	NR	GNR	R
Acetonitrile (ACN) <i>nitrile</i>	R	R	LTD	NR	R
Alconox, 1% <i>surfactant/detergent</i>	ND	TST	TST	ND	TST
Ammonium Hydroxide <i>caustic</i>	TST	GR	R	NR	TST
Ammonium Sulfate (saturated) <i>salt, aqueous solution</i>	R	GR	NR	ND	R
Amyl Acetate <i>ester</i>	TST	R	R	GR	TST
Amyl Alcohol <i>alcohol</i>	R	R	R	GR	TST
Benzene <i>HC, aromatic</i>	NR	R	R	NR	R
Benzyl Alcohol <i>HC aromatic/alcohol</i>	NR	R	R	ND	TST
Boric Acid (aqueous solution) <i>acid, inorganic</i>	R	GR	TST	GR	R
Butyl Acetate <i>ester</i>	TST	GR	TST	GNR	R
Butyl Alcohol <i>alcohol</i>	R	GR	R	GR	R
Carbon Tetrachloride <i>HC, halogenated</i>	NR	GR	R	GNR	TST
Cellosolve (Ethyl) <i>glycol ether</i>	R	GR	ND	GR	R
CHAPS (aqueous solution) <i>surfactant/detergent</i>	ND	TST	ND	ND	TST
Chloroform <i>HC, halogenated</i>	NR	R	R	GNR	NR
Cyclohexanone <i>ketone</i>	NR	R	NR	GNR	R
Diethyl Pyrocarbonate, 0.2% <i>carboxylic anhydride</i>	ND	ND	TST	ND	ND
Dimethyl Sulfoxide (DMSO) <i>sulfoxide</i>	R	R	NR	NR	R
Dimethylacetamide <i>amide</i>	R	GR	NR	NR	NR
Dimethylformamide <i>amide</i>	R	GR	NR	ND	R
Dioxane <i>ether</i>	R	GR	R	ND	R

Key to the Above Table

ND = No Data Presently Available

R = Recommended

GR = Generally Recommended

NR = Not Recommended

GNR = Generally Not Recommended

TST = Testing Recommended

LTD = Limited Recommendation

Chemical Compatibility

	Housing Materials		Filter Materials		
	PP	PTFE	PVDF	PES	nylon
Ethers <i>ether</i>	NR	R	R	ND	R
Ethyl Acetate <i>ester</i>	TST	R	R	GNR	R
Ethyl Alcohol <i>alcohol</i>	R	R	R	GR	TST
Ethylene Glycol <i>glycol</i>	R	R	R	GR	R
Formaldehyde <i>aldehyde</i>	R	R	R	ND	R
Formic Acid, 50% <i>acid, organic</i>	R	GR	R	ND	NR
Freon (TF or PCA) <i>HC, halogenated</i>	R	GR	R	ND	R
Gasoline <i>HC</i>	NR	R	R	GR	R
Glycerine (Glycerol) <i>glycol</i>	R	GR	R	GR	R
Guanidine Hydrochloride, 6M <i>salt, aqueous solution</i>	ND	GR	ND	ND	ND
Guanidine Thiocyanate, 5M <i>salt, aqueous solution</i>	ND	GR	ND	ND	ND
Helium <i>gas</i>	R	R	TST	ND	R
Hexane <i>HC, aliphatic</i>	NR	R	R	GR	R
Hydrochloric Acid, 1N (HCl) <i>acid, inorganic</i>	GR	R	R	GR	GR
Hydrochloric Acid, 6N (HCl) <i>acid, inorganic</i>	TST	R	TST	GR	TST
Hydrochloric Acid, conc. (HCl) <i>acid, inorganic</i>	NR	R	NR	ND	NR
Hydrofluoric Acid <i>acid, inorganic</i>	NR	R	NR	NR	NR
Hydrogen <i>gas</i>	R	R	R	ND	R
Hydrogen Peroxide, 3% <i>peroxide</i>	R	R	R	ND	R
Hydrogen Peroxide, 30% <i>peroxide</i>	TST	R	R	ND	TST
Hydrogen Peroxide, 90% <i>peroxide</i>	R	R	R	ND	NR
HYPO (aqueous solution) <i>salt, aqueous solution</i>	R	GR	R	ND	R
Isobutyl Alcohol <i>alcohol</i>	R	R	R	GR	TST

Key to the Above Table

ND = No Data Presently Available

R = Recommended

GR = Generally Recommended

NR = Not Recommended

GNR = Generally Not Recommended

TST = Testing Recommended

LTD = Limited Recommendation

Chemical Compatibility

	Housing Materials		Filter Materials		
	PP	PTFE	PVDF	PES	nylon
Isopropyl Acetate ester	TST	R	R	GNR	R
Isopropyl Alcohol alcohol	R	R	R	GR	TST
Kerosene HC	TST	LTD	R	GR	R
Lactic Acid, 50% acid, organic/alcohol	R	GR	TST	ND	TST
Lubrol PX (aqueous solution) surfactant/detergent	ND	TST	ND	ND	ND
Methyl Ethyl Ketone (MEK) ketone	R	R	NR	GNR	R
Mercaptoethanol, 0.1M alcohol/mercaptan	ND	ND	ND	ND	ND
Methyl Acetate ester	TST	R	NR	GNR	R
Methyl Alcohol alcohol	R	R	R	GR	TST
Methylene Chloride HC, halogenated	NR	R	NR	GNR	TST
Methyl Isobutyl Ketone (MIBK) ketone	NR	R	NR	GNR	R
Mineral Spirits HC	NR	R	R	GR	R
Nitric Acid, 6N acid, inorganic	TST	R	R	R	NR
Nitric Acid (concentrated) acid, inorganic	NR	ND	NR	ND	NR
Nitrobenzene HC, aromatic	NR	R	R	ND	R
Nitrogen gas	ND	R	R	ND	R
Nonidet-P40 (aqueous solution) surfactant/detergent	ND	ND	ND	ND	ND
Ozone gas	NR	GR	R	ND	NR
Paraldehyde aldehyde	TST	GR	TST	ND	R
Pentane HC, aliphatic	NR	GR	GR	GR	R
Petroleum Ether ether	ND	GR	R	ND	R
Phenol (aqueous solution) phenol	NR	GR	R	ND	NR
Potassium Hydroxide, 3N caustic	R	R	R	ND	R

Key to the Above Table

ND = No Data Presently Available

R = Recommended

GR = Generally Recommended

NR = Not Recommended

GNR = Generally Not Recommended

TST = Testing Recommended

LTD = Limited Recommendation

Chemical Compatibility

	Housing Materials		Filter Materials		
	PP	PTFE	PVDF	PES	nylon
Pyridine amine	R	GR	NR	NR	TST
Silicone Oils silicone	R	GR	R	ND	R
Sodium Carbonate (aqueous solution) salt, aqueous solution	R	R	R	ND	TST
Water (Brine) salt, aqueous solution	R	R	R	ND	R
Sodium Chloride (aqueous solution) salt, aqueous solution	R	R	R	ND	R
Sodium Dodecyl Sulfate surfactant/detergent	ND	ND	ND	ND	ND
Sodium Hydroxide, 3N caustic	R	R	R	R	R
Sodium Hydroxide (concentrated) caustic	R	R	R	R	NR
Sulfuric Acid (concentrated) acid, inorganic	NR	R	TST	GNR	NR
Tetrahydrofuran (THF) ether	NR	GR	NR	ND	R
Toluene HC, aromatic	NR	R	R	R	R
TCA (aqueous solution) acid, organic	R	GR	R	ND	TST
Trichloroethane HC, halogenated	NR	R	TST	GNR	TST
Trichloroethylene HC, halogenated	NR	R	R	GNR	TST
Tween 20 (aqueous solution) surfactant/detergent	ND	R	TST	ND	TST
Urea, 8M salt, aqueous solution	R	GR	R	ND	R
Xylene HC, aromatic	NR	R	R	ND	R

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Compound Compatibility

Filter to be used	PVDF	PES	PTFE	PES	PVDF
	.2µm	.2µm	.2µm	.45µm	.45µm
5-Fluorouracil			●		
(18F) Fluoromisondazole, Misiomidazole	●				
Acebutolol		●			
Acetylsalicylic acid		●			
Alpha1-Proteinase Inhibitor (Human)					●
Alprenolol		●			
Amiloride		●			
Amphotericin B for Injection USP					●
Atenolol		●			
Azathioprine				●	●
Azodicarbonamide		●			
Bleomycin Sulfate			●		
Caffeine		●			
Cetirizine				●	●
Chlorothiazide		●			
Chloramphenicol		●			
Cimetidine		●			
Ciprofloxacin		●			
Cisplatin, Cisplatin Injection			●		
Cyclosporine A	●				
Cytarabine			●		
Daunorubicin			●		
DE-310		●			
Diclofenac					●
Enalapril		●			
Ethionamide			●		
Factor IX Complex Heat-Treated					●
Gatifloxacin				●	●
Hydrochlorothiazide		●			

For more information and the references to this table please see htslabs.com.

Compound Compatibility

Filter to be used	PVDF	PES	PTFE	PES	PVDF
	.2µm	.2µm	.2µm	.45µm	.45µm
Ibuprofen				●	●
Iosniazid			●		
isonicotinic acid			●		
Ketamine		●			
Las 35917					●
Levofloxacin				●	●
Lomefloxacin				●	●
Methyl Gag; NSC-32946			●		
Metoprolol		●			
Mitomycin			●		
Morphazinamide			●		
Nadolol		●			
Nicotinic acid			●		
Paclitaxel	●				
p-Aminobenzoic acid (PABA)					●
p-aminosalicylic acid			●		
Pefloxacin				●	●
Pentoxifylline (PTX)	●				
Phenytoin					●
Pyrazinamide			●		
Pyrimethamine				●	●
Ranitidine		●			
Rifampicin				●	●
Sabeluzole					●
Streptokinase					●
Sulfadozine					●
Sulphasalazine		●			
Sulpiride		●			
Terbutaline		●			

For more information and the references to this table please see htslabs.com.

Compound Compatibility

Filter to be used	PVDF	PES	PTFE	PES	PVDF
	.2µm	.2µm	.2µm	.45µm	.45µm
Thiotepa Parenteral Sterile			●		
Timolol		●			
Tobramycin Vincristine Sulfate			●		
Tranexamic acid	●				
Triamcinolone Acetonide		●			
Triazinate; NSC-139105			●		
Tropicamide				●	
Vinblastine Sulfate			●		

For more information and the references to this table please see htslabs.com.

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