UCT

ENVIRONMENTAL METHODS

Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Solid Extraction and Gas Chromatography with Photoionization Detection

April 8, 2009

Method 506 Revison 1.1*

Background

Method 506 is an EPA analysis procedure for the determination of certain **phthalate and adipate esters.** The method can be applied to drinking water and ground water. Phthalate compounds efficiently partition from water using a C18 bonded silica solid-phase sorbent packed into cartridges. The following compounds determined by this method employ capillary column gas chromatography with a photoionization detector.

Analyte	Common Abreviation	CAS	GC Elution Order[1]
Bis(2-ethylhexyl) Phthalate	DEHP	117-81-7	6
Butyl benzyl Phthalate	BBP	85-68-7	4
Di-n-butyl Phthalate	DBP	84-74-2	3
Diethyl Phthalate	DEP	84-66-2	2
Dimethyl Phthalate	DMP	131-11-3	1
Bis(2-ethylhexyl) Adipate	DEHA	103-23-1	5
Di-n-octyl Phthalate	DnOP	117-84-0	7

[1]DB-5 fused silica capillary, 30mx0.32mm, 0.25 micron film

UCT Products Required:

ENVIRO-CLEAN® Universal C18 cartridge ECUNIC18

FLORISIL® Cleanup Cartridge (for phthalate esters, optional for dirty water)

Alumina Cleanup Cartridge (for phthalate esters, optional for dirty water)

Summary of Method

A 1-liter sample of water is extracted using an 83 mL C18 cartridge UCT ECUNIC18 then eluted with methylene chloride. The eluant is concentrated using a gentle stream of N_2 gas to reduce the volume to 1.0 mL. The concentration of analytes in the extract is determined with capillary column GC using photoionization detection.

Interferences

Care must be exercised to avoid sample contamination, as phthalate compounds are ubiquitous in the environment. Phthalate are used as plasticizers in PVC tubing and other common plastics found throughout the laboratory therefore the use of plastics must be avoided. Exhaustive clean up of reagents and glassware is required to eliminate background artifacts and prevent elevated GC baselines.

- Clean all glassware as soon as possible after use
- Rinse thoroughly with the last solvent used
- Heat glassware (except volumetric flasks) in a muffle oven at 400^{O}C for 1 hour
- Seal glassware with aluminum foil and store in a clean environment
- Use of high purity solvents will minimize interference problems

1. Sample Collection

a) Dechlorinate sample by adding 80 mg of sodium thiosulfate per liter. Mix until dissolved.
b) Samples must be refrigerated at 4^{O}C in a dark environment until analysis
c) Analyte stability may be effected by the matrix components

2. Cartridge Activation Procedure

a) Set-up a vacuum manifold system and mount cartridge on the glass adaptor. Various automated extraction systems may also be used.
b) Add a 10 mL aliquot of methylene chloride (CH_2Cl_2) and slowly draw through the cartridge
c) Repeat with a second 10 mL aliquot of methylene chloride. Hold for 1 minute.
d) Dry cartridge by drawing air through at full vacuum for 2-3 minutes

Note: The cartridge must not be allowed to go dry after the following steps, otherwise repeat

e) Add 10 mL of methanol to the cartridge and draw through until meniscus touches the surface of the cartridge frit
g) Add a second 10 mL portion of methanol and wait 1-2 minutes to activate sorbent
h) Add 10 mL reagent water to the cartridge and draw through until meniscus reaches the top of the frit
i) Cartridge is now ready for sample extraction

3. Sample Extraction

a) Add 5 mL of methanol to the 1-liter sample and mix well
b) Add the water sample to the cartridge and draw through over a period of about 20-30 minutes
c) Add 5 mL of acetonitrile (CH_3CN) to the sample bottle, shake then add to cartridge
d) After extraction, use full vacuum to dry the cartridge for 10 minutes
e) Place an eluate collection vial in the vacuum manifold
f) Add 5 mL of methylene chloride to the sample bottle and rinse
g) Using a disposable pipette transfer the methylene chloride to the cartridge and rinse the sides while adding
h) Repeat this procedure using another 5 mL aliquot of methylene chloride

4. Eluate Drying and Concentration

a) Pour eluant over a 3 gram bed of anhydrous sodium sulfate
b) Rinse vial and sodium sulfate with a 3 mL aliquot of methylene chloride
c) Repeat rinse using an additional 3 mL aliquot of methylene chloride
d) Place extract in a 28^{O}C heated evaporator and pass a stream of N_2 over solvent to evaporate
e) If sample is clean proceed to GC analysis

5. Florisil Column Cleanup for Phthalate Esters (if required)

Clean-up procedures are not required for clean drinking water. Under certain circumstances for dirty water, a Florisil or Alumina cleanup may be needed. If necessary, the following steps are used:

a) Add a 1 cm layer of anhydrous sodium sulfate to the top of a UCT Florisil cartridge
b) Flush cartridge with 40 mL of hexane, then discard but leave enough to cover frit
c) Add sample extract to the cartridge then rinse vial with 2 mL of hexane
d) Add 40 mL of hexane to the cartridge and elute. Discard this hexane solution
e) Elute using 100 mL of 20% diethyl ether in hexane (v/v) into a 500 mL K-D flask equipped with a 10 mL concentrator tube. Elute at a rate of about 2 mL/minute
f) No solvent exchange is required
g) Concentrate eluate in hot water bath at 85^{O}C

6. Alumina Column Cleanup for Phthalate Esters (if required)

a) Add a 1 cm layer of anhydrous sodium sulfate to the top of a UCT Alumina cartridge
b) Flush cartridge with 40 mL of hexane, then discard but leave enough to cover frit
c) Add sample extract to the cartridge then rinse vial with 2 mL of hexane
d) Add 35 mL of hexane to the cartridge and elute. Discard this hexane solution
e) Elute using 140 mL of 20% diethyl ether in hexane (v/v) into a 500 mL K-D flask equipped with a 10 mL concentrator tube. Elute at a rate of about 2 mL/minute
f) No solvent exchange is required
g) Concentrate eluate in hot water bath at 85^{O}C

7. Sample is now ready for GC analysis

Chromatographic Conditions

- Inject: 1-2 µL of sample extract or standard
- Injector: 295^{O}C
- Detector: 295^{O}C
- Program: 1 minute hold @ 60^{O}C
- 6^{O}C/minute to 260^{O}C, hold 10 minutes
- Splitless injection with 45-second delay
- Photoionization detection @ 10 eV

*The analyst should refer to EPA Method 506 “Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Solid Extraction and Gas Chromatography with Photoionization Detection”, Revision 1.1 Issued 1995, F.K. Kawahara and J.W. Hodgeson, Ed. By D. J. Munch US EPA, National Exposure Research Laboratory, Office of Research and Development, US Environmental Protection Agency, Cincinnati, Ohio 45268

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ENVIRO-CLEAN® Cartridges

Part Number: ECUNIC18

EPA Method 508.1 Rev 2.0

August 25, 2009

The UCT ECUNIC18 Universal cartridge is designed to provide a new level of performance for the solid-phase extraction (SPE) for the analysis of **chlorinated pesticides, herbicides and organohalides** using EPA method 508.1. With the cartridge's high capture efficiency, fast flow and excellent dry times, laboratory throughput is improved reducing sample preparation time.

Product Benefits

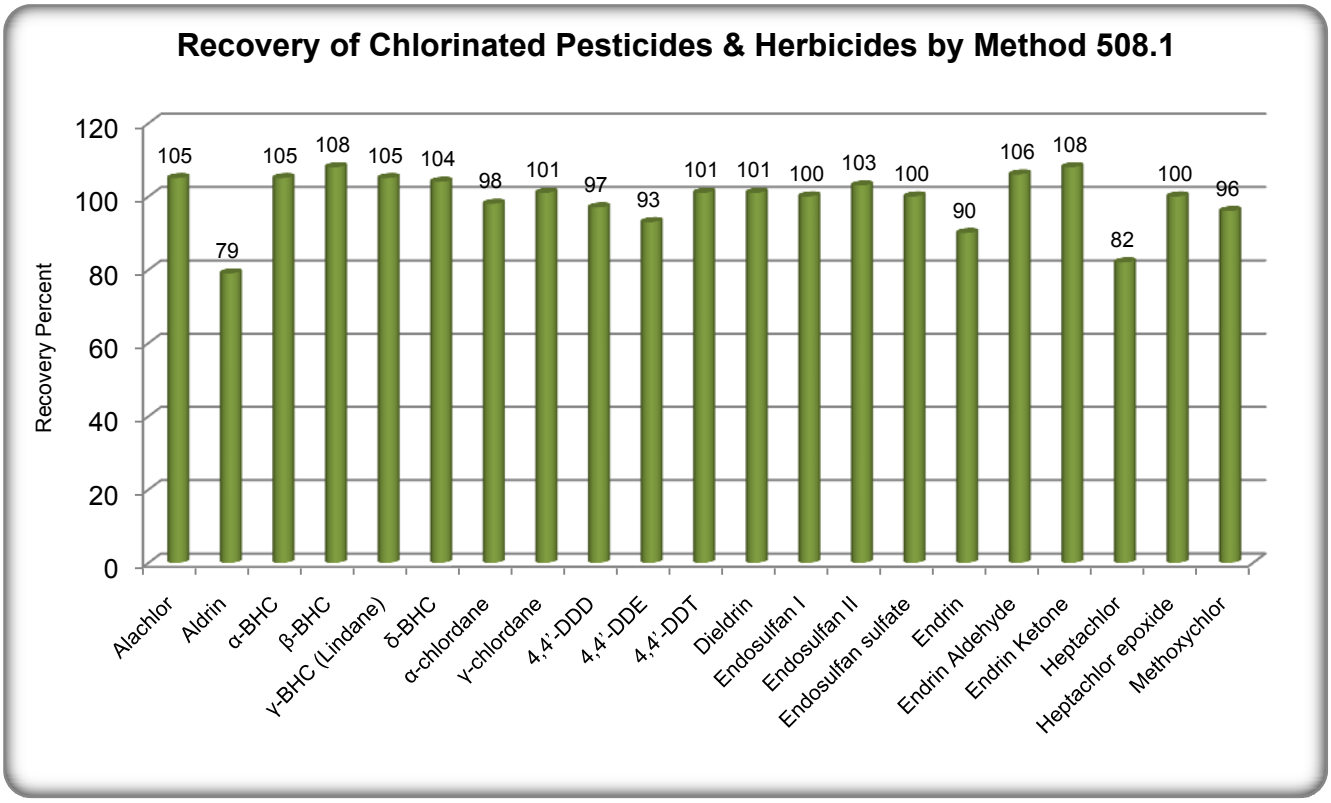
- SPE cartridge containing bonded C18 phase
- Excellent pH stability
- Fast flow rates for rapid analyte capture
- Works well at all levels of analyte loading
- Consistent results for excellent reproducibility
- PTFE frits eliminate potential contamination yielding clean extracts
- Packaged in metalized bags to maintain product cleanliness

Product Features

- Cartridges manufactured from special proprietary polypropylene
- Each cartridge contains 1100 mg endcapped C18 bonded ultra-clean silica sorbent
- Can be used on manual single or multi-station vacuum manifold systems
- Can be used with automated extraction systems

UCT Cartridge ECUNIC18 Shows Excellent Recovery with Replicate Samples of Laboratory Fortified Blanks (LFB)

Analyte	CAS	Retention Time[1]	Amt Spiked µg/L	Average Recovery %	Stdev
Alachlor	15972-60-8	22.86	0.1032	105	NA
Aldrin	309-00-2	24.81	0.0999	79	2.08
α-BHC	319-84-6	NA	0.1032	105	0.96
β-BHC	319-85-7	NA	0.1043	108	1.50
γ-BHC (Lindane)	58-89-9	NA	0.1038	105	0.82
δ-BHC	319-86-8	NA	0.1040	104	1.26
α-chlordane	5103-71-9	29.58	0.0969	98	0.50
γ-chlordane	5103-74-2	28.65	0.0969	101	1.71
4,4'-DDD	72-54-8	33.38	0.2056	97	2.08
4,4'-DDE	72-55-9	31.97	0.2006	93	2.38
4,4'-DDT	50-29-3	35.80	0.2014	101	0.96
Dieldrin	60-57-1	30.95	0.2046	101	0.82
Endosulfan I	959-98-8	29.36	0.1028	100	0.82
Endosulfan II	33213-65-9	32.81	0.2054	103	0.96
Endosulfan sulfate	1031-07-8	35.38	0.2124	100	2.06
Endrin	72-20-8	32.24	0.2016	90	7.33
Endrin Aldehyde	7421-93-4	33.96	0.2012	106	9.54
Endrin Ketone	53494-70-5	NA	0.2068	108	1.73
Heptachlor	76-44-8	NA	0.1032	82	5.29
Heptachlor epoxide	1024-57-3	27.20	0.1034	100	0.96
Methoxychlor	72-43-5	25.02	1.0016	96	1.71



Determination of Chlorinated Pesticides, Herbicides, Organohalides by Liquid-Solid Extraction and Electron Capture Gas Chromatography

Method 508.1 Rev 2.0

Method Summary*

A 1 liter sample of water is extracted by drawing through a **UCT C18 Universal cartridge ECUNIC18**. The analytes captured on the solid-phase are eluted from the cartridge using a small volume of ethyl acetate (EtAc) and methylene chloride ($MeCl_2$). The extract is concentrated by evaporation before analysis by injection into a gas chromatograph/electron capture system (GC/ECD) fitted with a high resolution fused silica capillary column

Sample Collection, Preservation and Storage

- Collect samples in glass containers
- Do not prerinse the container with the sample water

Preserve the sample by adding mercuric chloride ($MgCl_2$) to achieve a concentration of 10 mg/L. Other preservatives may be used if shown to be effective

Interferences

- Interfering contamination may occur when a sample of low concentration is analyzed immediately after a sample of high concentration. A laboratory blank should be inserted between low and high concentration samples to minimize this problem
- Other interferences may be caused by contaminants in solvents, reagents and sample processing apparatus that lead to anomalous GC peaks or elevated baselines.

1) Condition Cartridge

a) Insert a cartridge into the glass vacuum manifold or automated extraction system
b) Wash the cartridge with 5mL of a 1:1 mixture of ethyl acetate (EtAc) and methylene chloride ($MeCl_2$)
c) Draw the solvent through the cartridge with a low vacuum setting so that the solvent slowly drips through
d) Add 10 mL of methanol (MeOH) to the cartridge then slowly draw some of it through
e) Rinse the cartridge with 10 mL of reagent water and draw most of it through leaving a thin layer on the top of the sorbent

Do not let the cartridge go dry after addition of methanol otherwise repeat the steps d) and e)

2) Sample Addition

a) Add 5 mL of methanol to the 1 liter sample and mix well
b) Add 50 µL of the surrogate compound to the water sample and shake well
c) Draw the water sample through the cartridge under sufficient vacuum to require about 20 minutes or more for extraction
d) Dry the cartridge by drawing air or nitrogen through for about 10 minutes

3) Extract Elution

a) Insert an eluate collection tube into the vacuum manifold
b) Rinse the inside walls of the sample bottle using 10 mL EtAc then transfer solvent to the cartridge using a disposable pipette
c) Rinse the inside walls of the sample bottle using 10 mL of $MeCl_2$ then transfer to the cartridge using a disposable pipette
d) Using a disposable pipette rinse the cartridge and filter reservoir with two 3 mL portions of 1:1 EtAc:$MeCl_2$

e)

4) Sample Drying

a) Pour the combined elutes together through a drying tube (**UCT ECSS15M6**) which contains 5 grams anhydrous sodium sulfate
b) Rinse the drying tube with two 3 mL portions of 1:1 EtAc:$MeCl_2$
c) Concentrate the extract to about 0.8 mL under a gentle stream of nitrogen while heating in a water bath
d) Rinse the inside walls of the concentrator tube two or three times with EtAc during the evaporation
e) Add IS
f) Adjust the final volume of the extract to 1.0 mL

5) Analysis

a) Inject a 1-2 µL aliquot into a GC
b) Identify the analytes in the sample by comparison of the retention time to known reference chromatograms

6) GC Analysis Conditions

Retention time was determined with the following GC conditions:

- Injector temperature -- 250°C
- Detector temperature -- 320°C
- Injection volume -- 2 µL, splitless for 45 seconds
- Temperature program -- Inject at 60°C and hold one minute

-- program at 20°C/min. to 160°C hold three minutes
-- program at 3°C/min. to 275°C with no hold
-- program at 20°C/min. to 310°C with no hold

The IS retention time using these conditions is 21.15 minutes. The SUR retention time using these conditions is 28.18 minutes.

For complete details on Method 508.1 , rev 2.0 “Determination of Chlorinated Pesticides, Herbicides, and Organohalides by Liquid-Solid Extraction and Electron Capture Gas Chromatograph”, the analyst is referred to: J. W. Eichelberger rev 1.0, 1994 and J. Munch, rev 2.0, 1995, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

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EPA Method 509

Determination of Ethylene Thiourea (ETU) in Water Using Gas Chromatography with a Nitrogen-Phoshorus Detector

UCT Product CLEAN-ELUTE™ 25,000 mg diatomatious earth, 200 mL cartridge

October 1, 2009

Background

Method 509 is a gas chromatographic method for the determination of ethylene thiourea (CAS 96-45-7) a metabolic byproduct of the ethylene bisdithiocarbonate (EBDC) fungicides in water. Toxicological studies indicate that ETU may produce goitrogenic, tumorigenic, and teratogenic effects in laboratory animals, raising the concern that residues may be found in agricultural commodities. The method uses a packed column of diatomaceous earth to capture the analyte before elution with methylene chloride. Confirmation is made using a nitrogen-phosphorous detector or a mass spectrometer.

Method Summary

A 50 mL water sample is adjusted to ionic strength and pH by the addition of ammonium chloride (NH_4Cl) and potassium fluoride (KF). The sample is poured into a UCT CLEAN-ELUTE™ column and the ETU is eluted from the column using 400 mL of methylene chloride. An excess of a free radical scavenger is added to the eluate. The methylene chloride eluant is concentrated to 5 mL after exchange into ethyl acetate. GC analysis with a nitrogen-phosphorous detector or mass spectrometer is used for quantitation.

Safety

- ETU is a suspected carcinogen. Prepare all standards in a fume hood

Sample Collection and Preservation

- Grab samples must be collected in 60 mL glass containers fitted with Teflon-lined crew caps
- Do not pre-rinse with sample before collection
- After collection shake the sample bottle for 1 minute
- ETU may degrade in water even during refrigeration. Mercuric chloride has been used as a preservative but due to its toxicity and harm to the environment is not recommended
- Store sample on ice or in refrigerator at 4°C and protected from light. Extract as soon as possible after collection

Interferences

Method interferences arise from contaminated glassware, solvents, reagents and other laboratory apparatus in which the sample may come in contact. All reagents and glassware must be shown to be free from interferences under analysis conditions.

- Glassware must be scrupulously clean
- Clean glassware by rinsing with the last solvent used followed by hot water and detergent. Rinse with reagent water, dry and heat in an oven at 400°C for one hour. Do not heat volumetric flasks
- Always use high purity reagents and solvents
- Interfering contamination may occur when a low concentration sample is analyzed after a high concentration sample. Complete rinsing of the syringe using ethyl acetate may be required

Analysis Procedure

1) Sample Extraction

a) Pipette 50 mL of the water sample into a clean bottle
b) Add 1.5 grams ammonium chloride (NH_4Cl)
c) Add 25 grams potassium fluoride (KF)
d) Seal bottle and shake until salts are completely dissolved

2) Sample Extraction

a) Add 5 mL of 1000 g/mL of dithiothreitol (DTT, Cleland's Reagent) in ethyl acetate as a free radical scavenger to a 500 mL Kuderna-Danish K-D concentrator tube
b) Support a CLEAN-ELUTE™ 200 mL cartridge using a clamp over a (K-D) tube
c) Add the entire contents of the bottle from step 1) d) above
d) Do not use vacuum but allow the cartridge to stand for 15 minutes

3) Sample Collection

a) Add 400 mL of methylene chloride in 50 mL aliquots to the CLEAN-ELUTE™ column
b) Collect the eluant in the K-D apparatus

4) Extract Concentration

The following steps must be conducted in a fume hood

a) Add two boiling chips to the K-D apparatus and attach a macro Snyder column
b) Attach a condenser to the Snyder column to collect solvent
c) Place the K-D apparatus in a 65-70°C water bath so that the K-D tube is partially submerged in the water
d) Once liquid volume had been reduced to 5 mL remove from the water bath
e) Continue to reduce the liquid volume to < 1 mL in an analytical evaporator at 35-40°C under a stream of nitrogen
f) Dilute sample to 5 mL with ethyl acetate rinsing the walls of the K-D apparatus
g) Add 50 µL of internal standard and agitate
h) Transfer to a GC vial
i) Sample is ready for analysis

5) GC Analysis Conditions

Primary:

Column: 10 m long x 0.25 mm I.D. DB-Wax bonded fused

Carrier Gas: He @ 30 cm/sec linear velocity

Makeup Gas: He @ 30 mL/min flow

Detector Gases: Air @ 100 mL/min flow; H2 @ 3 mL/min flow

Injector Temperature: 220°C

Detector Temperature: 230°C

Oven Temperature: 220°C isothermal

Sample: 2 µL splitless; nine second split delay

Detector: Nitrogen-phosphorus

Confirmation Conditions:

Column: 5 m long x 0.25 mm I.D. DB-1701 bonded fused

Carrier Gas: He @ 30 cm/sec linear velocity

Makeup Gas: He @ 30 mL/min flow

Detector Gases: Air @ 100 m:/min flow; H2 @ 3 mL/min flow

Injector Temperature: 150°C

Detector Temperature: 270°C

Oven Temperature: 150°C isothermal

Sample: 2 µL splitless; nine second split delay

Detector: Nitrogen-phosphorus

Analyte	Primary Column RT (min)	Confirmation Column RT
ETU	3.5	4.5
THP internal standard	5.1	5.0
PTU surrogate standard	2.7	2.2

*The analyst should refer to EPA Method 509 “Determination of Ehtylene Thiourea (ETU) in Water Using Gas Chromatography with a Nitrogen-Phoshorus Detector”, Revision 1.0 Issued 1992, By DJ Munch and RL Graves, US EPA, National Exposure Research Laboratory, Office of Research and Development, US Environmental Protection Agency, Cincinnati, Ohio 45268
and TM Engel and ST Champagne, Battelle, Columbus Division

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ENVIRO-CLEAN® Carbon Cartridges

Part Number: EU52112M6

EPA Method 521

August 25, 2009

The UCT activated carbon cartridge is designed to provide a high level of performance in solid-phase extraction for the analysis of **nitrosamines** in finished drinking water.

Product Benefits

- Designed to meet US EPA specifications
- Free from interferences that cause false positives
- Double ringed to prevent fines
- No Lot to Lot variability
- Excellent analytical reproducibility
- Packaged in metalized, sealed pouches to maintain product cleanliness

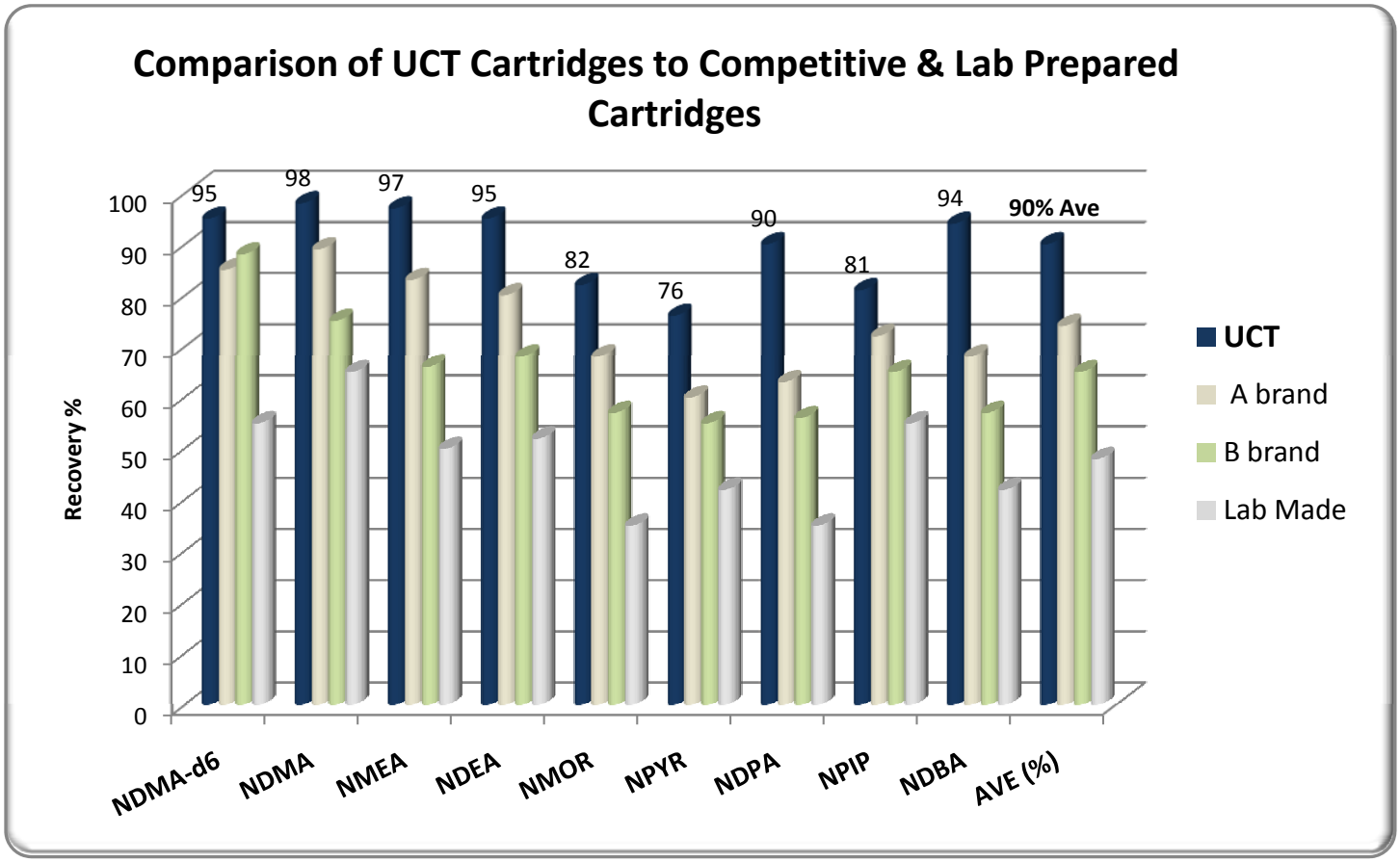
Product Features

- Each 6 mL cartridge contains 2000 mg of activated coconut carbon sorbent
- Can be used on manual single or multi-station manifold systems
- Cartridges may be used with automated extraction systems

Nitroaromatics, Nitramines and Nitrate Ester Analytes with CAS

UCT Product Number: ENVIRO-CLEAN® EU52112M6

Analyte	Abbreviation	CAS	% Recovery n=3
N-Nitrosodimethylamine	NDMA	62-75-9	95
N-Nitrosomethyldiethylamine	NMEA	10595-95-6	98
N-Nitrosodiethylamine	NDEA	55-18-5	95
N-Nitrosodi-n-propylamine	NDPA	621-64-7	90
N-Nitrosodi-n-butylamine	NDBA	924-16-3	94
N-Nitrosopyrollidine	NPYR	930-55-2	76
N-Nitrosopiperidine	NPIP	100-75-4	81



Data indicate the performance of **UCT** brand cartridges exceeds competitive brands **A** and **B,** and cartridges prepared in the laboratory

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EPA Method 521* Summary

Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS)

Scope and Application

Method 521 is a procedure using activated carbon for the determination of various nitrosamines in finished drinking water. The method can also be used for untreated source waters but has not been evaluated for these sources. Nitrosamines are sufficiently thermally stable and volatile for direct analysis by gas chromatography. Single laboratory LCMRL for the analytes in this method range from 1.2-2.1 ng/L.

Method Summary

Analytes and surrogates are readily extracted when a 500 mL water sample is drawn through a solid-phase extraction cartridge containing 2 grams of coconut activated carbon. The organic compounds eluted from the solid-phase with a small quantity of methylene chloride. The solvent is concentrated and an internal standard added. The sample components are identified after injection on a fused silica capillary column of a GC/MS/MS equipped with a large volume injection injector.

Interferences

- Major contaminant sources are reagents and water
- Nitrosamines may be present in trace amount in rubber products such as gloves and water systems. NDMA can leach from rubber products. These products must be avoided in the reagent water system. Analysis of a laboratory blank can provide information about the source of contamination
- Water stored in glass bottles with PTFE caps is recommended
- Rubber coated septa on injection vial may also introduce method analytes into the sample extracts giving false high readings

Sample Collection

- Filed sampling equipment must be free of plastic or rubber tubing
- All field samples must be dechlorinated with 80-100 mg of sodium thiosulfate per liter at time of collection
- Samples must iced during shipment and not exceed 10 °C
- Sample stored in the lab must be held at 6ºC
- Analyze with 14 days after collection
- Sample extracts can be stored up to 28 days in amber vials at -15°C or less and protected from light

Safety

The analytes in this method are classified as known human and mammalian carcinogens. Standard and stock solutions should be handled using suitable protection to skin and eyes.

Notes

- GC systems must be capable of temperature programming
- Deactivated post liners should be used
- Tandem mass spectrometers may be either triple quadrupole or ion trap

Procedure (manual or automated)

1) **Cartridge Conditioning**
   a) Add 3 mL of methylene chloride to the cartridge, then turn on the vacuum and slowly draw completely through the cartridge
   b) Add 3mL of methanol to the cartridge, turn on vacuum and draw through
   c) Add 3 mL of methanol again and draw through so that the methanol just covers the top layer of carbon.

Do not let the cartridge go dry after this step otherwise repeat starting at step 1- b)

   d) Add 3 mL of reagent water to the cartridge and draw through
   e) Repeat water rinse, step d) **5 additional times**

Proper conditioning of the cartridge is essential for good precision and accuracy

2) **Sample Extraction**
   a) Adjust the vacuum setting so that the flow rate is 10 mL/minute
   b) After sample extraction draw air through the cartridge for **10 minutes at full vacuum**
   c) After drying, proceed immediately to cartridge elution step 3)

3) **Cartridge Elution**
   a) Insert a clean collection tube in the manifold
   b) Fill the cartridge with methylene chloride
   c) Partially draw the methylene chloride through at low vacuum then turn vacuum off and allow cartridge to soak for 1 minute
   d) Draw the remaining methylene chloride through in dropwise fashion
   e) Continue to add methylene chloride to the cartridge as it is being drawn through until a total of 12-13 mL have been added

   Note: Small amounts of residual water from the sample container and SPE cartridge may form an immiscible layer with the extract. To eliminate the water a drying column packed with 5 grams of anhydrous sodium sulfate or use **UCT ECSS15M6** for drying. Wet the cartridge with a small volume of methylene chloride before adding extract. Rinse the drying column with 3 mL of methylene chloride.

   f) Concentrate the methylene chloride to about 0.9 mL in a water bath near room temperature. Do not concentrate less than 0.5 mL as loss of analyte may occur

4) **Sample Analysis**
   a) Calibrate the MS in EI mode using FC-43
   b) Inject into a GC/MS/MS
   c) Identify the product ion spectrum to a reference spectrum in a user created data base

Analyte	Retention Time (min)	Precursor Ion (m/z)	Product/Quantitation Ion (m/z)
NDMA	8.43	75	43(56)
NMEA	11.76	89	61(61)
NDEA	14.80	103	75(75)
NPYR	22.34	101	55(55)
NDPA	22.40	131	89(89)
NPIP	24.25	115	69(69)
NDBA	30.09	159	57(103)
NDMA-d6 surrogate	8.34	81	46(59)
NMEA-d10 internal std	14.63	113	81(81)
NDPA-d6 internal std	22.07	145	97(97)

Retention times were obtained on a Varian Saturn 4 GC/MS/MS using the following conditions:

Injector Program

Temp (°C)	Rate (°C/min)	Time (min)
37	0	0.72
250	100	2.13
250	0	40

Injector Split Vent Program

Time (min)	Split Status
0	Open
0.70	Closed
2.00	Open

GC Oven Temperature Program

Temperature (°C)	Rate (°C/min)	Hold Time (min)
40	0	3.0
170	4.0	0
250	20.0	3.0

Limits and Lowest Concentration Minimum Reporting Levels

Analyte	DL (ng/L)	LCMRL (ng/L)
NDMA	0.28	1.6
NMEA	0.28	1.5
NDEA	0.26	2.1
NPYR	0.35	1.4
NDPA	0.32	1.2
NPIP	0.66	1.4
NDBA	0.36	1.4

*For complete details on Method 521, September 2004, the analyst is referred to: J.W.Munch & M.V.Bassett, "Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS), National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

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Analysis of 1,4-Dioxane in 500 mL of Drinking Water by Gas Chromatography-Mass Spectrometry SIM Detection

Part Number: EU52112M6, 2 grams activated carbon in a 6 mL cartridge

February 24, 2009

1,4-dioxane (CAS 123-91-1), often referred to as dioxane, is a highly soluble non-biodegradable ether. This compound is used as solvent stabilizer to prevent the breakdown of chlorinated solvents.

1) Sample Preparation:

a) To dechlorinate the water sample add 25 mg of sodium sulfite, **then** acidify with 0.5 g of sodium bisulfate and swirl until dissolved.

2) Condition Cartridge:

a) Place a EU52112M6 cartridge on a single or multi-station vacuum manifold or automated extraction system
b) Add 3 mL of dichloromethane (methylene chloride) to the cartridge and draw through
c) Add 3 mL of methanol and draw through
d) Repeat step c)
e) Add 3 mL of DI water and draw through
f) Repeat step e) 4 times

3) Load Sample:

a) Add Internal standard * to the sample
b) Add sample to the cartridge. Adjust for a flow rate of 5 - 10 mL/minute
c) Dry cartridge by drawing air at full vacuum for 10 minutes

4) Elution:

a) Place a clean collection vial in the vacuum manifold
b) Add 3 mL of dichloromethane to the cartridge. Slowly draw through
c) Add a second 6 mL aliquot of dichloromethane and draw through
d) Add surrogate** (0.5 ppm) to the extract and bring to 10 mL final volume with dichloromethane
e) Dry extract by adding 2 g anhydrous sodium sulfate (Na_2SO_4) and vortex to mix the extract

5) Quantitate:

a) GC-MS-SIM (electron ionization)

6) Instrument & Conditions:

Column: Varian CP-Select 624 CB (6% cyanopropyl phenyl, 94& PDMS, 30 m 0.25mm x 1.4 um column) or equivalent
Injector: 200°C (Splitless mode)
Injector Volume: 1 µL
Flow: 1 mL minute
Oven: 30°C for 1 minute, 90°C at 7°C/minute, 200°C at 20°C/minute for 3 minutes

MS: quadrupole MS

SIM MODE

Dwell time 100 µs

Emission current 100 µA

***** Quantitation ions**

Segment 1 (0-8 minutes)	****THF-d_8**	**m/z 40***, 78, 80**
Segment 2 (8-16.5 minutes)	**1, 4 dioxane**	**m/z 58, 88*****
	***1, 4 dioxane-d_8**	**m/z 62, 64, 96*****

R 1.0 20090114

*Grinmmett, Paul E., Munch, Jean W., Method Development for the Analysis of 1,4-Dioxane in Drinking Water Using Solid-Phase Extraction and Gas Chromatography-Mass Spectrometry, Journal of Chromatographic Science, Vol. 47, January 2009. Office of Research and Development, National Exposure Laboratory, Cincinnati, OH.

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Method 525.2 Analytes

The analyte list for this method is comprised of over 120 compounds representative of several classes of pesticides, polynuclear aromatic hydrocarbons, PCBs, phthalates and adipates and other drinking water pollutants. Recovery ranges from 70-130%. Refer to the published method for compound specific MDL's.

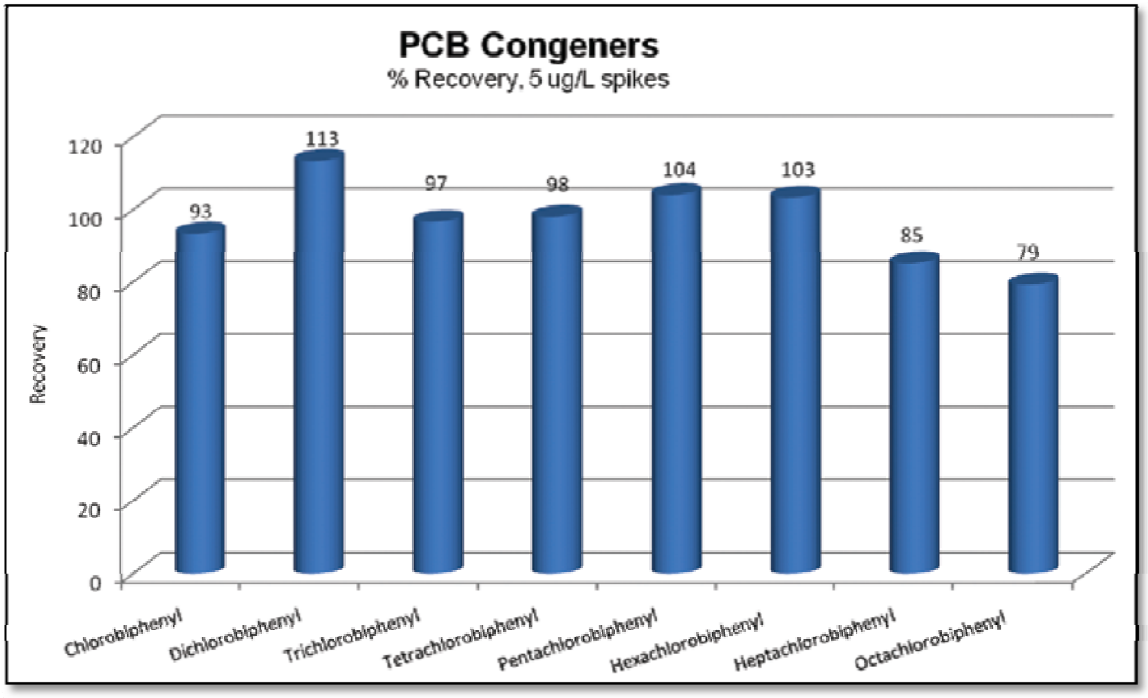
The validation data presented herein were determined on independent lots of UCT ENVIRO-CLEAN® Universal Cartridges. MDLs were not determined on all analytes as part of this validation. In addition to the listed method analytes, recovery data for an extended list of analytes is also included.

Table of Compounds
Tested using the UCT ENVIRO-CLEAN® Universal Cartridge 525

Analyte	Average 3 Replicates % Recovery	Std Dev
Acenaphthene	100	0.0
2,4-dinitrotoluene	83	NA
2,6-dinitrotoluene	78	NA
4,4"-DDE	91	4.2
4,4"-DDT	94	3.5
4,4'-DDD	94	4.5
Acenaphthylene	96.0	0.012
Acenaphthene	99.1	0.013
Acetochlor	115	0.01
Alachlor	99	0.007
Aldrin	77	4.4
Ametryn	95	4.6
Anthracene	80	0.0
Atraton	84	17.3
Atrazine	111	0.011
Benzo(a) anthracene	75.4	0.049
Benzo(a)pyrene	105	9.9
Benzo(b)fluoranthene	184.9	0.022
Benzo(k)fluoranthene	95.7	0.029
Benzo[g,h,i]perylene	83.1	0.05
BHC, alpha	108	6.9
BHC, beta	97	3.1
BHC,delta	109	7.9
BHC,gamma	102	11.9
bis- (2-ethylhexyl) adipate	95.1	0.033
bis 2 ethylhexyl phthalate	104	0.029
Bromacil	126	0.012
Butachlor	113	0.005
Butylate	103	4.6
Butylbenzylphthalate	97.1	0.02
Caffeine	90.0	0
Captan	86.9	0.273
Carboxin	103	12.9
Chlordane, alpha	97	4.6
Chlordane, gamma	94	2.5
Chlordane, trans nonachlor	115	11.0
Chlorneb	113	11.0
Chlorobenzilate	118	10.0
Chlorpropham	130	0
Chlorpyrifos (Dursban)	107	5.0
Chlorothalonil	117	12.1
Chrysene	100	0.012
Cyanazine (Bladex)	126	0.008
Cycloate	111	15.0

Analyte	Average 3 Replicates % Recovery	Std Dev
Dacthal (DCPA) methyl ester	118	13.1
Diazinon	135	0.031
Dibenzo[a,h]anthracene	77.4	0.051
Dichlorvos (DDVP)	127	9.5
Dieldrin	96	6.8
Diethylphthalate	99.1	0.071
Dimethoate	106	0.008
Dimethylphthlate	78.6	0.022
Di-n-butylphthalate	113	0.12
Diphenamid	119	0.008
Disulfoton	92.1	0.01
Disulfoton Sulfone	108	12.5
Endosulfan I	116	11.1
Endosulfan sulfate	114	6.8
Endrin	88	0.0
Endrin Aldehyde	97	3.6
Endrin Ketone	90	3.8
EPTC	102	0.005
Ethion	112	0.005
Ethoprophos	109	5.8
Etridiazole (terrazole)	97	1.2
Fenarimol	70	0.0
Fluoranthene	100	0.018
Fluorene	99.7	0.012
Heptachlor	79	8.2
Heptachlor Epoxide Iso A	116	16.3
Hexachlorobenzene	94	17.4
Hexachlorocyclopentadiene	82	8.4
Hexazinone (Velpar)	105	8.1
Indeno[1,2,3-cd]pyrene	77.4	0.16
Isophorone	91	NA
Lindane	127	4.8
methoxychlor	123	7.6
Methyl Paraxon (Parathion)	115	5.0
Metolachlor	111	0.004
Metribuzin	109	0.005
Mevinphos (phosdrin)	117	12.1
MGK 264	121	5.8
Molinate	114	0.013
Naphthalene	90.3	0.013
Napropamide (Devrinol)	115	2.3
Nonachlor, trans	116	11.1
Norflurazon	133	6.1
PCNB (carbaryl)	91.4	0.021
Pebulate	101	1.7
Pentachlorophenol	80	0.017
Permethrin, cis	124	2.1
Permethrin, trans	123	3.1
Perylene-d12	119	0.0
Phenanthrene	96.9	0.014
Phenanthrene-d10	99	6.6
Prometon	78.6	0.008
Prometryn	110	0.012
Pronamide (propyzamide)	101	1.2
Propachlor	113	15.0
Propazine	105	4.6
Pyrene	94.6	0.022
Simazine	91.4	0.005
Simetryn	93	4.6
Stirofos (tetrachlorvinphos)	126	6.9
Thiobencarb	112	0.008
Tebuthiuron	85	33.5
Terbacil	120	3.5
Terbutryn	103	2.3
Triademefon	98	6.9
Tricyclazole	107	5.0
Trifluralin	82	9.7
Trifluran	83	9.2
Trithion (carbofenothion)	101	0.004
Terbufos	95	7.0
Vernolate	107	1.2

PCB Congeners	Average	Std Dev
2-chlorobiphenyl	93	2.3
2.3-Dichlorobiphenyl	113	15.0
2,4,5-trichlorobiphenyl	97	3.1
2,2,4,4-tetrachlorobiphenyl	98	5.3
2,2,3,4,6-pentachlorobiphenyl	104	2.0
2,2,4,4,5,6-hexachlorobiphenyl	103	3.1
2,2,3,3,4,4,6-heptachlorobiphenyl	85	1.2
Octachlorobiphenyl (BZ#200)	79	1.2



EPA Method 525.2 Revision 2.0
Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry

Method Summary

A one liter water sample is adjusted to pH <2 using 6N HCL before passing through an 83 mL ENVIRO-CLEAN® Universal 525 UCT cartridge (ECUNI525). Analytes are eluted from the cartridge with ethyl acetate and methylene chloride. The extract is reduced in volume to 1.0 mL and analyzed by GC/MS.

Method

1. Assemble an all glass filtration apparatus using an 83 mL UCT Universal 525 cartridge.

a) Use of a vacuum manifold for multiple extractions or automated extractions is acceptable

2. Wash the extraction apparatus and cartridge

a) Add 10 mL of a 1: 1 mixture of ethyl acetate:methylene chloride (EtAc: $MeCl_2$) to the reservoir.
b) Draw a small amount through the cartridge with vacuum
c) Turn off the vacuum and allow the cartridge to soak for about one minute
d) Draw the remaining solvent through the cartridge to waste
e) Allow the cartridge to dry for 3 minutes under full vacuum

3. Condition the cartridge

a) Add 10 mL of methanol
b) Draw a small amount through the cartridge
c) Let soak for about one minute
d) Draw most of the remaining methanol through the cartridge, leaving 3 to 5 mm of methanol on the surface of the cartridge frit
e) Immediately add 20 mL of reagent water to the cartridge and draw most of the water through leaving 3 to 5 mm on the top of the cartridge frit

Note: Do not let the cartridge dry out after the addition of water

f) Add 5 ml of methanol to the water sample and mix well
g) Add the water sample to the cartridge and under vacuum, filter at a rate of approximately 50 mL per minute
h) Drain as much water from sample bottle as possible
i) Dry the cartridge under vacuum for 10 minutes

Note: Exceeding a 10 minute dry time could result in low recoveries. For faster drying, remove the cartridge and tapping the excess moisture from the bottom of the cartridge before continuing vacuum drying

5. Remove cartridge assembly

a) Insert a suitable sample tube for eluate collection
b) Add 10 mL of EtAc to the sample bottle
c) Rinse the sample bottle thoroughly
d) Transfer the solvent to the cartridge with a disposable pipette, rinsing sides of filtration reservoir
e) Draw half of solvent through cartridge then release the vacuum. Allow the remaining solvent to soak the cartridge for about one minute
f) Draw remainder through under vacuum
g) Repeat the solvent rinse of the sample bottle and apparatus using 10 mL of 1:1 EtAc:$MeCl_2$
h) Using a disposable pipette, rinse down the sides of the cartridge and bottle holder with another 10 mL aliquot of 1: 1 EtAc:$MeCl_2$
i) Add the rinse to the cartridge, then draw through

10. Dry the combined eluant

a) Use granular anhydrous sodium sulfate
b) Rinse the collection tube and sodium sulfate with two x 3 mL portions of $MeCl_2$ and place combined solvent in a concentrator tube
c) Draw through using vacuum
d) Concentrate the extract to 1 mL under gentle stream of nitrogen (may be warmed gently) being careful not to spatter the contents.

Note: Do not concentrate to <0.5 mL or loss of analytes could occur. Rapid extract concentration could result in loss of low molecular weight analytes

12. Analyze by GC/MS

Revison 2.0, 1995. Method authors: Eichelberger, J. W., Behymer,T .D. Budde, W L., Munch, J., National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

This summary highlights major steps in the 525.2 method. Complete details about the preparation and composition of reagent solutions can be found in method and should be referenced by anyone needing complete details. It is available as a part of Supplement 11 from National Technical Information Service (NTIS), Springfield, VA 22161; publication PB 92 207703. (800) 553-6847 or at www.epa.gov/safewater/methods/methods.html

DETERMINATION OF PHENOLS IN DRINKING WATER BY SOLID-PHASE EXTRACTION (SPE) AND CAPILLARY COLUMN GAS CHROMATOGRAPHY (GC/MS)

Part #: ECDVB156

February 3, 2009

Method 528 Revision 1.0

Reagents

Methylene Chloride
Methanol
Anhydrous Sodium Sulfate 5 g in a 6 mL cartridge ECSS25K
Enviro-Clean® 500mg in a 6 mL cartridge ECDVB156

Method Summary

A one liter sample of drinking water is extracted by drawing through a UCT Enviro-Clean® 6 mL cartridge containing 500 mg of polystyrene divinyl benzene copolymer. The phenolic compounds are eluted from the solid-phase using a small quantity of methylene chloride. An aliquot of the concentrated extract is injected into a high resolution fused silica capillary column of a GC/MS system. Respective phenols are identified by comparing their mass spectra and retention times by comparison to standards.

Condition Cartridge

- Adjust the pH of the water sample to 2 or less by the addition of 6N HCl acid
- Rinse the cartridge with three, 3 mL aliquots of methylene chloride, then draw to waste
- Rinse the cartridge with three, 3 mL aliquots of methanol then draw to waste. After the third rinse, leave enough methanol in the cartridge to cover the frit. **Do not let the cartridge dry out at this point.**
- Rinse the cartridge with three, 3 mL aliquots of 0.05N HCl. **Turn off the vacuum before the HCl solution drops below the level of the frit**

Sample Addition

- Add the water sample to the cartridge and adjust the vacuum such that the flow rate is about 20 mL/minute (50 minutes for a 1 liter sample). Allow the cartridge to dry for at least 10-15 minutes before proceeding to the next step. A dry cartridge is important for good recoveries.

Extract Elution

- Rinse the inside of the sample bottle with a 10 mL portion of methylene chloride. Add this to the cartridge and draw this through to the collection tube in a dropwise fashion.
- Add 2-3 mL of methylene chloride to the cartridge then slowly draw this through to the collection tube in a dropwise fashion.

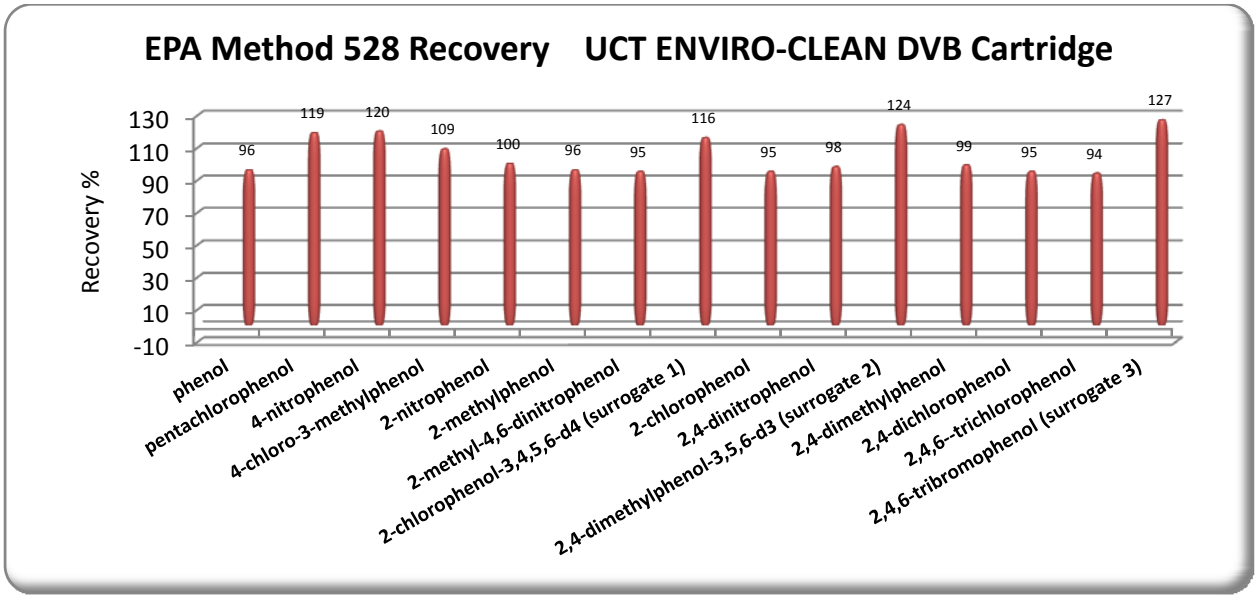
Eluate Drying

- Dry the eluate by passing through prerinsed anhydrous sodium sulfate column UCT Part number ECSS15M6 and collect eluate in a clean tube
- Rinse the sodium sulfate column with two, 3 mL aliquots of methylene chloride and collect in the tube
- Concentrate the extract to about 0.9 mL in a warm water bath (40^{O}C) under a gentle stream of nitrogen
- Adjust final volume to 1.0 mL with methylene chloride

Analysis

- Analyze the extract with GC/MS

Phenolic Analyte	CAS NUMBER
phenol	108-95-2
pentachlorophenol	87-86-5
4-nitrophenol	93951-79-2
4-chloro-3-methylphenol	59-50-7
2-nitrophenol	88-75-5
2-methylphenol (o-cresol)	95-48-7
2-methyl-4,6-dinitrophenol	534-52-1
2-chlorophenol-3,4,5,6-d4 (surrogate 1)	
2-chlorophenol	95-57-8
2,4-dinitrophenol	51-28-5
2,4-dimethylphenol-3,5,6-d3 (surrogate 2)	
2,4-dimethylphenol	105-67-9
2,4-dichlorophenol	120-83-2
2,4,6--trichlorophenol	88-06-2
2,4,6-tribromophenol (surrogate 3)	



UCT Product ENVIRO-CLEAN® ECDVB156

Results show that the UCT Product ENVIRO-CLEAN® ECDVB156

styrene divinyl benzene cartridge yields excellent recoveries of phenolic compounds

J. W. Munch, April 2000, National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

DCN-903020-123

ENVIRO-CLEAN®
EPA METHOD 535 GRAPHITIZED CARBON (90 m^2) CARTRIDGE

Part #: EC535156

February 3, 2009

The UCT EC535156 cartridge has been designed to provide a new level of performance in solid-phase extraction for the analysis of acetamide and acetanilide compounds. With its high capture efficiency, fast flow and excellent dry times, laboratory throughput can be significantly improved

Measurement of Chloroacetanilide and Other Acetamide Herbicide Degradates in Drinking Water by Solid-Phase Extraction and Liquid Chromatography/ Tandem Mass Spectrometry (LC/MS/MS)

Method Summary

A 250 mL water sample is drawn through and captured on a **UCT cartridge EC535156** containing 0.5 grams of nonporous graphitized carbon. Acetanilide and acetamide compounds are eluted from the cartridge using a small quantity of methanol containing 10 mM ammonium acetate. The methanol extract is concentrated to dryness by blow down with N_2 in a water bath at 65^{O}C then reconstituted with 1 mL of water containing 5 mM ammonium acetate. A 100 µL portion of the aqueous reconstitution is injected into an HPLC fitted with a C18 reverse phase analytical column. Detection occurs by tandem mass spectrometry and is compared to internal standards. A surrogate analyte of known concentration is measured with the same internal standard calibration procedure.

Interferences

Humic and/ or fulvic acid material if present in the water source is co-extracted with this method. High concentrations of these compounds can cause enhancement or suppression of the in the electrospray ionization source or low recoveries on the carbon SPE. Total organic carbon (TOC) is a good indicator of these interferences if present in the water sample.

Condition Cartridge

- Rinse the **UCT EC535156** cartridge with 20 mL of 10 mM ammonium acetate/methanol solution
- Rinse cartridge with 30 mL of reagent water. Do not let water drop below level of cartridge packing
- Add about 3 mL of reagent water to the top of the cartridge

Do not let the cartridge go dry during any step otherwise the conditioning process should be started over

Sample Addition

- Add sample water to the cartridge and adjust vacuum so the flow is about 10-15 mL/minute
- Rinse cartridge with 5 mL of reagent water
- Draw air or N_2 through the cartridge at high vacuum (10-15 in/Hg) for 3 minutes

Extract Elution

All glassware must be meticulously washed to avoid contamination

- Insert a clean collection tube into the extraction manifold
- Use 15 mL of 10 mM ammonium acetate/methanol and adjust vacuum to draw through at 5 mL/minute. Solvent will exit the cartridge in a drop wise fashion at this vacuum setting

Eluate Drying

- Concentrate the extract to dryness under a gentle stream of N_2 in a heated water bath at 60^{O}-70^{O}C to remove all of the ammonium acetate/methanol
- Reconstitute the dried eluate by adding 1 mL of 5 mM ammonium acetate/methanol solution

Extract Analysis

- Establish operating conditions for the liquid chromatograph and mass spectrometer according to Tables 1-4 in Section 17. See Table A below for RT and precursor ions
- If the analyte peak area exceed the range of the initial calibration curve, the extract may be diluted with 5 mM ammonium acetate/reagent water and adjusting internal standards to compensate for this dilution

*For complete details on Method 535 Version 1.1 the analyst is referred to: J. A. Shoemaker and M. V. Bassett, April 2005, National

Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

Table A

Triple Quadrupole MS/MS Method Conditions

Analyte	Retention Time	Precursor Ion	Product Energy	Collision Energy
Propachlor OA	7.33	206	134	8
Flufenacet OA	8.67	224	152	10
Propachlor ESA	10.01	256	80	25
Flufenacet ESA	10.81	274	80	25
Dimethenamid OA	13.25	270	198	10
Dimethenamid ESA	14.87&15.11	320	80	25
Alachlor OA	15.86	264	160	10
Acetochlor OA	16.34	264	146	10
Alachlor ESA	18.46	314	80	25
Metolachlor OA	18.60	278	206	8
Acetochlor ESA	19.12	314	80	30
Metolachlor ESA	20.95	328	80	25
Dimethachlor ESA (sur)	12.18	300	80	25
Butachlor ESA (IS)	36.95	356	80	25

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UCT ENVIRO-CLEAN® Cartridges
EPA Method 548.1 Endothall

Part Number: EC548006

September 28, 2009

The UCT EC548006 cartridge has been designed to provide a new level of performance for endothall (CAS 145-73-3) analysis using ion-exchange solid-phase extraction. Endothall can be easily captured without difficult derivatization techniques for faster analysis. With the cartridge's high capture efficiency, fast flow and excellent dry times, laboratory throughput can be significantly improved.

Product Benefits

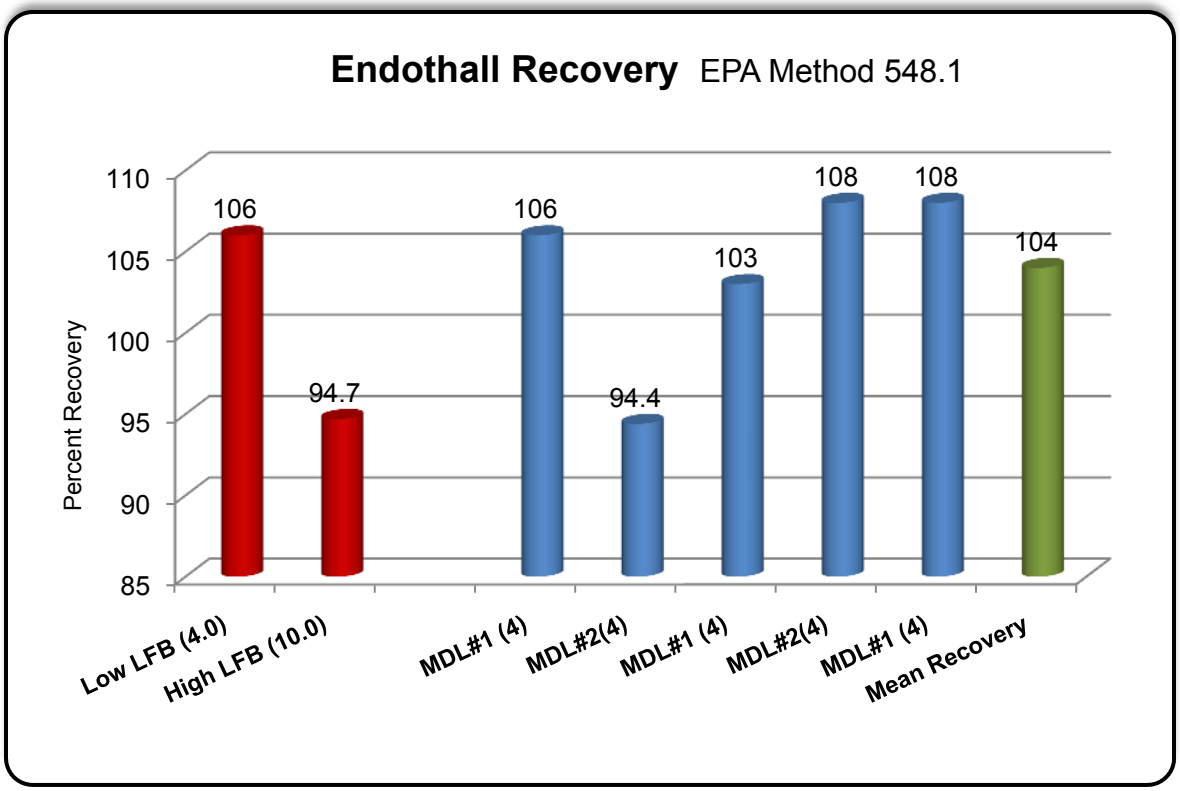
- Pre-packed SPE columns using anion capture resin
- No sample channeling due to improper cartridge packing
- Fast flow rates for rapid analyte capture
- Polypropylene frits eliminate potential contamination yielding clean extracts
- Works well at all levels of analyte loading
- Consistent results for excellent reproducibility
- Cartridges sealed at both ends to maintain product integrity
- Packaged in polypropylene bags to maintain product cleanliness
- Cartridge provides an effective sample clean-up for potential organic matrix interferences

Product Features

- 6 mL cartridges manufactured from special proprietary polypropylene
- Each cartridge contains the EPA prescribed 3.5 ± 0.1 cm bed of slurry packed sorbent
- Anion resin fully compliant with Method 548.1
- Can be used on manual single or multi-station manifold systems
- Can be used with automated extraction systems

Pre-packed UCT column is sealed at both ends to assure product integrity

The UCT column is fully compliant with EPA Method 548.1



UCT Cartridge EC548006 Shows Excellent Recovery with Laboratory Fortified Blanks (LFB) and Replicate Samples

Determination of Endothall in Drinking Water by Ion-Exchange Extraction, Acidic Methanol Methylation and Gas Chromatography/ Mass Spectrometry

Method 548.1 Revision 1.0

Method Summary*

The endothall molecule contains two dissociable carboxylic acid functional groups. Endothall is captured using liquid-solid cartridges containing a primary tertiary amine anion exchange resin. A 100 mL water sample is passed through the cartridge and the analyte is eluted with 8 mL of acidic methanol. After a small amount of methylene chloride is added as co-solvent to the extract and heated for 60 minutes at 50°C, the dimethyl ester of endothall is formed. The ester is partitioned into 8-10 mL of methylene chloride by the addition of salted water. The extract is reduced to 1 mL volume with nitrogen purge for a concentration factor of 100. The extract is analyzed by GC/MS or GC/FID using a megabore capillary column.

Interferences

- Glassware must be scrupulously clean by rinsing with the last solvent used in it
- Bake all glassware except volumetric flasks at 400°C for several hour prior to use
- The use of high purity solvents is essential
- Major potential interferences in this ion-exchange procedure are other naturally occurring ions such as calcium, magnesium and sulfate. Calcium and magnesium (>100 mg/L) can complex with the endothall anion and make it unavailable for capture as an anion
- Sulfate anions (>250 mg/L) can act as a counter ion displacing anionic endothall on the ion exchange column. Elevated levels of these ions may contribute to reduced recovery of the primary analyte

One or both of the following remedies may be used reduce these interferences:

- Sample dilution to reduce the concentration of these ions (10:1)
- Ethylenediamine tetraacetic acid (EDTA) addition to complex the cations (186 mg/100 mL sample)

It is critical that the following extraction steps be followed exactly in order for the cartridge to effectively function in sample clean up and extraction

1) Condition Cartridge

a) Remove the seal caps on each end of the cartridge and place it on the vacuum manifold system
b) Leave a 1 cm layer of reagent water over the resin bed between each liquid addition
c) Add 10 mL of methanol and draw through
d) Add 10 mL of reagent water and draw through
e) Add 10 mL of 10% H_2SO_4 in methanol and draw through
f) Add 10 mL of reagent water and draw through
g) Add 20 mL 0f 1 N NaOH and draw through
h) Add 20 mL of reagent water and draw through
i) Draw each reagent through the cartridge at a rate of 10 mL/minute

Do not allow the cartridge to become dry between steps otherwise repeat steps starting with c)

2) Sample Addition

a) Fill the reservoir with 60 mL of sample and adjust sample flow rate to 3 mL/minute. Add the remaining sample to keep the reservoir from going dry
b) After the sample has been drawn through the cartridge add 10 mL of methanol and draw through
c) Dry cartridge for 5 minutes under 10-20 in Hg vacuum
d) Place a culture tube inside the manifold to collect the eluant

3) Extract Elution

- Elute the cartridge with 8 mL of 10% H_2SO_4 in methanol followed by 6 mL of methylene chloride (CH_2Cl_2) over a 1 minute period

4) Sample Derivatization and Partition

- Place the cap on the culture tube and hold for 1 hour at 50°C
- Pour the contents of the culture tube into a 125 mL separatory funnel rinsing the tube with 2 x 0.5 mL aliquots of methylene chloride. Add the rinse to the separatory funnel
- Add 20 mL of 10% $NaSO_4$ in reagent water to the separatory funnel. Vigorously shake the separatory funnel three times venting the funnel each time
- Allow the phases to separate then drain the organic layer into a 15 mL graduated centrifuge tube
- Repeat the above extraction with two additional 2 mL aliquots of methylene chloride adding the methylene chloride to the organic phase in the centrifuge tube

5) Analysis

a) Analyze the extract by injecting 2 µL of the concentrated extract into a GC/MS
b) Identify endothall by comparison of its mass spectrum to a reference sample

Retention Times and Method Detection Limits

	Retention Time (min)			Method Detection Limits		
Compound	**Column A**	**Column B**	**Column C**	**GC/MS**	**2 g/L spike**	**FID**
Endothall	16.02	19.85	18.32	1.79	**µg/L**	0.7
D10-Acenapththene	14.69					

Column A: DB-5 fused silica capillary for GC/MS, 30 m x 0.25 mm, 0.25 micron film
MS inlet temperature = 200°C
Injector temperature = 200°C
Temperature Program: Hold five minutes at 80°C, increase to 260°C at 10°/min, hold 10 minutes

Column B: FID primary column, RTX Volatiles, 30 m x 0.53 mm I.D., 2 micron film
Detector temperature = 280°C
Injector Temperature = 200°C
Carrier gas velocity = 50 cm/sec
Temperature program: Same as Column A.

Column C: FID confirmation column, DB-5, 30 m x 0.32 mm ID, 0.25 micron film.
Carrier Gas velocity = 27 cm/sec
Same injector, detector and temperature program as Column A.

*For complete details on Method 548.1 "Determination of Endothall in Drinking Water by Ion-Exchange Extraction, Acid Methanol Methylation and Gas Chromatography/Mass Spectrometry", the analyst is referred to: J. W. Hodgeson, August 1992, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

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UCT ENVIRO-CLEAN® CARTRIDGES
EPA METHOD 549.2

Part #: EEC08156

February 3, 2009

The UCT cartridge (EEC08156) for Diquat / Paraquat Analysis has been designed to provide a new level of performance in solid-phase extraction. With its high capture efficiency, fast flow and excellent dry times, laboratory throughput can be significantly increased.

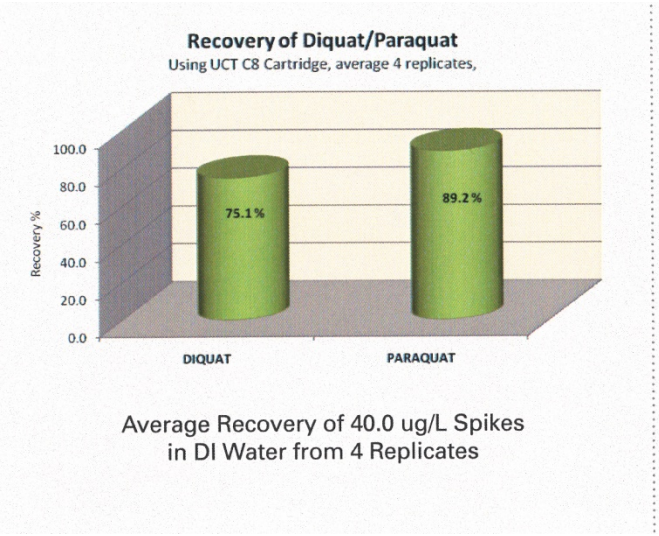
Product Benefits

- Fast flow rates for rapid analyte capture
- SPE using bonded endcapped C8
- Excellent pH stability (1-14)
- Teflon frits eliminate potential contamination
- Works well at all levels of analyte loading
- No lot to lot variability (EPA has noted significant variability with some brands)*
- Excellent reproducibility (MDL diquat 1.6 µg/L, paraquat 1.7 µg/L)
- Specially packaged to maintain product cleanliness

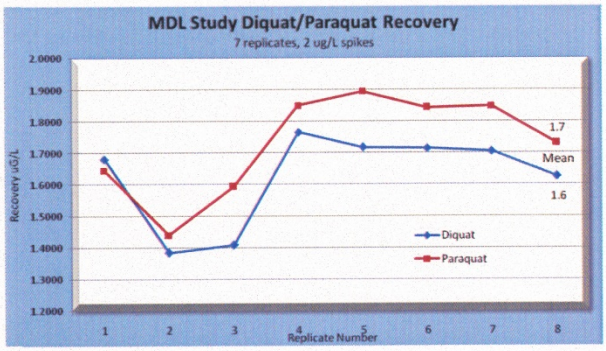
Product Features

- 6 mL polypropylene cartridge packed with 500 mg of endcapped C8
- Can be used on manual single or multi-station manifolds system
- Can be used with automated extraction systems
- Not all bonded sorbents are capable of achieving acceptable recoveries using method 549.1

The C8 used in UCT cartridges has been tested for both diquat and paraquat recoveries.



Average Recovery of 40.0 ug/L Spikes in DI Water from 4 Replicates



Results of an MDL Study Using 7 Replicates
2.0 ug/L spikes
- Mean Recovery = Diquat 1.6255 µg/L and Paraquat 1.7310 µg/L
- Standard Dev = Diquat 0.1587 µg/L and Paraquat 0.1725 µg/L

EPA Method 549.2
Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and High Performance Liquid Chromatography with Ultraviolet Detection

Method Summary

This method determines diquat (1,1'-ethylene-2,2'-bipyridilium dibromide salt) and paraquat (1,1'-dimethyl-4,4'-bipyridilium dichloride salt) in drinking water and drinking water sources using HPLC with photodiode array UV detection. The analytes are extracted from 250 mL of water adjusted to pH 7.0 to 9.0 using 6 mL C8 solid phase extraction cartridges with ion-pairing. Using an acidic aqueous solvent, analytes are eluted from the cartridge and detected at 308nm and 257nm, respectively.

This summary highlights major steps in the 549.2 method. Complete details about the preparation and composition of reagent solutions can be found in method and should be referenced by anyone needing complete details. It is available as a part of Supplement 11 from National Technical Information Service (NTIS), Springfield, VA 22161; publication PB 92 207703. (800) 553-6847 or at www.epa.gov/safewater/methods/methods.html

- Mean Recovery = Diquat 1.6255 µg/L and Paraquat 1.7310 µg/L
- Standard Dev = Diquat 0.1587 µg/L and Paraquat 0.1725 µg/L

Spike level for both compounds 2.0 µg/L; n=7

Preparation

- Since diquat and paraquat are ionic, all glassware should be silanized prior to use to deactivate the glass surface. The use of plastic lab ware is preferable.[1]
- Adjust a 250 mL of sample to pH 7.0 to 9.0 with 10 % aqueous sodium hydroxide or 10 % aqueous hydrochloric acid solution
- Assemble a C8 bonded silica extraction cartridges in an all-glass or plastic filtration apparatus.

Condition the Cartridge

- Add 3 mL of methanol to the cartridge soaking for about one minute. Apply vacuum to pull most of the methanol through the cartridge leaving a 3 to 5 mm layer of methanol on top of the cartridge
- Add 3 mL reagent water to the cartridge. Using vacuum, draw most of the water through the cartridge leaving 3 to 5 mm of water on the surface of the cartridge.
- Apply 5 mL of conditioning **Solution A**[2] to the cartridge. Using vacuum draw a small amount through then allow the cartridge to soak for one minute. After one minute, draw most of the remaining solution through the cartridge leaving 3-5 mm layer on top.
- Using two 10 mL aliquots of reagent grade water, rinse Solution A from the cartridge. Allow 3-5 mm of water to remain on the cartridge surface.
- Repeat the above procedure a second time using conditioning **Solution B.**[3] Return solution in cartridge.
- Cartridges may be prepared in advance and stored up to 48 hour prior to use if capped and stored at 40^{O}C. Do not let the cartridge dry out.

Sample Extraction

- Add the water sample to the reservoir and start the vacuum. Draw the sample through the cartridge. Drain as much of the water from the sample bottle as possible.
- Rinse the cartridge with 5 mL methanol.
- Remove the filtration assembly and insert a silanized 5 mL volumetric flask for collection of the eluate.

Cartridge Elution

- Add 4 mL of the cartridge eluting solution[4] to the cartridge and allow to soak for one minute. Draw any remaining solution through the cartridge, leaving 3 to 5 mm on the cartridge. The cartridge eluting solution contains acid and diethylamine which disrupts the ion-pair interactions releasing the analytes.
- Add another 4 mL of cartridge eluting solution to the remaining solution on the cartridge, and draw it completely through.
- Using cartridge **ion-pair solution,**[5] bring the eluate to a known volume. The extract is now ready for HPLC analysis.

Notes and Working Solutions

1. Since diquat and paraquat tend to adsorb onto glass surfaces, all glassware that comes into contact with samples, sample extracts, or standard solutions should be deactivated by silanization. The use of PVC lab ware circumvents this step.

2. Solution A contains cetyl trimethyl ammonium bromide to deactivate residual silanol groups on the C8

3. Solution B contains 1-hexanesulfonic acid which adsorbs to C8 forming a cation exchange sorbent.

4. The aqueous elution solution contains orthophosphoric acid and diethylamine in DI water

5. Ion-pair concentrate contains hexanesulfonic acid

* EPA Method 549.2 Revision 1.0 Issued June, 1997 as a part of "Methods for the Determination of Organic Compounds in Drinking Water," Hodgeson, J.W., Bashe, W.J., (Technology Applications, Inc.) and Eichelberger, J., Environmental Monitoring Systems Laboratory, Office of Research and Development, US Environmental Protection Agency, Cincinnati, Ohio 45268

DCN-903020-116

Bisphenol A Analysis in Water by GC/MS Using an ENVIRO-CLEAN® 200 mg C18 Extraction Cartridge

Part #: EEC1812Z

February 5, 2009

1. **Prepare Sample**
   a) Using 100 mL of sample water, adjust the pH to 7 or less using 100mM acetic acid
   b) Add internal standard to water sample

Note: Bisphenol A has a pKa value of approximately 9.5

2. **Condition ENVIRO-CLEAN® Extraction Cartridge**
   a) Place a cartridge(s) on a multistation vacuum manifold or automated extraction system
   b) Condition the cartridge by adding 3 mL of methanol
   c) Partially draw the methanol through until the surface of the liquid reaches the top of the cartridge frit
   d) Wait 1 minute then add 3 mL of DI water to the cartridge.
   e) Add 1 mL of 100 mM acetic acid
   f) Draw liquid through until it touches the top of the frit
   g) Cartridge is now ready for sample extraction

Note: Do not allow the sorbent to completely dry out after the addition of methanol, otherwise repeat procedure.

3. **Apply Sample**
   a) Add sample to cartridge at a rate of approximately 5 mL/minute by adjusting vacuum

4. **Wash Cartridge**
   a) Wash by drawing through 5 mL of deionized water
   b) Dry sorbent (5 minutes at > 10 inches Hg)

5. **Elute**
   a) Insert a collection vial in the vacuum manifold
   b) Rinse sample bottle with 3 mL of methanol
   c) Add the methanol to the cartridge
   d) Elute at 5 mL per minute
   e) Add 3 mL of methanol to the cartridge
   f) Elute at 5 mL per minute

6. **Evaporate**
   1. Evaporate methanol eluate using gentle N_2 (< 40°C) to dryness
   2. Add 50 µL of ethyl acetate to dissolve
   3. Add 50 µL of reagent MTBSTFA* or BSTFA** to derivatize. Vortex
   4. Heat mixture for 20-30 minutes @ 70°C
   5. Cool. Sample is now ready for GC injection

*MTBSTFA-- N-(t-butyldimethylsilyl)-N-methyltrifluoroacetamide
**BSTFA-- *N,O* -Bis(trimethylsilyl)trifluoroacetamide

7. **Instrument Conditions**

- **Column:** Rtx-5MS, 30m, 0.25 mm ID, 0.50 µm df (5% diphenyl/95% dimethyl polysiloxane)
- **Injector Temperature:** 250°C
- **Detector Temperature:** 250° C
- **Oven Program:** Initial 70°C, ramp @ 20°C/minute to 320°C, hold 3.0 min
- **Purge Flow:** Initially Off. On at 0.75 minutes
- **Split Flow:** 30 mL/minute
- **Inject:** 2 µL

8. **Quantitate**

MS in EI (+) mode:

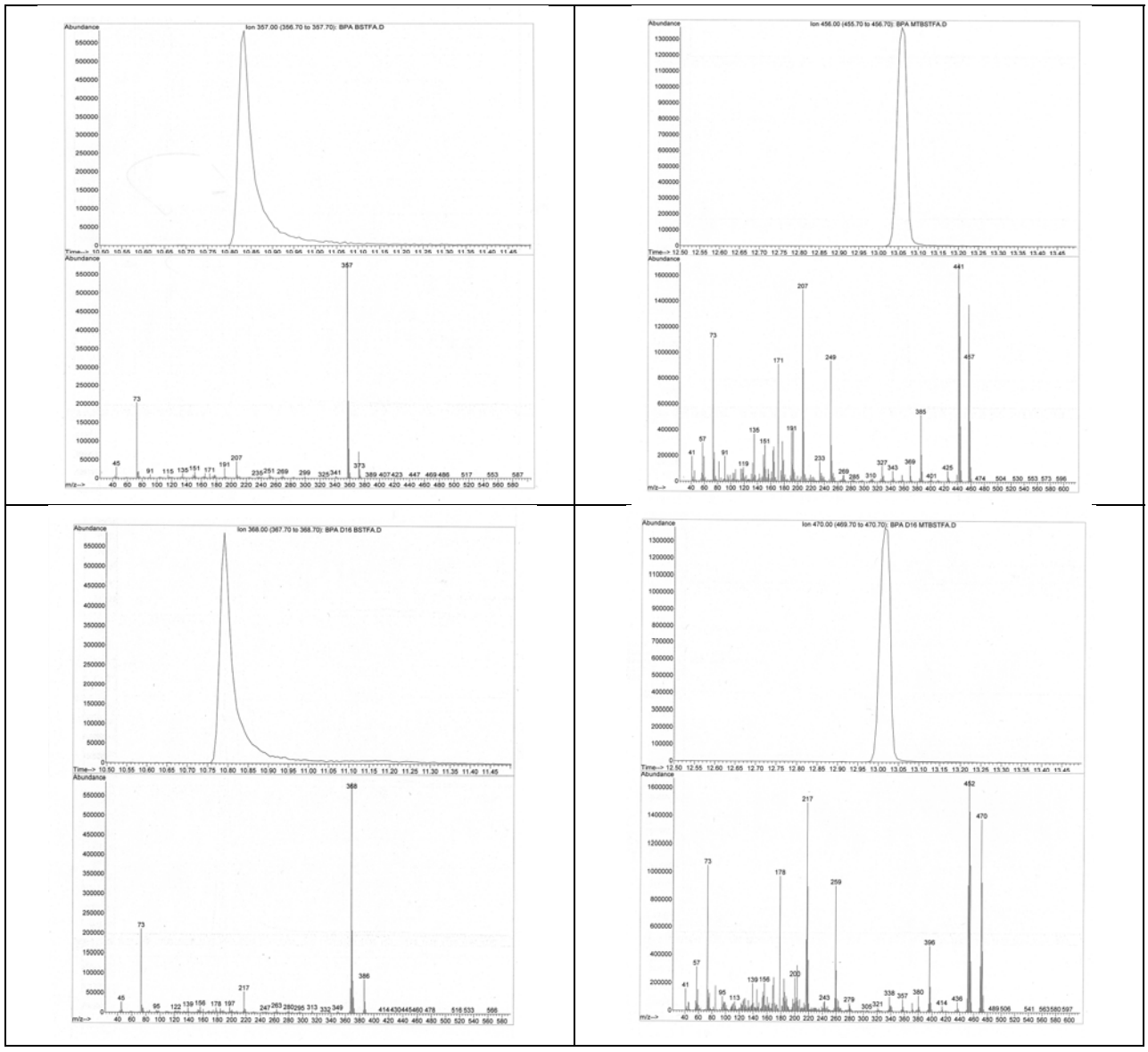
BSTFA

	Primary ion	Secondary ion	Tertiary ion
BPA	357	373	207
BPA-D16	368	386	217

MTBSTFA

BPA	441	457	207
BPA-D16	452	470	217

Chromatogram Showing Retention Time for Bisphenol A in Water



Spike Concentration: 0.15 µg/L
Calc Concentration: 0.157 µg/L
Recovery: 105%

DCN-905020-132

Bisphenol A Analysis in Water by LC/MS/MS Using an ENVIRO-CLEAN®
200 mg C18 Extraction Cartridge

Part #: EEC1812Z

February 5, 2009

1. **Prepare Sample**
   a) Using 100 mL of sample water, adjust the pH to 7 or less using 100mM acetic acid
   b) Add internal standard to water sample

   Note: Bisphenol A has a pKa value of approximately 9.5

2. **Condition ENVIRO-CLEAN® Extraction Cartridge**
   a) Place a cartridge(s) on a multistation vacuum manifold or automated extraction system
   b) Condition the cartridge by adding 3 mL of methanol
   c) Partially draw the methanol through until the surface of the liquid reaches the top of the cartridge frit
   d) Wait 1 minute then add 3 mL of DI water to the cartridge.
   e) Add 1 mL of 100 mM acetic acid
   f) Draw liquid through until it touches the top of the frit
   g) Cartridge is now ready for sample extraction

   Note: Do not allow the sorbent to completely dry out after the addition of methanol, otherwise repeat procedure.

3. **Apply Sample**
   a) Add sample to cartridge at a rate of approximately 5 mL/minute by adjusting vacuum

4. **Wash Cartridge**
   a) Wash by drawing through 5 mL of deionized water
   b) Dry sorbent (5 minutes at > 10 inches Hg)

5. **Elute**
   a) Insert a collection vial in the vacuum manifold
   b) Rinse sample bottle with 3 mL of methanol
   c) Add the methanol to the cartridge
   d) Elute at 5 mL per minute
   e) Add 3 mL of methanol to the cartridge
   f) Elute at 5 mL per minute

6. **Evaporate**
   a) Evaporate methanol eluate using gentle N_2 (< 40°C) to less than 500 µL
   b) Bring sample volume to 500 µL
   c) Sample is ready for injection

7. **Quantitate**
   a. LC-MS/MS MRM transition **(**negative ion mode)

Instrumental & Conditions:

Column: 100 x 2.1 (3 µm) Selectra® Phenyl, UCT, LLC
Instrument: Applied Biosytems Triplequad LC/MS/MS (other systems may be used)

- Detector: API3200 QTrap
- bisphenol A Precursor ion mass 226.9, product ion mass 109.1
- *bisphenol A standard deuterated A-D16. Precursor ion mass 241.1, Product ion mass 223.1

Inject: 5-10 µL

Mobile Phase: acetonitrile/0.1% formic acid

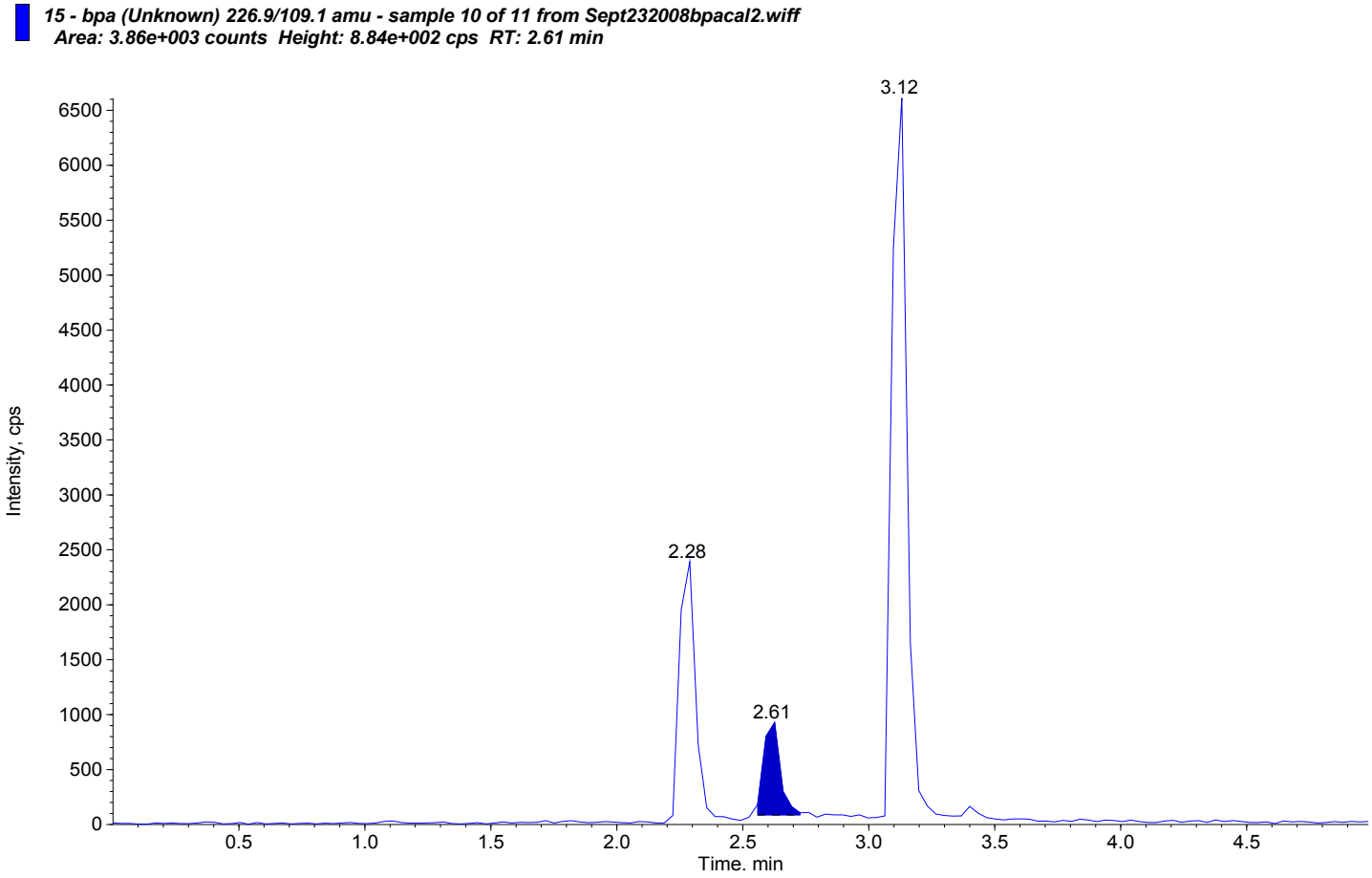
Flowrate: 0.5 mL/ minute

Flow Program

Time in minutes	**% Acetonitrile**	**% 0.1% Formic Acid**
0	30	70
3.0	90	10
3.5	30	70
5.0	30	70

Chromatogram Showing Retention Time for Bisphenol A in Water

Spike concentration: 15 µg/L
Calc Recovery: 15.70 µg/L
Recovery: 105%



DCN-905020-131

Extraction of Diesel Range Organics

Part Number: ENVIRO-CLEAN® Universal DRO cartridge (# ECUNIPAH)*
Anhydrous Sodium Sulfate (# ECSS50K)
August 25, 2009

1. Condition Cartridge

a) Add 10 mL of methylene chloride ($MeCl_2$) to the cartridge. Let the methylene chloride soak on the cartridge for about 1-2 minutes
b) Draw the methylene chloride through the cartridge to waste
c) Add 10 mL of acetone to the cartridge. Let the acetone soak for about 2 minutes
d) Draw the acetone to waste
e) Air dry the cartridge with full vacuum for a few seconds
f) Add 10 mL of methanol to the cartridge.
g) Draw some of the methanol through the cartridge leaving a layer just covering the frit
h) Allow the methanol to soak for about 2 minutes

Note: Do not allow the cartridge to dry after addition of methanol

i) Add 20 mL of deionized water to the cartridge. Draw most of the water through the cartridge to waste

2. Sample Addition

a) Add 5 mL of 1:1 HCl to the sample
b) Add 5 mL of methanol (optional) and any surrogates to the sample. Mix.
c) Add the sample to the cartridge using vacuum
d) Draw the sample through the cartridge in 15 – 20 minutes
e) Allow the cartridge to dry under vacuum for 10 minutes**

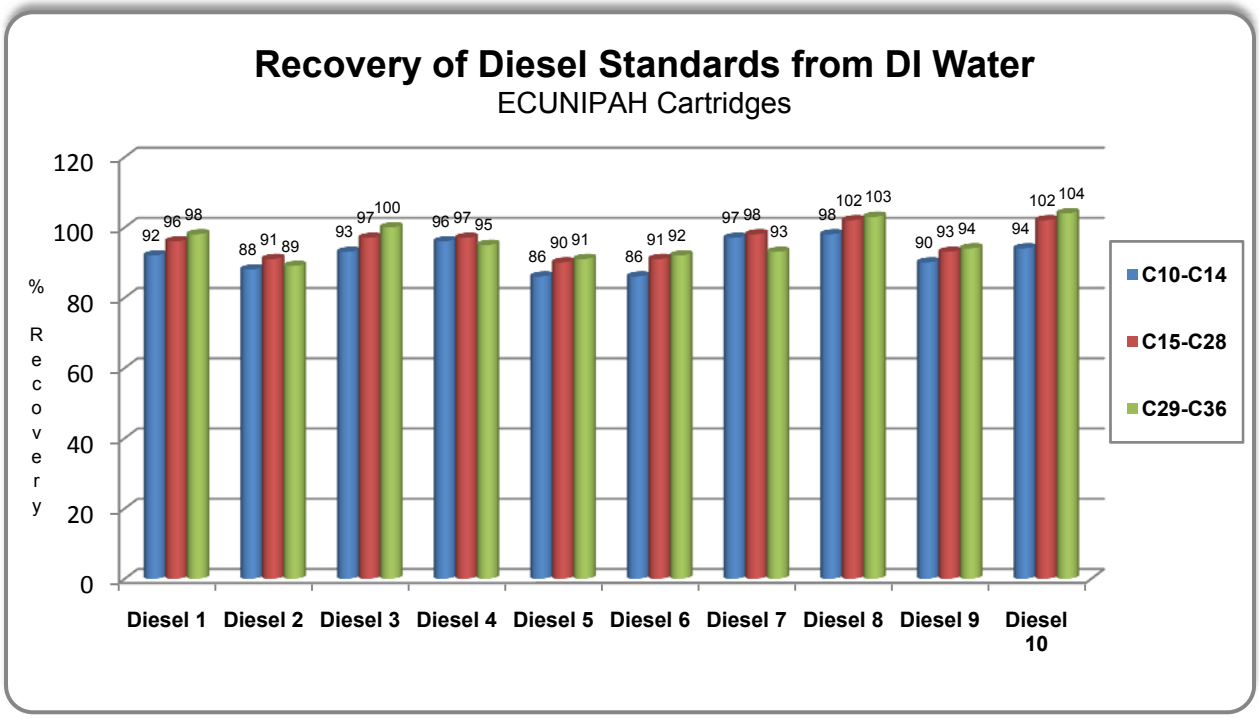
3. Extract Elution

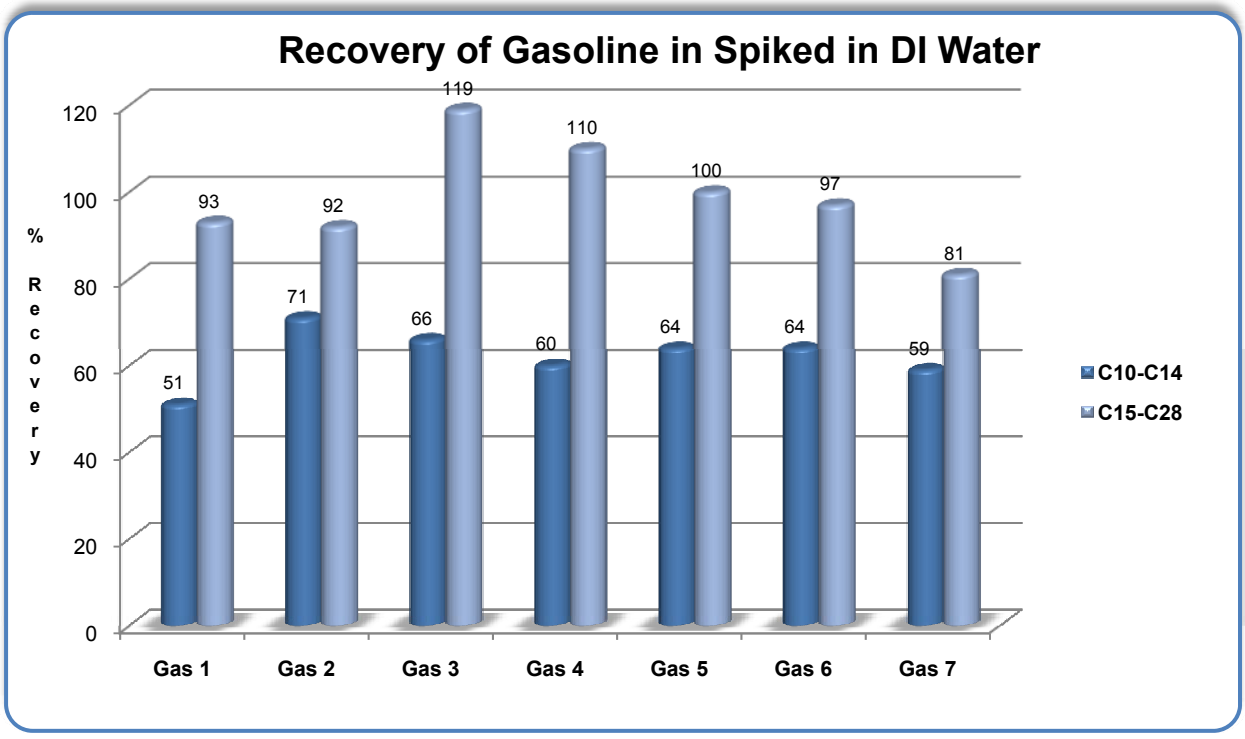
a) Place a collection tube or vial under the cartridge
b) Add 5 mL of acetone to the sample bottle to remove any residue
c) Add 5 mL of acetone to the cartridge. Allow the solvent to soak for 1 minute and draw the acetone into the collection vial
d) Repeat this procedure 3 times using 10 mL aliquots of methylene chloride
e) Dry the extract by passing through anhydrous sodium sulfate
f) Carefully rinse the collection vial with methylene chloride and add the solvent to the sodium sulfate

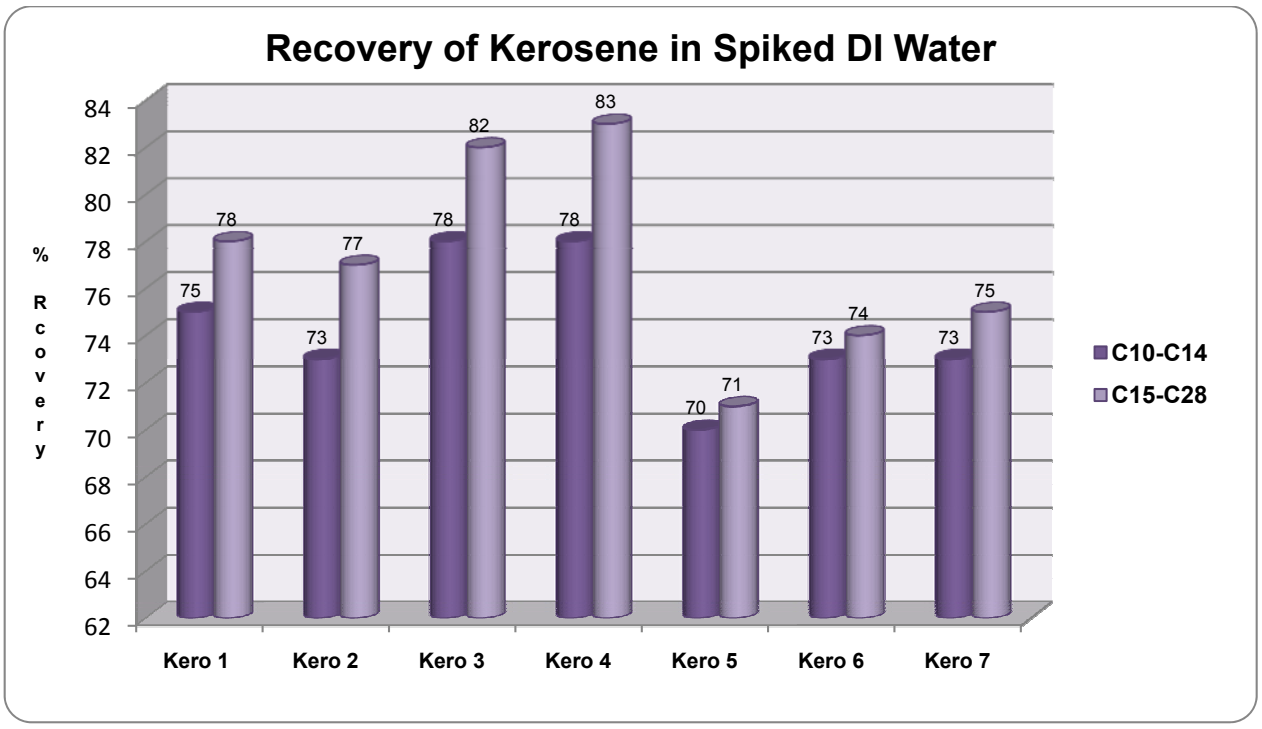
4. Concentration and Analysis

a) Carefully concentrate the extract to a final volume. A micro-KD followed by micro-Snyder column concentration is recommended

Note: Most extraction errors are caused by poor concentration technique. Do not concentrate below 0.5 ml. or low recoveries will result







*The ENVIRO-CLEAN® Universal DRO cartridge may be used on standard vacuum manifolds (# VMFF016GL), standard disk manifolds (#ECUCTVAC6) (with adapter part # ECUCTADP). The cartridge is specifically designed to fit the Horizon SPE DEX 4790® made by Horizon Technologies, Inc.

**Faster drying results are obtained by removing the cartridge during drying and shaking or tapping the excess moisture from the bottom of the cartridge. Drying times are approximate. Do not over dry as low recoveries may result.

DCN-905290-121

POLYCYCLIC AROMATIC HYDROCARBONS FROM A WATER MATRIX

Part #: ECUNIPAH

February 3, 2009

Reagents:
Methanol
Acetonitrile**
Methylene Chloride**
Anhydrous Sodium Sulfate (# ECSS25K)
ENVIRO-CLEAN® Universal PAH cartridge (# ECUNIPAH)

Condition Cartridge
Add 10 mL of methylene chloride to the cartridge and let it soak for 1 minute. Draw through to waste. Add 10 mL of acetone to the cartridge and let it soak for 1 minute. Draw through to waste. Add 10 mL of methanol to the cartridge and let it soak for 1 minute. Pull most of the methanol to waste but do not allow the sorbent to dry. Add 10 mL of deionized water to the cartridge and let it soak for 1 minute. Draw most of the water to waste but do not allow the sorbent to dry.

Sample Addition
Add 5 mL of methanol (optional) and any surrogates to the sample. Mix. Add the sample to the cartridge under vacuum. Ideally, the sample should pass through the cartridge in approximately 15 – 20 minutes. Allow the cartridge to dry under vacuum for 10 minutes.***

Extract Elution
Place a collection tube or vial under the cartridge. Add 5 mL of acetone to the sample bottle to remove any sample residue. Add the acetone to the cartridge. Allow to soak for 1 minute and draw the solvent into the collection device. Repeat this procedure three more times using 10 mL aliquots of methylene chloride. Dry the extract by passing it through anhydrous sodium sulfate. Thoroughly rinse the collection device with methylene chloride and add this solvent to the sodium sulfate.

Concentration and Analysis
Carefully concentrate the extract to a final volume. **Note:** Most extraction errors are caused by poor concentration technique. Do not concentrate below 0.5 mL or low recoveries will result.

• The ENVIRO-CLEAN® Universal PAH cartridge can be used on standard vacuum manifolds (# VMFF016GL), standard disk manifolds (#ECUCTVAC6) (with adapter part # ECUCTADP). The cartridge is specifically designed to fit the Horizon SPE DEX 4790® made by Horizon Technologies, Inc.

** Acetonitrile may be substituted for acetone and methylene chloride.
***Faster drying results can be obtained by removing the cartridge during drying and shaking or tapping the excess moisture from the bottom of the cartridge. Drying times are approximate. Do not over dry. Low recoveries could result.

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EXTRACTION OF ORGANOCHLORINE PESTICIDES AND PCBs

Part #: ECUNIC18

February 3, 2009

Reagents:

Methylene Chloride
Acetone
Methanol
Anhydrous Sodium Sulfate (# ECSS25K)
ENVIRO-CLEAN® Universal C18 cartridge (# ECUNIC18)

Condition Cartridge

Rinse cartridge with 10 mL of methylene chloride and let the methylene chloride soak on the cartridge for approximately 1.5 min. Pull the methylene chloride through the cartridge to waste. Add 10 mL of acetone to the cartridge and let the acetone soak for approximately 1.5 minutes. Pull the acetone to waste and air dry the cartridge with full vacuum for a few seconds. Add approximately 10 ml of methanol to the cartridge and allow the methanol to soak for approximately 1.5 min. From this point until sample addition the cartridge must not go dry. Pull some of the methanol through the cartridge leaving a layer just covering the frit. Add approximately 20 mL of deionized water to the cartridge and pull most of the water through the cartridge to waste but do not allow the sorbent to dry.

Sample Addition

Adjust the pH of the sample to 2 using sulfuric acid. Do not use pH paper to test the pH of deionized water as poor recoveries may result. Add 5 mL of methanol (optional) and any surrogates to the sample. Mix. Add the sample to the cartridge under vacuum. Ideally, the sample should pass through the cartridge in approximately 15 – 20 minutes. Allow the cartridge to dry under full vacuum for 10 minutes.**

Extract Elution

Place a collection tube or vial under the cartridge. Add 5 mL of acetone to the sample bottle to remove any residue. Add the acetone to the cartridge. Allow the solvent to soak for 1 minute and pull the acetone into the collection device. Repeat this procedure 3 more times using a 10 mL aliquot of methylene chloride. Dry the extract by passing it through anhydrous sodium sulfate. Carefully rinse the collection device with methylene chloride and add the solvent to the sodium sulfate.

Concentration and Analysis

Carefully concentrate the extract. Solvent exchange if necessary. Note: Most extraction errors are caused by poor concentration technique.

• The ENVIRO-CLEAN® Universal C18 cartridge can be used on standard vacuum manifolds (# VMF016GL), standard disk manifolds (# ECUCTVAC6) (with adapter part # ECUCTADP). The cartridge is specifically designed to fit the Horizon SPE DEX 4790® made by Horizon Technologies, Inc.

**Faster drying results can be obtained by removing the cartridge during drying and shaking or tapping the excess moisture from the bottom of the cartridge. Drying times are approximate. Do not over dry. Low recoveries could result.

Results:

spikes in DI water

Compound	Amount Spiked	% Recovery Ave n=4	STDEV
4,4'-DDD	0.2056	**97.0**	2.08
4,4'-DDE	0.2006	**93.0**	2.38
4,4'-DDT	0.2014	**101.0**	0.96
Aldrin	0.0999	**79.0**	5.10
Dieldrin	0.2046	**101.0**	0.82
Endosulfan I	0.1028	**100.0**	0.82
Endosulfan II	0.2054	**103.0**	0.96
Endosulfan sulfate	0.2124	**100.0**	2.06
Endrin	0.2016	**90.0**	7.33
Endrin aldehyde	0.2012	**106.0**	9.54
Endrin ketone	0.2068	**108.0**	1.73
Heptachlor	0.1032	**82.0**	5.29
Heptachlor epoxide	0.1034	**100.0**	0.96
Methoxychlor	1.0016	**96.0**	1.71
alpha-BHC	0.1032	**105.0**	0.96
alpha-Chlordane	0.099	**98.0**	0.50
beta-BHC	0.1043	**108.0**	1.50
delta-BHC	0.104	**104.0**	1.26
gamma-BHC (Lindane)	0.1038	**105.0**	0.82
gamma-Chlordane	0.0969	101.0	1.71

The recovery data shows excellent analyte recovery using UCT cartridge product ECUNIC18

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EPA Method 1664, Revision A
N-Hexane Extractable Material (HEM) (Oil & Grease) by
Solid-Phase Extraction & Gravimetry

ENVIRO-CLEAN® Universal Oil and Grease Cartridges
Part Number ECUNIOGXF
August 3, 2009

Method Summary

A sample of water, pH adjusted to <2, is extracted using a UCT Universal Oil and Grease Cartridge containing the solid-phase sorbent C18.

1) **Sample Collection**
   a) All samples must be acidified prior to analysis
   b) Adjust the pH of a 1-liter sample to 2 or lower by adding 5 mL of 6N HCl or 2.5 mL of concentrated H_2SO_4. A smaller volume of sample may be used provided all quality control requirements are met
   c) If the acid was added to the sample in the field, it is not necessary to repeat this step unless the pH has increased during storage
   d) Refrigerate sample to 0-6ºC if analysis is delayed more than 4 hours from collection

2) **Assemble**
   a) Assemble the UCT cartridge adapter(s) on a vacuum manifold
   b) Add the **ENVIRO-CLEAN® Universal Oil and Grease Cartridge** (Note 1)
   c) Place a cartridge in an automated extraction system if used in instead of a vacuum manifold
   d) Connect the vacuum manifold to a suitable trap and attach to a vacuum system capable of attaining a minimum of 25” Hg (635 mm) of vacuum

3) **Sample Spikes**
   a) Prepare a matrix spike by adding 40 mg/L of a PAR (precision and recovery) standard (Note 2)
   b) A concentration of 20 mg/L may also be used as long as the spike concentration is higher than the background concentration

4) **Condition the Cartridge**
   a) Wash the cartridge, including the sides, with 10 mL of hexane
   b) Allow to soak for 1 minute
   c) Draw the hexane through the cartridge to waste using vacuum. Discard the hexane
   d) Draw full vacuum through the cartridge for 2 minutes to remove the hexane
   e) Add 10 mL of methanol to the cartridge and slowly draw the methanol through the cartridge leaving a layer on the surface of the cartridge.
      Do not let the sorbent dry out.
   f) Soak for one minute then add 30 mL of DI water to the cartridge
   g) Draw the water through the cartridge to waste
   h) Do not allow the sorbent to completely dry to a powder before adding the sample, otherwise repeat this step starting with the addition of methanol

5) Sample Addition

a) Add the water sample directly to the cartridge and draw through under vacuum. This may take several minutes for complete flow through the cartridge depending upon the level of solids in the sample. Do not allow the flow rate to exceed 500 mL per minute. Note 3
b) Remove the cartridge before drying and tap the excess water from the bottom of the cartridge
c) Allow the cartridge to dry under full vacuum for 10 minutes to remove any residual water

6) Elution

a) Remove any water remaining in the bottom support of the cartridge with a paper towel
b) Place an extract collection vial in the manifold. Note 4
c) Rinse the sample bottle with 10 mL of hexane. Add the hexane rinse to the cartridge, washing the sides of the cartridge and the bottle holder if used. Soak for 1 minute. **Do not use any other solvents except hexane.**
d) Slowly draw the hexane through the cartridge and into the collection vial.
e) Do not allow the vacuum to blow air over the extract
f) Immediately, repeat this procedure 2 additional times using 10 mL of hexane for each rinse
g) While collecting the hexane do not allow it to splash out of the collection vial
h) Add another 10 mL of hexane to the cartridge, rinsing the bottle holder. Soak for 2 minutes. Draw the hexane through the cartridge and collect

7) Dry the Extract

a) Remove the collection vial from the manifold and cover with a screw cap
b) Shake the extract to form a water/hexane emulsion and immediately pour the extract through a sodium sulfate funnel or column containing approximately 40 g of anhydrous sodium sulfate. Do not use filter paper to hold the sodium sulfate. Glass wool is acceptable.
c) Collect the extract in a clean, tared vessel
d) Rinse the collection vial with 5 mL of hexane and add it to the sodium sulfate. Note 5

8) Gravimetric Analysis

a) Carefully evaporate the hexane using an analytical evaporator or similar device at $40^{O}C$ until a constant weight is obtained
b) Alternate concentration techniques and containers such as glass beakers or aluminum pans may be used
c) Do not dry on a hot plate or in an oven
d) Allow to cool in a desiccator before weighing
e) Record this weight as the mass per unit volume of oil and grease and report HEM as mg/L. Note 6

Notes

1) This cartridge is designed to fit the Horizon SPE-DEX® 4790 automated extraction system. The cartridge will also fit a standard 3 (# ECUCTVAC3) or 6 station (#ECUCTVAC6) disk manifold with our optional adapter (#ECUCTADP). The cartridge also fits a standard vacuum manifold (#VMF016GL).

2) PAR standards may be prepared by dissolving 20 mg of stearic acid and 20 mg of hexadecane in 5 mL of acetone.

3) To achieve good flow if very high solids are present, add glass wool to the cartridge prior to extraction to prevent clogging. The glass wool must be thoroughly rinsed with hexane as part of the cartridge during the elution step.

4) Procedures for drying the hexane extract:
   - Place a plug of glass wool in the bottom of a small funnel, then add 3-5 grams of sodium sulfate to the top.
   - Record the weight of a clean vial or weigh pan and place under the funnel
   - Pour the hexane from the eluate onto the bed of sodium sulfate
   - Rinse the sides of the vial and the sodium sulfate bed with clean hexane and collect in the weighed vial
   - Evaporate the hexane and report the results as mg/L HEM

5) Gloves are recommended when handling the vial as skin oils may affect the actual sample weight.

6) It is important that the extract not be over dried or dried at high temperatures as low recoveries may result by evaporation of volatile oils.

For Method 1664 updates see: http://www.epa.gov/waterscience/methods/

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ENVIRO-CLEAN® DVB Cartridge

Part Number: **ECDVB156**

EPA Method 8330B

September 16, 2009

The UCT ECDVB156 styrene divinylbenzene cartridge is designed to provide a new level of performance in solid-phase extraction for the analysis of **nitroaromatics, nitramines and nitrate ester compounds--explosive and explosive residue compounds**. With its high capture efficiency, fast flow and excellent dry times, laboratory throughput can be significantly improved using this solid-phase product

Product Benefits

- SPE using styrene divinylbenzene polymeric gel
- SPE has been shown to provide equal or superior results as compared to liquid-liquid extraction LLE*
- No hydrolysis of the solid-phase DVB
- Fast flow rates for rapid analyte capture
- Teflon frits in the cartridge eliminate particle fines yielding clean extracts
- Works well at all levels of analyte loading
- No Lot to Lot variability
- Excellent analytical reproducibility
- Packaged in metalized, sealed pouches to maintain product purity

Product Features

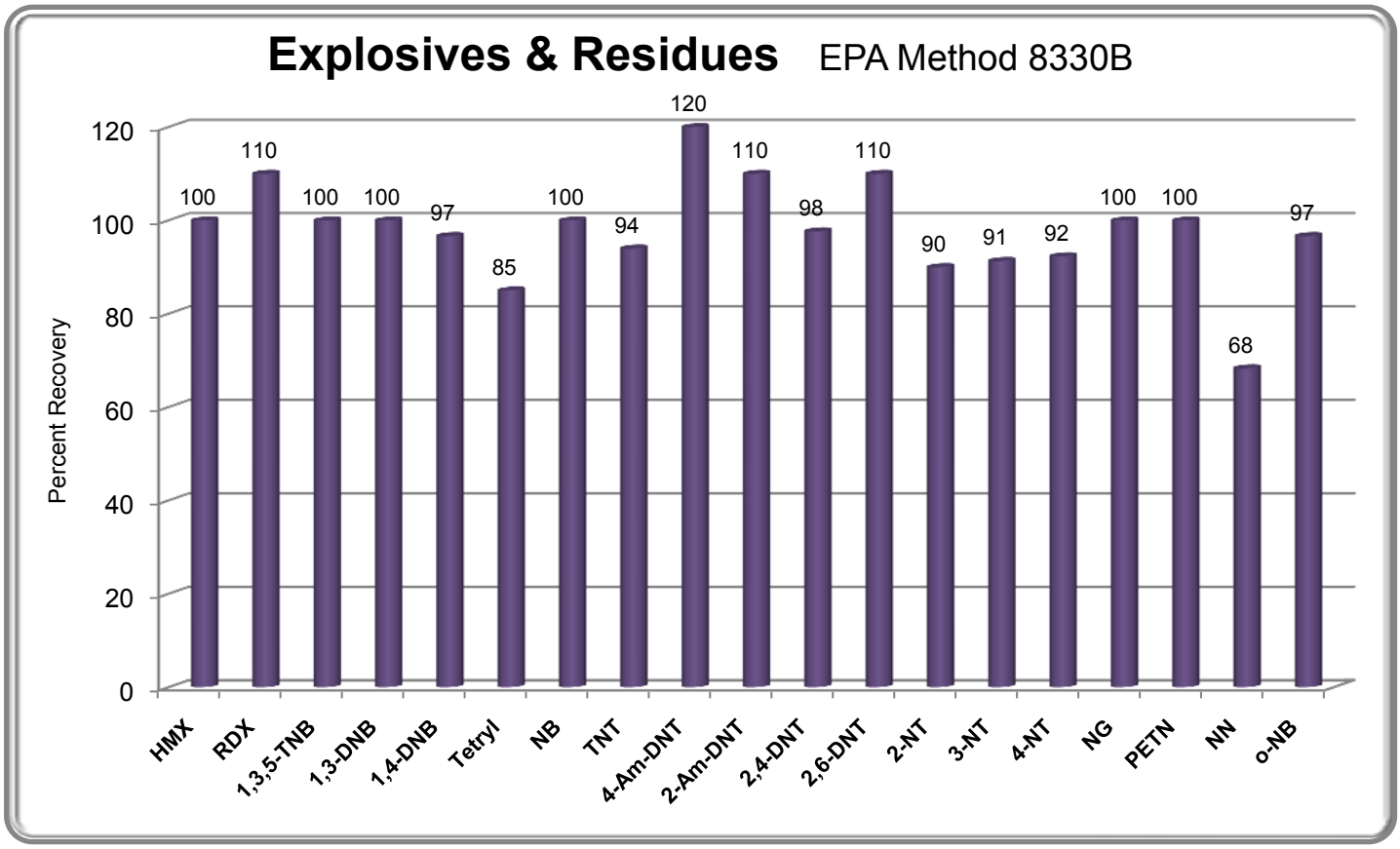
- 6 mL cartridge body manufactured from special polypropylene
- Each cartridge contains 500 mg of styrene divinylbenzene DVB sorbent
- Can be used on manual single or multi-station manifold systems
- Cartridges may be used with automated extraction systems

Nitroaromatics, Nitramines and Nitrate Ester Analytes and CAS Number

The following RCRA compounds have been determined by this method in water, soil & sediment matrices

Analyte	Abbreviation	CAS	% Recovery n=3
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0	100
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4	110
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4	100
1,3-Dinitrobenzene	1,3-DNB	99-65-0	100
1,4-Dinitrobenzene	1,4-DNB	10025-4	97
Methy-2,4,6-trinitrophenylnitramine	Tetryl	47945-8	85
Nitrobenzene	NB	98-95-3	100
2,4,6-Trinitrotoluene	TNT	118-96-7	94
4-Amino-2,6-dinitrotoluene	4-Am-DNT	19406-51-0	120
2-Amino-2,6-dinitrotoluene	2-Am-DNT	35572-78-2	110
2,4-Dinitrotoluene	2,4-DNT	121-14-2	98
2,6-Dinitrotoluene	2,6-DNT	606-20-2	110
2-Nitrotoluene	2-NT	88-72-2	90
3-Nitrotoluene	3-NT	99-08-1	91
4-Nitrotoluene	4-NT	99-99-0	92
Nitroglycerin	NG	55-63-0	100
Pentaerythritol tetranitrate	PETN	78-11-5	100
3,5-Dinitroaniline	3,5-DNA	618-87-1	68
1-Nitronaphthalene	NN	86-57-7	97
o-Dinitrobenzene	o-NB	528-29-0	100

Method 8330B Recovery Values



The UCT ECDVB156 styrene divinyl benzene cartridge for EPA Method 8330B shows excellent recovery of explosive and explosive residue analytes

Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC)

Method 8330B

Method Summary

This method is used for the trace analysis (ppb) of explosive and propellant residues in water, soil and sediment matrices using high performance liquid chromatography (HPLC) and a dual wavelength UV or diode array detector. It is an updated method from 8330 promulgated September 1996. In this method aqueous samples are preconcentrated using the UCT styrene divinylbenzene SPE sorbent cartridge ECDVB156 as described in Method 3535 then eluted with acetonitrile or other appropriate solvent. The final extract is diluted with water as appropriate to bring the concentration into an analytical range suitable for HPLC analysis.

Interferences

- Solvents, reagents, glassware and other sample processing hardware may show interferences in sample analysis. All material must be demonstrated to be free from interferences under conditions of the analysis by analyzing method blanks

- 2,4-DNT and 2,6-DNT elute at similar retention times on C18 columns using method separation conditions. If it is not apparent that both isomers are present or are not detected an isomeric mixture should be reported

- Tetryl is thermally labile (decomposed with heat at temperature above room temperature) and decomposes in methanol/water solutions. All aqueous samples expected to contain tetryl should be diluted with acetonitrile and acidified with sodium bisulfate to pH <3 prior to filtration

- Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak when using C18 columns

Note

All samples should be stored at 2° to 4° C prior to extraction and should be extracted within 14 days of collection

I Sample Preparation--Solid Matrices, (e.g. soil) (from Method 3535)

A soil sample is placed in a glass vial and dried with sodium sulfate. Acetonitrile (ACN) is added to the sample then mixed by vortex to suspend the soil in the solvent. The sample vial is then placed in a chilled ultrasonic bath for sonication. After 18 hours of sonication the sample is centrifuged for 15 to 20 minutes and the ACN solvent portion is removed from the vial. The volume of the removed aliquot is doubled by adding an equal volume of calcium chloride solution. The extract is then filtered through 1 μm Teflon filters.

1) Procedure (Ultrasonic Preparation)

a) Weigh 2.0 ± .04 g of solid sample into a 25 mL glass vial
b) Add 2 g of sodium sulfate and mix
c) Add 0.1-mL explosives soil surrogate to all samples, blanks, and spikes
d) Using a syringe or pipette, add 0.5 mL explosives spike to the LCS, LCSD if applicable, matrix spike, and matrix spike duplicate samples
e) Using a graduated cylinder, add 10 mL of ACN and vortex swirl the sample for approximately 1 minute to suspend the soil in the ACN
    a. Place the sample in a cooled ultrasonic bath (<10°C). Make sure the water level in the sonicator is at least as deep as the level of solvent in the vial. Sonicate for 18 hours
    b. After sonication, centrifuge the sample for 15 to 20 minutes to separate the solids from the solvent

2) Final Preparation

a) Using a pipette, add 5 mL of a (5.0 gram/L) calcium chloride ($CaCl_2$) solution to a 10 mL volumetric flask
b) Using a disposable pipette, bring to volume using the solvent layer of the centrifuged sample
c) Mix thoroughly then allow the mixture to stand 15 minutes
d) Filter the sample through 1 μm Teflon filters using a disposable syringe
e) Discard the first 3 mL and retain the remainder in an appropriately labeled 12 mL vial
f) Store in a refrigerator until analysis

II Sample Preparation--Aqueous matrices, (e.g. water) (from Method 3535)

A measured volume of the aqueous sample is adjusted to a specified pH then extracted using the UCT styrene divinylbenzene SPE sorbent cartridge **ECDVB156**. Two challenges are noted for aqueous sample preparation. First, any particulate matter in the original sample must be included in the sample aliquot that is extracted. Second, the sample container must be rinsed with solvent as the majority of organic analytes are hydrophobic and may adhere to the sample container surfaces.

Note

- Do not concentrate explosives residue to dryness as they may DETONATE
- For explosives and nitramines or nitroaromatics the extraction pH should be as received in the sample
- Using a graduated cylinder, measure 1 liter of sample water. A smaller sample size may be used when analytical sensitivity is not a concern
- Add 5.0 mL of methanol and surrogate standards to all samples and blanks
- Add matrix spikes standards to representative sample replicates

Note: Adjustment of sample pH may result in precipitation or flocculation reactions and potentially remove analytes from the aqueous portion. The analyst should note the formation of such precipitates or floc and transfer any such material with rinses to the SPE extraction cartridge. Do not let the cartridge dry out after cartridge conditioning with acetonitrile (ACN)

A. Glass Apparatus Washing:

Analyte	1st solvent wash	2nd solvent wash	3rd solvent wash
Explosives	5 mL acetone	15 mL isopropanol	15 mL methanol
Nitramines, Nitroaromatics	5 mL ACN	15 mL ACN	

Draw solvents through the cartridge under low vacuum

B. Cartridge Conditioning:

Analyte	Condition Step 1	Step 2	Step 3	Step 4
Explosives	20 mL ACN, 3 min*	20 mL ACN	50 mL DI water	50 mL DI water
Nitramines, Nitroaromatics	15 mL ACN, 3 min*	30 mL DI water		

*Soak time

Draw solvents through the cartridge under low vacuum

1) **Initial Preparation**
   a) Assemble a DVB extraction cartridge **UCT ECDVB156** in an all-glass manifold.
   b) Use of a manifold for multiple extractions or automated extraction equipment is acceptable

2) **Cartridge Conditioning**
   a) Follow the 4 steps in Table **Cartridge Conditioning for** solvent quantities

Do not let the cartridge dry out once the cartridge is conditioned as this may affect analyte recovery

b) **Sample Extraction**
   a) Add the contents of the sample bottle to the cartridge
   b) Adjust vacuum to about 10-15 mm Hg to obtain a uniform flow rate of approximately 10 ml per minute. This will require about 1 hour for sample extraction
   c) After all the sample is drawn through, draw air through the cartridge for 15 minutes to dry it
   d) Do not dry for longer than 20 minutes as lower recovery may result

c) **Cartridge Elution**
   a) Insert a collection tube in the base of the vacuum manifold

Explosives

   b) Add 4 mL of ACN and soak for 3 minutes
   c) Draw through using gravity flow or very low vacuum into a collection tube
   d) Store extract in freezer until analysis

Nitramines and Nitroaromatics

   b) Add 5 mL ACN, soak for 3 minutes
   c) Draw through using a gravity flow or very low vacuum into a collection tube
   d) Store extract in freezer until analysis

d) **Extract Concentration**
   a) Concentrate the extract to 0.7 mL under a gentle stream of nitrogen in a warm bath at 40^{O} C
   b) Transfer the extract to a 1 mL volumetric flask
   c) Add internal standard for a extract concentration of 5 µg/mL
   d) Extract is now ready for analysis by HPLC

RP-HPLC Columns for the Analysis of Explosive Residues	
Primary Columns	C-18 reversed-phase HPLC column, 25-cm x 4.6-mm, 5 µm C8 reversed-phase HPLC column, 15-cm x 3.9-mm, 4 µm
Secondary Columns	CN reversed-phase HPLC column, 25-cm x 4.6-mm, 5 µm Luna Phenyl-Hexyl reversed-phase HPLC column, 25-cm x 3.0-mm, 5 µm

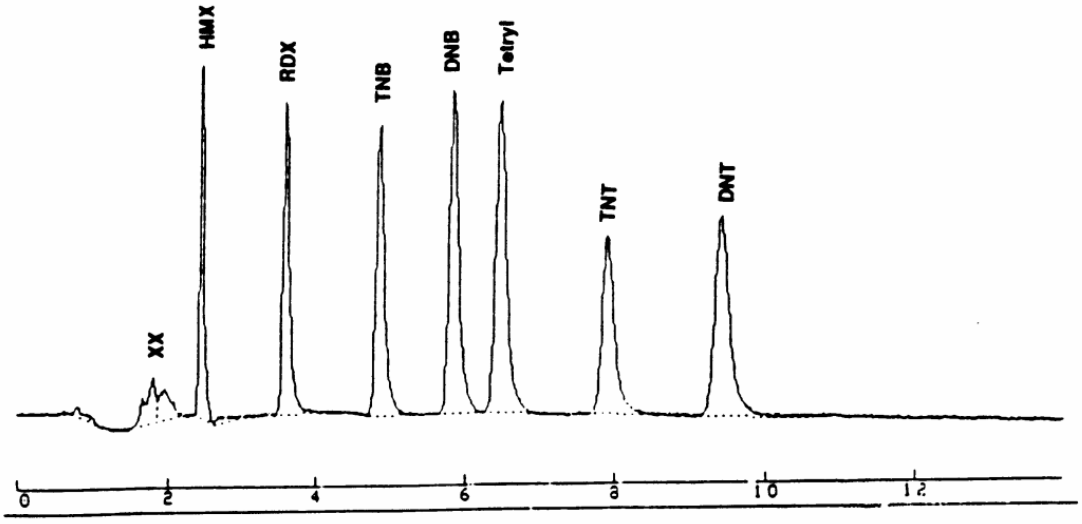
Injection volume: 100 µL

UV Detector: Dual 254nm & 210nm or Photodiode Array

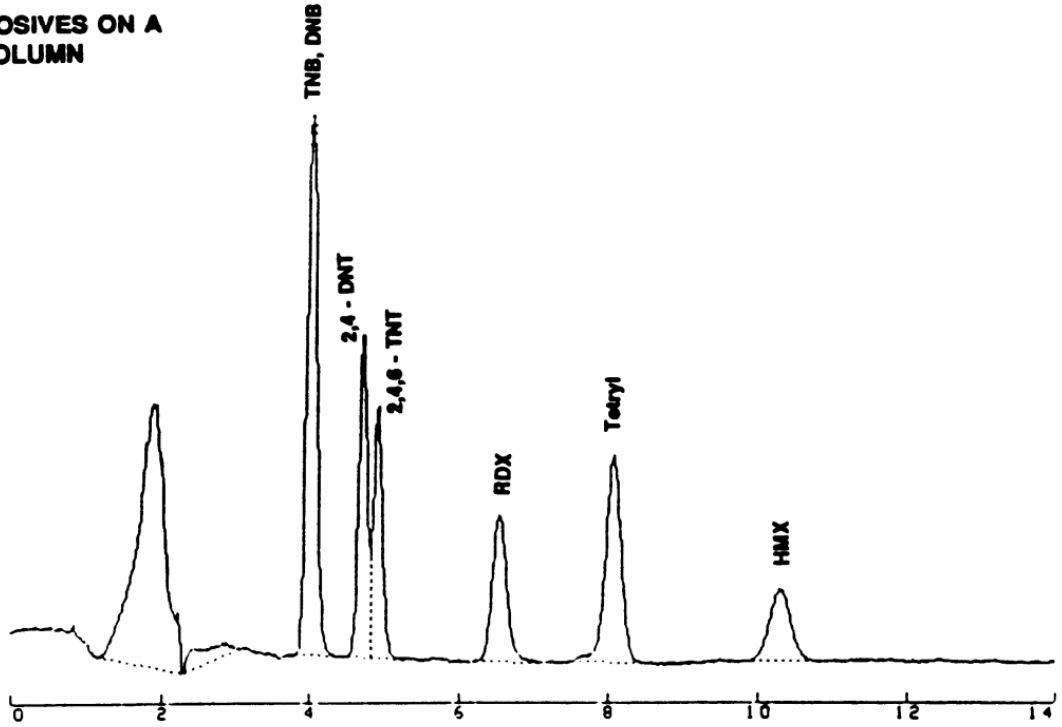
Mobile phase: For C18 & CN column, 50:50 methanol:water

Energetic Compounds Currently Not Target Analytes of Method 8330	
Compound	**Symbol**
picric acid (PA)/Ammonium picrate	AP
2,4-diamino-6-nitrotoluene	
2,6-diamino-4-nitrotoluene	
hexanitro-hexaazaisowurtzitane	CL-20
1,3,3-trinitroazetidine	TNAZ
hexahydro-1-nitroso-3,5-dinitro-1,3,5- triazine	MNX
hexahydro-1,3,5-trinitroso-1,3,5-triazine	TNX
nitrocellulose	NC
nitroguanidine	NQ
diphenylamine	DPA
n-nitroso-diphenylamine	NDPA
2-nitrodiphenylamine	
4-nitrodiphenylamine	
2,4-dinitrodiphenylamine	

EXPLOSIVES ON A C18 COLUMN



EXPLOSIVES ON A CN COLUMN



RETENTION TIMES AND CAPACITY FACTORS ON LC-18 AND LC-CN COLUMNS

|Analyte	LC-18 RT minutes	LC-CN RT minutes
HMX	2.44	8.35
RDX	3.78	6.15
1,3,5-TNB	5.11	4.05
1,3-DNB	6.16	4.18
3,5-DNA	6.90	NA
Tetryl	6.93	7.36
NB	7.23	3.81
NG	7.74	6.00
2,4,6-TNT	8.42	5.00
4-Am-DNT	8.88	5.10
2-Am-DNT	9.12	5.65
2,6-DNT	9.82	4.61
2,4-DNT	10.05	4.87
2-NT	12.26	4.37
4-NT	13.26	4.41
PETN	14.10	10.10
3-NT	14.23	4.45

*For complete details on Method 8330 “Nitroaromatics and Nitramines by High performance Liquid Chromatography” Revision 2 October 2006, the analyst is referred to: National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268 and Method 3550 Revision 0, December 1996

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Recovery of Various Herbicides in Water Using UCT ENVIRO-CLEAN® Universal C18 Solid-Phase Extraction Cartridges

Part #: ENVIRO-CLEAN® Universal C18 cartridge ECUNIC18

April 8, 2009

The GC analysis of select phenoxy acid based herbicides found in various surface waters may be easily determined by the use of this procedure. Herbicides DCPA (Dacthal), MCPP (Mecoprop), Dicamba, MCPA, Dichloroprop, 2,4-D and 2,4,5-TP (Silvex), 2,4,5-T, Dinoseb, 2,4-DB, common household weed killers are evaluated using this method.

1. **Vacuum Manifold Set-Up**
   a) Assemble a disk manifold vacuum extraction apparatus
   b) Install an 83 mL C18 ENVIRO-CLEAN® Universal cartridge

   Note: All glassware must be acid rinsed with dilute sulfuric acid

2. **Sample Preparation**
   a) Adjust the pH of a 500-1000 mL water sample to 2.0 ±0.5 with 6N HCl
   Use of a pH meter is highly recommended over pH paper

3. **Cartridge Conditioning**
   a) Add 5 mLs of methanol to the cartridge
   b) Draw a few drops through until methanol touches top of frit
   c) Hold for 1 minute
   d) Rinse cartridge with 15 mLs of water adjusted to pH 2 with 6N HCl

Note: Do not let the cartridge dry out at this point otherwise repeat conditioning step

4. **Sample Extraction**
   a) Adjusting the vacuum setting such that flow is about 25 mLs per minute
   b) Draw the water sample through the cartridge
   c) Draw air through the cartridge under full vacuum for 5 minutes to dry the cartridge
   d) Tap the cartridge to help remove excess water. **DO NOT OVERDRY**

5. **Elution**
   a) Insert a collection tube containing 2 grams of acidified sodium sulfate
   b) Add 10 mLs of methanol to the sample bottle and swirl
   c) Add to cartridge

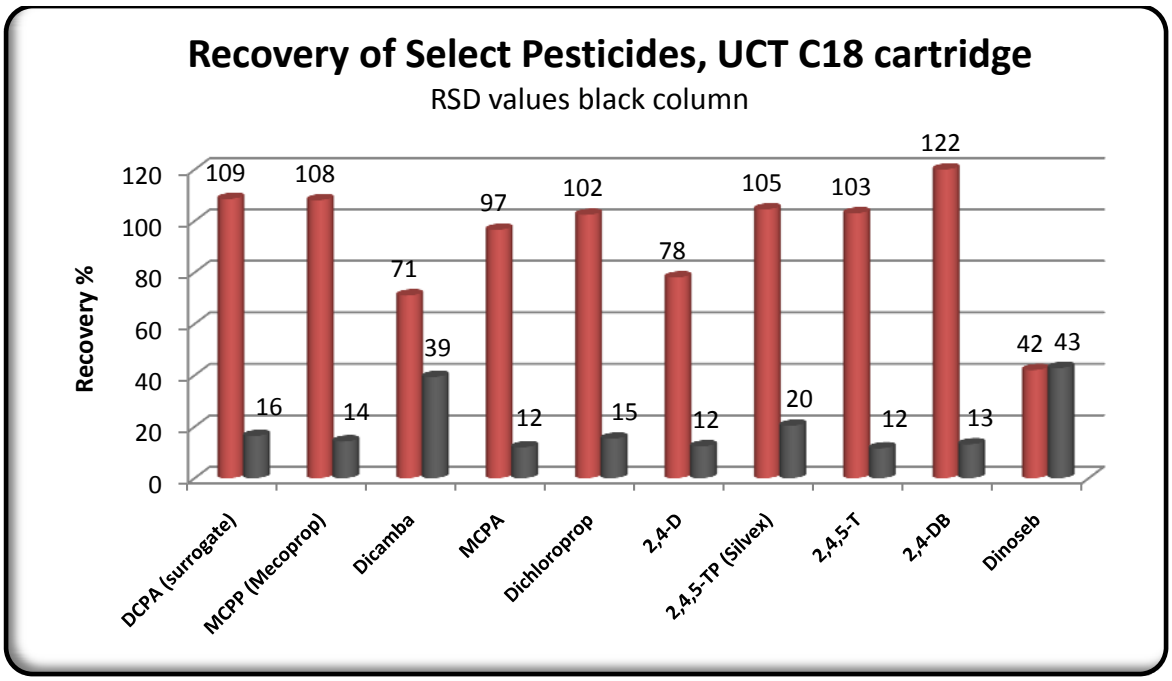
6. **Analyte Elution**
   a) Elute cartridge using 8 mL of methanol
   b) Repeat with a second aliquot of methanol
   c) Add 5 mLs of methylene chloride to the sample bottle and rinse
   d) Add to cartridge
   e) Elute cartridge using 5 mL of methylene chloride (CH_2Cl_2)
   f) Repeat with a second aliquot of methylene chloride and add to e)

7. **Concentration and Exchange**
   a) Derivitize 1 mL from step 6.f) with diazomethane
   b) Exchange into 1 mL hexane

Methylation is detailed in EPA Method 8151 or 515. Extract must be very dry otherwise methylation may be incomplete

Herbicide Data Table

Pesticide	CAS	UCT C18	% SD
DCPA (surrogate)	1861-32-1	109	16
MCPP (Mecoprop)	93-65-2	108	14
Dicamba	62610-39-3	71	39
MCPA	94-74-5	97	12
Dichloroprop	120-36-5	102	15
2,4-D (amine salt)	2008-39-1	78	12
2,4,5-TP (Silvex)	93-72-1	105	20
2,4,5-T	93-76-5	103	12
2,4-DB	94-82-6	122	13
Dinoseb	88-85-7	42	43



* **Procedure for Preparing Acidified Sodium Sulfate Anhydrous from EPA Method 8151A 5.10**,
Product Number: UCT brand sodium sulfate ECSS25K

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A Method for the Extraction of Imidachloprid from Pond Water

Part #: EUBCX1M6 1000 mg of Benzenesulfonic acid in 6 mL cartridge

April 8, 2009

1. **Prepare Sample**
   a) Adjust the sample pH to less than 7 using 0.1N HCl
   b) If solids are present, settle by centrifugation. Do not use filter paper

2. **Condition ENVIRO-CLEAN® Extraction Cartridge**
   a) Wash the BCX cartridge with 5 mL of methanol and 5 mL of deionized water.
   Do not allow the sorbent to dry out

3. **Add Sample**
   a) Add water sample to the BCX cartridge
   b) Adjust vacuum for a flow rate of 1-3 mL per minute. One drop every 3 seconds is ideal
   c) Wash cartridge using deionized water or other pH neutral solvent through the BCX cartridge at high vacuum
   d) Dry cartridge under full vacuum for 10 minutes

4. **Sample Elution**
   a) Add a collection tube to the vacuum manifold
   b) Elute the cartridge with 6 mL of 4% ammonium hydroxide in methanol at a rate of 1 mL per minute*

5. **Evaporate**
   a) Concentrate under gentle N_2

6. **Analyze by LC**

*Only use fresh ammonium hydroxide. The 4% ammonium hydroxide in methanol must be fresh daily.

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EPA Method 608 ATP*

Part #: ECUNIC18

August 25, 2009

The EPA has accepted the use of C18 bonded phases in packed cartridge format expanding the method from a disk only approach. This method is used in place of liquid-liquid extraction. The UCT ECUNIC18 Universal cartridge has been designed to provide a high level of performance for the solid-phase extraction (SPE) and analysis of certain **organochlorine pesticides and PCB's** in municipal and industrial discharges. With the cartridge's high capture efficiency, fast flow and rapid dry times, laboratory throughput can be significantly improved and sample preparation time reduced.

Product Benefits

- SPE cartridge containing bonded C18 phase
- Excellent pH stability under acidic conditions
- Fast flow rates for rapid analyte capture
- Works well at all levels of analyte loading
- Consistent results for excellent reproducibility
- PTFE frits eliminate potential contamination yielding clean extracts
- Packaged in metalized bags to maintain product cleanliness

Product Features

- Cartridges manufactured from clean proprietary polypropylene
- Each cartridge contains 1100 mg endcapped C18 bonded ultra-clean silica sorbent
- Can be used on manual single or multi-station vacuum manifold systems
- Can be used with a variety of automated extraction systems

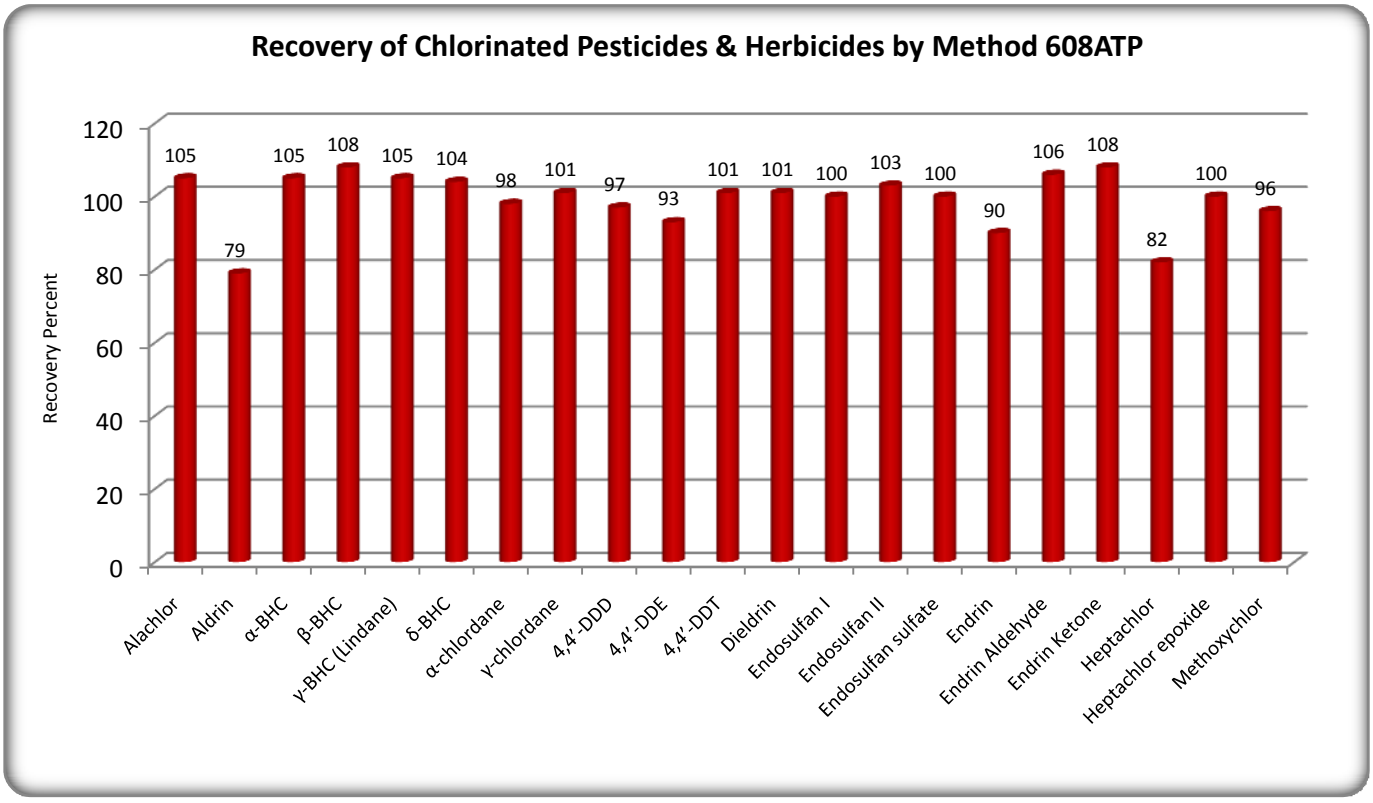
UCT Products Required:

1) Universal Cartridge ECUNIC18
2) Florisil PR® column cleanup (optional) **EUFLS1M6**
3) $NaSO_4$ Drying Tube UCT ECSS15M6

Recovery of Method 608ATP Analytes

Analyte	CAS	Amt Spiked µg/L	Average Recovery %	Stdev
Aldrin	309-00-2	0.0999	79	2.08
α-BHC	319-84-6	0.1032	105	0.96
β-BHC	319-85-7	0.1043	108	1.50
γ-BHC (Lindane)	58-89-9	0.1038	105	0.82
δ-BHC	319-86-8	0.1040	104	1.26
α-chlordane	5103-71-9	0.0969	98	0.50
γ-chlordane	5103-74-2	0.0969	101	1.71
4,4'-DDD	72-54-8	0.2056	97	2.08
4,4'-DDE	72-55-9	0.2006	93	2.38
4,4'-DDT	50-29-3	0.2014	101	0.96
Dieldrin	60-57-1	0.2046	101	0.82
Endosulfan I	959-98-8	0.1028	100	0.82
Endosulfan II	33213-65-9	0.2054	103	0.96
Endosulfan sulfate	1031-07-8	0.2124	100	2.06
Endrin	72-20-8	0.2016	90	7.33
Endrin Aldehyde	7421-93-4	0.2012	106	9.54
Endrin Ketone	53494-70-5	0.2068	108	1.73
Heptachlor	76-44-8	0.1032	82	5.29
Heptachlor epoxide	1024-57-3	0.1034	100	0.96
Methoxychlor	72-43-5	1.0016	96	1.71

UCT Cartridge ECUNIC18 Shows Excellent Recovery with Laboratory Fortified Blanks (LFB)



An Alternative Test Procedure for the Measurement of Organochlorine Pesticides and Polychlorinated Biphenyls in Waste Water

Method 608ATP

Federal Register Vol. 60, # 148, August 2, 1995

Method Summary*

A 1-liter sample of water is extracted by drawing through a **UCT C18 Universal cartridge ECUNIC18**. The analytes captured on the solid-phase are eluted from the cartridge using a small volume of acetone followed by methylene chloride ($MeCl_2$). The eluant is dried and exchanged into hexane for analysis by injection into a gas chromatograph with electron capture detection system (GC/ECD) fitted with a high-resolution fused silica capillary column.

Interferences

- Interferences may generally be attributed to contamination from solvents, glassware or other laboratory equipment leading to anomalous GC peaks. Glassware must be scrupulously cleaned and high purity solvents used.
- Interfering contamination may occur when a sample of low concentration is analyzed immediately after a sample of high concentration. A laboratory blank should be inserted between low and high concentration samples to minimize this potential problem
- Phthalate esters may pose a problem in pesticide analysis when using electron capture detection. This results from contact with common laboratory plastics such as PVC

Sample Collection

- Samples must be collected in glass containers following conventional practices **except** the bottle must not be prerinsed with sample before collection
- Samples must be refrigerated at 4°C from collection to analysis and extracted within 72 hours of collection
- If samples are to be held longer than 72 hours the pH must be adjusted to 5.0-9.0 with sodium hydroxide or sulfuric acid depending upon initial pH

Procedure

1) Condition Cartridge

a) Insert a cartridge into the glass vacuum manifold or automated extraction system
b) Wash the cartridge with 10 mL of methylene chloride ($MeCl_2$). Apply solvent for 1 minute then draw through to waste
c) Draw air under full vacuum to completely dry cartridge
d) Add 10 mL of methanol (MeOH) to the cartridge then slowly draw some of it through
e) Allow the cartridge to soak for 1 minute in methanol
f) Do not let the cartridge go dry after addition of methanol otherwise repeat the addition of methanol addition step
g) Rinse the cartridge with 10 mL of reagent water and draw most of it through leaving a thin layer on the top of the sorbent

2) Sample Addition

a) Adjust sample pH to < 2 using sulfuric acid
b) Adjust the vacuum and draw the sample water through the cartridge over a 20-30 minute time period
c) If sample water is high in suspended solids, allow particulates to settle then slowly decant the water in the bottle. Once most of the water passes through the cartridge add the solids portion
d) Dry the cartridge by drawing air through for about 5-10 minutes

3) Extract Elution

a) Insert an eluate collection tube into the vacuum manifold
b) Add 5 mL of acetone to the sample bottle then swirl
c) Add this to the cartridge
d) Soak for 1 minute and slowly collect eluate
e) Add 20 of methylene chloride to the sample bottle, cover and shake. Add this to the cartridge
f) Soak for 2 minutes and slowly collect eluate
g) Rinse the inside walls of the sample bottle using 10 mL of methylene chloride then transfer solvent to the cartridge using a disposable pipette rinsing the inside of the cartridge
h) Soak for 2 minutes then collect eluate

4) Sample Drying

a) Pour the combined elutes together through a drying tube (**UCT ECSS15M6**) which contains 5 grams anhydrous sodium sulfate. Alternatively, use 5 grams of sodium sulfate over a bed of glass wool in a funnel
b) Rinse the drying tube or sodium sulfate bed with 2 x 3 mL portions of 1 methylene chloride
c) Concentrate sample using a Kuderna-Danish (KD) concentrator while performing solvent exchange into hexane. Other drying techniques may be used
d) Concentrate sample under a gentle stream of N_2 while gently heating in a water bath
e) Rinse the inside walls of the concentrator tube two or three times with hexane during the evaporation
f) Adjust the final volume of the extract to 10 mLs

Florisil PR® Clean-up (if needed)

Clean-up procedures may not be needed for relatively clean samples. If required the following procedure is used and is designed to remove polar interferences from organochlorine pesticide and PCB extracts in hexane eluants prior to analysis

UCT EUFLSA1M6 – 1000 mg small particle Grade A Florisil® for slower gravity flow

UCT EUFLS1M6 – 1000 mg regular particle PR Grade Florisil® for more viscous samples

1) Procedure

a) Place a cartridge in a vacuum manifold

b) Prerinse the Florisil® column with 10 mL of 90:10 hexane/acetone using gravity flow (A low vacuum may be necessary to start flow)

c) Discard solvent

d) Add a collection tube under the column

e) Add a 2 mL aliquot of the sample extract (in hexane) to the column

f) Collect extract by gravity

g) Add 10 mL of 90:10 hexane/acetone to the column

h) Continue to collect by gravity or low vacuum

i) Gently evaporate the extract to a volume of 1 mL

j) Adjust eluate to a final volume of 2 mL with hexane

k) Sample is now ready for analysis

Sulfur Clean-up (if needed)

UCT ECCU01K – 1 kG copper granules

1) Procedure

a) Post Sample Extraction

a) Place 4 grams of copper bead in a glass vial
b) Add 2 mL of liquid sample extract to the vial

b) Sulfur Removal

a) Seal the glass vial and mix sample with copper beads for 2 minutes
b) Allow to stand for approximately 10 minutes
c) If sample contains high levels of sulfur, repeat process with 4grams of fresh copper beads

Note: For the analysis of PCB type analytes, copper may reside in the extract

c) Analysis, GC/MS or LC/MS

a) Transfer clean extract to autosample vial
b) Inject 1-2 µL for GC
c) Inject 5-10 µL for LC

d) Sample is now ready for 6081tp analysis

Sample Analysis by 608ATP

5) Analysis

a) Inject a 1-2 µL aliquot into a GC
b) Identify the analytes in the sample by comparison of the retention time to known reference chromatograms

*For complete details on Method 607ATP, the analyst is referred to: "An alternative test procedure for the measurement of organochlorine pesticides and polychlorinated biphenyls in waste water", Federal register/Vol.60, No.148, August 2, 1995, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

Florisil® is a registered trademark of U.S. Silica

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EXTRACTION OF METALS

Part #: EUTAX15Z

February 3, 2009

Metals: Tin, Nickel, Mercury, Copper, Chromium, Ruthenium

Matrix: Water, Blood, Biological Fluids, Organic Solvents & Tissue Homogenates

1) **Sample Pre-treatment**

The primary concern using ion exchangers is to adjust the pH of the compound of interest so that it is totally ionized. Adjust the sample to pH 7 with buffer or ammonium hydroxide.

Aqueous or Organic Solvent Samples:

Adjust sample to pH 7.0 with 100 mM dibasic sodium phosphate buffer and vortex.

Whole Blood, Serum or Plasma:

To 1 ml of sample add 4 ml of D.I. H20 and vortex. Let stand 5 minutes and centrifuge for 10 minutes at 2000 rpm and discard pellet. Adjust to pH 9.0 with 100 mM dibasic sodium phosphate buffer or ammonium hydroxide.

2) **Column Conditioning**

Add 3 ml of methanol followed by
Add 3 ml of water.
Add 3 ml of buffer pH 7.0

3) **Sample Application**

Apply the sample to the column at a rate of 1 ml per minute.
A faster rate of application may exceed the rate of ion-exchange.

4) **Analyte Purification**

Wash the column with 2 ml of pH 7.0 buffer used in column equilibration.

5) **Elution**

Elute with 3 ml of acidic methanol (2% HCl, pH 2.0).

Alternative elutions:

Elute with 3 ml of acidic methanol (Formic acid to pH 2.0).
Elute with 3 ml of 0.1 M Nitric Acid (pH 2.0).

DCN-903020-119

Ion Exchange Sorbents for Metals Extraction-Analysis & Sorbent Use Selection Guide

UCT ENVIRO-CLEAN® (Ion-Exchange Cartridges)*

January 5, 2010

The determination of trace metals in aqueous environmental samples or other matrices often require sample pretreatment and cleanup procedures prior to analysis by using specific ion-exchange sorbents. The sorbents are used to eliminate matrix interferences and achieve high concentrations of metal ions for good analytical accuracy. They are important when using such techniques as AA, IES and ICP-AES.

The use of ion-exchange sorbents for the preconcentration, separation and determination of metal ions for trace analysis is well established in the literature. Selection of an appropriate sorbent ensures both high efficiency in metal chelating while minimizing the mass of sorbent required for a particular analytical task. A high efficiency sorbent means that a smaller bed mass may be used thereby reducing the quantity of solvent required for elution yielding greater analytical sensitivity.

Recommendations in this application note include the following metal ions:

Zinc (II)	**Arsenic (V)**	**Tin (IV)**	**Selenous IV)**
Mercury (II)	Chromium (III)	Copper (II)	Platinum (0)

Other metal ions may be extracted by the use of these ion-exchange sorbents

Sorbent Selection for Metals Extraction

Solid-phase sorbents have differing capacity and selectivity for various metal ions due to the specific nature of the ion-exchange functional group, the metal species and the valence state of the metal of interest. Depending on the specific metal ion of interest, elution of the cartridge may be most efficient using both the acid followed by the base elution procedure. This can be determined by looking at the following Extraction Protocol Tables. For example, when eluting Hg(II) from PSA the highest recovery is obtained using acid elution (green box) followed by base elution (yellow box).

Sample Analysis

1) Sample Extraction

a) Assemble an all glass extraction apparatus

b) Place a UCT ENVIRO-CLEAN® cartridge on the apparatus

Note: Cartridge selection will depend on the volume of sample or the concentration of metal to be extracted

c) Condition 1mL cartridge by adding 3 mL of methanol. (Larger cartridges will require a larger volume of solvent and water wash volume in steps c) and d))

d) Add 3 mL of reagent water and allow to drip through the cartridge

Note: Do not allow the cartridge to dry out after addition of water, otherwise repeat step d)

Add 10-50 mL of sample water to the cartridge. A larger sample volume may be used depending on metal concentration or suspended solids content

e) Adjust vacuum setting so that the water flows at 1-3 mL/minute until sample has passed completely through the cartridge

f) Allow the cartridge to air dry for about 1 minute under full vacuum

2) Elution--<u>Acid</u>

a) Prepare a 100 mM nitric acid elution solution

b) Place a collection vial in the vacuum manifold

c) Add 3 mL of the nitric acid solution to the cartridge

d) Adjust flow rate for a flow of 1-3 mL/minute

e) Dilute eluant to an appropriate volume for detection using reagent water

f) Sample is ready for analysis

3) Elution--<u>Base</u>

a) Prepare a 100 mM triethylamine elution solution

b) Place a collection vial in the vacuum manifold

a) Add 3 mL of the triethylamine solution to the cartridge

b) Adjust flow rate for a flow of 1-3 mL/minute

c) Dilute eluant to an appropriate volume for detection using reagent water

d) Sample is ready for analysis

4) Analysis

a) Prepare calibration curves for use with atomic absorption (AA) or Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) using appropriate metals standards

Extraction Protocol Tables

How to use these tables: When choosing an ion-exchange sorbent to capture arsenic (V) for example, all ion-exchange sorbents will capture a small quantity of metal ions, however, only a base extraction would elute metal ions from these sorbents. For extraction of zinc ions, all sorbents would have moderate to high capacity but elution could only occur from the sorbent using acidic elution conditions.

Acid Extraction Protocol

Sorbent	Cu (II)	Zn (II)	As (V)	Sn (IV)	Se (IV)	Hg (II)	Cr (III)	Pt (0)
PSA								
BCX-HL								
CCX								
TAX								
THX								
NAX								

Base Extraction Protocol

Sorbent	Cu (II)	Zn (II)	As (V)	Sn (IV)	Se (IV)	Hg (II)	Cr (III)	Pt (0)
PSA								
BCX-HL								
CCX								
TAX								
THX								
NAX								

Good to High Capacity
Moderate Capacity
Little or No Capacity

Ion-Exchange Sorbent Key

PSA	Primary secondary amine
BCX-HL	Benzene sulfonic acid –high load
CCX	Carboxylic acid
TAX	Triacetic acid
THX	Sulfhydryl (thiopropyl)
NAX	Aminopropyl

Primary Secondary Amine (PSA)

The PSA ion-exchange sorbent has a significant capacity for Hg(II) Se(IV) followed by a lesser capacity for Sn(IV), Cu(II), Zn(II) and Cr(III). Metal ions are readily eluted from PSA by the use of weak acid solutions such as 100mM nitric acid solution. Additional recovery for selenium can be obtained by following the acid elution by the use of 100 mM triethylamine solution

Benzenesulfonic Acid-High Load (BCX-HL)

The BCX-HL ion-exchange sorbent is the least selective of all ion-exchange sorbents and has significant capacity for Hg(II) and Sn(IV) thus ensuring high extraction efficiency for trace analysis. It is also a strong sorbent for Cu(II), Zn(II), Cr(III) and small amounts of Pt. In most cases, metal ions are readily eluted from BCX-HL by the use of 100mM nitric acid solution. Improvement in Hg(II) recovery yield and Sn(IV) can be achieved when eluting with 100 mM triethylamine solution.

Carboxylic Acid (CCX)

The CCX sorbents have high selectivity for Sn(IV) and Hg(II). Metal ions are readily eluted from CCX by the use of weak acid solutions such as 100mM nitric acid solution. Sn(IV) is eluted using 100 mM triethylamine solution. Additional Hg(II) is released under basic elution.

Triacetic Acid (TAX)

TAX sorbents have the highest affinity for Sn(IV) and Hg(II) followed by lesser amounts of Cu(II) and Zn(II). Metal ions are readily eluted from TAX by the use of weak acid solutions such as 100 mM nitric acid solution. Sn(IV) is eluted using 100 mM triethylamine solution. Additional Hg(II) is released under basic elution.

Sulfhydryl THX (thiopropyl)

THX sorbents have the highest affinity for Hg(II) and Sn(IV), and approximately equal weights of Sn(IV) and Cu(II). Metal ions are readily eluted from THX by the use of weak acid solutions such as 100mM nitric acid solution. Sn(IV), Se(IV) and Hg(II) are eluted using 100 mM triethylamine solution.

Aminopropyl (NAX)

The NAX ion-exchange sorbent has a significant capacity for Hg(II) followed by Se(IV). Metal ions are readily eluted from NAX by the use of weak acid solutions such as 100mM nitric acid solution. Additional Hg(II) is released under basic elution.

For further data and specific information and discussion of each sorbent see separate UCT publications: **Topics in Solid-Phase Extraction: Metals Analysis.**

*UCT ENVIRO-CLEAN® Ion-exchange cartridges are available in a variety of cartridge sizes, sorbent mass and particle size to most analytical requirements. For further information, contact UCT.

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PESTICIDES IN FATTY MATRICES EXTRACTION PROCEDURE REAGENTS:

February 3, 2009

Reagents:

Acetone
Toluene
Acetonitrile
Ethyl acetate
Magnesium sulfate anhydrous (UCT #ECMAG00D)
Sodium chloride (UCT #ECNACL05K)
UCT QuEChERS product (CUMPSC1815CT)
UCT product (ECPSAC1856)

1) Weigh 20.0 ± 0.10 grams (g) of homogenized sample into a 250 mL plastic centrifuge bottle that has been tared on a weigh balance capable of weighing to 0.01 grams.

2) Fortify each sample with process control spiking (PCS) solution.

3) Add 50 mLs of Ethyl Acetate (EtAc) to each tube containing a sample.

4) Fortify each sample with internal standard (ISTD) spiking solution.

5) Reduce sample material particle size by using a high speed disperser for approximately 1 minute.

6) Add 2 g of anhydrous magnesium sulfate (MgSO4) **(#ECMAG00D)** and 0.5 g anhydrous sodium chloride (NaCl) **(#ECNACL05K)**. {Note: Do not get the powders in the threads or rims of the tubes.}

7) Seal the tube and shake vigorously for approximately 1 minute mechanically or by hand, making sure that the solvent interacts well with the entire sample and that the crystalline agglomerates are broken up sufficiently.

8) Cool the sample in a -20 ºC freezer for approximately 30 minutes.

9) Centrifuge at 10,000 RCF for 5 minutes.

10) Decant > 50 mL of the EtAc layer into a 50 mL glass graduated centrifuge tube using a funnel and filter paper. Allow the extract to come to room temperature and adjust the volume to 50 mL with a Pasteur pipette.

11) Concentrate the extract under a stream of nitrogen with a 70 ºC water bath until the volume remains constant (this will be ~ 3 mLs and will take ~ 1 hour).

12) Dilute to 20 mLs with acetonitrile (MeCN) and cap with a glass stopper, vortex for 1 minute, and freeze at -70 ºC for 30 minutes.

13) Centrifuge the extract while frozen for 3 minutes (The MeCN will thaw during centrifugation).

14) Directly after centrifugation in step 13, filter > 15 mLs of the MeCN layer of the extract with a 0.45 µm syringe filter into a 15 mL glass centrifuge tube.

15) Allow the extract to come to room temp, adjust the volume to 15 mL, and concentrate to 2.25 mL under a stream of nitrogen with a 70 ºC water bath.

16) For LC/MS/MS analysis, transfer 1 mL of extract to a 2 mL mini-centrifuge tube that contains 0.05 g PSA, 0.05 g C18, and 0.15 g MgSO4. **(#CUMPSC18CT)**

17) Vortex for 1 minute and centrifuge.

18) Transfer to auto sampler vial and analyze by LC/MS/MS.

19) For GC analyses, use the dual layer cartridge **(#ECPSAC1856)** containing 500 mg of C18 sorbent and 500 mg of primary secondary amine (PSA) sorbent with approximately 0.75 – 0.80 grams (~ 0.6 cm = 0.25 inches) of anhydrous MgSO4 added to the top of the cartridge.

20) Condition the SPE cartridge by adding one cartridge volume (4.0 mLs) of acetone/toluene (3:1; v/v) using a UCT positive pressure SPE manifold and eluting to waste.

21) Place a properly labeled 15 mL graduated disposable plastic centrifuge tube below the cartridge in the positive pressure SPE manifold.

22) Quantitatively transfer 1 mL of the sample extract from step 15 to the SPE cartridge.

23) Elute SPE cartridge in a drop wise manner (Regulated Flow Pressure = 35 psi) into a properly labeled 15 mL graduated glass centrifuge tube with acetone/toluene (3:1; v/v), collecting the eluate while washing the SPE cartridge **three times** with **4 mLs of eluant**. Do not allow the cartridge to go dry until step 24.

24) After the last 4 mL portion of eluant has passed through the cartridge move the switch of the positive pressure SPE manifold from “Regulated Flow” to “Full Flow/Dry” to dry the SPE cartridge for approximately 1 minute.

25) Using an N-Evap with the water bath set at 50°C and nitrogen flow set at <10 liters per minute (LPM) {typical setting in 2 – 6 LPM}, evaporate the sample to approximately 0.5 mL.

26) Add 3 mL of toluene to the centrifuge tube containing the sample.

27) Evaporate again to < 0.5 mL. (This is to insure all other solvents have been removed from the sample.)

28) Bring the volume to 1.0 mL with toluene and Vortex to mix solvent into sample.

29) Analyze by GCMS-EI and GCMS-NCI.

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ANALYSIS OF TOBACCO ALKALOIDS

Part #: EUBCX1H2Z

February 3, 2009

A strong cation exchange column (benzene sulfonic acid) is used to capture tobacco alkaloids which have been ionized by the use of acid. Non-polar and other extraneous compounds are removed from the extract yielding cleaner chromatograms without loss of target alkaloids.

Sample Preparation

- Weigh 100 mg of fine ground tobacco in a screw cap vial, add 6 mL 0.1M sodium acetate buffer (pH 4.5) and 100 µL internal standard (d4-nornicotine, 1 µg/µL).
- Mix on rotating shaker for 10 minutes, then filter extract through 20 micron frit filter column.
- Add 300 µL glacial acetic acid and mix.
- Condition SPE column, UCT Part #: EUBCX1H2Z (200 mg benzene sulfonic acid sorbent) with 3 mL of MeOH:1.0M acetic acid (80:20).
- Pour sample onto column, aspirate for 1-2 mL/min by vacuum.
- Wash column with 3 mL of MeOH: 1.0M acetic acid (80:20).
- Dry column for 5-10 min with full vacuum.
- Elute alkaloids with 3 mL CH_2Cl_2/isopropanol/NH_4OH (70:26:4) by gravity.
- Evaporate eluant to dryness with nitrogen and low heat (< 40° C).
- Reconstitute with 200 µL ethyl acetate.
- Analyze on GC/FID/NPD or GC/MSD.

Results:

Alkaloid	Pka	Flue Cured Tobacco	Burkey Lamina Tobacco
	n=15/mean	mg/gram/CV	mg/gram/CV
Mysomine	NA	48/6.2	189/7.9
Nicotine	7.94	39406/6.2	39119/8.8
Nornicotine	9.46	1381/3.5	5429/5.2
Anatasine	9.20	229/5.2	183/8.7
Anatabine	8.23	1932/2.3	1774/2.3
2,3'-dipyridyl	4.25	54/8.9	30/11.2
Cotinine	4.88	20/11.2	52/12.4
Formylnornicotine	NA	31/11.9	145/12.4

Results show the average of 15 separate runs and indicate that reproducibility is excellent

Sample Chromatogram

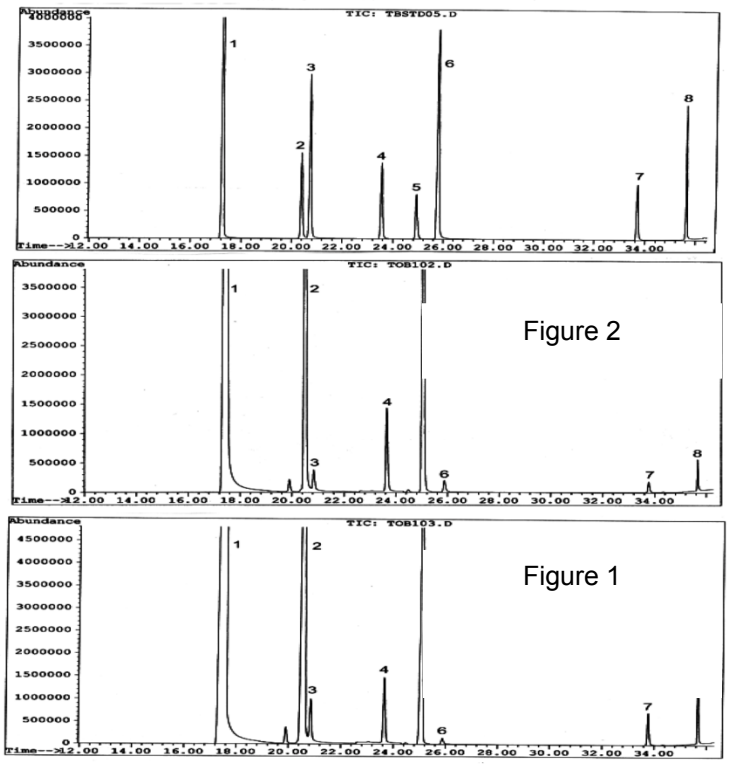

Figure 2

Figure 1

(1) nicotine, (2) nornicotine, (3) myosmine, (4) anabasine,(5) anatabine, (6) 2,3'-dipyridyl, (7) cotinine, (8) formylnornicotine

Instrument: Agilent 5890GC/5971MSD
GC column: Rtx-5 Amine, 30 m x 0.25 mm i.d. x 1.0 µm film
Injector: 1 µL sample at 10:1 split, 250º C
Temp program: Initial 120º C, hold 1 min, ramp 2.5º C/min to 200º C, ramp 20º C/min to 280º C, hold 1 min.
MSD conditions: SIM monitoring, EI mode, 295º C

DCN-903020-60

SAMPLE PREPARATION OF GLYPHOSATE (N-PHOSPHONOMETHYL GLYCINE) AND GLUFOSINATE (RS)-2-AMINO-4-(HYDROXYL-METHYL-PHOSPHORYL) BUTANOIC ACID BY SOLID-PHASE ANION EXCHANGE EXTRACTION

Part #: EUQAX2M6

February 3, 2009

Glyphosate, (CAS 1071-83-6) known principally as Roundup®, and glyfosinate (CAS 51276-47-2) (Basta®, Challenge®) are known as broad spectrum, nonselective systemic herbicides that are absorbed through the leaves of plants. It is used in many countries throughout the world because of its effectiveness at killing grass, broadleaf and woody plants. Sample preparation can be achieved using the UCT Enviro-Clean® anion exchange cartridge EUQAX2M6. For this procedure, a water sample is raised to pH 6 or more to ionize the analyte. The sample is drawn through the cartridge followed by elution with acidified methanol.

Sample Conditioning

- Adjust water sample pH to 6 or higher.

Cartridge Conditioning

- Add 5 mL of methanol to the cartridge
- Draw methanol through with vacuum leaving enough to cover surface of sorbent
- Rinse the cartridge using 10 mL of reagent water

Note: Do not let the cartridge dry out after addition of methanol

Extraction Protocol

- Draw a known volume of pH adjusted sample water through the cartridge

Note: Sample volume is determined by the quantitation limit

- Adjust vacuum so that flow is approximately 1 - 3 mL per minute
- Rinse cartridge using 10 ml of reagent water
- Dry the cartridge by drawing full vacuum for 10 minutes

Analyte Elution

- Elute using 4 mL of 1 mol/L HCl/methanol solution (4/1)
- Add eluant to the cartridge then draw through at 1 mL/minute
- Evaporate to dryness with N_2 flow in a water bath heated to 50^{O}C

Analysis

Analysis of glyphosate is conducted using GC/MS

- Add 50 µL of N-methyl-N-(tert-butyldimethylsilyl) triflouroacetamide (MTBSTFA) and 50 µL of dimethylformamide
- Sonicate at room temperature for 2 minutes
- Transfer to GC vial insert and cap
- Heat to 80^{O}C for 30 minutes
- Cool to room temperature and analyze by GC/MS

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QuEChERS

Quick, **E**asy, **Ch**eap, **E**ffective, **R**ugged and **S**afe

September 3, 2009

Solid-Phase Method for Extraction of Pesticide Residues

QuEChERS, the Multiresidue Method of Choice

QuEChERS (pronounced *Catchers*), an acronym for **Q**uick, **E**asy, **C**heap, **E**ffective, **R**ugged and **S**afe, is a sample preparation and clean-up technique for the analysis of multiple pesticide residues in high moisture food samples. Since the development and publication of the method by Anastassiades and Lehotay, et al in 2003, **QuEChERS** has been gaining significant popularity. It is the method of choice for food analysis because it combines several steps and extends the range of pesticides recovered over older, more tedious extraction methods. The method has undergone various modifications and enhancements over the years since its first introduction. These have been designed to improve recovery for specific types of pesticides. Although Schenck and Vega published a clean-up method in 2001 prior to the introduction of **QuEChERS,** its techniques may be incorporated in the current method enhancing **QuEChERS** utility.

While primarily used for the analysis of fruits and vegetables, the **QuEChERS** method is also finding utility as the sample preparation method of choice for a full range of food products such as honey, nut meats, soybeans, animal feeds, foliage and other foods as well. Organic acids, plant pigments and other potential contaminants are removed during the cleanup process yielding cleaner chromatograms. The method offers the advantages of high recoveries, accurate results, high sample throughput and low non-chlorinated solvent usage. This reduces reagent costs and staff exposure to hazardous solvents. Additionally, glassware usage and labor costs are reduced since sample requirements are small and less bench space is required. The broad utility and ease of use makes the method an excellent choice for residue analysis.

The Need for QuEChERS

Consensus has been growing within the scientific community that small doses of pesticides and other chemicals can have adverse health effect on humans and animals. In the last few years, pesticide residues in foods have become a major consumer safety issue since application of chemical pesticides for food products is widely used. Also, as large quantities of fruits and vegetables are now imported, concerns have arisen as to their safety versus those grown domestically. To address these concerns, regulatory agencies have resorted to the use of various analytical methods to monitor these food stocks increasing both the scope of residue analysis and the number of samples analyzed.

The analysis of pesticide residues in food and environmental samples has been practiced for over 40 years by laboratories throughout the world. The method of extracting pesticide residues from food samples and preparing them for analysis is a time consuming, expensive, and labor intensive process. To address this problem, new multiresidue methods such as the **QuEChERS** method have been developed to accommodate the increase in

sample loads. This new multiresidue method has yielded an increase in laboratory throughput while also improving analytical sensitivity. Improved throughput has been accomplished primarily by enhanced sample cleanup products that reduce potential interferences to yield cleaner chromatograms and reduced potential instrument downtime.

Multiresidue methods cover a broad scope of pesticides (see Appendix I) and offer the advantages of being cost-effective, rapid, sensitive, and sufficiently accurate for regulatory purposes. The **QuEChERS** method streamlines analysis and makes it easier and less expensive for analytical chemists to examine high moisture foods where water content may present problems with extraction of pesticides. Even dried vegetation can be rehydrated prior to extraction to facilitate the use of the **QuEChERS** method.

How Does QuEChERS Work?

QuEChERS is known as a multiclass, multiresidue method (MRM) for analysis of pesticides from high water content (80-95%) matrices. Multi-residue pesticide analysis of food and environmental samples can be problematic due to the wide range of chemical properties encountered with pesticide residues. Also, the complex sample matrix may contain abundant quantities of chlorophyll, lipids, sterols and other components that can interfere with good sample analysis. Use of the **QuEChERS** method reduces these problems.

The **QuEChERS** method now published as AOAC method **2007.01 "Determination of Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate"** consists essentially of a liquid-liquid micro extraction. This is followed by sample clean-up to remove unwanted matrix materials that interfere with chromatographic analysis. After shaking a sample with acetonitrile buffered with sodium acetate, partitioning is aided by the addition of $MgSO_4$. The preferred solvent is acetonitrile because it has been shown to provide extraction of the broadest range of organic compounds without co-extraction of large amounts of lipophilic material. Although other solvents can be employed depending upon the residues to be extracted, acetonitrile is highly compatible with GC/MS and LC/MS applications showing the fewest interferences.

Some modifications to the original **QuEChERS** method have been introduced to ensure efficient extraction of pH dependent compounds (e.g. phenoxyalcanoic acids), to minimize degradation of susceptible compounds (e.g. base and acid labile pesticides) and to expand the spectrum of food matrices amenable by the method. Buffering with citrate salts has been introduced in the first extraction/partitioning step to adjust the pH to a compromise value of 5 to 5.5, where most acid and base labile pesticides are sufficiently stabilized. To improve stability of base-labile compounds in the sample extracts, a small amount of formic acid is added to the final extract after cleanup using a primary-secondary amine (PSA) sorbent. Acidic pesticides are directly analyzed from the raw extract before PSA cleanup. In another modification introduced by Schenck, graphitized carbon black (GCB) is used to remove plant pigments.
Currently there are three variations of the **QuEChERS** method being used in the United States

1) **The original QuEChERS method.** Introduced in 2003, this method used sodium chloride to enhance extraction.

2) **Dispersive AOAC 2007.01.** Uses sodium acetate as a buffer replacing sodium chloride.

3) **The dual phase column:** This method variation introduces the use of PSA and GCB to remove high levels of chlorophyll and plant sterols in the final extract without the loss of planar pesticides (polar aromatics) using an acetone:toluene solvent blend (3:1).

Dry commodities such as cereals, dried fruits, tobacco or teas require the addition of water prior to extraction to weaken interactions of pesticides with the matrix and to ensure adequate partitioning. Even commodities with high lipid content such as avocados or other high oil load plants can be analyzed by this method. However, due to a partitioning to the lipid phase, highly non-polar pesticides may give relatively low yet consistent recoveries of up to 70%. These co-extracted lipids in the can be reduced by a freezing-out step or the use of a C18 cleanup step.

The **QuEChERS** method gives at least fourfold lower material costs with significantly greater sample throughput per analyst than traditional methods. By combination of several different steps there is less chance for the introduction of error at each step. A polypropylene (PP) tube is the only consumable item required eliminating all glassware used in conventional methods. Furthermore, less than 10 mL of solvent waste is generated, much less than the 75-450 mL generated by other methods. Key to the new approach is the re-discovery of a rapid procedure called dispersive solid-phase extraction. This technique quickly removes residual moisture with magnesium sulfate. Other potential interferences are reduced by employing a primary-secondary amine sorbent to capture acidic components.

How is QuEChERS Conducted?

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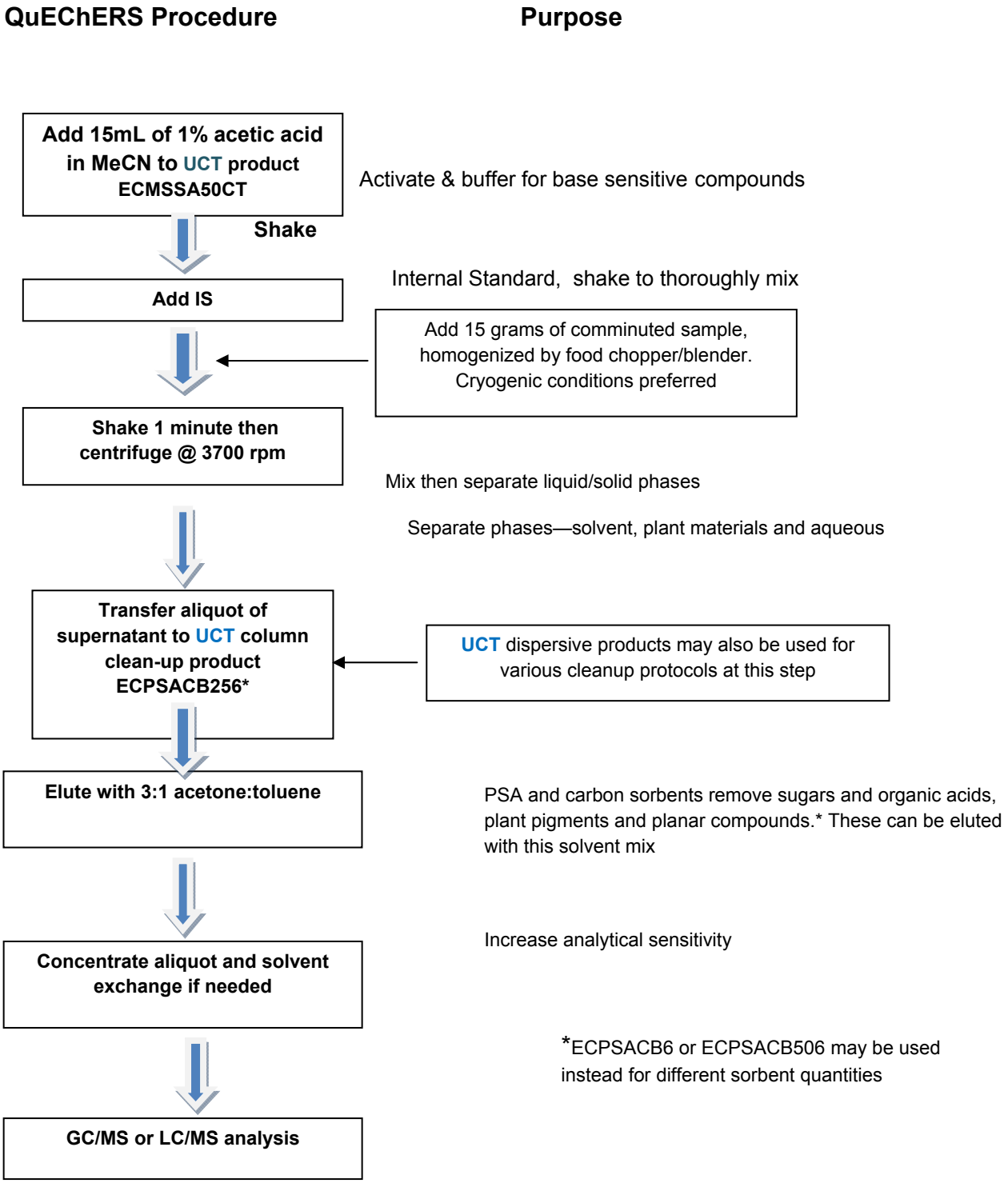
Procedural steps in the QuEChERS analysis for base sensitive compounds can be outlined in the schematic representation below:



QuEChERS Methods

Dispersive Methods

AOAC 2007.01 in brief (if base sensitive compounds are present)

1. To product ECMSSA50CT containing 6 grams anhydrous magnesium sulfate and 1.5 grams of anhydrous sodium acetate in a 50 mL polypropylene centrifuge tube, add 15 ml of 1% acetic acid in acetonitrile
2. Shake to mix contents
3. Add surrogate or internal standards if desired
4. Add 15 grams homogenized hydrated sample to the centrifuge tube
5. Shake for I minute
6. Centrifuge for 1 minute at 3700 rpm
7. Add an aliquot of the supernatant to the appropriate dispersive clean-up product: **UCT** CUMPSCB2CT, CUMPS2CT, CUMPSC18CT, ECMPSCB15CT, or ECMPSC1815CT
8. Shake for 1 minute
9. Centrifuge for 1 minute at 3700 rpm
10. Analyze extract

For compounds that are not base sensitive the following procedure provides a cleaner extract. This procedure is also necessary for acid labile compounds.

1. To product ECMSSC50CT containing 6 grams anhydrous magnesium sulfate and 1.0 gram of sodium chloride in a 50 mL polypropylene centrifuge tube, add 15 ml of acetonitrile
2. Shake to mix contents
3. Add surrogate or internal standards if desired
4. Add 15 grams of homogenized hydrated sample to the centrifuge tube
5. Shake for 1 minute
6. Centrifuge for 1 minute at 3700 rpm
7. Add a aliquot of the supernatant to the appropriate dispersive solid-phase cleanup tube: **UCT** CUMPSCB2CT, CUMPS2CT, CUMPSC18CT, ECMPSCB15CT, or ECMPSC1815CT (See Product List and Use Description below)
8. Shake for 1 minute
9. Centrifuge for 1 minute at 3700 rpm
10. Analyze extract

Matrix plant pigments often interfere with analysis. To reduce these interferences, graphitized carbon can be added to the dispersive solid-phase clean-up tubes. However, the use of carbon may result in a loss of planar (polar aromatic) pesticides. Cleanup of plant pigments without loss of planar pesticides can be accomplished by using the UCT Dual-Phase Cartridge Clean-Up Procedure.

Dual Phase Cartridge Clean-Up Procedure (elution for planar [polar aromatic] compounds)

1. Pre-rinse cartridge with 5 mL of toluene
2. Add an aliquot of the supernatant to the cartridge
3. Start collection
4. Elute with 6-12 mL of 3:1 acetone:toluene
5. Concentrate for GC/MS analysis or
6. Concentrate to dryness and reconstitute in mobile phase for LC analysis

Cartridge product selection used for this analysis: UCT ECPSACB6, ECPSACB256 or ECPSACB506 depending upon sorbent mass required.

Effect of Solvent Volume on Extraction

In a study designed to evaluate the effect of sample mass to solvent ratio (1:1 & 2:1MeCN) on recovery in a spiked fruit sample, Table 1 shows that the most polar pesticides did not partition into the MeCN phase as readily with the use of a lower solvent volume. However recovery still remained above 75% which is suitable for most analytical purposes. This indicates that the use of a minimum quantity of solvent for increased sensitivity will still yield good recovery values.

Average Recovery (%) of Selected Pesticides from a 10 gram Fruit Sample

Table 1

Pesticide	MeCN, 5 mL	MeCN, 10 mL
Dichlorvos	95	96
Methamidophos[b]	76	95
Mevinphos	96	100
Acephate[b]	84	99
o-Phenylphenol	94	94
Omethoate[b]	85	100
Diazinon	95	99
Chlorothalonil	94	95
Metalaxyl	94	100
Carbaryl	93	99
Dichlofluanid	97	97
Captan	97	100
Thiobendazole[b]	88	99
Folpet	92	94
Imazalil	92	102

[b] most polar Anastassiades, M. & Lehotay, S. J of AOAC International, Vol., 86, 2003

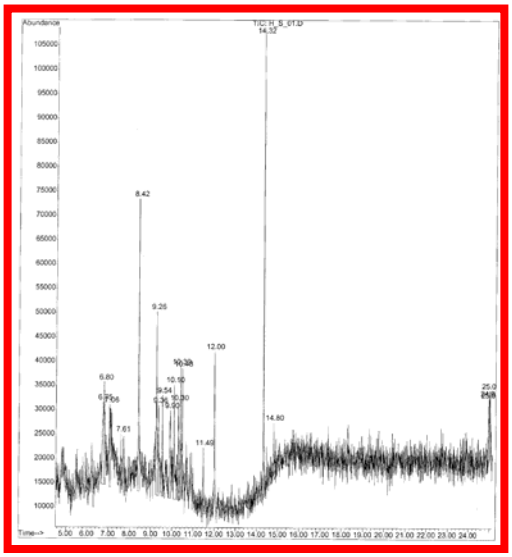
UCT Products Reduce Contamination Often Found in Laboratory Preparations

Many laboratories assemble their own clean-up products for the **QuEChERS** analysis. However contamination may be inadvertently introduced in the final extract complicating analyte peak identification. In a study conducted at the USDA ARS Eastern Regional Research Center, commercially prepared **QuEChERS** products were compared to those products prepared in the USDA lab. Bulk anhydrous magnesium sulfate, PSA and endcapped C18 sorbents provided by **UCT**, were assembled in the USDA lab then compared to **UCT** manufactured products using the same lot of bulk sorbents. The ratio of magnesium sulfate, PSA and C18 was 3:1:1 for this test. The clean-up products were evaluated on extracts of milk, honey and soybean and the efficacy of clean-up was determined by GC/MS analysis. Comparison of the extracts was made by counting the number of GC peaks above the threshold. Results clearly showed that the commercially prepared product provided superior clean-up to the product prepared in the lab. This result was confirmed in all three matrices. The extra peaks observed in the lab prepared product were probably caused by contamination from within the lab environment. The **UCT** assembled products were prepared under controlled manufacturing conditions that eliminated potential contamination typically encountered in lab environments. These results, coupled with obvious time and labor savings for assembly, indicate that **QuEChERS** products preassembled at **UCT** are preferable to products made "in-house".

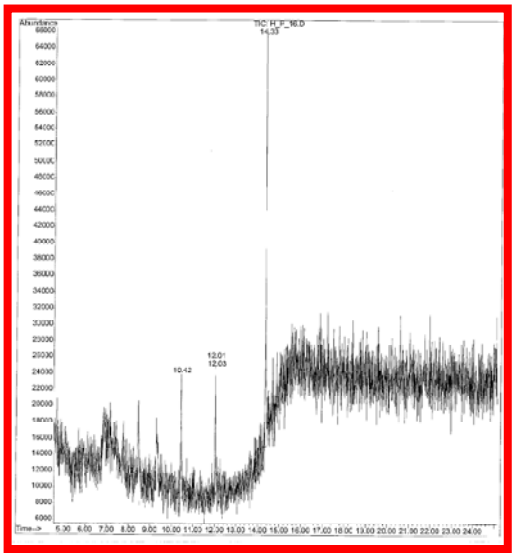
The results from these tests are summarized below for in tabular and graphic format for soybeans, honey and milk products. Chromatograms are shown for honey.

Chromatograms

Honey Extracted with "In-House" Product



Honey Extract Cleaned with UCT Products



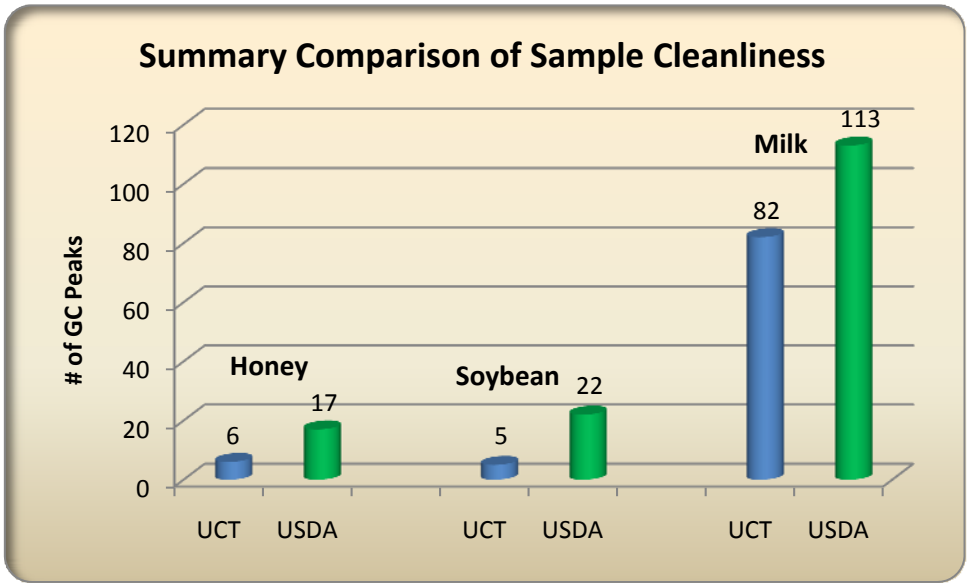
The chromatogram on the left is representative of those obtained when clean-up materials were prepared in-house. The chromatogram on the right was obtained using **UCT** prepackaged **QuEChERS** products and shows a significant reduction in background interferences. This difference in background interference is thought to be due to sorption of contaminants from the laboratory environment

Chromatograms for soybean and milk products showed similar improved clean-up when using UCT manufactured vs. "in-house" prepared products.

Table Showing the Total Number of Peaks as Seen in a GC Chromatogram

Data indicate that the use of UCT prepared products results in cleaner chromatograms

Matrix	HONEY		SOYBEAN		MILK	
	# of peaks	# of peaks	# of peaks	# of peaks	# of peaks	# of peaks
Replicate	UCT	USDA	UCT	USDA	UCT	USDA
1	7	20	7	17	43	91
2	9	12	8	15	49	103
3	7	21	5	20	52	108
4	8	24	2	12	43	121
5	5	18	6	8	46	117
6	5	22	2	13	45	104
7	8	8	7	11	49	89
8	4	13	4	10	103	117
9	5	18	4	7	107	127
10	8	12	3	9	106	127
11	6	15	2	31	116	120
12	6	12	8	28	126	118
13	6	19	6	35	104	119
14	6	21	4	51	106	108
15	5	20	4	43	100	118
16	4	14	7	43	109	113
Average	**6**	**17**	**5**	**22**	**81**	**113**



Why use UCT SPE QuEChERS products?

- Save valuable laboratory time in preparation
- Reduced variability due to consistent product and rigorous quality control
- Cleaner extracts from cleaner products
- Extraneous GC peak counts are significantly lower using **UCT** prepared **QuEChERS** product
- Variability of GC peak counts on replicate samples were significantly lower using **UCT QuEChERS** products
- Dual layer columns are packaged in Mylar to eliminate potential sorbent contamination
- $MgSO_4$ is specially treated in a muffle furnace to remove organic contaminates typically encountered with in-house preparation

UCT provides a variety of solid-phase QuEChERS clean-up products that contain the proper sorbents for optimum extraction, clean-up and separation of analytes from complex matrices

UCT Products Used in the Micro Extraction Step

Cartridge product *ECMSSA50CT* is a 50 mL extraction tube that contains 6 grams of anhydrous magnesium sulfate and 1.5 grams of anhydrous sodium acetate. It complies with AOAC Method 2007.01. It is designed to allow the extraction of "base sensitive" compounds such as chlorothalonil, dichlofluanid, tolyfluanid, folpet, captafol, captan from non-acidic matrices.

Cartridge product *ECMSSC50CT* is a 50 mL extraction tube contains 4 grams of anhydrous magnesium sulfate and 1 gram of sodium chloride. It is designed for use where base sensitive compounds are not present or are not of analytical interest. Eliminating the buffer allows a cleaner extract, and the sodium chloride aids in the extraction of the analytes.

Cartridge product EUMIV50CT is a 50 mL extraction tube that contains 6 grams anhydrous magnesium sulfate, 1.5 grams of sodium chloride and 0.75 grams of sodium citrate sesquihydrate.

UCT Cartridge Products Used for Sample Clean-Up

Several cartridge products are offered for use in sample clean-up. **UCT** provides a variety of **QuEChERS** products in SPE cartridge format which include PSA and GCB. These sorbents are used to remove various polar organic acids, polar pigments, some sugars and fatty acid co-extractables from **QuEChERS** extracts. These sorbents may be additionally combined with C18 for the removal of fatty plant lipids and sterols. Graphitized carbon black is used to remove sterols and pigments such as chlorophyll. Magnesium sulfate or other salts are used to enhance extraction as well as the removal of water and the partitioning of residues into the solvent phase. Because carbon has a strong affinity to retain planar molecules, Schenck et al have reported that the use of a 3:1 acetone:toluene solvent blend performed well at eluting these compounds from carbon sorbents. Bulk sorbents are also available from the **UCT** catalog.

Products List and Use Description

Part Number	Contents and Use Description

ChloroFiltr™ Dispersive Products

ChloroFiltr™ is a revolutionary new QuEChERS sorbent for the removal of chlorophyll without the loss of analytes.

CUMPSGG2CT	**2mL micro-centrifuge tubes with 150mg magnesium sulfate, 50mg PSA and 50mg ChloroFiltr™**
ECMPSGG15CT	**15mL centrifuge tube with 900mg magnesium sulfate, 300mg PSA and 150mg ChloroFiltr™**

Dispersive Products

CUMPSCB2CT	**2mL micro-centrifuge tubes with 150mg Anhydrous Magnesium Sulfate, 50mg PSA, 50mg Carbon** *A dispersive SPE product for removing polar organic acids, some sugars and lipids which may cause some loss of planar pesticides. Designed for use with a 2mL aliquot of supernatant.*
CUMPS2CT	**2mL micro-centrifuge tubes with 150mg Anhydrous Magnesium Sulfate, 50mg PSA** *A dispersive SPE product for removing polar organic acids, some sugars and lipids. Designed for use with a 2mL aliquot of supernatant.*
CUMPSC18CT	**2mL micro-centrifuge tubes with 150mg Anhydrous Magnesium Sulfate, 50mg PSA, 50mg endcapped C18** *A dispersive SPE product for removing polar organic acids, sterols, some sugars and lipids. esigned for use with a 2mL aliquot of supernatan*t.
CUMPS15C18CT	**2mL micro-centrifuge tubes with 150mg Anhydrous Magnesium Sulfate, 150mg PSA & 50mg endcapped C18**
ECMPSCB15CT	**15mL centrifuge tubes with 900mg Anhydrous Magnesium Sulfate, 300mg PSA, 150mg carbon** *A dispersive SPE product for removing polar organic acids, some sugars and lipids. This product will cause the loss of planar pesticides. Designed for use with a 10mL aliquot of supernatant.*
ECMPSC1815CT	**15mL centrifuge tubes with 900mg Anhydrous Magnesium Sulfate, 300mg PSA, 150mg endcapped C18** *A dispersive SPE product for removing polar organic acids, sterols, some sugars and lipids from a 10mL aliquot.*
ECMS12CPSA415CT	**15mL centrifuge tube with 1200mg magnesium sulfate, 400mg PSA**
ECMNAX15CT	**15mL centrifuge tube with 900mg magnesium sulfate, 150mg of aminopropyl** *Florida-Modified QuEChERs for State Program Fruits and Vegetables*
ECMPSA50CT	**50mL centrifuge tube with 1200mg magnesium sulfate, 200mg PSA**

Cartridge Products

Dual phase cartridges are available as an alternative to traditional QuEChERS dSPE clean-up.

ECPSACB6 **6mL cartridges with 200mg Graphitized Carbon on top, 400mg PSA on bottom, separated by a Teflon frit***

Used in the Schenck variation of QuEChERS, this product removes pigments, polar organic acids, some sugars and lipids from an aliquot of extract.

ECPSACB256 **6mL cartridges with 250mg Graphitized Carbon on top, 500mg PSA on the bottom, separated with a Teflon frit***

Used for the same application as ECPSACB6 but with a different quantity of sorbents. When in doubt use ECPSACB256.

ECPSACB506 **6mL cartridges with 500mg Graphitized Carbon on top, 500mg PSA on the bottom, separated with a Teflon frit***

Used for the same application as ECPSACB6 but with a different quantity of sorbents. When in doubt use ECPSACB256.

ECNAXCB506 **6mL cartridges with 500mg Aminopropyl and 500mg Graphitized Carbon**

***Products available with Polyethylene or Teflon frits. Your choice will depend on your application and price requirements.**

European QuEChERS Method EN 15662

ECQUEU12CT **2mL centrifuge tube with 150mg magnesium sulfate, 25mg PSA**

ECQUEU22CT **2mL centrifuge tube with 150mg magnesium sulfate, 25mg C18, 25mg PSA**

ECQUEU32CT **2mL centrifuge tube with 150mg magnesium sulfate, 25mg PSA, 2.5mg GCB**

ECQUEU42CT **2mL centrifuge tube with 150mg magnesium sulfate, 25mg PSA, 7.5mg GCB**

ECQUEU415CT **15mL centrifuge tube with 4000mg magnesium sulfate, 1000mg NaCl, 500mg sodium citrate dibasic sesquihydrate, 1000mg sodium citrate tribasic dihydrate**

ECQUEU215CT **15mL centrifuge tube with 6000mg magnesium sulfate, 1500mg sodium acetate**

ECMPS15CT **15mL centrifuge tube with 900mg magnesium sulfate, 150mg PSA**

ECQUEU315CT **15mL centrifuge tube with 900mg magnesium sulfate, 150mg PSA, 150mg C18**

ECQUEU515CT **15mL centrifuge tube with 900mg magnesium sulfate, 150mg PSA, 15mg GCB**

ECQUEU615CT **15mL centrifuge tube with 900mg magnesium sulfate, 150mg PSA, 45mg GCB**

ECQUEU750CT **50mL centrifuge tube with 4000mg magnesium sulfate, 1000mg NaCl, 500mg sodium citrate dibasic sesquihydrate, 1000mg sodium citrate tribasic dihydrate**

QuEChERS Multi-Packs

QuEChERS extraction reagents for all of the popular QuEChERS methods are available in individual metalized pouches for your convenience. Each pack of 50 pouches comes with racks of 50 empty centrifuge tubes with plug seal caps.

EC4MSSA50CT-MP	Each pouch will contain 4000 mg magnesium sulfate and 1g of Sodium Acetate	**50**
ECMSNA50CT-MP	Each pouch will contain 8000 mg magnesium sulfate and 3500mg of sodium chloride	**50**
EUMIV50CT-MP	Each pouch will contain 6000 mg magnesium sulfate, 1500 mg sodium chloride, 1500mg of sodium citrate dihydrate, 750mg of disodium citrate sesquihydrate	**50**
ECMSSA50CT-MP	**Each pouch will contain 6000 mg of magnesium sulfate and 1500 mg of sodium acetate**	**50**
ECMSSC50CT-MP	Each pouch will contain 4000 mg of magnesium sulfate and 1000 mg of sodium chloride	**50**
ECMSSC50CTFS-MP	Each pouch will contain 6000 mg of magnesium sulfate and 1500 mg of sodium chloride	**50**
ECQUVIN50CT-MP	Each pouch will contain 8000 mg of magnesium sulfate and 2000 mg of sodium chloride	**50**
ECQUEU750CT-MP	Each pouch will contain 4000 mg of magnesium sulfate, 1000 mg of sodium chloride, 500 mg of sodium citrate dibasic sesquihydrate, and 1000mg sodium citrate tribasic dihydrate	**50**

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F.J. Schenck and J.E. Hobbs, “Evaluation of the Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) approach to pesticide residue analysis” Bull. Environ. Contam. Toxicol., 73, 24-30 (2004)

Appendix I

List of possible pesticide analytes that have been shown to yield >90% (or >70 %*) recoveries using the QuEChERS method. GC-amenable pesticides are capitalized; those preferentially analyzed by LC/MS-MS are not capitalized; those that can be analyzed by either technique are underlined**

Pesticide Analyte

acephate*	acetamiprid	Acrinathrin	aldicarb	aldicarb sulfone
aldicarb sulfoxide	Aldrin	azaconazole	azamethiphos	azinphos-methyl
azoxystrobin	Bifenthrin	bitertanol	Bromopropylate	bromuconazole
Bupirimate	buprofezin	butocarboxim	butocarboxim sulfone	butocarboxim sulfoxide
Cadusafos	carbaryl	carbendazim	carbofuran	3-hydroxy-carbofuran
chlorbromuron	(α-, γ-)Chlordane	(α-,β-Chlorfenvinphos	Chlorpropham	Chlorpyrifos
Chlorpyrifos-methyl	Chlorthaldimethyl	Chlorothalonil*	Chlozolinate	clofentezine
Coumaphos	cycloxydim*	(λ-)Cyhalothrin	cymoxanil	Cypermethrin
cyproconazole	cyprodinil	(2,4'-4,4'-)DDE	(2,4'-4,4'-)DDT	Deltamethrin
demeton	demeton-O-sulfoxide	demeton-S-methyl	demeton-S-methyl sulfone	desmedipham
Diazinon	dichlofluanid*	Dichlorobenzophenone	dichlorvos	diclobutrazole
Dicloran	dicrotophos	Dieldrin	Diethofencarb	difenoconazole
Diflufenican	dimethoate	dimethomorph	diniconazole	Diphenyl
Diphenylamine	disulfoton	disulfoton sulfone	diuron	dmsa
dmst	dodemorph	α- Endosulfan	β-Endosulfan	Endosulfan sulfate
EPN	epoxiconazole	Esfenvalerate	etaconazole	ethiofencarb sulfone
ethiofencarb sulfoxide	Ethion	ethirimol	Ethoprophos	etofenprox
Etridiazole	Famoxadone	fenamiphos	fenamiphos sulfone	Fenarimol
Fenazaquin	fenbuconazole	fenhexamid*	Fenithrothion	fenoxycarb
Fenpiclonil	Fenpropathrin	Fenpropidine	fenpropimorph	fenpyroximate
Fenthion	fenthion sulfoxide	Fenvalerate	florasulam*	Flucythrinate I & II
Fludioxonil	flufenacet	Flufenconazole	flusilazole	Flutolanil
Fluvalinate	Fonophos	fosthiazate	Furalaxyl	furathiocarb
furmecyclox	Heptachlor	Heptachlor epoxide	Heptenophos	Hexachlorobenzene
hexaconazole	hexythiazox	imazalil	imidacloprid	Iprodione
iprovalicarb	isoprothiolane	isoxathion	kresoxim-methyl	Lindane
linuron	Malathion	malathion oxon	Mecarbam	mephosfolan
Mepronil	Metalaxyl	metconazole	methamidophos*	Methidathion
methiocarb	methiocarb sulfone*	methiocarb sulfoxide	methomyl	methomyl-oxime

metobromuron	metoxuron	Mepanipyrim	Mevinphos	monocrotophos
monolinuron	myclobutanil	nuarimol	Ofurace	omethoate
oxadixyl	oxamyl	oxamyl-oxime	oxydemeton-methyl	paclobutrazole
Parathion	Parathion-methyl	penconazole	pencycuron	*cis*- Permethrin
***trans*-Permethrin**	phenmedipham	*o*-Phenylphenol	Phorate	phorate sulfone
Phosalone	Phosmet	Phosmet-oxon	phosphamidon	Phthalimide
picoxystrobin	Piperonyl butoxide	pirimicarb	pirimicarb-desmethyl	Pirimiphos-methyl
prochloraz	Procymidone	profenofos	Prometryn	Propargite
Propham	propiconazole	propoxur	Propyzamide	Prothiofos
pymetrozine	Pyrazophos	pyridaben	pyridaphenthion	pyrifenox
pyrimethanil	Pyriproxyfen	Quinalphos	Quinoxyfen	Quintozene
sethoxydim	spinosad	spiroxamine	tebuconazole	tebufenozide
Tebufenpyrad	tetraconazole	Tetradifon	Tetrahydrophthalimide	Terbufos
Terbufos sulfone	thiabendazole	thiacloprid	thiamethoxam	thiodicarb
thiofanox	thiofanox sulfone	thiofanox sulfoxide	thiometon	thiometon sulfone
thiometon sulfoxide	thiophanate-methyl	Tolclofos-methyl	tolylfluanid	triadimefon
triadimenol	Triazophos	trichlorfon	tricyclazole	tridemorph
trifloxystrobin	trifluminazole	Trifluralin	Triphenylphosphate	vamidothion
vamidothion sulfone	vamidothion sulfoxide	Vinclozolin		

**from “Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) Approach for Determining Pesticide Residues”, Steven J. Lehotay, U.S. Department of Agriculture, Agricultural Research Service, Eastern Regional Research Center; 600 East Mermaid Lane; Wyndmoor, Pennsylvania 19038; USA

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1. Sample Pretreatment

a) Prepare an acid washed beaker*
b) Add 10-100 grams of soil sample
c) Add enough DI H_2O to form a loose slurry
d) Insert a magnetic stir bar and extract for 15 minutes
e) Adjust pH to 2 using 50% aqueous sulfuric acid (H_2SO_4)
f) Continue extraction for 15 minutes adjusting pH as needed
g) Filter sample through previously acidified filter media

Note: Acid washed glassware must be used in this procedure. Soda lime glassware must be avoided as it may interfere with the analysis

2. Condition C18 SPE Cartridge

a) Add 5 mL CH_3OH and wait 1 minute
b) Add 5 mL DI H_2O

Note: Aspirate at low vacuum setting. Do not let cartridge dry out otherwise repeat steps a) and b)

3. Add Sample

a) Adjust vacuum and load cartridge at 10 mL/minute flow rate

4. Dry Cartridge

b) Dry cartridge for 10 minutes at full vacuum

5. Elute Phenoxyacetic acid Herbicides

a) Place a clean collection vial in manifold
b) Add 5 mL of CH_3OH and wait 1 minute
c) Add a second 5 mL volume of CH_3OH
d) Adjust vacuum and collect at 1-2 mL/ minute

6. Dry Eluate

a) Evaporate to dryness at < 40˚C using N_2
b) Reconstitute in 100 µL of mobile phase for **LC-MS/MS**
- Inject 10-100 µL

HPLC Analysis and Instrumentation Requirements

Guard Column: C18 10mm x 2.6mm with 0.5 µm frit

Analytical Column:

- C18 100 mm x 2 mm 5 µm particle ODS-Hypersil
- C18 100 mm x 2 mm 3 µm particle MOS2-Hypersil or equivalent

HPLC/MS Interface:

- Micromixer 10-µL interface HPLC column system with HPLC post-column addition solvent

Interface:

- Thermospray ionization interface and source capable of generating both positive and negative ions and have a discharge electrode or filament

Mass Spectrometer System:

- A single quadrupole mass spectrometer capable of scanning from 1 to 1000 amu
- Scanning from 150 to 450 amu in 1.5 sec. or less using 70 volts (nominal) in positive or negative electron modes
- Capable of producing a calibrated mass spectrum for polyethylene glycol (PEG 400, 600, or 800, average mol. wts.) or other compounds used as a calibrant
- Use PEG 400 for analysis of chlorinated phenoxyacid compounds. PEG is introduced via the Thermospray interface circumventing the HPLC

Thermospray Temperatures:
Vaporizer Control: 110°C to 130°C
Vaporizer Tip: 200°C to 215°C
Jet: 210°C to 220°C
Source Block: 230°C to 265°C

Recommended HPLC Chromatographic Conditions

Chlorinated Phenoxyacid Compounds

Initial Mobile Phase %	**Initial Time** minutes	**Final** minutes	**Final Mobile Phase %**	**Time** minutes
75A/25	2	15	40/60	
40A/60	3	5	75/25	10

A=0.1 M ammonium acetate/methanol

Limits of Detection in the Positive and Negative Ion Modes for HPLC Analysis of Chlorinated Phenoxyacid Herbicides and Esters

Compound	Positive Ion Mode Quantitation LOD		Negative Ion Mode Quantitation LOD	
	Ion	ng	Ion	ng
Dalapon	Not detected		141 $(M^- H)^-$	11
Dicamba	238 $(M^+NH_4)^+$	13	184 $(M^-HCl)^-$	3.0
2,4-D	238 $(M^+NH_4)^+$	2.9	184 $(M^-HCl)^-$	50
MCPA	218 $(M^+NH_4)^+$	120	199 $(M^-1)^-$	28
Dichloroprop	252 $(M^+NH_4)^+$	2.7	235 $(M^-1)^-$	25
MCPP	232 $(M^+NH_4)^+$	5.0	213 $(M^-1)^-$	12
2,4,5- T	272 $(M^+NH_4)^+$	170	218 $(M^-HCl)^-$	6.5
2,4,5-TP Silvex	286 $(M^+NH_4)^+$	160	269 $(M^-1)^-$	43
Dinoseb	228 $(M^+NH_4^-NO)^+$	24	240 (M-)	19
2,4-DB	266 $(M^+NH_4)^+$	3.4	247 (M-1)-	110
2,4,5-D, butoxy ethanol ester	321 $(M^+H)^+$	1.4	185 $(M^-C_6H_{13}O_1)^-$	
2,4,5-T,butoxy ethanol ester	372 $(M^+NH_4)^+$	0.6	195 $(M^-C_8H_{15}O_3)^-$	
2,4,5-T, butyl ester	328 $(M^+NH_4)^+$	8.6	195 $(M^-C_6H_{11}O_2)^-$	
2,4-D, ethyl hexyl ester	350 $(M^+NH_4)^+$	1.2	161 $(M^-C_{10}H_{19}O_3)^-$	

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1. Sample Preparation

a) Weigh out 1 g of homogenized beef kidney sample, kidney juice or serum in a 50 mL FEP (fluorinated ethylene propylene) centrifuge tube. A disposable polypropylene 50 mL centrifuge tube can also be used

b) Add 100 µL of 1 µg/ml internal standard consisting of ^{13}C-sulfamethazine, penicillin-V and cefadroxil in water. Penicillin and cephalosporin are used for method performance control

c) Add 2 mL water and 8 mL acetonitrile

d) Vortex briefly, then shake for 5 minutes

e) Centrifuge at 3450 rpm for 5 minutes

f) Decant the supernatant into a 50 mL centrifuge tube containing 500 mg of UCT EEC1800X sorbent

g) Vortex briefly, then shake for 30 seconds

h) Centrifuge at 3450 rpm for 1 minute

i) Transfer 5 mL aliquot of the supernatant into a graduated tube

2. Evaporate

a) Reduce extract volume to < 1 mL using a stream of N_2

b) Readjust extract volume to 1 mL with DI water

c) Filter the extract through a 0.45µm PVDF syringe filter into a clean vial

3. Analysis

a) Extract is now ready for analysis using LC-MS/MS

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1. **Sample Preparation**
   a) Weigh 1-5 grams of sample in a clean beaker
   b) Add 1-2 volumes of 5% methanol/water
   c) Stir for 15 minutes on a magnetic stir plate
   d) Adjust sample pH to 2 using 50% aqueous sulfuric acid (H_2SO_4)
   e) Continue extraction for a additional 15 minutes
   f) Adjust pH if necessary to maintain pH 2
   g) Filter or centrifuge sample
   h) Decant supernatant or collect filtrate
   i) Add appropriate internal standards and surrogates

2. **Condition Cartridge**
   Add 10 mL methanol and wet sorbent
   Add 20 mL DI water and draw through cartridge to remove methanol

   Note: Do not allow the cartridge to dry out otherwise repeat steps a) and b)

3. **Extract Sample**
   Draw sample through cartridge at 10 mL/ minute.

4. **Dry Cartridge**
   Dry cartridge for 10 minutes at full vacuum

5. **Elute Brodificoum, Daphacinone, and Hydramethylnon**
   Add 10 mL of ethyl acetate to sample bottle and shake
   Add to cartridge
   Adjust vacuum and collect eluate at 1-2 mL/ minute flowrate
   Add 10 mL of methylene chloride (CH_2Cl_2) to sample bottle
   Add to cartridge
   Collect eluate at 1-2 mL/ minute flowrate
   Pass combined organic phases through anhydrous sodium sulfate tube

6. **Dry Eluate**
   Using N_2, evaporate sample to less than 0.5 mL
   Bring to 1.0 mL with ethyl acetate in a volumetric flask

7. **Analyze Sample**
   Inject 1-2 µL onto GC, GC/MS

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Analysis of Malachite Green and Metabolite Leucomalachite Green by HPLC

Part #: EUBCX256

April 8, 2009

Malachite Green CAS 569-64-2

1. Sample Preparation

a) Add 1 g of sample to a 50 mL centrifuge tube
b) Add 50uL TMPD* solution at 1mg/mL
c) Spike using malachite green and leucomalachite green at 0.1 µg/mL
d) Add internal standard
e) Allow to sit for 10 minutes
f) Add 10 mL of Mcilvaines** Buffer/Methanol 1:1
g) Shake for 1 minute
h) Centrifuge at 5000 rpm for 10 minutes
i) Collect the supernatant
j) Repeat steps f) through i) until a volume of 20 mL of supernatant is obtained

2. Condition Cartridge

a) Add 5 mL of methanol to cartridge EUBCX256 and soak for 1 minute
b) Add 5 mL of water and draw through
c) Add 5 mL of Mcilvaines buffer

Note: Do not let the cartridge go dry otherwise repeat steps a) through c)

3. Extraction

a) Load supernatant from step 1j
b) Adjust vacuum for flow of 1–3 mL per minute

4. Wash Cartridge

a) Add 5 mL 0.1N HCl and slowly draw through
b) Add 10 mL water and draw through
c) Add 5 mL 1:1 MeOH: water and draw through
d) Add 10 mL hexane and draw through
e) Dry under vacuum for 10 minutes

5. **Elute cartridge**
   a) Elute at 1–3 ml per minute using a solution of 50% ethyl acetate, 45% methanol, 5% ammonium hydroxide
   b) Carefully evaporate to dryness at 50°C using N_2
      a) Reconstitute with LC mobile phase (50% acetonitrile and water containing 0.05 M p-toluene sulfonic acid (TSA) as a counter ion

6. **Analyze by LC/MS**
   a) Use a reverse-phase C18 analytical column

Solutions:

* TMPD – N,N,N',N'-tetramethyl-1,4-phenylenediamine dihydrochloride, CAS 637-01-4

**Mcilvaines buffer pH 2.6 – mix equal parts of 0.1M citric acid monohydrate with 0.2M disodium hydrogen phosphate dihydrate (Na_2HPO_4)•H_2O

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Extraction of Bentazone and Acifluorfen from Solid Matrices Using C_{18} SPE with GC/MS Analysis

Part #: ECUNIC18 or EEC181M6

April 8, 2009

1. Sample Pretreatment

Homogenize 5-10 grams of solid sample using 3-4 volumes of 5% aqueous methanol (CH_3OH)
Centrifuge sample
Transfer supernatant to appropriate sized sample bottles
Note: Adjust methanol to pH 2 using 0.1N HCl

8. Condition C_{18} Cartridge

Add 5 mL CH_3OH
Add 15 mL DI H_2O adjusted to pH2 using 0.1N HCl
Note: Do not let the cartridge dry out otherwise repeat steps a) and b)

9. Extract Sample

Adjust vacuum and draw water sample at 25 mL/ minute

10. Dry C_{18} SPE

Dry column for 10 minutes at full vacuum

11. Elute Bentazone/ Acifluorfen

a) Prepare a clean test tube by adding 2g of acidified sodium sulfate*
b) Add 10 mL of ethyl acetate to sample bottle and shake
c) Add to C_{18} cartridge
d) Soak for 1 minute
e) Adjust vacuum and collect eluate in the tube containing sodium sulfate
f) Add 10 mL of methylene chloride (CH_2Cl_2) to the sample bottle and swirl
g) Add to C_{18} column
h) Soak for 1 minute and collect
i) Repeat using another 10 mL of aliquot of CH_2Cl_2
j) Adjust vacuum and collect at 1-2 mL/ minute

12. Dry Eluate

Pass extract through 10 g of acidified sodium sulfate
Collect sample in a clean vial (Do not use soda lime glass)
Add appropriate internal standard

13. Evaporate

Concentrate to desired final volume

14. Derivatize sample

Use trimethylsilyldiazomethane (TMSD) by EPA Method 515 or diazomethane by EPA Method 8151.
Note: The extract must be completely dry or incomplete methylation will occur.

15. Analysis

Inject 1-2 µL onto a GC/MS

*See Procedure for Preparing Acidified Sodium Sulfate Anhydrous, UCT, Inc., Revision 1.1, EPA Method 8151A, 5.10

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QuEChERS Extraction and Clean-Up of Pesticides from Olive Oil

Part #: CUMPS2CT (150 mg anhydrous $MgSO_4$ & 50 mg PSA)

April 8, 2009

1. Sample Extraction

a) In a suitable vial, add 1.5mL of olive oil

b) Add 1.5 mL of hexane

c) Add 6 mL of acetonitrile

d) Shake for 30 minutes

e) Allow layers to phase separate for 20 minutes

f) Collect acetonitrile layer (top layer)

g) Repeat steps c) through f) and combine acetonitrile layers

2. Sample Clean-up

a) Add 1 ml of combined acetonitrile to UCT product CUMPS2CT

b) Shake for 2 minutes by hand

c) Centrifuge at 3000 rpm for 2 minutes

d) Remove solvent layer

e) Analyze by HPLC using MS detection

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QuEChERS Multiresidue Pesticide Method for The Determination of Multiple Pesticides in Wines*

Part Number:

ECQUVIN50CT (50 mL centrifuge tube, 8.0 grams anhydrous $MgSO_4$ & 2 grams NaCl)

ECMPSCB15CT (900 mg anhydrous $MgSO_4$, 300 mg PSA &150 mg GCB)

February 11, 2010

This method summary describes a multi-residue pesticide method for the determination of 72 pesticides in wines. Pesticides are extracted using acetonitrile saturated with magnesium sulfate and sodium chloride followed by a dispersive solid-phase cleanup with primary-secondary amine (PSA) and graphitized carbon black (GCB) sorbents.

Analysis is performed using ultra-performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) in ESI (positive) mode.

1. Sample Preparation

a) Add 20 mL acetonitrile (ACN) and *internal standard* 250 uL Fluconazole (10 µg/L) to **ECQUVIN50CT**
b) Quantitatively add 20.0 mL of wine
c) Shake for approximately 2 minutes
d) Centrifuge at 4500 rpm for 5 minutes (use refrigerated centrifuge if available)
e) Transfer 9.0 mL of top layer and add to **ECMPSCB15CT** (900 mg anhydrous $MgSO_4$, 300 mg PSA &150 mg GCB)
f) Vortex tube for approximately 10 seconds
g) Open tube and add 3.0 mL of toluene and shake for 1 minute
h) Centrifuge the tube for 5 minutes @ 4500 rpm
i) Quantitatively transfer 2.0 mL of supernatant to a glass centrifuge tube
j) Evaporate to dryness at < 40 ˚C using N_2
k) Add 500 µL of ACN and 25 µL of *surrogate standard* (benzanilide - 20.0 µg/L) for QC and 500 µL of 20 mM ammonium acetate in 1% ACN to the dried extract
l) Vortex for approximately 5 seconds and filter into autosampler vial using 17mm, 0.2 µm nylon membrane cartridges attached to a disposable syringe

2. UPLC/MS/MS Analysis

UPLC Conditions:

Column: Water's Acquity UPLC BEH C_{18} column 100 x 2.1 mm, 1.7 µm particle or equivalent

Flowrate: 0.2 mL/minute

Injection volume: 3 µL

Analytical Standards: Matrix Matched

Gradient Program:

Time	% Acetonitrile	% 10mM Ammonium Acetate
0	10	90
10	90	10
14.5	90	10
14.6	10	90
20.1	10	90

Triple Quadrupole MS Conditions--electrospray ionization mode (ESI+)

Capillary Voltage: 1.5 kV

Source Temperature: 120 °C

N_2 Flow: cone 50 L/h, desolvation 800 L/h

Collision Gas: Argon

Dwell Time: 10 µS for multiple reaction monitoring (MRM) experiments

Collision Cell Pressure: 5.9 x 10^{-3} mbar

Summary of MS/MS Conditions

Pesticide	Molecular Weight	CV (V)	Quantification Transition
Acephate	183.17	20	184.0→143.0
Acetamiprid	222.67	30	223.4→126.1
Acibenzolar S-methyl	210.27	35	211.1→136.0
Aldicarb	190.27	12	208.1→116.0
Aldicarb sulfone	222.27	15	240.0→222.9
Aldicarb sulfoxide	206.26	15	224.2→206.9
Atrazine	215.69	35	215.9→173.85
Avermectin B_{1b}	873.09	20	876.6→553.4
Avermectin B_{1a}	873.09	20	890.7→567.5
Azoxystrobin	403.30	25	404.0→372.1
Benalaxyl	325.41	26	326.1→148.1
Benfuracarb	410.53	20	411.2→190.0
Benzanilide	197.24	30	198.1→105.1
Bifenazate	300.35	20	301.3→170.2
Bitertanol	337.42	20	338.2→ 99.1
Buprofezin	305.44	25	306.3→201.2
Carbaryl	201.22	22	202.1→145.1
Carbendazim	191.19	30	192.0→160.0
Carbofuran	221.26	26	222.1→123.1
Chloroxuron	290.75	35	291.0→ 72.2
Cyprodinil	225.29	45	226.1→ 93.0
Cyromazine	166.19	25	167.2→ 85.1
Diclobutrazol	328.24	30	328.1→70.2
Dimethoate	229.26	20	230.1→199.0
Dimethomorph	387.86	35	388.0→301.1
Dimoxystrobin	326.39	20	327.1→206
Dinotefuran	202.20	20	203.5→14.0
Diuron	233.10	30	233.0→72.1
Ethofumesate	286.35	30	286.9→258.9
Famoxadone	374.39	-32	373.2→ 282
Fenamidone	311.40	25	312.2→236.2
Fenbuconazole	336.82	35	337.1→125.0
Fenhexamid	302.20	65	301.9→261.9
Fenpropimorph	304.49	40	304.4→147.1
Fluconazole	306.27	30	307.2→220
Fludioxinil	248.19	-45	247.0→180.0
Furathiocarb	382.48	30	383.2→195.1
Hexaconazole	314.21	35	314.0→ 70.2
Imazalil	297.18	35	297.1→159.0
Imidacloprid	255.65	25	256.1→175.0
Ipconazole	333.86	35	334.1→70.2
Iprovalicarb	320.43	24	321.2→119.0
Kresoxim-methyl	313.35	20	314.1→116.0
Mepanipyrim	223.28	30	224.4→ 77.3
Metalaxyl	279.34	25	280.1→220.1
Methamidophos	141.13	22	142.0→ 94.0

Methomyl	162.21	20	163.0→ 88.0
Methoxyfenozide	368.47	15	369.5→149.0
Mevinphos	224.15	22	225.1→192.8
Myclobutanil	288.78	35	289.1→ 70.2
Omethoate	213.14	20	214.1→183.0
Oxadixyl	278.31	20	279.1→219.1
Piperonyl butoxide	338.45	17	356.2→177.0
Prochloraz	376.67	20	376.1→308.0
Propamocarb	188.27	30	189.1→102.1
Propargite	350.48	20	368.1→231.0
Propiconazole	342.22	35	342.0→159.0
Propoxur	209.24	20	210.0→111.0
Pyraclostrobin	387.83	23	388.0→194.0
Pyridaben	364.94	22	365.3→309.1
Pyrimethanil	199.25	40	200.1→107.0
Quinoxyfen	308.14	50	307.8→196.8
Rotenone	394.42	40	395.3→213.2
Simazine	201.66	30	202.2→131.4
Spinosyn A	731.97	40	732.6→142.2
Spinosyn D	746.00	30	746.6→142.2
Spiroxamine	297.48	30	298.2→144.0
Tebuconazole	307.82	30	308.2→ 70.2
Thiabendazole	201.25	35	202.0→175.0
Triadmimefon	293.75	30	294.0→197.1
Trifloxystrobin	408.38	25	409.0→186.0
Triflumizole	345.75	20	346.0→278.1
Vamidothion	287.34	20	288.1→146.0
Zoxamide	336.54	35	336.0→187.0

Schematic Diagram of Sample Preparation Steps

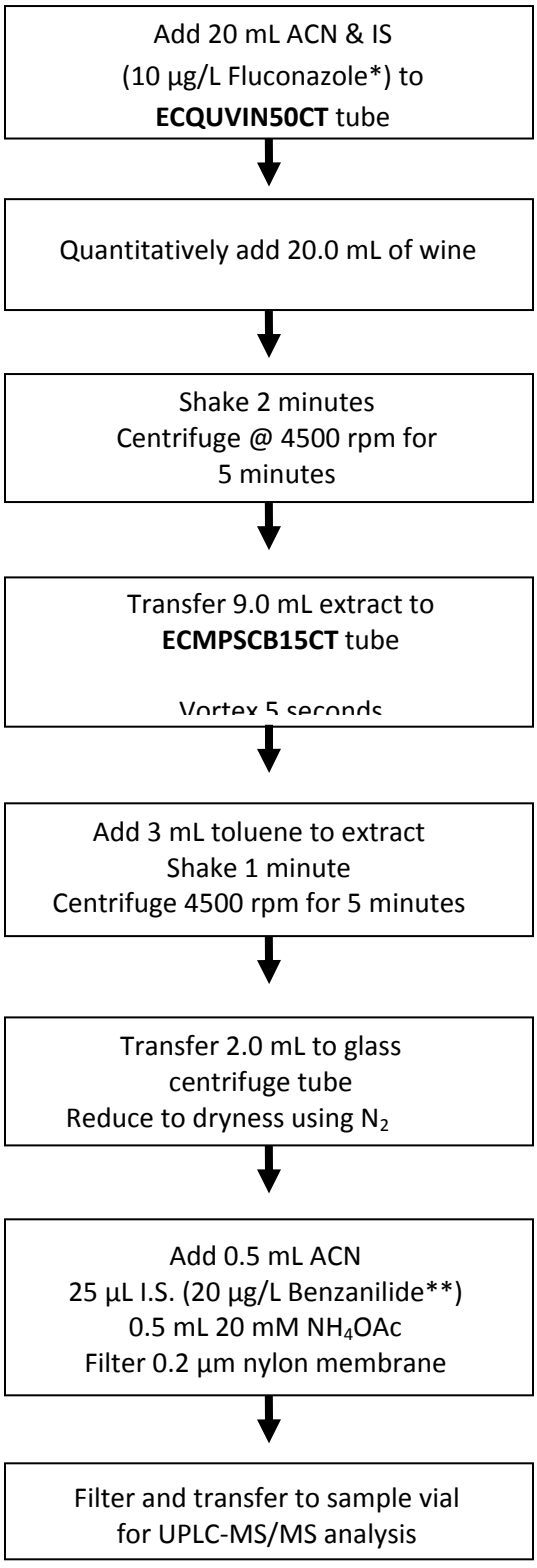

Table of Average Pesticide Recoveries at 100 g/L Spike

(average values with SD, n=4)

	Pesticide Recovery	
	Red Wine @ 100 µg/L	White Wine @ 100 µg/L
Acephate	84±4	79±3
Acetamiprid	83±8	97±7
Acibenzolar S-methyl	80±15	45±5
Aldicarb	92±5	82±5
Aldicarb sulfone	91±7	83±4
Aldicarb sulfoxide	83±8	80±1
Atrazine	92±5	83±5
Avermectin B_{1b}	94±12	107±13
Avermectin B_{1a}	82±8	80±6
Azoxystrobin	93±5	86±4
Benalaxyl	92±5	84±4
Benfuracarb	ND	ND
Benzanilide	69±7	70±8
Bifenazate	86±4	86±11
Bitertanol	92±5	86±4
Buprofezin	91±4	88±6
Carbaryl	77±4	76±4
Carbendazim	126±7	106±7
Carbofuran	90±4	86±4
Chloroxuron	75±5	72±2
Cyprodinil	38±2	56±5
Cyromazine	89±6	83±5
Diclobutrazol	89±6	82±4
Dimethoate	88±7	84±4
Dimethomorph	95±5	85±4
Dimoxystrobin	85±5	74±6
Dinotefuran	88±4	78±5
Diuron	74±12	90±1
Ethofumesate	92±10	95±14
Famoxadone	87±3	86±5
Fenamidone	88±5	80±5
Fenbuconazole	133±21	90±11
Fenhexamid	91±5	83±4
Fenpropimorph	86±6	84±4
Fluconazole	112±4	101±2
Fludioxinil	91±2	87±8
Furathiocarb	81±4	77±4
Hexaconazole	90±2	77±7
Imazalil	89±5	83±5
Imidacloprid	94±6	87±4
Ipconazole	89±5	83±5
Iprovalicarb	94±6	87±4
Kresoxim-methyl	85±5	86±5
Mepanipyrim	76±6	94±12
Metalaxyl	94±5	85±5
Methamidophos	82±6	74±5

Methomyl	90±4	81±4
Methoxyfenozide	102±5	89±5
Mevinphos	84±5	71±4
Myclobutanil	96±8	90±4
Omethoate	82±4	75±4
Oxadixyl	94±3	88±4
Piperonyl butoxide	94±5	87±4
Prochloraz	84±3	84±5
Propamocarb	80±3	80±5
Propargite	93±6	86±2
Propiconazole	94±4	86±5
Propoxur	89±5	82±4
Pyraclostrobin	77±6	76±4
Pyridaben	85±4	83±4
Pyrimethanil	79±6	75±4
Quinoxyfen	70±5	68±3
Rotenone	81±3	85±9
Simazine	85±9	88±7
Spinosyn A	88±7	83±4
Spinosyn D	87±4	80±3
Spiroxamine	92±5	84±4
Tebuconazole	90±4	83±5
Thiabendazole	71±3	75±5
Triadmimefon	89±8	84±7
Trifloxystrobin	90±8	84±4
Triflumizole	88±6	86±3
Vamidothion	86±4	83±6
Zoxamide	86±4	80±4

*Adapted from Kai Zhang, Jon W. Wong et al, Multiresidue Pesticide Analysis of Wines by Dispersive Solid-phase Extraction and Ultra-High Performance Liquid Chromatography-Tandem Mass Spectrometry *Journal of Agricultural and Food Chemistry*

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Analysis of Anthelmintics in Animal Tissue Using QuEChERS and LC/MS/MS

1) Extraction

a) To 10 g of homogenized/hydrated sample in a 50 mL centrifuge tube add 10 mL acetonitrile
b) Add internal standard (Cyprodinil + 2,4D)
c) Shake for 1 minute
d) Add contents of ECMSSC50CT-MP pouch (4 g of anhydrous magnesium sulfate and 1 g sodium chloride) to the centrifuge tube
e) Immediately shake for 1 minute
f) Centrifuge for 5 minutes at 3450 rcf

2) Sample Clean-Up

a) Add an aliquot of supernatant from (step 1 f) to product ECMSC1850CT (50 mL centrifuge tube containing 1500 mg anhydrous magnesium sulfate and 500 mg C18)
b) Shake for 1 minute
c) Centrifuge for 1 minute at 3450 rcf

3) Analysis

a) Place 0.5 mL into auto sampler vial
b) Add QC spike (TPP)
c) Inject onto LC-MS/MS
d) Use mode ESI + or ESI- depending upon specific analyte of interest

39 Flukicides/Anthelmintics

ESI+		ESI-
IS Triphenylphosphate	**QC Spike** Cyprodinil	**IS** 2,4D
Abamectin	Albendazole	Bithionol
Doramectin	Albendazole sulfoxide	Clorsulon
Emamectin	Albendazole sulfone	Closantel
Eprinomectin	Albendazole amino sufone	Niclosamide
Moxidectin	Cambendazole	Nitroxynil
Lyermectin	Flubendazole	Oxyclozanide
Selemectin	Flubendazole, amino	Rafoxanide
Diclorvos	Flubendazole, hydroxy	Triclabendazole
Coumaphos	Fenbendazole	
Coumaphos-Oxon	Fenbendazole sulfone	
Haloxon	Oxfendazole	
Morantel	Mebendazole	
Levemisole	Mebendazole, amino	
	Mebendazole, hydroxy	
	Oxibendazole	
	Thiabendazole	
	Thiabendazole, 5-hydroxy	
	Triclabendazole	
	Triacleabendazole suldxide	

Adapted from Kinsella, Lehotay et al, "New method for the Analysis of Anthelmintics in Animal Tissue"

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Antibiotics in Beef or Serum by QuEChERS

Part Number: ENVIRO-CLEAN® ECMSC1850CT

November 23, 2009

This is a streamlined sample preparation method for the analysis of several classes of antibiotics in beef, kidney juice or serum

1) Extraction

a) Weigh 1 g of homogenized beef kidney sample, kidney juice or serum in a 50 mL FEP (fluorinated ethylene propylene) tube or disposable polypropylene tube

b) Add 100 µL of 1 µg/mL composite internal standard solution of ^{13}C-sulfamethazine (to compensate for volume change), penicillin-V and cefadroxil (for method performance) in water

c) Add 2 mL water

d) Add 8 mL acetonitrile

e) Shake for 5 minutes

f) Centrifuge at 3450 rcf for 5 minutes

2) Clean-Up

d) Transfer the supernatant into a 50 mL tube with 500 mg C18 (ECMSC1850CT) (50 mL centrifuge tube containing 1500 mg anhydrous magnesium sulfate and 500 mg C18)

e) Shake for 30 seconds

f) Centrifuge at 3450 rcf for 1 minute

g) Place 5 mL aliquot of the supernatant into a graduated tube

h) Evaporate down to < 1 mL

i) Bring volume to 1 mL with reagent water

j) Transfer the extract into vials by filtering through PVDF 0.45 m membrane filter syringes

k) Sample is now ready for analysis by LC-MS/MS

Table of some antibiotics that were analyzed using this procedure

Sulfonamides	Macrolides	Fluoroquinolones	Tetracyclines
sulfathiazole	erythromycin	ciprofloxacin	oxytetracycline
sulfamethazine	lincomycin	danofloflaxin	tetracycline
sulfachloropyridazine	tytosin	difloxacin	
sulfadoxine		orbifloxacin	
sulfamethazole		sarafloxacin	
sulfadimethoxine			

B-Lactams			
amoxicillin	ampicillian	cefadroxil	cefezolin
cloxacillin	DCCD	dicloxacillin	oxacillin
nafcillin	Penicillin G	Penicillin V	

CCD desfuroylcenftiofur cysteine disulfide

* adapted from work done by Kate Mastovska at USDA

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Optimized QuEChERS Method For Acrylamide Analysis*

UCT Products: ECMSSC50CT-MP
CUMPS2CT

October 21, 2009

1) Extraction

a) Add 1.0 gram of sample to a 50 mL centrifuge tube

b) Add contents of ECMSSC50CT-MP pouch (4g $MgSO_4$ and 1g NaCl)

c) Add 500 ng/g d_3-acrylamide to the tube

d) Add 5 mL of hexane

e) Vortex for 1 minute

f) Add 10 mL of reagent water and 10 mL of acetonitrile

g) Shake vigorously for 1 minute

h) Centrifuge for 5 minutes at 3450 rcf

2) Clean-Up

a) Discard the hexane top layer

b) Add 1 mL of the acetonitrile layer to a CUMPS2CT tube (150 mg $MgSO_4$, 50 mg PSA)

c) Mix for 30 seconds

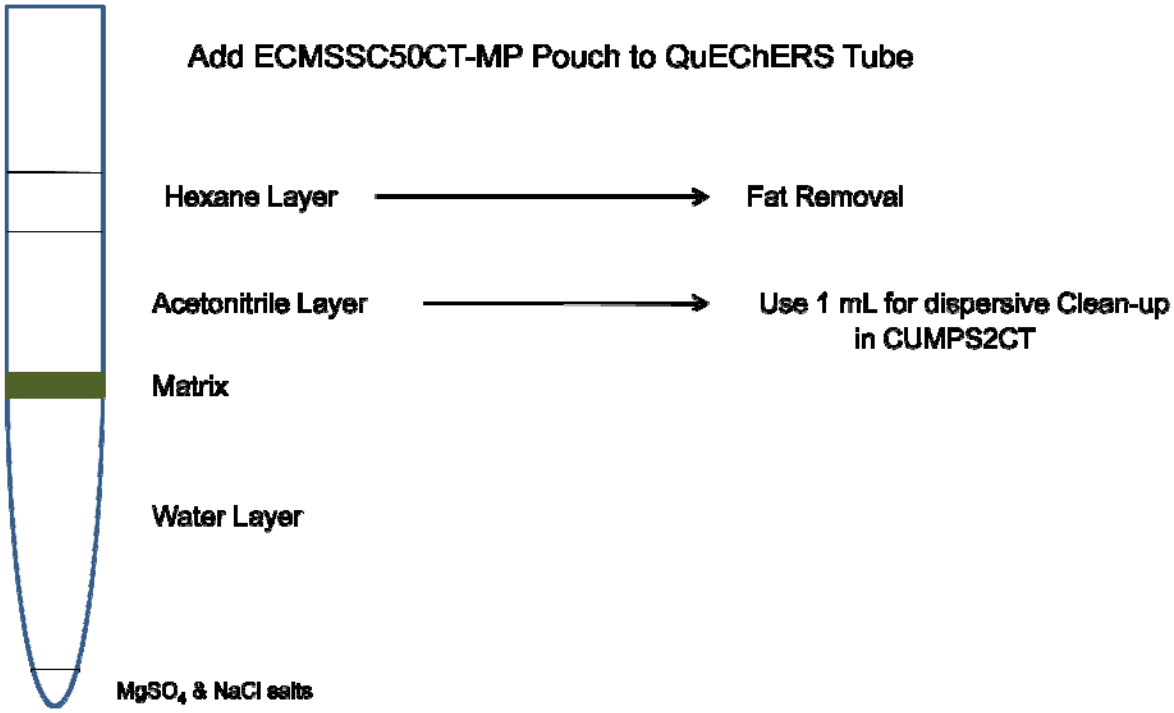
d) Centrifuge at 3450 rcf for 1 minute

e) Transfer liquid portion to an injection vial

3) Analysis

a) Inject 5-10 µL into an LC/MS/MS

Acrylamide Extraction Step



*Adapted from Kate Mastovska, USDA-ARS

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Trichothecene Type A & B Analysis in Wheat and Corn Using the QuEChERS Approach*

Part Number:
ECMSSC50CT-MP (50 mL centrifuge tube, 4 g anhydrous magnesium sulfate, 1 g NaCl)
CUMPS2CT (150 mg anhydrous magnesium sulfate and 50 mg PSA
January 22, 2010

Introduction

An extraction and purification method for the simultaneous LC-MS determination of five mycotoxins is described including three type A, diacetoxyscirpenol (DAS), T-2 toxin and HT-2 toxin, and two type B trichothecenes, deoxynivalenol (DON) and nivalenol (NIV). The analysis has been optimized using a modified QuEChERS approach. These mycotoxins are responsible for a wide range of disorders in animals. They have been found to inhibit proteins synthesis and to have immunosuppressive and cytotoxic effects. Health risks associated with human exposure to *Fusarium* toxins are recognized worldwide and depend on concentration in a particular diet. The major dietary sources of trichothecenes are cereal products wheat and corn.

Procedure

1) Sample Preparation

a) Thoroughly homogenize a sample of grain products using a laboratory mill
b) Weigh 5 g of sample into the 50 ml centrifuge tube
c) Add 10 mL of methanol:acetonitrile (85:15) into 50 mL centrifuge tube
d) Shake to disperse solvent
e) Add the contents of the **ECMSSC50CT-MP** pouch containing 4 g anhydrous magnesium sulfate, 1 g sodium chloride to the centrifuge tube
f) Vortex for 1 minute then centrifuge @ 4,000 rpm for 10 minutes

2) Sample Cleanup

a) Transfer a 1 mL aliquot to a 2 mL **CUMPS2CT** tube (150 mg anhydrous magnesium sulfate and 50 mg PSA)
b) Shake for 1 minute
c) Centrifuge for 10 minutes @ 4,000 rpm
d) Filter extract through a 0.45 µm filter into an LC injection vial if supernatant is not clear
e) Sample is now ready for analysis

3) Analysis

a) MSD detection with atmospheric pressure ionization (API) configured for electrospray positive ion mode

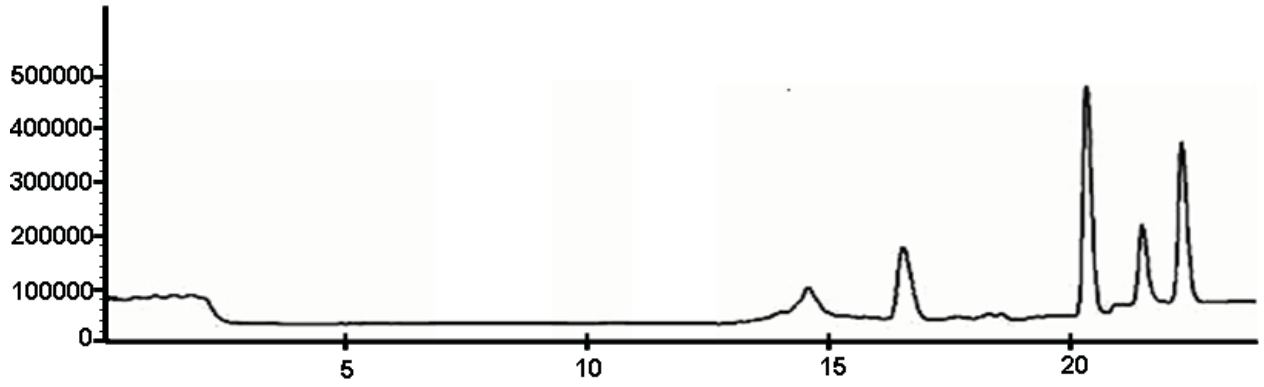
b) Analytical column: Luna C18 (250mm x 4.6 mm x 5 µm) or equivalent may be used but may change elution times

c) Mobile phase A: 1% formic acid in water, B: 1% formic acid in methanol

d) Gradient, Flow 0.5 mL/minute, Initial 40%B, 10 minutes 90% B until 25 minutes

Mass Ions for Mycotoxins [Na+M]	
Ion	**M/Z**
NIV	355
DON	319
DAS	389
HT2	447
T2	489

Chromatogram Showing Elution of Mycotoxins
Peaks in order of elution: NIV, DON, DAS, HT-2, T-2



*Modified from Sospedra et al, "Use of the Modified Quick, Easy, Cheap, Effective, Rugged and Safe Sample Preparation Approach for the Simultaneous Analysis of Type A and B Trichothecenes in Wheat Flour," J of Chromatography A

DCN-102201-182

CLEAN-UP OF ORGANOCHLORINE PESTICIDES AND PCB EXTRACTS USING FLORISIL®

Part #: EUFLSA1M6 or EUFLS1M6

February 3, 2009

This application is designed to remove polar interferences from organochlorine pesticide and PCB extracts in hexane prior to analysis.

REAGENTS:

Hexane
Acetone

Product Description:

EUFLSA1M6 – 1000 mg small particle Grade A Florisil® for slower gravity flow.
EUFLS1M6 – 1000 mg regular particle PR Grade Florisil® for more viscous samples.

PROCEDURE:

1. Prerinse a column with 9 mL of 90:10 hexane/acetone by gravity. (A low vacuum may be necessary to start flow)
2. Discard solvent.
3. Add a collection tube under the column.
4. Add a 2 mL aliquot of the sample extract (in hexane) to the column.
5. Collect extract by gravity.
6. Add 9 mL of 90:10 hexane/acetone to the column.
7. Continue to collect by gravity.
8. Gently evaporate the extract to a volume of 1 mL.
9. Bring to a final volume of 2 mL with hexane.

Florisil® is a registered trademark of U.S. Silica.

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FRACTIONATION OF ALIPHATIC AND AROMATIC HYDROCARBONS USING ENVIRO-CLEAN® TPH SILICA

(Developed in cooperation with Lancaster Laboratories, Inc.)

Part #: XRSIHT13M15

February 3, 2009

Background:

The composition of petroleum is a complex mixture of hundreds of different hydrocarbon compounds. The resultant makeup of hydrocarbons released into the environment is variable and dependent on the conditions to which it is subsequently exposed. While in the environment, petroleum composition is influenced by a number of factors including volatilization, leaching and/or biological degradation. These environmental effects yield a mixture whose toxicological properties can be vastly different than the parent product. Based on the known toxicological properties of petroleum products we can assume that:

- aromatic compounds are more toxic than aliphatic compounds
- the toxicity of aliphatic compounds is dependent upon their molecular weight with low molecular weight compounds showing relatively higher toxicity

The fractionation of the total petroleum hydrocarbon extract is necessary to determine the concentration of the aliphatic versus aromatic compounds. The Massachusetts Department of Environmental Protection (MADEP) has taken the approach of fractionating the C9-C18 aliphatics (n-nonane to n-octadecane), C19-C36 aliphatics (n-nonadecane to hexatriacontane), and the C11-C22 aromatics (naphthalene to benzo(g,h,i)perylene). These compounds are associated with the release of hydrocarbons in the environment. The aromatics are considered the most toxic form of hydrocarbon.

Procedure:

Prepare Extract

1. Solvent exchange the hydrocarbon extract from methylene chloride to hexane using a K-D apparatus.

Prepare Cartridge

2. Thoroughly rinse cartridge with two, 10 mL aliquots of pentane.

3. Add 1 mL of the extract to the cartridge.

4. Elute aliphatic fraction with pentane by gravity and collect everything in an ampoule. A total of 10 mL should be collected.

5. Place a fresh ampoule under the cartridge and elute the aromatic fraction with methylene chloride by gravity. A total of 10 mL should be collected.

6. Concentrate each fraction separately to a final volume on a steam bath using an ampoule and micro-Snyder column combination. Other techniques may be used but the loss of C9-C18 hydrocarbons may result.

It is very important to keep the silica cartridges dry and away from room air prior to use. Moisture and contaminants in the air will reduce the effectiveness of the silica and may cause contamination of the extract. Pre-rinsing the cartridges with acetone may reduce this problem.

Results:

Classification	Range	Percent Recovery
Aromatics	**C11-C22**	**88**
surrogates	2-fluorobiphenyl	**123**
surrogates	o-terphenyl	**100**
Aliphatics	**C9-C18**	**85**
	C19-C36	**89**
surrogates	1-chlorooctadecane	**58**

MA EPH DATA from Lancaster Labs

UCT in cooperation with Lancaster Laboratories, Inc., has developed a fractionation product that provides consistent and accurate results free from contamination

DCN-903020-124

1. **Post Sample Extraction**
   c) Place 4 grams of copper beads in a glass vial
   d) Add 2 mL of liquid sample extract to the vial

2. **Sulfur Removal**
   d) Seal the glass vial and mix sample with copper beads for 2 minutes
   e) Allow to stand for approximately 10 minutes
   f) If sample contains high levels of sulfur, repeat process with 4 grams of fresh copper beads

   Note: For the analysis of PCB type analytes, copper may reside in the extract

3. **Analysis, GC/MS or LC/MS**
   d) Transfer clean extract to autosampler vial
   e) Inject 1-2 µL for GC
   f) Inject 5-10 µL for LC

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