# **Applications**

## **SPE Applications**



#### Hypericins from St. John's Wort



#### Procedure Using GracePure™ C18-Max, 1000mg:

Sample Treatment – Pulverize 300mg St. John's Wort powder into five, 3mL aliquots of methanol:water (80:20). Combine and filter extracts to produce 15mL filtrate. Dilute to 30mL with water.

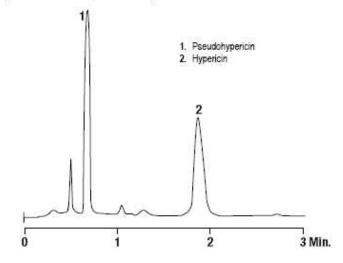
Conditioning – Rinse device with 5mL methanol followed by 5mL deionized water.

Sample Application – Apply 2mL filtrate to the top of the SPE device and draw through the C18 packed bed.

Wash\* - Wash device with 3mL of deionized water.

Elution - Elute with 2mL methanol.

\*Repeat load and wash steps consecutively until filtrate is consumed.



Column: Alltima™ C18, 3µm 53 x 7mm Rocket™ HPLC Column, (Part No. 50605)

Mobile Phase: MeOH:0.2% HsPO4 (95:5)

Flow Rate: 4.0mL/min Detector: VIS at 585nm

#### Phenolic Acids from Echinacea

CHROM 9110

#### Procedure Using GracePure™ C18-Max, 1000mg:

Sample Treatment – Pulverize 300mg echinacea powder into five, 3mL aliquots of methanol. Combine and filter extracts to produce 15mL filtrate. Dilute to 30mL with water.

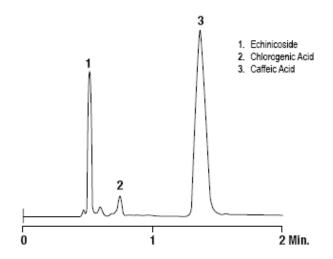
Conditioning – Rinse device with 5mL methanol followed by 5mL deionized water.

Sample Application – Apply 2mL filtrate to the top of the SPE device and draw through the C18 packed bed.

Wash\* - Wash device with 2mL of deionized water.

Elution - Elute with 5mL of methanol:water (50:50).

\*Repeat load and wash steps consecutively until filtrate is consumed.



Column: Alltima™ C18, 3µm 53 x 7mm Rocket™ HPLC Column, (Part No. 50605)

Mobile Phase: ACN:10mM K2HPO4, pH 2.6 (20:80)

Flow Rate: 3.5mL/min Detector: UV at 330nm



#### Sedatives from Kava Kava

(CHROM-9125)

#### Procedure using GracePure™ C18-Max, 1000mg:

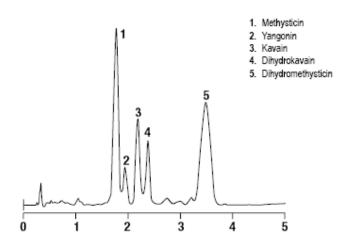
Sample Treatment – Pulverize 300mg commercial kava kava root powder into four, 2mL aliquots of methanol:water (80:20). Combine and filter extracts to produce 8mL filtrate. Dilute to 10mL with water.

Conditioning – Rinse device with 5mL methanol followed by 5mL water. Sample Application\* – Apply 2mL filtrate.

Wash\* - Wash with 2mL water.

Elution - Elute with 3mL methanol.

\*Before elution step, repeat load and wash steps until filtrate is consumed.



Column: Alltima™ C18, 3µm, 53 x 7mm Rocket™ HPLC Column (Part No. 50605)

Mobile Phase: ACN:IPA:0.2% Acetic Acid (17:23:60)

Flow Rate: 3.5mL/min
Detector: UV 220nm
Inj. Vol.: 5µL

#### Antioxidants from Chamomile

CHROM 9133

#### Procedure Using GracePure™ C18-Max, 1000mg:

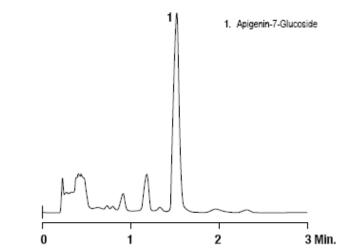
Sample Treatment – Pulverize 1g commercial chamomile tea grounds into 6mL dioxane:methanol (50:50). Filter extract and dilute to 20mL with water.

Conditioning – Rinse device with 5mL methanol followed by 5mL water. Sample Application\* – Apply 1mL filtrate.

Wash\* - Wash with 1mL water.

Elution – Elute first with 2mL methanol:water (50:50). Elute second, fraction and use for this analysis, with 2mL methanol.

\*Before elution step, repeat load and wash steps until filtrate is consumed.



Column: Alltima™ C18, 3µm 53 x 7mm Rocket™ HPLC Column, (Part No. 50605)

Mobile Phase: ACN:20mM K<sub>2</sub>HPO<sub>4</sub>, pH 7.3 (65:35)

Flow Rate: 3mL/min Detector: UV 340nm Inj. Vol.: 5µL

#### Vasodilators from Dong Quai

CHROM-9132

#### Procedure using GracePure™ C18-Max, 1000mg:

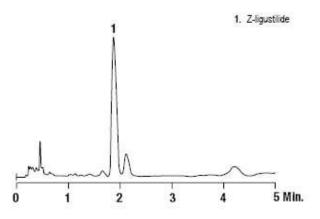
Sample Treatment – Pulverize 1500mg commercial dong quai root powder into three, 5mL aliquots of methanol. Combine and filter extracts to produce 15mL filtrate. Dilute to 30mL with water.

Conditioning – Rinse device with 5mL methanol followed by 5mL water. Sample Application\* – Apply 1mL filtrate.

Wash\* - Wash with 2mL water.

Elution - Elute with 2mL methanol.

\*Before elution step, repeat load and wash steps until filtrate is consumed.



Column: Alltima\*\* C18, 3µm 53 x 7mm Rocket\*\* HPLC Column (Part No. 50605)

Mobile Phase: Methanol: 0.2% Acetic Acid (65:35)

Flow Rate: 4.5mL/min Detector: UV 270nm Inj. Vol.: 5µL

# **Applications - Pharma**

#### Benzodiazepines from Human Plasma

CHROM 10606 10608

#### Procedure using GracePure™ C18-Aq, 500mg:

Sample Treatment – Spike 500μL plasma with 10μL of a 0.1mg/mL standard solution. Combine with 100μL of 0.1M sodium carbonate buffer. Vortex and centrifuge for 10 minutes.

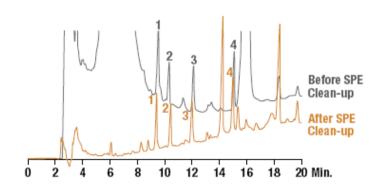
Conditioning - Rinse device 2mL water.

Sample Application - Apply entire sample.

Wash - Wash with 2mL water and then 50µL methanol.

Elution - Elute with 600µL methanol.

- Oxazepam
- Clonazepam
- 3. Temazepam
- 4. Diazepam



Column: Alltima™ HP C18, 5µm, 250 x 4.6mm HPLC Column (Part No. 87680)

Mobile Phase: A: Water B: Acetonitrile Gradient: (Time, %B): (0,30), (30,80)

Flow Rate: 1mL/min
Detector: UV at 254nm
Temperature: Ambient



# **Applications - Pharma**

#### Diuretics from Urine

CHROM 10289 10290

#### Procedure using GracePure™ C18-Max, 1000mg:

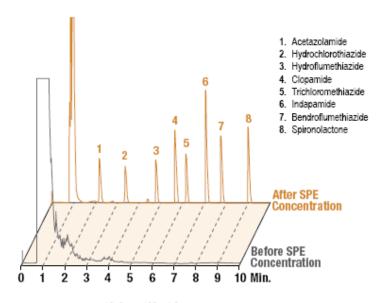
Sample Treatment – Spike synthetic urine with 8 diuretics to a concentration of 1.25µg/mL each.

Conditioning - Rinse device 5mL methanol, followed by 5mL water.

Sample Application - Apply 15mL spiked urine sample at 1mL/min.

Wash - Wash with 5mL water.

Elution – Elute with 2mL methanol. Evaporate solvent and reconstitute in 250 $\mu$ L HPLC mobile phase.



Column: Alltima" C18, 3µm, 100 x 4.6mm HPLC Column (Part No. 81382)

Mobile Phase: A: 25mM Ammonium Acetate, 0.1%TFA B: Acetonitrile, 0.1%TFA

Gradient: (Time, %B): (0,20,), (10,90)

Flow Rate: 1.0mL/min Detector: ELSD



# Applications – Food/Ag

#### Fungicides from Red Wine

CHROM 10637 10638

#### Procedure using GracePure™ C18-Max, 500mg:

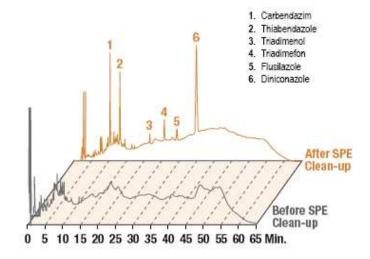
Sample Treatment – Add 0.167mg/mL each of carbendazim, thiabendazolem, triadimenol, triadimefon, flusilazole, diniconazole into 1mL water. Combine with 1mL Beaujolais red wine.

Conditioning - Rinse device with 3mL methanol followed by 3mL water.

Sample Application - Apply 2mL red wine mixture.

Wash - No wash.

Elution - Elute with 3mL methanol.



Column: Alltima" HP C18 Amide, 5µm, 250 x 4.6mm HPLC Column (Part No. 87734)

Mobile Phase: A: Water B: Acetonitrile

Gradient: (Time, %B): (0,15%), (15,45%), (50,45%), (65,15%)

Flow Rate: 1mL/min Detector: UV at 254nm



# **Applications – Food-Ag**

#### Preservatives from Fruit Punch

CHROM 2576

#### Procedure using GracePure™ Anion-X, 500mg:

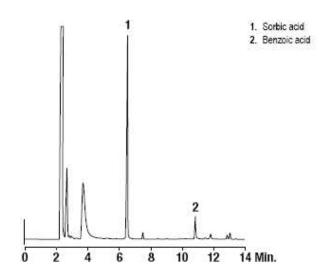
Sample Treatment – Use 8mL fruit punch and adjust pH to 10 using potassium hydroxide.

Conditioning - Rinse device 10mL water.

Sample Application - Apply 8mL pH adjusted fruit punch sample.

Wash - Wash with 20mL water.

Elution - Elute with 1mL 1.0N hydrochloric acid followed by 1mL methanol.



Column: Heliflex® AT® AquaWax-DA, 30m x 0.25mm x 0.25µm Capillary GC Column

(Part No. 14537)

Temp: 200°C (5 min hold) to 230°C (4 min hold) at 5°C/min

Carrier: Helium at 0.75mL/min (25cm/sec)

Detector: FID at 250°C



## **Applications - Environmental**

#### Nitroaromatics and Naphthols from Soil

CHROM 10597 10598

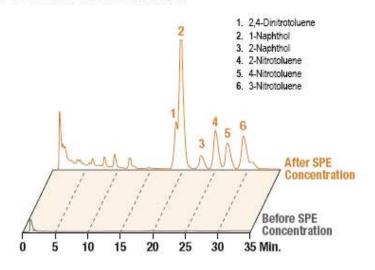
#### Procedure using GracePure™ C18-Max, 500mg:

Sample Treatment – Spike 100g soil with 7.5μg/g of each analyte. Combine soil with 1000mL deionized water, shake for 10 minutes, and filter.

Conditioning – Rinse device with 5mL methanol followed by 5mL water. Sample Application – Aspirate 1000mL water sample through SPE at flow rate 1-5mL/min.

Wash - No wash. Air dry for 15 seconds.

Elution – Elute with three 1mL aliquots of methanol:water (50:50). Air dry for 15 seconds between each elution.



Column: Adsorbosphere\* UHS C18, 5µm, 150 x 4.6mm HPLC Column (Part No. 288118)

Mobile Phase: Methanol:Water (50:50)

Flow Rate: 1.0mL/min
Detector: UV at 254nm
Temperature: Ambient



# **Applications - Environmental**

#### Chlorinated Pesticides from Water

CHROM-1668

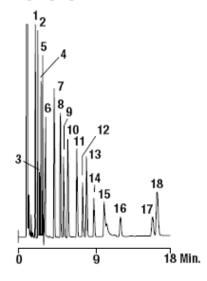
#### Procedure Using GracePure™ C18-Fast, 1000mg:

Conditioning – Rinse device with 5mL of methanol followed by 5mL deionized water.

Sample Application – Pass 100mL–500mL (containing 1% methanol) of water sample through the device at 20mL/minute.

Wash – Wash device with 10mL of deionized water then 10mL of methanol:deionized water (20:80). Remove excess by passing air through the device for two minutes.

Elution – Elute with 2mL of hexane:ethyl acetate (70:30). Pass extract through 2g–3g sodium sulfate to remove residual water.



#### Recovery (as % Standard)

- α-BHC (104%)
- 2. Lindane (109%)
- 3. β-BHC (106%)
- 4. Heptachlor (92%)
- 5. ō-BHC (110%)
- 6. Aldrin (98%)
- 7. Heptachlor Epoxide (107%)
- 8. α-Endosulfan (105%)
- p,p'-DDE (79%)
- 10. Dieldrin (102%)
- 11. Endrin (104%)
- 12. p,p'-DDD (92%)
- 13. β-Endosulfan (103%)
- 14. p,p'-DDT
- 15. Endrin Aldehyde (102%)
- 16. Endosulfan Sulfate (103%)
- 17. Methoxychlor (99%)
- 18. Endrin Ketone (104%)

Column: AT\*-Pesticide 20m x 0.53mm x 0.60µm Capillary GC Column, (Part No. 16846)

Temperature: 210°C Carrier Gas: Helium, 35cm/sec

Detector: ECD



## Polyaromatic Hydrocarbos (PAH's) in Water

#### **SPE Conditions**

Procedure using GracePure™ C18-Max SF tube, 500mg/6mL (part #5138767)

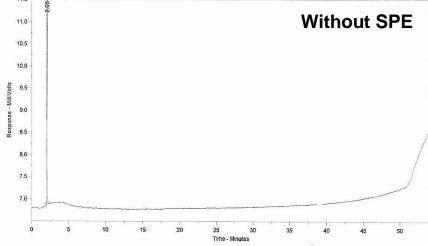
Sample Treatment-500mL of water and add 75mL of 2-propanol, spiked with 20uL of PAH mixture at 2000ug/mL of each PAH, which corresponds to 40ug/500mL water, o 80ppb of each PAH.

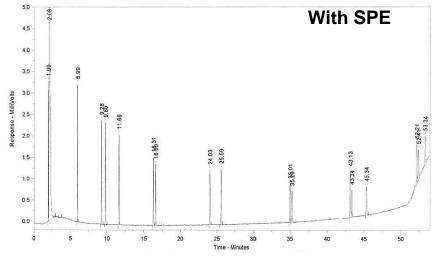
Conditioning-2 X 5mL of methanol, then 5m of 85% water/15% 2-propanol.

Sample Application-Attach 75mL reservoir (part #210575) with a syringe adapter (part #210705)

and apply entire sample. Dry columns for 3 minutes at full vacuum.

Elution-Elute PAHs with 3 X 1mL CH2Cl2 and adjusted the volume to 1mL by evaporating under a gentle nitrogen stream.





Compound (PAHs from water)	PAH Standard (20uL Restedk standard/ 1mL CH2CI2)	12:51 run Run#1	% Recovery
Naphthalene	7243	6277	86.66%
Acenaphthylene	7277	6948	95.48%
Acenaphthene	7285	7110	97.60%
Fluorene	7304	7098	97.18%
Phenanthrene	7286	7109	97.57%
Anthracene	6786	6518	96.05%
Fluoranthene	6928	6751	97.45%
Pyrene	7295	7385	101.23%
Benzo(a)anthracene	7263	5389	74.20%
Chrysene	7331	4426	60.37%
Benzo(b)fluoranthene	7446	6873	92.30%
Benzo(k)fluoranthene	7542	4250	56.35%
Benzo(a)pyrene	7273	4548	62.53%
Indeno(1,2,3-cd)pyrene	10961	5675	51.77%
Dibenzo(a,h)anthracene	8886	4172	46.95%
Benzo(ghi)perylene	11357	3688	32.47%

# SRACE

## GC Conditions

AT-5ms, part #15807, as follows: oven=  $80^{\circ}$ C (1min hold) to  $160^{\circ}$ C at  $25^{\circ}$ C/min, to  $300^{\circ}$ C at  $3^{\circ}$ C/min, to  $325^{\circ}$ C (3 min hold) at  $25^{\circ}$ C/min, injector= $270^{\circ}$ C, FID= $340^{\circ}$ C, split 50:1,  $14.0^{\circ}$ psig, helium,

linear velocity=28cm/sec (at 80°C). 1.0uL injections were done via 7683B autosampler.

## Polyaromatic Hydrocarbos (PAH's) in Soil

#### **SPE Conditions**

Procedure using GracePure™ C18-Max SPE tube, 500mg/6mL (part #5138767)

Sample Treatment-Weighed out 5 grams soil, spike with 80µg of each PAH, add 30mL 2-propanol and homogenize. Filter with 75mL filter tube (part #210775). Bring up to 200mL with deionized water and shake.

Conditioning-2 X 5mL of methanol, then 5mL of 85% water/15% 2-propanol.

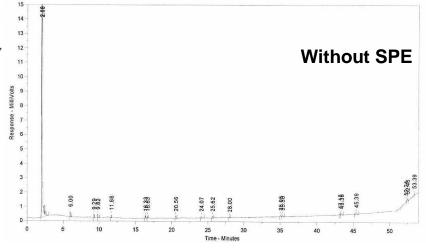
Sample Application-Attach 75mL reservoir (part #210575) with a syringe adapter (part #210705) and apply entire sample.

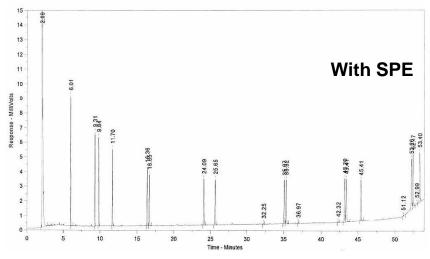
Wash-2 X 500uL of 85% water/15% 2-propanol. Dry columns for 10 minutes at full vacuum.

Elution-Elute PAHs with 2 X 1mL CH2Cl2 and adjusted the volume to 1mL by evaporating under a gentle nitrogen stream.

#### **GC Conditions**

AT-5ms, part #15807, as follows: oven=  $80^{\circ}$ C (1min hold) to  $160^{\circ}$ C at  $25^{\circ}$ C/min, to  $300^{\circ}$ C at  $3^{\circ}$ C/min, to  $325^{\circ}$ C (3 min hold) at  $25^{\circ}$ C/min, injector= $270^{\circ}$ C, FID= $340^{\circ}$ C, split 50:1, 14.0psig, helium, linear velocity=28cm/sec (at  $80^{\circ}$ C).





Compound (PAHs from water)	Retention Time (min.)	PAH Standard Peak Area (40uL Restedk standard/1mL CH2Cl2)	Soil-40uL PAH in 5grams soil-Peak Area	% recovery
Naphthalene	6.01	17598	16196	92.0%
Acenaphthylene	9.31	18061	17211	95.3%
Acenaphthene	9.84	18180	16687	91.8%
Fluorene	11.70	18483	16537	89.5%
Phenanthrene	16.36	18570	16450	88.6%
Anthracene	16.65	17263	14943	86.6%
Fluoranthene	24.09	17746	15669	88.3%
Pyrene	25.65	18830	16600	88.2%
Benzo(a)anthracene	35.07	18997	16882	88.9%
Chrysene	35.32	18941	17427	92.0%
Benzo(b)fluoranthene	43.20	19502	17976	92.2%
Benzo(k)fluoranthene	43.41	19686	18038	91.6%
Benzo(a)pyrene	45.41	19245	17272	89.7%
Indeno(1,2,3-cd)pyrene	52.26	22230	20893	94.0%
Dibenzo(a,h)anthracene	52.47	20755	19331	93.1%
Benzo(ghi)perylene	53.40	21730	18809	86.6%



## Polyaromatic Hydrocarbos (PAH's) in Cereal

#### **SPE Conditions**

Procedure using GracePure<sup>™</sup> C18-Max SPE tube, 500mg/6mL (part #5138767)

Sample Treatment-Weighed out 5 grams crushed toasted oat cereal, spike with 20µg of each PAH, add 30mL 2-propanol and homogenize. Filter with 75mL filter column (part #210775). Bring up to 200mL with deionized water and shake.

Conditioning-2 X 5mL of methanol, then 5mL of 85% water/15% 2-propanol.

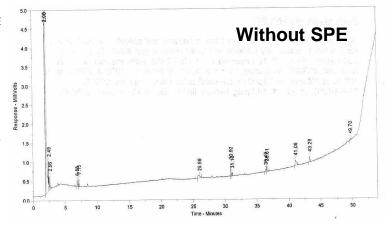
Sample Application-Attach 75mL reservoir (part #210575) with a syringe adapter (part #210705) and apply entire sample.

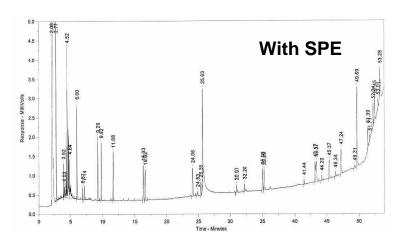
Wash-2 X 500uL of 85% water/15% 2-propanol. Dry columns for 10 minutes at full vacuum.

Elution-Elute PAHs with 2 X 1mL CH2Cl2 and adjusted the volume to 1mL by evaporating under a gentle nitrogen stream.

#### **GC Conditions**

AT-5ms, part #15807, as follows: oven= 80°C (1min hold) to 160°C at 25°C/min, to 300°C at 3°C/min, to 325°C (3 min hold) at 25°C/min, injector=270°C, FID=340°C, split 50:1, 14.0psig, helium, linear velocity=28cm/sec (at 80°C).





Compound	PAH Standard (20uL Restek standard/1mL CH2Cl2)	Cheerios (5- grams) + 10uL Restek PAH standard	%_ Recovery
Naphthalene	8076	(per 0.5mL) 4576	56.7%
•			
Acenaphthylene	8114	4422	54.5%
Acenaphthene	8123	4333	53.3%
Fluorene	8144	3991	49.0%
Phenanthrene	8124	3898	48.0%
Anthracene	7566	3466	45.8%
Fluoranthene	7725	4340	56.2%
Pyrene	8134	12862	158.1%
Benzo(a)anthracene	8098	3079	38.0%
Chrysene	8174	2970	36.3%
Benzo(b)fluoranthene	8302	3117	37.5%
Benzo(k)fluoranthene	8409	5228	62.2%
Benzo(a)pyrene	8109	3137	38.7%
Indeno(1,2,3-cd)pyrene	12222	4634	37.9%
Dibenzo(a,h)anthracene	9908	2998	30.3%
Benzo(ghi)perylene	12663	2031	16.0%



### PCB's in Salmon

#### **SPE Conditions**

Procedure using GracePure<sup>™</sup> C18-Fast SPE tube, 1000mg/6mL (part #5138759)

Sample Treatment-Weigh out 2.5 grams salmon, spike with 2.5µg Aroclor 1242, add 25mL acetonitrile, homogenize for 30 seconds. Filter with 75mL filter tube (part #210775). Rinse salmon again with 5 mL acetonitrile. Bring up to 100mL with deionized water.

Conditioning-Rinse with 6mL hexanes, 6mL acetone, 2 X 6mL methanol, 2 X 6mL water.

Sample Application-Attach 75mL reservoir, apply entire sample.

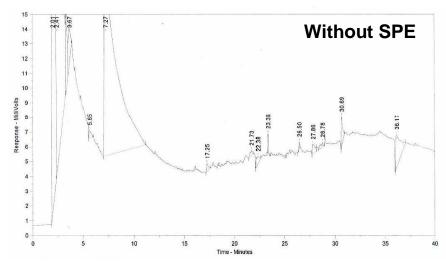
Wash-2 X 5mL water. Dry columns for 10 minutes at full vacuum.

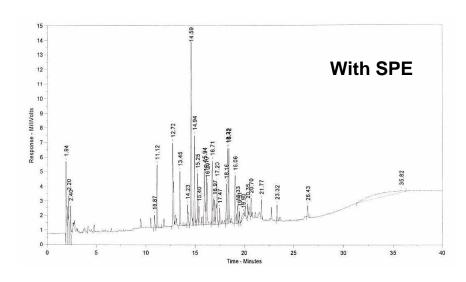
Elution-Elute PCBs with 2 X 3mL 3% toluene/97% n-hexane. Evaporate solvent and reconstitute in 1mL n-hexane.

#### **GC Conditions**

AT-5ms, 30m X 0.25mm ID, 0.25um film, part #15807 Oven=150℃ (2 min)-300℃ (8 minutes) at 5℃/minute Injector=275℃, head pressure=15psig (per EPC-1000), linear velocity=25cm/second, flow=0.74mL/minute, split ratio=64:1, SGE FocusLiner with glass wool.

Detector: ECD=310°C





Retention	Aroclor		
Time	1242 Std.	C18HF	
(min) for	(2.5ug/mL)	(215430)	C18HF
Std.	Peak Area	Extract	Recovery
11.12	17031	2717	16.0%
12.72	31669	12253	38.7%
13.45	17950	7839	43.7%
14.59	57650	38061	66.0%
14.94	25207	16434	65.2%
15.25	13684	8928	65.2%
16.71	17749	15523	87.5%
17.20	18229	16186	88.8%
18.16	10481	10427	99.5%
18.32	20478	20802	101.6%
18.42	21631	21665	100.2%
19.06	17292	17849	103.2%
20.70	4475	5868	131.1%
21.77	4810	7920	164.7%
23.32	5293	4909	92.7%



## **Triazine Herbicides in Water**

#### **SPE Conditions**

Procedure using GracePure™ C18-Fast, 500mg/3mL, part #5138758

Sample Treatment-Spiked 500mL of water spiked with 10ug each triazine herbicide (simazine, atrazine, propazine, terbuthylazine, and cyanazine)..

Conditioning-2 X 2mL acetone, 2 X 2mL methanol, 2 X 2mL water. Column must not run dry!

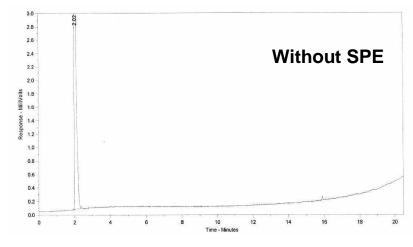
Sample Application- Attached a 75mL reservoir (part #210575) to the SPE tube with a syringe adapter (part # 210705) and aspirate the sample through the tube.

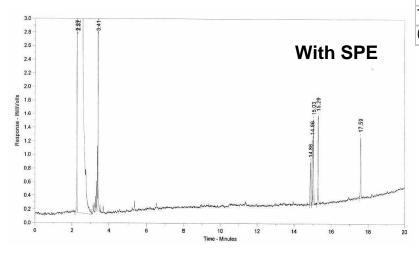
Wash-5mL DI water. Dry column for 25 minutes at full vacuum.

Elution-Elute triazine herbicides with 3 X 1mL acetone. Adjust sample volume to 1mL by evaporating off acetone with a warm nitrogen stream.

#### **GC Conditions**

Injected 1.6uL of above sample onto AT-5ms column, 30m X 0.25mm ID, 0.25µm film thickness (part #15807) under the following conditions: oven=80℃ (0 minutes)-280℃ (0 minutes) at 10℃/min, Inj=250℃, FID=310℃, 14.5psig helium, linear velocity=29cm/sec, flow=1.1ml/minute, split ratio=50:1, SVF=55mL/minute (all per Agilent 6890).





			High Flow	
			C18	
		Standard	Extract	
	Retention Time	<u>Peak</u>	Peak	Percent
Triazine Herbicide	(minutes)	<u>Area</u>	<u>Area</u>	Recovery
Simazine	14.86	2344	1277	54%
Atrazine	14.96	2301	1947	85%
Propazine	15.03	2537	2434	96%
Terbuthylazine	15.29	2606	2694	103%
Cyanazine	17.59	2374	1967	83%



## **Chlorinated Pesticides in Vegetables**

#### **SPE Conditions**

Procedure using GracePure™ C18-Max SPE tube, 500mg/6mL (part #5138767)

Sample Treatment-Homogenize 5 grams green bell pepper with 25mL methanol, spike with 0.5µg each of 16 chlorinated pesticides. Filter with 75mL filter column (part #210775). Dilute to 200mL with DI water and shake.

Conditioning-2 X 5mL methanol, then 5mL DI water.

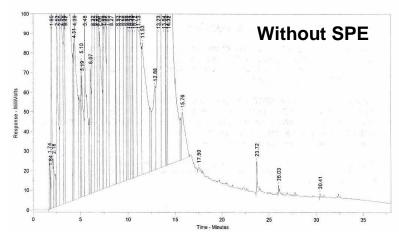
Sample Application-Attach 75mL reservoir (part #210575) with a syringe adapter (part #210705) and apply entire sample.

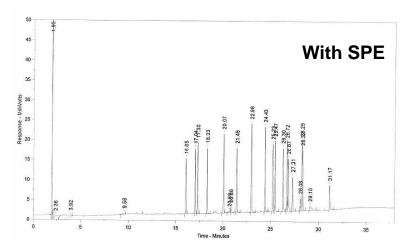
Wash-5mL DI water. Dry column for 10 minutes at full vacuum.

Elution-Elute chlorinated pesticides with 2 X 1mL n-hexane/CH2Cl2 (70:30), evaporated this to dryness and reconstituted with 1mL n-hexane.

#### **GC Conditions**

Capillary column part #15807, AT5ms, 30m X 0.25mm ID, 0.25 $\mu$ m film thickness. Oven=80°C (1min) to 100°C at 15°C/min, to 160°C at 10°C/min, to 275°C at 5°C/min, column head pressure at 14.0psi on the EPC-1000, dead time of 1.91 minutes, linear velocity of 26cm/sec, flow=0.77mL/minute. The split vent flow 50 mL/minute, split ratio of 65:1, injector-250°C, ECD=310.





	Green Pepper Extract				
Peak ID	(0.5ug/5g pepper)		Standard Mix (0.5ng/uL)		
				Peak Area	
	Retention		Retention	per 1uL	Percent
	Time (min)	Peak Area	Time (min)	injection	Recovery
a-BHC	16.05	46063	16.01	88061	52.31%
b-BHC	17.04	59975	17.00	68282	87.83%
g-BHC	17.30	60575	17.26	91262	66.37%
d-BHC	18.33	59755	18.29	75807	78.83%
Heptachlor	20.07	74949	20.03	114932	65.21%
Aldrin	21.46	60142	21.42	91516	65.72%
Heptachlor					
Epoxide	22.98	88131	22.94	101043	87.22%
Endosulfan I	24.43	83820	24.39	95358	87.90%
p,p'-DDE	25.23	67348	25.19	80951	83.20%
Dieldrin	25.47	72783	25.43	84646	85.99%
Endrin	26.30	70607	26.26	101810	69.35%
Endosulfan II	26.72	72973	26.68	88101	82.83%
p,p'-DDD	26.87	56557	26.83	65089	86.89%
Endrin Aldehyde	27.31	37431	27.27	79733	46.95%
p,p'-DDT	28.25	71065	28.20	75198	94.50%
Endosulfan					
Sulfate	28.32	58904	28.28	69970	84.18%



#### **SPE Conditions**

Procedure using GracePure™ C18-Max SPE tube, 500mg/6mL (part #5138767)

Sample Treatment-Weighed out 5 grams dry soil, spike with 1µg each of 16 chlorinated pesticides, add added 25mL methanol and homogenize. Filter with 75mL filter column (part #210775). Dilute to 200mL with DI water and shake.

Conditioning-2 X 5mL methanol, then 5mL D water.

Sample Application-Attach 75mL reservoir (part #210575) with a syringe adapter (part #210705) and apply entire sample.

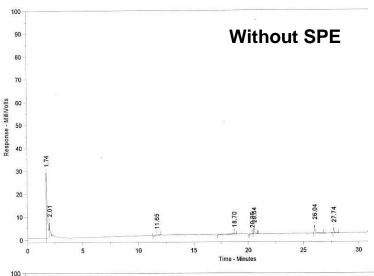
Wash-5mL DI water. Dry column for 10 minutes at full vacuum.

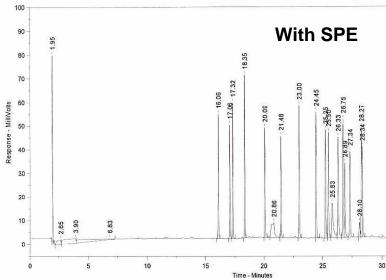
Elution-Elute chlorinated pesticides with 2 X 1mL n-hexane/CH2Cl2 (70:30), evaporate to dryness and reconstitute with 1mL iso-octane.

#### **GC Conditions**

Capillary column part #15807, AT-5ms,  $30m \times 0.25mm$  ID,  $0.25\mu m$  film thickness. Oven= $80^{\circ}C$  (1min) to  $100^{\circ}C$  at  $15^{\circ}C$ /min, to  $160^{\circ}C$  at  $10^{\circ}C$ /min, to  $275^{\circ}C$  at  $5^{\circ}C$ /min, column head pressure at 14.0psi on the EPC-1000, dead time of 1.91 minutes, linear velocity of 26cm/sec, flow=0.77mL/minute. The split vent flow 50 mL/minute, split ratio of 65:1, injector- $250^{\circ}C$ , ECD=310.

## **Chlorinated Pesticides in Soil**





		Soil extract- C18-HC,	Chlorinated	
	Retention	500mg/8mL	Pesticide	
	Time	tube (part	Standard	
Peak ID	(minutes)	#255350)	(1ng/uL)	
				Percent
		Peak Area	Peak Area	Recovery
a-BHC	16.06	158448	286020	55.4%
b-BHC	17.06	165957	194744	85.2%
g-BHC	17.32	183511	287341	63.9%
d-BHC	18.35	221120	256677	86.1%
Heptachlor	20.09	169849	284632	59.7%
Aldrin	21.48	152652	254393	60.0%
Heptachlor Epoxide	23.00	206506	269834	76.5%
Endosulfan I	24.45	202110	258071	78.3%
p,p'-DDE	25.25	170622	235132	72.6%
Dieldrin	25.50	175712	226833	77.5%
Endrin	26.33	195974	207536	94.4%
Endosulfan II	26.75	208941	235161	88.9%
p,p'-DDD	26.89	130127	167842	77.5%
Endrin Aldehyde	27.34	158593	199430	79.5%
p,p'-DDT	28.27	187277	196553	95.3%
Endosulfan Sulfate	28.34	141277	187194	75.5%

