Project No. _____
Book No. 5751 TITLE Extractions of Pavilion Samples

4/20/12 (pod) Samples from Pavilion

		Initial pH	vol HCl added	vol NH4OH	Final pH	10 ng/µl d3-AA	10 ng/µl d4-NP	1 ng/µl d2-OP	10 ng/µl 7 AEOs	10 ng/µl OP/NP	25 ng Neod.
1	Blank	7	0	0	7	50 µl	50 µl	250 µl	25 µl	0	0
2	Water Spike	7	0	0	7					50	40
3	LV12WAT056	7	0	0	7					0	0
4	LV12WAT058	7	0	0	7						
5	LV12WAT059	8	10 µl	0	7						
6	LV12WAT061	7	0	0	7						
7	LV12WAT063 #1	12	400 µl	70 µl	7						
8	LV12WAT063 #2	12	400 µl	70 µl	7						
9	LV12WAT065	7	0	0	7						

All samples = 500 mL

HLB cartridges
Lot # 108A31354B

10 ng/µl d3-AA = 5752-36-2
10 ng/µl AA = 5752-36-2
10 ng/µl d4-NP = Made 4/20/12 from 100 ng/µl d4-NP (5752-29-1)
1 ng/µl d2-OP = Sigma Analytical Std (Fluka S150607 Lot # S2B A018X)
10 ng/µl 7 AEOs = 5752-38-1
10 ng/µl OP/NP = Made 4/20/12 from stock solutions (5752-22-10 and 5752-22-11)
25 ng/µl Neodol = 5752-38-2
10 ng/µl NPEO = 5752-38-3

1) Conditioned Autotraces to remove air bubbles from solvent lines (AH)

2) Ran method "alkylphenol" on old Autotrace and "alkylphenol_b" on New Autotrace.
(Elutes once with 5 mL 2:2:1 MeOH/acetone/ethyl acetate and once with 90:10 MTBE/MeOH)

3) Collected sample runthrough for acylamide extractions
~~LV12WAT059~~ Autotrace Only loaded 469 mL (out of 500) of LV12WAT059 and 488 mL of LV12WAT061

TITLE Extractions of Pavilion samples Project No. Book No. 5751

71

042312 AD

Continued extractions from 04/20/12 (70). Extracted collected runthrough for acrylamides. Using Biotage activated carbon cartridges, LOT# 11251306GB.

old autotrace	New autotrace
1 Blank	1 LV12WAT058
2 Waterspike	2 LV12WAT059
3 LV12WAT056	3 LV12WAT061
	4 LV12WAT063#1
	5 LV12WAT063#2
	6 LV12WAT065

>> *See pg 70 for detailed spike info.* <

1. conditioned autotraces to remove air bubbles from solvent lines (2x).

2. Ran "07251 acrylamide" on old autotrace and "Acrylamide" on new autotrace to extract samples for acrylamides

3. Eluted 10 mL off column w/ MeOH.

Samples: 540 mL (out of 550) of LV12WAT059 & LV12WAT061. were loaded onto autotrace.

Turbovap

~~BA~~ concentrated eluates from 4-20-12 to 0.5 mL w/ MeOH.

AD 04/23/12

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Project No. _____
Book No. 5751 TITLE Extraction of Pavilion Samples

(1) 042512 AD

TurboVap

concentrated eluates from 4-20-12 (pg 70) and 4-23-12 (pg 71) to 0.5mL with MeOH.

(2) 042512AD Extractions of samples from Pavillion. HLB cartridges Lot#108A31354B

	Samples	pH@7	*Spike sol'n	*Internal std	* 1ng/uL d2-DP
old 1	Blank	↓	~~0~~	50uL ↓	250uL ↓
old 2	LV12WAT060 SPK		50uL		
old 3	LV12WAT062 SPK		50uL		
new 4	LV12WAT066 SPK		50uL		
new 5	LV12WAT067		~~0~~		
new 6	LV12WAT069		~~0~~		
new 7	LV12WAT071		~~0~~		
new 8	LV12WAT073		~~0~~		
new 9	LV12WAT073 SPK		50uL		

*Spike sol'n: 4-25-12 5752-42-1
*Internal std: 4-25-12 5752-41-3
*1ng/uL d2-DP: Sigma Analytical Std (Fluka 51506 07 Lot # SZBA018X)

Autotrace
* conditioned both autotraces w/ "condition" method to remove air bubbles from solvent lines

*Ran method "alkylphenol" on old autotrace & "alkylphenol-b" on new autotrace to elute 500mL of the samples. Eluted 10mL of samples w/ 2:2:1 MeOH/acetone/Ethyl acetate (5mL) & 90:10 MeOH/acetone
* collected runthrough for acrylamide extractions

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TITLE Extraction of Pavilion Samples Project No. _____ Book No. 5751

042612 AD Extraction of Pavillion Samples. Using Blotage activated carbon Cartridges, Lot # 11251306GB.

Samples

see pg 72 for spike info

1 Blank
2 LV12WAT060 SPK
3 LV12WAT062 SPK
4 LV12WAT066 SPK
5 LV12WAT067
6 LV12WAT069
7 LV12WAT071
8 LV12WAT073
9 LV12WAT073 SPK

Autotrace

* Conditioned both autotraces w/ "condition" method to remove air bubbles from solvent lines.

* Ran "072511 acrylamide" method on old autotrace, & "Acrylamide" method on new autotrace to elute 500mL of the samples, Eluted 10mL of samples w/ MeOH.

Turbovap

* Concentrated eluates from alkylphenol run on 4-25-12 (pg 72) to 0.5mL w/ MeOH.

AD
05 03 12

Recorded by: [signature] Date 05 07 12 Verified by: Date

TITLE Extraction of Pavillion Samples

Project No.

Book No. 5751

050412A

Turbovap: Concentrated samples (DI water spikes see page 74, to 0.5mL with MeOH.

050712A Extraction of samples from Pavillion. HLB cartridges Lot #108A3135

	Samples	initial pH	vol HCL added	final pH	*spike sol'n	*internal
old 1	Blank	7	0	7	0	50
old 2	Water spike	7	0	7	50uL	
old 3	LV12WAT080	7	0	7	0	
New 4	LV12WAT082	7	0	7	0	
New 5	LV12WAT084	7	0	7	0	
New 6	LV12WAT086#1	7	0	7	0	
New 7	LV12WAT086#2	7	0	7	0	
New 8	LV12WAT088	11	200uL	7	0	
New 9	LV12WAT088-SPK	11	200uL	7	50uL	

*spike sol'n: 4-25-12 5752-42-1
* Internal std: 4-25-12 5752-41-3

Autotrace
ran "condition" method twice on each autotrace.
ran "alkylphenol" method on each autotrace.
Loaded 500mL of each water sample onto columns, collected sample from run through to run ~~one~~ for acrylamides. Eluted 10mL off column w/ 5mL 2:2:1 MeOH/Acetone/Ethyl Acetate and 5mL 90:10 MTBE/MeOH.

Sample
82 - loaded 512 mL
84 - loaded 494 mL
86#2 - loaded 497 mL
88 ~~#A~~ - loaded 517 mL

Note: Samples 4, 5 & 6 eluted more than 10mL off column. Samples 7, 8 & 9 eluted 7mL off column.

Recorded by: | Date | Verified by: | Date

Project No.
Book No. 5751 TITLE Extraction of Pavilion Samples

050812 AD

Extraction of water samples from pavillion to recover acrylamide. Using Biotage activated carbon cartridges Lot # 11251306GB.

Ran collected run through from Ethoxylate run on 050712 (see pg. 75).

Samples

1 Blank
2 Waterspike
3 LV12WAT080
4 LV12WAT082
5 LV12WAT084
6 LV12WAT086#1
7 LV12WAT086#2
8 LV12WAT088
9 LV12WAT088-spike

} See pg. 75 for spike info.

autotrace

* ran "condition" method twice.
* ran "072511 acrylamide" for samples 1-3. Loaded 550mL of sample through columns. Eluted 10mL off column w/ MeOH.
* ran "acrylamide" method for samples 4-9. Loaded 517mL onto column. Eluted 10mL off column w/ MeOH.

AD 050912

TITLE Extraction of pavilion samples Project No. Book No. 5751

050812 AD

Turbovap.

Concentrated samples from Ethoxylate run on pg 75 (050712), to 0.5mL w/ MeOH.

050912 AD

Extraction of water samples from Pavilion WY. Recovery of ~~all~~ AD Ethoxylates using HLB cartridges, Lot # 108A31354B.

	Samples	initial pH	vol HCl added	final pH	*Internal Std	*d2-OP
1	Blank	7	0	7	50 µL	250 µL
2	LV12WAT091	7	0	↓	↓	↓
3	LV12WAT093	7	0	↓	↓	↓
4	LV12WAT095	11	150 µL	↓	↓	↓
5	LV12WAT097	11	↓	↓	↓	↓
6	LV12WAT099	11	↓	↓	↓	↓

*internal std: 4-25-12, 5752-41-3
* d2-OP: 1ng/µL, Sigma analytical std (fluka-S150607, Lot SZBA018X)

Autotrace

Ran "condition" method twice.
Ran "alkylphenol-b" method. Loaded 500mL of sample onto column. Collected run through to run for acrylamides.
Eluted 10mL off column w/ 5mL 2:2:1 MeOH/acetone/ethylace and 5mL 90:10/MTBE/MeOH.

Turbovap: concentrated acrylamide sample eluates from run on 050812 (pg 76) to 0.5mL w/ MeOH.

see pg 78 for notes

Recorded by: [signature] Date 05-09-12 Verified by: Date

78

Project No.

Book No. 5751 TITLE Extraction of Pavilion samples

05 09 12 AD

NOTE

samples didn't all ~~elute~~ load AD 500 mL. + 50 mL.

Blank = 548 mL
LV12WAT091 = 490 mL
LV12WAT093 = 513 mL
LV12WAT095 = 500 mL
LV12WAT097 = 517 mL
LV12WAT099 = 517 mL.

---

05 10 12 AD

Turbovap: concentrated samples from alkylphenol run (050912 (pg 77), to 0.5 mL. Washed sides w/ MeOH.

Autotrace: Extraction of Pavilion samples that were collected from the run through of the alkylphenol run, on 5-9-12 (pg 77), to recover acrylamide using Biotage activated carbon cartridges, Lot # 1125130 GB.

Samples
Blank
LV12WAT091
LV12WAT093
LV12WAT095
LV12WAT097
LV12WAT099

} see pg 77 for spike info.

Ran condition method twice.
Ran acrylamide method to extract 550 mL of sample, to elute 10 mL off column w/ MeOH.

Recorded by: [illegible] Date 5-10-12 Verified by: Date

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TITLE Extraction of Pavilion Samples

Project No.

Book No. 5751

05/1/12 Turbovap: Concentrated samples from acrylamide run on 05/1/12 (pg 78), to 0.5mL, washing sides w/ MeOH.

*Extraction of pavilion samples for ethoxylates recoveries. Using HLB cartridges, lot ~~#~~ 108A31354

	Samples	initial pH	HCl added	final pH	*Spike sol'n	*internal std	*1ng/µL d2-OP
1	water spike	7	Ø	7	50µL	50µL	250µL
2	LV12WAT101	10	75µL	↓	Ø	↓	↓
3	LV12WAT103	10	75µL	↓	Ø	↓	↓
4	LV12WAT105	7	Ø	↓	Ø	↓	↓
5	LV12WAT105 spike	7	Ø	↓	50µL	↓	↓

Autotrace

"conditioned" method to clean solvent lines twice.

"alkylphenol-6 method to load 500mL of the samples, eluted 10mL off column w/ 5mL 2:2:1 MeOH/acetone/ethyl acetate and 5mL 90:10 MTBE. Collected run through of samples to run acrylamide method.

*spike sol'n: 4.25.12, 5752-42-1

*internal std: 4.25.12, 5752-41-3

*1ng/µL d2-OP: Sigma analytical std (fluk9-S150607, lot SZ1BAØ18X)

Note water spike Ø

1. ~~Wastewater~~: loaded 550mL total
2. LV12WAT101: loaded 530mL total
3. LV12WAT103: loaded 530mL total
4. LV12WAT105: loaded 525mL total
5. LV12WAT105spk: loaded 536mL total.

*All samples eluted 8mL off column.

Recorded by: [signature] Date 5-11-12 Verified by: Date

Project No.
Book No. 5751 TITLE Extraction of Pavilion Samples

05/4/12 A

Turbovap: concentrated eluates from ethoxylate extraction on 5-11-12 (pg 79), to 0.5mL w/ MeOH.

Extraction of collected samples from ethoxylate run on 5-11-12 (pg 79) to test for acrylamide recoveries w/ Biotage Activated Carbon cartridges, Lot # M2513066B.

Samples
water spike
LV12WAT101
LV12WAT103
LV12WAT105
LV12WAT105 spike

} see pg. 79 for spike info

autotrace
- ran "Condition" method twice to remove air bubbles from solvent lines.
- ran "Acrylamide" method to load 550mL of sample onto column. Eluted 10mL of the column w/ MeOH.

05/5/12 A

Patrick DeArmond concentrated samples to 0.5mL w/ MeOH.

A 05/6/12

Recorded by: Date: Verified by: Date:

81

TITLE Extraction of Pavilion Samples

Project No.

Book No. 5751

052112 AD

Extraction of Pavilion WY samples to test for ~~acrylamide~~ alkylphenol recoveries (AEOs). This is a repeat run of selected samples. Using Rinsed HLB Cartridges, Lot # 10843135 4B

	Samples	initial pH@	added HCL	final pH@	*spike soln	*internal std
1	waterspike	7	0	7	50uL	50uL
2	LV12WAT058	7	0		0	
3	LV12WAT060spk	7	0		~~NO~~ 50uL	
4	LV12WAT062spk	7	0		~~NO~~ 50uL	
5	LV12WAT080	7	0		0	
6	LV12WAT089spk	11	150uL		50uL	
7	LV12WAT082	7	0		0	
8	LV12WAT084	7	0		0	
9	LV12WAT087#1	7	0		0	
10	LV12WAT087#2	7	0		0	
11	LV12WAT089	11	150uL		0	

Autotrace's

*Ran "Condition" method twice on old & new autotrace.

*Ran "Alkylphenol" method on old autotrace for samples 1-6, loaded 500mL of sample onto column.

*Ran "alkylphenol_b" method on new autotrace for samples 7-11, loaded 500mL of sample onto column.

*Eluted 10mL off each column (old & new) w/ 90:10 MTBE, for every sample.

*Spike sol'n: 4.25.12, 5752-42-1

*internal std: 4.25.12, 5752-41-3

*d2-op: 1ng/uL, Sigma analytical std (fluka-S150607, lot SZ BA018X)

See pg 82 continued Note →

Recorded by: [signature] Date 5-21-12 Verified by: Date

82 Project No. _____ Concentration of

Book No. 5751 TITLE Samples w/ Turbo Vap.

052312 AD

(pg 81)

Concentrated samples to 0.5mL w/ MeOH.

Note: samples didn't completely load 500mL, +50mL

LW2WAT058: 529mL
LW2WAT080: 523mL
LW2WAT082: 518mL
LW2WAT084: 498mL
LW2WAT087#1: 541mL
LW2WAT087#2: 500mL
LW2WAT089: 517mL

052312

AD

Recorded by: | Date | Verified by: | Date

TITLE Extraction of pavilion samples

Project No.

Book No. 5751

060112~~A~~

Extraction of pavilion samples to test for alkylphenol recoveries, using HLB cartridges, Lot # 108A3B54B.

	Samples	initial pH	added HCl	final pH	*internal std	* d2-OP
1	Blank	7	~~0~~	7	50 uL	250 uL
2	LV12WAT064	11	35 uL	↓	↓	↓
3	LV12WAT071	7	~~0~~			
4	LV12WAT101	10	12.5 uL			

autotrace

- ran "condition" method twice to clean solvent lines and remove air bubbles.
- ran "alkylphenol_L" method, loaded 50 uL of sample onto columns.
- Eluted 10mL off column w/ 90:10 MtBe/MeOH.

* Internal std: 042512, 5752-41-3

* 1ng/uL d2-OP: Sigma analytical std, (fluka-S1586007, lot SZBA018X).

Turbovap: concentrated samples to 0.5mL while solvent exchanging w/ MeOH.

060612

Recorded by: | Date | Verified by: | Date