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5/30/12 Calibrated/Tuned in Negative Mode usi
gdd Instrument Optimization feature and
PPG 3000 std. provided by AB Sciex.

Instrument Settings saved to D:\Analyst Data\Projects\API Instrument Data\Instrument Optimization\2012-5-30_9.59

Optimized instrument in negative mode for nonylphenol (
octylphenol (OP), nonylphenol-d4 (NP-d4), and octylphenol-d2 (O

Mobile phase during optimization: 300 µl/min 50/50
2mM NH_4OAc in H_2O / 2 mM NH_4OAc

Teed in each compound (1-10 ng/µl) at 10 µl/min.
(d2-NP at 20 µl/min)

OP-d2 was optimized manually because its sens
was too low.
(Changed the mobile phase composition
85% B)

OP-d2 parameters

207 → 135
↑ product ion

parameters used for all compounds

Curtain gas = 25
Needle Voltage = -4500
Temp = 200
Ion Source gas 1 = 90
Ion Source gas 2 = 50
Declustering potential = -80
Entrance Potential = -11

CAD = 9
CE = -33 V
CXP = -6 V

Witnessed & Understood by me, | Date | Invented by: | Date
[signature] | 5/30/12 | Recorded by:

TITLE _____ Project No. _____

Book No. 511

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Wednesday, May 30, 2012, 12:00

NP

Infusion Compound Optimization
MS/MS Analysis, negative
Precursor ion: MW Ion Search Window: ± 1.000 (Da)
Product ion: Auto Select, Criteria:
From the most intense: 3 peaks. Build final method using 3 most intense peaks.
Exclude Product Ions within ± 5.000(Da) of Precursor Ion m/z.
Min. mass for product ion: 40.000(Da). Threshold for product ion > 0.0(cps)
Quad 1 Resolution: Unit resolution, Quad 3 Resolution: Unit resolution

Target Compound	MW(neutral, Da)	No. Charges
Nonylphenol	220.200	1

Initial Q1 Scan

Target Compound	Mass(Da)	Intensity(cps) (5 MCA Average)
Nonylphenol	219.059	683260

Parameter: DP Start: -400.0 Stop: -1.0 Step: 5.0

Masses(Da)	Current Value	New Value	Intensity(cps)
219.059	-90.0	-75.0	170496

Final Q1 Scan

Target Compound	Mass(Da)	Intensity(cps) (5 MCA Average)
Nonylphenol	219.061	186600

Final Q1MI Method: Nonylphenol_CCpt_FinalQ1MI_Neg.dam
Target Compound: Nonylphenol

Initial Product Mass	Initial Product Ion Intensity(cps) (26 MCA Sum)
133.136	71000
116.844	14000
118.882	13000

Parameter: CE Start: -130.0 Stop: -5.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
219.061/116.844	-54.0	-76.0	755
219.061/118.882	-54.0	-46.0	1002
219.061/133.136	-54.0	-36.0	7858

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
219.061/116.844	-5.0	-7.0	780

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
219.061/118.882	-5.0	-5.0	910

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
219.061/133.136	-5.0	-9.0	6977

Final Product Ion Mass(Da)	Final Product Ion Intensity(cps) (10 MCA Average)
117.100	950
118.900	1140
132.900	8120

Optimal Product Ion Mass: 132.900 Optimal Product Ion Intensity: 8120
Optimal Product Ion Mass: 118.900 Optimal Product Ion Intensity: 1140
Optimal Product Ion Mass: 117.100 Optimal Product Ion Intensity: 950
Final MRM Method: Nonylphenol_CCpt_FinalMRM_Neg.dam
Compound optimization completed successfully.

~~Curtain G~~
Curtain gas 30
IonSpray Voltage -4500
Temp 400
Ion Source Gas 1 90
Ion Source Gas 2 40

Needle x = 5.50
y = 2.00

conditions at 50% B, 0.300 mL/min

Later changed to 85% B, 0.25 mL/min

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Witnessed & Understood by me, | Date 5-31-12 | Invented by: | Date | Recorded by

EPAPAV0050818

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Book No. 5111 TITLE _____
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Wednesday, May 30, 2012, 13:23 OP

Infusion Compound Optimization
MS/MS Analysis, negative
Precursor ion: MW Ion Search Window: ± 1.000 (Da)
Product ion: Auto Select, Criteria:
From the most intense: 3 peaks. Build final method using 3 most intense peaks.
Exclude Product Ions within ± 5.000(Da) of Precursor Ion m/z.
Min. mass for product ion: 40.000(Da). Threshold for product ion > 0.0(cps)
Quad 1 Resolution: Unit resolution, Quad 3 Resolution: Unit resolution

Target Compound	MW(neutral, Da)	No. Charges
Octylphenol	206.200	1

Initial Q1 Scan

Target Compound	Mass(Da)	Intensity(cps) (5 MCA Average)
Octylphenol	204.990	838880

Parameter: DP Start: -400.0 Stop: -1.0 Step: 5.0

Masses(Da)	Current Value	New Value	Intensity(cps)
204.990	-90.0	-85.0	216555

Final Q1 Scan

Target Compound	Mass(Da)	Intensity(cps) (5 MCA Average)
Octylphenol	205.019	249660

Final Q1MI Method: Octylphenol_QOpt_FinalQ1MI_Neg.dam
Target Compound: Octylphenol

Initial Product Mass	Initial Product Ion Intensity(cps) (26 MCA Sum)
133.101	43000
134.200	38000
117.000	6000

Parameter: CE Start: -130.0 Stop: -5.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
205.019/117.000	-54.0	-84.0	545
205.019/133.101	-54.0	-34.0	6772
205.019/134.200	-54.0	-26.0	5125

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
205.019/117.000	-5.0	-7.0	497

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
205.019/133.101	-5.0	-7.0	6435

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
205.019/134.200	-5.0	-1.0	5547

Final Product Ion Mass(Da)	Final Product Ion Intensity(cps) (10 MCA Average)
116.900	530
132.900	6880
134.200	5830

Optimal Product Ion Mass: 132.900 Optimal Product Ion Intensity: 6880
Optimal Product Ion Mass: 134.200 Optimal Product Ion Intensity: 5830
Optimal Product Ion Mass: 116.900 Optimal Product Ion Intensity: 530
Final MRM Method: Octylphenol_QOpt_FinalMRM_Neg.dam
Compound optimization completed successfully.

Invented by: Date
Recorded by:

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Wednesday, May 30, 2012, 13:37

NP-d4

Infusion Compound Optimization
MS/MS Analysis, negative
Precursor ion: MW Ion Search Window: ± 1.000 (Da)
Product ion: Auto Select, Criteria:
From the most intense: 3 peaks. Build final method using 3 most intense peaks.
Exclude Product Ions within ± 5.000(Da) of Precursor Ion m/z.
Min. mass for product ion: 40.000(Da). Threshold for product ion > 0.0(cps)
Quad 1 Resolution: Unit resolution, Quad 3 Resolution: Unit resolution

infused at 20 µl/min

Target Compound	MW(neutral, Da)	No. Charges
Nonylphenol-d4	224.200	1

Initial Q1 Scan

Target Compound	Mass(Da)	Intensity(cps) (5 MCA Average)
Nonylphenol-d4	223.056	61200

Parameter: DP Start: -400.0 Stop: -1.0 Step: 5.0

Masses(Da)	Current Value	New Value	Intensity(cps)
223.056	-90.0	-95.0	60938

Final Q1 Scan

Target Compound	Mass(Da)	Intensity(cps) (5 MCA Average)
Nonylphenol-d4	223.019	69580

Final Q1MI Method: Nonylphenol-d4_QOpt_FinalQ1MI_Neg.dam
Target Compound: Nonylphenol-d4

Initial Product Mass	Initial Product Ion Intensity(cps) (26 MCA Sum)
110.026	19000
122.200	6000
88.800	5000

Parameter: CE Start: -130.0 Stop: -5.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
223.019/88.800	-54.0	-122.0	63
223.019/110.026	-54.0	-30.0	5955
223.019/122.200	-54.0	-126.0	60

Intensity = 60 (cps) is too low. Min. Intensity = 100 (cps)

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
223.019/88.800	-5.0	-1.0	67

Intensity = 67 (cps) is too low. Min. Intensity = 100 (cps)

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
223.019/110.026	-5.0	-5.0	5878

Parameter: CXP Start: -55.0 Stop: 0.0 Step: 2.0

Masses(Da)	Current Value	New Value	Intensity(cps)
223.019/122.200	-5.0	-55.0	67

Intensity = 67 (cps) is too low. Min. Intensity = 100 (cps)

Final Product Ion Mass(Da)	Final Product Ion Intensity(cps) (10 MCA Average)
90.200	100
110.000	6410
123.800	80

Optimal Product Ion Mass: 110.000 Optimal Product Ion Intensity: 6410
Optimal Product Ion Mass: 90.200 Optimal Product Ion Intensity: 100
Optimal Product Ion Mass: 123.800 Optimal Product Ion Intensity: 80
Final MRM Method: Nonylphenol-d4_QOpt_FinalMRM_Neg.dam
Compound optimization completed successfully.

Witnessed & Understood by me, | Witnessed by:

DVD 5

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5-30-12

Created LC-MS Method for alkylphenols.
Saved in D:\Analyst Data\Projects\HF\Acquisition Methods\ as alkylphenols_20120530.dam

Used optimized parameters from p. 10-13 for OP, OP-d2, NP,

OP-d2 : 207 → 135
OP : 205 → 133
NP : 219 → 133
NP-d4 : 223 → 110

LC:

Time	% B	Flow rate
0	40	0.25 ml/min
2	40	↓
8	95	
10	95	
12.5	40	
15	40	

5-30-12

Witnessed & Understood by me,	Date	Invented by:	Date
[illegible]	5-30-12	Recorded by:	

111

TITLE Pavilion WY Samples

Project No.

Book No: 5111

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5-30-12

Data File	Sample Name	Method	Sample	Inj
20120530_PavilionAPs	20120530_001	allaylphenols_20120530	MeOH	5
	_002		0.05 ng/µl APs 5/1/12	
	_003		0.2 ng/µl	
	_004		0.3 ng/µl	
	_005		0.5 ng/µl	
	006		0.8 ng/µl	
	007		1.0 ng/µl	
	008		MeOH	
	009		# water spike 5/21/12	
	010		# 1 blank 5/9/12	
	011		# 2 LV12WAT058 5/21/12	
	012		# 3 LV12WAT060 5/21/12 spike	
	013		# 4 LV12WAT062 5/21/12 spike	
	014		# 3 LV12WAT062 spike 4/25/12	
	015		# 2 LV12WAT060 spike 4/25/12	
	016		# 5 LV12WAT067 4/25/12	
	017		# 6 LV12WAT069 4/25/12	
	018		# 7 LV12WAT071 4/25/12	
	019		# 8 LV12WAT073 4/25/12	
	020		# 9 LV12WAT073 spike 4/25/12	
	021		# 5 LV12WAT080 5/21/12	
	022		# 7 LV12WAT082 5/21/12	
	023		# 8 LV12WAT084 5/21/12	
	024		# 9 LV12WAT087 #1 5/21/12	
	025		# 10 LV12WAT087 #2 5/21/12	
	026		# 6 LV12WAT089 spike 5/21/12	
	027		# 11 LV12WAT089 5/21/12	
	028		# 2 LV12WAT091 5/9/12	
	029		# 3 LV12WAT093 5/9/12	
	030		# 4 LV12WAT095 5/9/12	
	031		MeOH	
	032		CCV 0.5 ng/µl APs 5/1/12	
	033		# 1 Blank 4/25/12	
	034		# 5 LV12WAT097 5/9/12	
	035		# 6 LV12WAT099 5/9/12	

Witnessed & Understood by me, | Date 5-30-12 | Invented by: | Date

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Project No.
Book No. 5111 TITLE Pavilion WY Samples

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5-30-12 POP

Data File	Sample Name	Method	Sample ID
20120530_PavilionAPs	20120530_036	alkylphenols_20120530	#2 LV12WAT101 5/10/12
	037		#3 LV12WAT103 5/10/1
	038		#4 LV12WAT105 5/10/1
	039		#5 LV12WAT105 spk 5/1
	040		#2 water spike 4/20/12
	041		#3 LV12WAT056 4/20
	042		#7 LV12WAT063 #1 4/
	043		#8 LV12WAT063 #2 4/2
	044		#9 LV12WAT065 4/20/
	045		MeOH
	046		CCV 0.3 ng/µl APs 5/1/1

5/31/12 POP

Data File	Sample Name	Method	Sample ID
20120531_PavilionAPs	20120531_001	alkylphenols_20120530	MeOH
	002		0.05 ng/µl APs 5-1-12
	003		0.5
	004		1.0
	005		#5 LV12WAT059 4/20/12
	006		#6 LV12WAT061 4/20/12
	007		MeOH
	008		CCV 0.3 ng/µl APs 5-1-12

5-31-12

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[signature]	5-31-12	Recorded by:	

TITLE Pinellas alkylphenols

Project No. _____

Book No. 511

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6/4/12 PPH

Re-extracted some Pinellas samples that were previously out of calib. range

Extracted 50 ml sample.

Datafile	Sample Name	Method	Sample ID	Vo
20120604_AP	20120604_001	alkylphenols_20120530	MeOH	50
	002		0.3 ng/µl APs 5/1/12	
	003		0.5 ng/µl APs 5/1/12	
	004		1.0 ng/µl APs 5/1/12	
	005		MeOH	
	006		#1 Blank 6/1/12	
	007		#2 LV12WAT064 6/1/12	
	008		#3 LV12WAT071 6/1/12	
	009		#4 LV12WAT101 6/1/12	
	010		MeOH	
	011		CCV 0.8 ng/µl APs 5/1/12	

Needle position was adjusted to x = 5.5 and y = prior to injecting samples

Mobile phases A = 2 mM NH4OAc in Wa
B 2 mM NH4OAc in ACN

Column = Waters BEH C18

PPH 6-4-12

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Witnessed & Understood by me, | Date | Invented by: | Date

EPAPAV0050824