Summary

Test Name:	Popcorn Emission Phase 1 Popcorn 1 Test (POP-Phase1P1)
Purpose:	Popcorn chamber background check and VOC emissions from popcorn in the chamber
Reporter:	Xiaoyu Liu
Medium:	TENAX
Method:	ATD/GC/MSD
Instrument:	Perkin-Elmer ATD 400/HP6890/5973
Reporting Format:	nnn — In calibration ***nnn*** — Estimated value above quantification limit (bold italics) ~~**nnn**~~ — Estimated value below quantification limit (bold strikethrough) **nnn** — Estimated value below detection limit (bold underlined)
Test Dates:	February 3, 4, 2004
Analysis Dates:	February 3, 4, 2004
Review Status:	Preliminary review by Xiaoyu Liu was completed on February 9, 2004
Final Review:	

VERSION 1

POP-Phase1P1.xls

Popcorn Emission Phase 1 Popcorn 1 Test (POP-Phase1P1)

GC/MS was calibrated on 12/10-11/03

The calibration of butyric acid was invalid. It can not be used for quantification of samples.
Carboxylic acids react readily with alcohols in the presence of catalytic amounts of mineral acids to yield compounds called esters.

Analytical method POP6-7.M &POP6-7D.M (Different quantitation methods due to different solvent)

Purge time	5 min
Primary Purge Flow/Desorption Flow	39 ml/min
Primary Purge Flow/Desorption Temp	250°C
Primary Desorption Time	10 min
Valve Temp	200°C
Low Flow Cold Trap Temp	- 30°C
Secondary Desorption Temp	275°C
Secondary Desorption Time	30 min
Inlet Split	Not used
Outlet Split	22.3 ml/min split ratio of 10:1
Transfer Line Temp	225°C
GC Column	DB-624, 30 m with 0.25 mm ID and 1.4 mm film thickness
Oven Temperature Program	45°C for 4 min, to 130°C at 6°C/min, to 220°C at 35°C/min, hold 20 min, total time 40.74 mins
Carrier flow	2.2 ml/min measured at 45°C
Detector Type	5973 MSD
Solvent delay	3 min

POP6-7.M was used for p-xylene and butyric acid calibration, POP6-D.M was used for diacetyl calibration.

ATD 400 GC/MS Analysis of VOCs

ID	Description	Date	Time	Operator
DCC2621	DCC 120803STDB-3	2/3/2004	22:03	XLIU
DCC2634	DCC 121103STD-3	2/3/2004	23:02	XLIU
ID2637	Popcorn chamber inlet A	2/4/2004	0:02	XLIU
ID2638	Popcorn chamber inlet B	2/4/2004	1:02	XLIU
ID2635	Popcorn chamber outlet A	2/4/2004	2:02	XLIU
ID2636	Popcorn chamber outlet B	2/4/2004	3:01	XLIU
FB2639	Popcorn chamber field blank	2/4/2004	4:01	XLIU
DCC2647	DCC 120803STDB-3	2/4/2004	18:39	XLIU
DCC2648	DCC 121103STD-3	2/4/2004	19:39	XLIU
BKG2640	Phase1P1-BKG	2/4/2004	20:39	XLIU
FB2643	Phase1P1-FB	2/4/2004	21:39	XLIU
ID2641	Phase1P1-POPPING	2/4/2004	22:38	XLIU
ID2642	Phase1P1-OPENING	2/4/2004	23:37	XLIU

ATD400 GC/MS Calibration Performed on 12/10-11/03

Two calibration solutions are made, one is 2,3-butanedione in CH2Cl2 (121103STD),
the other is p-xylene and butyric acid in MeOH (120803STDB)

g/mL = mg/uL
Conc (ug/mL = ng/uL) = Volume(uL)*Density(mg/uL)*Purity*1000(ug/mg)/Solution Volume (mL)
1g = 1000 mg = 1000,000 ug = 1000,000,000 ng
1 mL = 1000 uL

Stock Solution 120803STDB

Analytes	Formula	%Purity	MW	Density(mg/uL)	Volume (uL)	Concentration (ng/uL)
Butyric Acid	CH3CH2CO2H	99	88.11	0.964	110	10497.96
p-Xylene	C6H4(CH3)2	99	106.17	0.866	100	8573.4

Diluted Stock Solution from 120803STDB

	120803STDB-00 (ng/uL)
120803STDB (uL)	100
CH3OH (uL)	900
Total Volume (uL)	1000
Analytes	
Butyric Acid	1049.796
p-Xylene	857.34

Calibration Standards

Concentrations are ng/2uL

	120803STDB-1	120803STDB-6	120803STDB-2	120803STDB-3	120803STDB-4	120803STDB-5
120803STDB-00(uL)	5	10	30	100	250	420
CH3OH (uL)	995	990	970	900	750	580
Total Volume (uL)	1000	1000	1000	1000	1000	1000
Analytes						
Butyric Acid	10.49796	20.99592	62.98776	209.9592	524.898	881.82864
p-Xylene	8.5734	17.1468	51.4404	171.468	428.67	720.1656

Calibration Calculation (8-881 ng/tube)

Relative Response Factor (RRF) = Comp's (Area/ng)/ IS's (Area/ng)
2 uL injection
d8-Toluene (IS) concentration 430.35ng/injection, RT=8.84 min
No IAP was analyzed after calibration since this calibration will be used as semi-quantification.

Butyric acid	ng	Area (Response)	IS Area (Response)	RF	RF Average	RF STDS	%RF RSD	Data File
RT=12.20	10.50	11260	18167656	0.0254				CAL2619.D
	10.50	13996	17871016	0.0321				CAL2618.D
	10.50	15799	17815993	0.0364	0.031	0.01	17.64	CAL2617.D
	21.00	39202	17778418	0.0452				CAL2623.D
	21.00	36705	17977700	0.0418				CAL2622.D
	21.00	44287	17746161	0.0512	0.046	0.00	10.23	CAL2620.D
	62.99	162818	18544467	0.0600				CAL2471.D
	62.99	164189	18608064	0.0603				CAL2477.D
	62.99	106317	17760204	0.0409	0.054	0.01	20.67	CAL2616.D
	209.96	1750685	18607916	0.1928				CAL2478.D
	209.96	2115579	18462101	0.2349				CAL2479.D
	209.96	1989524	18531914	0.2200	0.216	0.02	9.87	CAL2480.D
	524.90	6665556	18476192	0.2958				CAL2498.D
	524.90	7292075	18816084	0.3177				CAL2491.D
	524.90	6319477	18795574	0.2757	0.296	0.02	7.10	CAL2493.D
	881.83	12660111	18749774	0.3295				CAL2495.D
	881.83	12652864	18488440	0.3340				CAL2497.D
	881.83	12228476	18862198	0.3164	0.327	0.01	2.80	CAL2499.D
AVG				0.153				
STD				0.124				
%RSD				81.59				

p-Xylene	ng	Area (Response)	IS Area (Response)	RF	RF Average	RF STDS	%RF RSD	Data File
RT=12.58	8.57	436508	18167656	1.206				CAL2619.D
	8.57	397963	17871016	1.118				CAL2618.D
	8.57	408565	17815993	1.151	1.158	0.04	3.85	CAL2617.D
	17.15	783484	17778418	1.106				CAL2623.D
	17.15	737115	17977700	1.029				CAL2622.D
	17.15	739831	17746161	1.046	1.060	0.04	3.81	CAL2620.D
	51.44	2395201	18544467	1.081				CAL2471.D
	51.44	2452749	18608064	1.103				CAL2477.D
	51.44	2294170	17760204	1.081	1.088	0.01	1.17	CAL2616.D
	171.47	7435046	18607916	1.003				CAL2478.D
	171.47	7677160	18462101	1.044				CAL2479.D

	171.468	7769112	18531914	1.052	1.033	0.03	2.55	CAL2480.D
	428.67	18881676	18476192	1.026				CAL2498.D
	428.67	20268581	18816084	1.081				CAL2491.D
	428.67	19523782	18795574	1.043	1.050	0.03	2.71	CAL2493.D
	720.17	31008316	18749774	0.988				CAL2495.D
	720.17	30833043	18488440	0.997				CAL2497.D
	720.17	30855688	18862198	0.978	0.987	0.01	0.97	CAL2499.D
AVG				1.068				
STD				0.057				
%RSD				5.37				

Calibration Range of the Instrument

Analyte	PQL(ng/tube)	Hi Cal(ng/tube)
Butyric Acid	10.50	881.83
p-Xylene	8.57	720.17

120803STDB-3 was analyzed as DCC

Stock Solution 120803STDB

Analytes	Formula	%Purity	MW	Density(mg/uL)	Volume (uL)	Concentration (ng/uL)
2,3-Butanedione	CH3COCOCH3	97	86.09	0.981	100	9515.7

Diluted Stock Solution from 121103STD

	121103STD-00 (ng/uL)
121103STDA (uL)	100
Methylene Chloride (uL)	900
Total Volume (uL)	1000
Analytes	
2,3-Butanedione	951.57

Calibration Standards 121103STD

	Concentrations are ng/2uL				
	121103STD-1	121103STD-2	121103STD-3	121103STD-4	121103STD-5
12110303STD-00(uL)	5	30	100	250	420
Methylene Chloride (uL)	995	970	900	750	580
Total Volume (uL)	1000	1000	1000	1000	1000
Analytes					
2,3-Butanedione	9.5157	57.0942	190.314	475.785	799.3188

Calibration Calculation (9-799 ng/tube)

Relative Response Factor (RRF) = Comp's (Area/ng)/ IS's (Area/ng)
2 uL injection
d8-Toluene (IS) concentration 430.35ng/injection, RT=8.84 min
No IAP was analyzed after calibration since this calibration will be used as semi-quantification.

2,3-Butanedione	ng	Area (Response)	IS Area (Response)	RRF	RRF Average	RF STDS	%RF RSD	Data File
RT=3.95	9.52	171708	18482164	0.420				CAL2600.D
	9.52	106560	18576436	0.259				CAL2601.D
	9.52	141136	18485421	0.345	0.342	0.080	23.54	CAL2602.D
	57.09	954751	18344068	0.392				CAL2603.D
	57.09	926991	17632075	0.396				CAL2625.D
	57.09	966978	17856580	0.408	0.399	0.008	2.07	CAL2605.D
	190.31	3252807	17944440	0.410				CAL2606.D
	190.31	3011078	18016084	0.378				CAL2607.D
	190.31	3476213	17663982	0.445	0.411	0.034	8.16	CAL2608.D
	475.79	7487533	18135113	0.373				CAL2609.D
	475.79	6728293	18178935	0.335				CAL2610.D
	475.79	7847609	17723643	0.400	0.370	0.03	8.94	CAL2611.D
	799.32	10995383	18080840	0.327				CAL2612.D
	799.32	16415001	17755155	0.498				CAL2613.D
	799.32	13095536	17923327	0.393	0.406	0.09	21.15	CAL2624.D
AVG				0.385				
STD				0.055				
%RSD				14.31				

Calibration Range of the Instrument

Analyte	PQL(ng/tube)	Hi Cal(ng/tube)
2,3-Butanedione	9.52	799.32

121103STD-3 was analyzed as DCC

Daily Calibration Check (120803STDB-3, 121103STD-3)

VOCs	AVG RF	Response	IS Response	Measured (ng/2uL)	Injected (ng/2uL)	%Recovery	Data File	Run Date	Pass/Fail
p-Xylene	1.068	7296325	17804676	165.146	171.468	96.3	DCC2621	2/3/2004	Pass
2,3-Butanedione	0.385	2589804	17807623	162.374	190.314	85.3	DCC2634	2/3/2004	Pass
p-Xylene	1.068	7643433	18649650	165.164	171.468	96.3	DCC2647	2/4/2004	Pass
2,3-Butanedione	0.385	3051849	17954478	189.777	190.314	99.7	DCC2648	2/4/2004	Pass

	%Recovery		AVG	STD	%RSTD
Date	2/3/2004	2/4/2004			
p-Xylene	96.3	96.3	96	0.01	0.01
2,3-Butanedione	85.3	99.7	93	10.18	11.01

VOCs Measured per Tenax Tube for the Test

ID	Description	Analyte	AVG RF	Response	IS Response	Per Tube (ng)
ID2637	Popcorn chamber inlet A	p-Xylene	1.068	6305	17817663	~~0.143~~
		2,3-Butanedione	0.385	2869		~~0.180~~
		d10-o-Xylene		633434969		
ID2638	Popcorn chamber inlet B	p-Xylene	1.068	4588	18147264	~~0.102~~
		2,3-Butanedione	0.385	4170		~~0.257~~
		d10-o-Xylene		639463631		
ID2635	Popcorn chamber outlet A	p-Xylene	1.068	123114	18574213	~~2.671~~
		2,3-Butanedione	0.385	5374		~~0.323~~
		d10-o-Xylene		628249715		
ID2636	Popcorn chamber outlet B	p-Xylene	1.068	131929	18752920	~~2.835~~
		2,3-Butanedione	0.385	8660		~~0.516~~
		d10-o-Xylene		631419388		
FB2639	Popcorn chamber field blank	p-Xylene	1.068	2362	19057884	~~0.050~~
		2,3-Butanedione	0.385	6552		~~0.384~~
		d10-o-Xylene				
BKG2640	Phase1P1-BKG	p-Xylene	1.068	73	18389002	~~0.002~~
		2,3-Butanedione	0.385	1487		~~0.090~~
		d10-o-Xylene		166753162		
FB2643	Phase1P1-FB	p-Xylene	1.068	63	18273241	~~0.001~~
		2,3-Butanedione	0.385	775		~~0.047~~
		d10-o-Xylene				
ID2641	Phase1P1-POPPING	p-Xylene	1.068	340822	18411142	~~7.460~~
		2,3-Butanedione	0.385	590510		35.810
		d10-o-Xylene		181186125		
ID2642	Phase1P1-OPENING	p-Xylene	1.068	2750777	18528446	59.829
		2,3-Butanedione	0.385	1937291		116.737
		d10-o-Xylene		176021745		

Concentrations of VOCs Measured from the Gas Phase during the Test

ID	Description	Sample Volume (L)	Concentration (ng/L = ug/m3) p-Xylene	2,3-Butanedione
ID2637	Popcorn chamber inlet A	3.70	~~0.039~~	~~0.049~~
ID2638	Popcorn chamber inlet B	3.77	~~0.027~~	~~0.068~~
ID2635	Popcorn chamber outlet A	3.70	~~0.722~~	~~0.087~~
ID2636	Popcorn chamber outlet B	3.77	~~0.752~~	~~0.137~~
BKG2640	Phase1P1-BKG	0.28	~~0.006~~	~~0.317~~
ID2641	Phase1P1-POPPING	0.28	~~26.213~~	125.825
ID2642	Phase1P1-OPENING	0.25	237.988	464.356

d10-o-xylene Response/Sample Volume

ID	Description	Sample Volume (L)	Response/Sample volume
ID2637	Popcorn chamber inlet A	3.70	171314393
ID2638	Popcorn chamber inlet B	3.77	169619000
ID2635	Popcorn chamber outlet A	3.70	169912026
ID2636	Popcorn chamber outlet B	3.77	167485249
BKG2640	Phase1P1-BKG	0.28	585921160
ID2641	Phase1P1-POPPING	0.28	636634311
ID2642	Phase1P1-OPENING	0.25	700175334

Identification of Compounds Using NIST 98 MS Library

ID2637 Chamber Inlet A

RT (min)	ID	Matching Quality
6.47	trichloroethene	97
8.792	d8-toluene	IS
13.298	d10-o-xylene	IS
16.078	decane	95

ID2635 Chamber Outlet A

RT (min)	ID	Matching Quality
6.474	trichloroethene	97
8.786	d8-toluene	IS
13.329	d10-o-xylene	IS
16.091	decane	95
18.771	4-cyanocyclohexene	97
21.221	dodecamethyl-cyclohexasiloxane	91
22.515	1,6-dichloro-1,5-cyclooctadiene	90

FB2643 Phase1P1-FieldBlank

RT (min)	ID	Matching Quality
8.784	d8-toluene	IS
16.649	benzaldehyde	91
19.466	acetophenone	74
21.246	2,5-dimethyl benzaldehyde	87
25.761	diethyl phthalene	91

BKG2640 Phase1P1-Background

RT (min)	ID	Matching Quality
8.777	d8-toluene	IS
13.246	d10-o-xylene	IS
16.064	decane	95
16.642	benzaldehyde	95
18.752	4-cyanocyclohexene	97
18.982	phenol	93
19.459	acetophenone	91
19.633	decamethyl-cyclopentasiloxane	50
20.771	decanal	91
21.221	dodecamethyl-cyclohexasiloxane	91
25.763	diethyl phthalate	93

ID2641 Phase1P1-POPPING

RT (min)	ID	Matching Quality
3.92	diacetyl	9
6.076	acetic acid	90
8.49	3-hydroxy-2-butanone	72
8.774	d8-toluene	IS
8.903	toluene	49
10.61	hexanal	91
12.179	butanoic acid	91
12.518	p-xylene	95
13.243	d10-o-xylene	IS
13.574	2-propenoic acid, butyl ester	64
16.061	decane	90
16.639	benzaldehyde	94
18.052	1-(2-methoxypropoxy)-2-propanol	78
18.759	4-cyanocyclohexene	97
18.979	phenol	90
19.63	decamethyl-cyclopentasiloxane	87
20.768	decanal	91
21.319	dodecamethyl-cyclohexasiloxane	91
22.099	2-butoxy-1-methyl-2-oxoet, butanoic acid	90
22.282	triacetin	90

ID2641 Phase1P1-OPENING

RT (min)	ID	Matching Quality
3.923	diacetyl	9
5.612	3-methyl-butanal	91
5.86	2-methyl-butanal	87
6.006	acetic acid	90
7.025	1-hydroxy-2-propanone	9
8.484	3-hydroxy-2-butanone	9
8.778	d8-toluene	IS
8.906	toluene	70
10.613	hexanal	91
10.824	propylene glycol	86
12.283	butanoic acid	91
12.522	p-xylene	97
12.623	furfural	83
13.238	d10-o-xylene	IS
13.577	2-propenoic acid, butyl ester	64
15.771	4-methyl-2-trimethylsilylo	72
17.523	1-(2-methoxy-1-methoxypropoxy)-2-propanol	90
17.652	1-(2-methoxy-1-methoxyethoxy)-2-propanol	83
18.046	1-(2-methoxypropoxy)-2-propanol	64
18.753	4-cyanocyclohexene	97
19.203	tetramethyl pyrazine	91
19.634	decamethyl-cyclopentasiloxane	53
21.323	dodecamethyl-cyclohexasiloxane	91
22.103	2-butoxy-1-methyl-2-oxoet, butanoic acid	90
22.295	triacetin	90
23.552	beta-humulene	89
25.764	diethyl phthalate	93

Sample Time / Volume for the test

Popcorn Emission Phase 1 Popcorn 1 Test (POP-Phase1P1)

Sorbent Type	Tube ID	Sample ID	Date	Sample Start Time	Sample End Time	Sample Time	Pump	Sampling Flow Rate (mL/min)	Sampling Duration (min)	Volume (L)	Temp (C)
TENAX	2637	Popcorn chamber inlet A	2/3/2004	17:00	17:25	17:12	97308-1	147.9	25	3.6975	24.4
TENAX	2638	Popcorn chamber inlet B	2/3/2004	17:00	17:25	17:12	97308-2	150.8	25	3.7700	24.4
TENAX	2635	Popcorn chamber outlet A	2/3/2004	16:26	16:51	16:38	97308-1	147.9	25	3.698	24.4
TENAX	2636	Popcorn chamber outlet B	2/3/2004	16:26	16:51	16:38	97308-2	150.8	25	3.770	24.4
TENAX	2639	Popcorn chamber field blank	2/3/2004	FIELD BLANK							
TENAX	2640	Phase1P1-BKG	2/4/2004	15:37	15:39	15:38	97308-1	142.3	2	0.285	24.4
TENAX	2643	Phase1P1-FB	2/4/2004	FIELD BLANK							
TENAX	2641	Phase1P1-POPPING	2/4/2004	16:11	16:13	16:12	97308-1	142.3	2	0.285	24.4
TENAX	2642	Phase1P1-OPENING	2/4/2004	14:33	14:35	14:34	97308-1	142.3	1.77	0.251	24.4

Data Summary of Duplicates/Triplicates for the Test

ID	Description	Elapsed Time, (h)	Sample Volume (L)	Concentration (ug/m3) p-Xylene	2,3-Butanedione
2637	Popcorn chamber inlet A		3.698	~~0.04~~	~~0.05~~
2638	Popcorn chamber inlet B		3.770	~~0.03~~	~~0.07~~
		AVG		0.03	0.06
		STDS		0.01	0.01
		%RSD		0.00	23.55

ID	Description	Elapsed Time, (h)	Sample Volume (L)	p-Xylene	2,3-Butanedione
2635	Popcorn chamber outlet A		3.698	~~0.72~~	~~0.09~~
2636	Popcorn chamber outlet B		3.770	~~0.75~~	~~0.14~~
		AVG		0.74	0.11
		STDS		0.02	0.03
		%RSD		0.00	31.17

Chamber Background Measured for the Test

		Concentration (ug/m3)	
ID	Description	p-Xylene	2,3-Butanedione
2640	Phase1P1-BKG	~~0.01~~	~~0.32~~

Field Blanks Measured for the Test (ng/tube)

ID	Description	Date	p-Xylene	2,3-Butanedione
2640	Popcorn chamber field blank	2/3/2004	~~0.05~~	~~0.38~~
2643	Phase1P1-FB	2/4/2004	~~0.00~~	~~0.05~~

Field Controls Measured for the Test (ng/tube)

There were no field controls for this test.