Summary

Test Name:	Popcorn Emission Phase 1 Popcorn 8 (#46) Test (POP-Phase1P8)
Purpose:	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 23, 2004
Analysis Dates:	February 24, 2004
Review Status:	Preliminary review by Xiaoyu Liu was completed on March 2, 2004
Final Review:	

VERSION 1

POP-Phase1P8.xls

Popcorn Emission Phase 1 Popcorn 8 (#46) Test (POP-Phase1P8)

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
DCC2679	DCC 120803STDB-3	2/24/2004	16:40	XLIU
DCC2676	DCC 121103STD-3	2/24/2004	17:39	XLIU
FB2680	Phase1P7&P8-FieldBlank	2/24/2004	18:39	XLIU
BKG2682	Phase1P8-Background	2/24/2004	22:35	XLIU
ID2684	Phase1P8-POPPING	2/24/2004	23:34	XLIU
ID2687	Phase1P8-OPENING	2/25/2004	0:33	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 RT=12.20	ng	Area (Response)	IS Area (Response)	RF	RF Average	RF STDS	%RF RSD	Data File
	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 RT=12.58	ng	Area (Response)	IS Area (Response)	RF	RF Average	RF STDS	%RF RSD	Data File
	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	7371790	17837579	166.546	171.468	97.1	DCC2679	2/24/2004	Pass
2,3-Butanedione	0.385	3180199	17604789	201.687	190.314	106.0	DCC2676	2/24/2004	Pass

	%Recovery
Date	2/24/2004
p-Xylene	97.1
2,3-Butanedione	106.0

VOCs Measured per Tenax Tube for the Test

ID	Description	Analyte	AVG RF	Response	IS Response	Per Tube (ng)
FB2680	Phase1P7&P8-FieldBlank	p-Xylene	1.068	6208	18247095	~~0.137~~
		2,3-Butanedione	0.385	224		~~0.014~~
		d10-o-Xylene				
BKG2682	Phase1P8-Background	p-Xylene	1.068	95969	18307296	~~2.113~~
		2,3-Butanedione	0.385	93208		~~5.684~~
		d10-o-Xylene		55171559		
ID2684	Phase1P8-POPPING	p-Xylene	1.068	113566	18361254	~~2.493~~
		2,3-Butanedione	0.385	112340		~~6.831~~
		d10-o-Xylene		20591664		
ID2687	Phase1P8-OPENING	p-Xylene	1.068	3829826	18280240	84.430
		2,3-Butanedione	0.385	1769482		108.073
		d10-o-Xylene		20726009		

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
BKG2682	Phase1P8-Background	0.79	~~**2.666**~~	~~**7.174**~~
ID2684	Phase1P8-POPPING	0.32	~~**7.865**~~	~~**21.554**~~
ID2687	Phase1P8-OPENING	0.32	266.396	340.997

d10-o-xylene Response/Sample Volume

ID	Description	Sample Volume (L)	Response/Sample volume
BKG2682	Phase1P8-Background	0.79	69631753
ID2684	Phase1P8-POPPING	0.32	64971594
ID2687	Phase1P8-OPENING	0.32	65395485

Identification of Compounds Using NIST 98 MS Library

BKG2682	Phase1P8-Background		ID2684	Phase1P8-POPPING		ID2687	Phase1P8-OPENING	
RT (min)	ID	Matching Quality	RT (min)	ID	Matching Quality	RT (min)	ID	Matching Quality
			3.24	hexane	87	3.249	hexane	87
			3.956	diacetyl	9	3.947	diacetyl	9
						5.892	2-methyl-butanal	43
			6.214	acetic acid	78	6.039	acetic acid	90
						7.021	pentanal	86
						7.076	1-hydroxy-2-propanone	9
8.501	3-hydroxy-2-butanone	40	8.526	3-hydroxy-2-butanone	64	8.535	3-hydroxy-2-butanone	78
8.802	d8-toluene	IS	8.802	d8-toluene	IS	8.801	d8-toluene	IS
			8.93	toluene	55	8.939	toluene	70
						9.352	octane	91
10.153	1,3-bis(trimethylsilyl) benzene	78	10.151	decamethyl tetrasiloxane	78	10.16	hexamethyl cyclotrisiloxane	90
						10.27	ethyl ester butanoic acid	91
			10.646	hexanal	78	10.637	hexanal	91
10.897	propylene glycol	80	10.921	propylene glycol	50	10.848	propylene glycol	86
						12.169	butanoic acid	91
12.548	p-xylene	87	12.546	p-xylene	94	12.546	p-xylene	97
						12.647	furfural	86
13.252	d10-o-xylene	IS	13.252	d10-o-xylene	IS	13.252	d10-o-xylene	IS
						14.152	heptanal	95
						14.289	a-pinene	87
15.778	octamethyl cyclotetrasiloxane	72	15.794	octamethyl cyclotetrasiloxane	90	15.875	octamethyl cyclotetrasiloxane	78
16.1	decane	76						
						16.381	2-pentyl furan	76
16.669	benzaldehyde	94	16.675	benzaldehyde	95	16.666	benzaldehyde	97
			17.41	octanal	80	17.418	octanal	95
						18.07	hexanoic acid	83
18.779	4-cyanocyclohexene	97	18.777	4-cyanocyclohexene	97	18.786	4-cyanocyclohexene	97
						19.226	tetramethyl pyrazine	95
			19.52	2-nonanone	97	19.52	2-nonanone	95
19.651	decamethyl-cyclopentasiloxane	76	19.649	nonanal	49	19.658	nonanal	98
19.66	nonanal	53						
20.78	carbonic acid, butyl ester octyl ester	46	20.787	decanal	91	20.786	decanal	91
			21.255	3,5-dimethyl benzaldehyde	91	21.245	3,5-dimethyl benzaldehyde	90
21.34	dodecamethyl-cyclohexasiloxane	91	21.337	dodecamethyl-cyclohexasiloxane	94	21.337	dodecamethyl-cyclohexasiloxane	91
22.533	2-chloro-1,3-butadiene dimer	42						
22.918	dodecamethyl pentasiloxane	40						
						23.576	a-muurolene	53
24.836	2H-pyran-2-one, tetrahydro-6-pentyl	96						
			25.798	diethyl phthalate	96	25.797	diethyl phthalate	93

Sample Time / Volume for the test

Popcorn Emission Phase 1 Popcorn 8 Test (POP-Phase1P8)

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	2680	Phase1P7&P8-FieldBlank	2/23/2004	FIELD BLANK							
TENAX	2682	Phase1P8-Background	2/23/2004	16:18	16:23	16:20	97308-1	158.5	5.0	0.792	21.7
TENAX	2684	Phase1P8-POPPING	2/23/2004	16:40	16:42	16:41	97308-1	158.5	2.0	0.317	21.7
TENAX	2687	Phase1P8-OPENING	2/23/2004	16:43	16:45	16:44	97308-1	158.5	2.0	0.317	21.7

Data Summary of Duplicates/Triplicates for the Test

No duplicate samples for this test.

Chamber Background Measured for the Test

		Concentration (ug/m3)	
ID	Description	p-Xylene	2,3-Butanedione
2682	Phase1P8-Background	~~2.67~~	~~7.17~~

Field Blanks Measured for the Test (ng/tube)

ID	Description	Date	p-Xylene	2,3-Butanedione
2680	Phase1P7&P8-FieldBlank	2/23/2004	~~0.14~~	~~0.01~~

Field Controls Measured for the Test (ng/tube)

There were no field controls for this test.