Summary

Test Name:	DCC Record in Popcorn Emission Phase 1 Tests After Popcorn 9 Test
Purpose:	Record DCC Results
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:	March -April, 2004
Analysis Dates:	March - April, 2004
Review Status:	Preliminary review by Xiaoyu Liu was completed on , 2004
Final Review:	

VERSION 1

PoP-DCC-Phase1afterP9.xls

DCC Record in Popcorn Emission Phase 1 Tests After Popcorn 9 Test

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.

This notebook records DCC results for popcorn emission tests in Phase1 after P9 since EPA staff process data after P9 test.

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
DCC2686	DCC 120803STDB-3 (P10-P13)	3/5/2004	15:18	XLIU
DCC2705	DCC 121103STD-3 (P10-P13)	3/5/2004	16:17	XLIU
DCC2741	DCC 120803STDB-3 (P14-P19)	3/12/2004	15:48	XLIU
DCC2744	DCC 121103STD-3 (P14-P19)	3/12/2004	16:47	XLIU
DCC2745	DCC 120803STDB-3 (P14-P19)	3/15/2004	23:35	XLIU
DCC2746	DCC 121103STD-3 (P14-P19)	3/16/2004	0:34	XLIU
DCC2759	DCC 120803STDB-3 (P20-P25)	3/19/2004	15:44	XLIU
DCC2758	DCC 121103STD-3 (P20-P25)	3/19/2004	16:43	XLIU
DCC2765	DCC 120803STDB-3 (P20-P25)	3/20/2004	16:08	XLIU
DCC2766	DCC 121103STD-3 (P20-P25)	3/20/2004	17:07	XLIU
DCC2782	DCC 120803STDB-3 (P26-P36)	3/23/2004	19:39	XLIU
DCC2783	DCC 121103STD-3 (P26-P36)	3/23/2004	20:39	XLIU
DCC2786	DCC 120803STDB-3 (P26-P36)	3/24/2004	17:04	XLIU
DCC2787	DCC 121103STD-3 (P26-P36)	3/24/2004	18:02	XLIU
DCC2796	DCC 120803STDB-3 (P26-P36)	3/25/2004	11:40	XLIU
DCC2802	DCC 121103STD-3 (P26-P36)	3/25/2004	12:38	XLIU
DCC2832	DCC 120803STDB-3 (P37-P42)	4/1/2004	17:21	XLIU
DCC2833	DCC 121103STD-3 (P37-P42)	4/1/2004	18:19	XLIU
DCC2834	DCC 120803STDB-3 (P37-P42)	4/2/2004	17:44	XLIU
DCC2835	DCC 121103STD-3 (P37-P42)	4/2/2004	18:43	XLIU
DCC2854	DCC 120803STDB-3 (P43-P44)	4/12/2004	13:33	XLIU
DCC2855	DCC 121103STD-3 (P43-P44)	4/12/2004	14:32	XLIU
DCC2847	DCC 120803STDB-3 (P45-P50)	4/14/2004	18:42	XLIU
DCC2844	DCC 121103STDB-3 (P45-P50)	4/14/2004	19:40	XLIU
DCC2890	DCC 120803STDB-3 (P45-P50)	4/15/2004	17:08	XLIU
DCC2891	DCC 121103STDB-3 (P45-P50)	4/15/2004	18:07	XLIU
DCC2911	DCC 120803STDB-3 (P51-P57)	4/21/2004	21:38	XLIU
DCC2913	DCC 121103STDB-3 (P51-P57)	4/21/2004	22:37	XLIU
DCC2914	DCC 120803STDB-3 (P51-P57)	4/23/2004	6:11	XLIU
DCC2915	DCC 121103STDB-3 (P51-P57)	4/23/2004	7:10	XLIU
DCC2939	DCC 120803STDB-3 (P57-P61)	4/27/2004	12:55	XLIU
DCC2938	DCC 121103STDB-3 (P57-P61)	4/27/2004	1:55	XLIU
DCC2949	DCC 120803STDB-3 (P62-P65)	5/1/2004	12:12	XLIU
DCC2943	DCC 121103STDB-3 (P62-P65)	5/1/2004	13:11	XLIU
DCC2947	DCC 120803STDB-3 (P62-P65)	5/3/2004	13:09	XLIU
DCC2950	DCC 121103STDB-3 (P62-P65)	5/3/2004	14:07	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	7556314	17785710	171.213	171.468	99.9	DCC2686	3/5/2004	Pass
2,3-Butanedione	0.385	3074447	17013530	201.756	190.314	106.0	DCC2705	3/5/2004	Pass
p-Xylene	1.068	7611083	18138008	169.104	171.468	98.6	DCC2741	3/12/2004	Pass
2,3-Butanedione	0.385	2955937	17389260	189.788	190.314	99.7	DCC2744	3/12/2004	Pass
p-Xylene	1.068	7654381	17776075	173.529	171.468	101.2	DCC2745	3/15/2004	Pass
2,3-Butanedione	0.385	2608422	17188766	169.429	190.314	89.0	DCC2746	3/16/2004	Pass
p-Xylene	1.068	7476969	18000477	167.394	171.468	97.6	DCC2759	3/19/2004	Pass
2,3-Butanedione	0.385	2770668	17308735	178.720	190.314	93.9	DCC2758	3/19/2004	Pass
p-Xylene	1.068	7622261	17884081	171.757	171.468	100.2	DCC2765	3/20/2004	Pass
2,3-Butanedione	0.385	2690524	17822161	168.551	190.314	88.6	DCC2766	3/20/2004	Pass
p-Xylene	1.068	7432734	17859715	167.715	171.468	97.8	DCC2782	3/23/2004	Pass
2,3-Butanedione	0.385	2468259	17433158	158.077	190.314	83.1	DCC2783	3/23/2004	Pass
p-Xylene	1.068	7520229	17505040	173.127	171.468	101.0	DCC2786	3/24/2004	Pass
2,3-Butanedione	0.385	2397629	17145503	156.130	190.314	82.0	DCC2787	3/24/2004	Pass
p-Xylene	1.068	7246883	17239550	169.404	171.468	98.8	DCC2796	3/25/2004	Pass
2,3-Butanedione	0.385	3243381	16441641	220.246	190.314	115.7	DCC2802	3/25/2004	Pass
p-Xylene	1.068	7323779	17330223	170.306	171.468	99.3	DCC2832	4/1/2004	Pass
2,3-Butanedione	0.385	3077826	17043828	201.619	190.314	105.9	DCC2833	4/1/2004	Pass
p-Xylene	1.068	7212397	16817717	172.827	171.468	100.8	DCC2834	4/2/2004	Pass
2,3-Butanedione	0.385	2762808	16600208	185.820	190.314	97.6	DCC2835	4/2/2004	Pass
p-Xylene	1.068	7760339	17505170	178.654	171.468	104.2	DCC2854	4/12/2004	Pass
2,3-Butanedione	0.385	3308279	17160005	215.248	190.314	113.1	DCC2855	4/12/2004	Pass
p-Xylene	1.068	7529468	17603390	172.372	171.468	100.5	DCC2847	4/14/2004	Pass

2,3-Butanedione	0.385	2858083	17355300	183.864	190.314	96.6	DCC2844	4/14/2004	Pass
p-Xylene	1.068	6895207	16750717	165.887	171.468	96.7	DCC2890	4/15/2004	Pass
2,3-Butanedione	0.385	2983246	16341588	203.821	190.314	107.1	DCC2891	4/15/2004	Pass
p-Xylene	1.068	6772977	16883649	161.663	171.468	94.3	DCC2911	4/21/2004	Pass
2,3-Butanedione	0.385	2640383	16695415	176.573	190.314	92.8	DCC2913	4/21/2004	Pass
p-Xylene	1.068	6960131	17273698	162.379	171.468	94.7	DCC2914	4/23/2004	Pass
2,3-Butanedione	0.385	3070529	17002651	201.628	190.314	105.9	DCC2915	4/23/2004	Pass
p-Xylene	1.068	6949091	17084448	163.917	171.468	95.6	DCC2939	4/27/2004	Pass
2,3-Butanedione	0.385	3165327	16555472	213.467	190.314	112.2	DCC2938	4/27/2004	Pass
p-Xylene	1.068	6745386	16967654	160.208	171.468	93.4	DCC2949	5/1/2004	Pass
2,3-Butanedione	0.385	3023404	16504801	204.522	190.314	107.5	DCC2943	5/1/2004	Pass
p-Xylene	1.068	6879151	16532850	167.682	171.468	97.8	DCC2947	5/3/2004	Pass
2,3-Butanedione	0.385	3042353	16020735	212.022	190.314	111.4	DCC2950	5/3/2004	Pass

	%Recovery								
Date	3/5/2004	3/12/2004	3/15/2004	3/19/2004	3/20/2004	3/23/2004	3/24/2004	3/25/2004	4/1/2004
p-Xylene	99.9	99	101.20	97.62	100.2	97.8	101.0	98.8	99.3
2,3-Butanedione	106.0	100	89.03	93.91	88.6	83.1	82.0	115.7	105.9

4/2/2004	4/12/2004	4/14/2004	4/15/2004	4/21/2004	4/23/2004	4/27/2004
100.8	104.2	100.5	96.7	94.3	94.7	95.6
97.6	113.1	96.6	107.1	92.8	105.9	112.2