LABORATORY DATA AND DATA PACKAGE REVIEW

(Technical and Administrative Review)

Project number: RP1581	Analyst: (b) (7)(C) [redacted]	Reviewer: (b) (7)(C) [redacted]
Project name: Freedom Industries		
Technique: HS-GC-MS	PAC: (b) (7)(C) [redacted]	Date submitted for review: 6/16/14

Administrative and Technical Review

Data package includes:		Verification:	
✓	Objective of work	✓	Proofread for content
✓	List of samples analyzed	✓	Check spelling and punctuation in text
✓	Method reference(s)	✓	Check for data transcription errors
✓	Description of sample prep/subsampling	✓	Check spelling of compounds/analytes
✓	Instrument identification	✓	Project number, analyst's initials and date on each NEIC generated record
✓	Software (include version number)	✓	Instrument Logbook entry by analyst
✓	QC requirements/data quality summary	✓	Reference to and description of tables
✓	Date of analysis		
✓	Summary of results		

Administrative and technical findings summary and actions requested, if any. Minor corrections, for example things noted by sticky note, need not be listed:*

Reviewer's signature: **Date:**

Response to Action(s) Requested*

Analyst's signature: **Date:**

Final Statement of Reviewer

I have reviewed this data package and it meets the documentation requirements of the NEIC Quality System. The data support the results being reported.

Reviewer's signature: (b) (7)(C) [redacted] **Date:** 6/23/14

I have presented a summary of QC data from these analyses to a Branch QA representative.
Data were provided by: email _____ hardcopy _____ LIMS X

Analyst's signature (b) (7)(C) [redacted] **Date:** 6/23/14

*Attach additional sheets as necessary.

Analysis Summary (Use n/a if item is not applicable to your analysis)

1	Project Code	RP1581
2	Project Name	Freedom Industries
3	Project Location	Charleston, WV
4	Analyst	(b) (7)(C) [redacted]
5	Date of Summary	06/12/14
6	Time Frame of Analysis	May – June 2014
7	Objective of Analysis	Determine methanol concentration in samples
8	Number of Samples/Aliquots for analysis	8 samples / 11 aliquots for separate phases
9	Physical Phase(s) [how many of each phase]	11 liquid phases
10	Analysis Method /Procedure	*Volatile Organic Analysis by Gas Chromatography/Mass Spectrometry Appendix A*, NEICPROC/00-002R4, as guidance
11	Sample Preparation Method/Procedure	NEICPROC/00-002R4 and NEICPROC/00-049R3 as guidance, and see spreadsheet
12	Analysis Matrix/Solvent	Methanol:water
13	Instrument Identification	GC-MS with Combipal (#4915, 4784, 4785)
14	Software Identification	Environmental ChemStation MSD ChemStation E.02.00.493
15	Instrument Method	MeOH 052014.M
16	Additional Analysis Information	Matrix Modifying Solution did not have the pH lowered
17	Data Processing Method	Microsoft Office Excel 2007
18	Under Scope of Accreditation (yes/No)?	Yes
19	Blank(s) evaluation (Yes/No)?	Yes and blanks were clean
[redacted]		
20	Initial Tune	No new tune was done
21	Internal Standard Recovery	Stable
22	Surrogate Recovery	N/A
23	Initial Calibration Verification	Initial calibration was done 02/13/14
24	Continuing Calibration Verification	Analyzed CCVs at beginning of all sequences and calibration curve remained stable
25	Replicate % Relative Standard Deviation	Station #1, L-1: 4.98% Station #1, L-2: 1.11%
26	Spike % Recovery	Station #1, L-1: 95.8% Station #1, L-2: 89.4%
27	Control Samples % Recovery (or %Deviation)	CCV: -3.97% SCV: -8.28%
28	QC results outside of acceptance Criteria:	Based on the Criteria in the NEIC procedures the QC results were within the acceptance limits.
29	Approach for estimated Measurement Uncertainty	Relative standard error of the mean, the highest error is reported as the uncertainty.
30	Estimate of Measurement Uncertainty	2.88%
31	Comment(s)	

Methanol Concentrations

Station #	LIMS Tag #	$[MeOH]_{sample}$ (µg/mL)	Reporting Limit (µg/mL)
Discharge Sample, L-2	N405006-02	< 500	500
Station #2	N405006-09	< 2000	2000
Station #3	N405006-10	< 1000	1000
Station #4, L-2	N405006-12	7210	2000
RDC-777 Tank 203, L-1	N405006-05	< 500	500
Discharge Sample, L-1	N405006-01	< 1000	1000
Station #4, L-1	N405006-11	1140	1000
Storage Tank 393*	N405006-03	< 500	500
MCHM Tank	N405006-04	2710	1000
Station #1, L-1 (n=3)	N405006-07	3660	1000
Station #1, L-2 (n=3)	N405006-08	16300	2500

Quality Control for Sample Triplicates

Station #	LIMS Tag #	$[MeOH]_{obs}$ (µg/mL)	$[MeOH]_{sample}$ (µg/mL)	$[Mean]_{obs}$ (µg/mL)	$[Mean]_{sample}$ (µg/mL)	Std Dev_{sample} (µg/mL)	% RSD_{sample}	SEM_{sample}	$RSEM_{sample}$
Station #1, L-1	N405006-07	17.26	3452	18.29	3657	182.3	4.98	105.2	2.88
Station #1, L-1	N405006-07DUP1	19.00	3800						
Station #1, L-1	N405006-07DUP2	18.60	3720						
Station #1, L-2	N405006-08	32.77	16385	32.63	16315	180.5	1.11	104.2	0.639
Station #1, L-2	N405006-08DUP3	32.90	16450						
Station #1, L-2	N405006-08DUP4	32.22	16110						

Sample Concentrations

Station #	LIMS Tag #	Data File	$[MeOH]_{obs}$ (µg/mL)	$[MeOH]_{sample}$ (µg/mL)
Discharge Sample, L-2	N405006-02	052214_RP1581_AMH_003	< RL	< RL
Station #2	N405006-09	052214_RP1581_AMH_008	< RL	< RL
Station #3	N405006-10	052214_RP1581_AMH_009	< RL	< RL
Station #4, L-2	N405006-12	052214_RP1581_AMH_010	18.02	7208
RDC-777 Tank 203, L-1	N405006-05	052214_RP1581_AMH_012	< RL	< RL
Discharge Sample, L-1	N405006-01	052214_RP1581_AMH_013	< RL	< RL
Station #4, L-1	N405006-11	052214_RP1581_AMH_015	5.68	1136
Storage Tank 393*	N405006-03	052214_RP1581_AMH_017	4.41	441
MCHM Tank	N405006-04	052214_RP1581_AMH_018	13.56	2712

*N405006-03 shows methanol in sample. However, due to amount of other compounds in sample the decision was made to not analyze with higher sample amount due to overload on column.

Triplicate Data

Station #	LIMS Tag #	Data File	$[MeOH]_{obs}$ (µg/mL)	$[MeOH]_{sample}$ (µg/mL)	$[Mean]_{obs}$ (µg/mL)	$[Mean]_{sample}$ (µg/mL)	Std Dev_{sample} (µg/mL)	% RSD_{sample}	SEM_{sample}	$RSEM_{sample}$
Station #1, L-1	N405006-07	052814_RP1581_AMH_003	17.26	3452	18.29	3657	182.3	4.98	105.2	2.88
Station #1, L-1	N405006-07DUP1	052814_RP1581_AMH_004	19.00	3800						
Station #1, L-1	N405006-07DUP2	052814_RP1581_AMH_005	18.60	3720						
Station #1, L-2	N405006-08	052814_RP1581_AMH_007	32.77	16385	32.63	16315	180.5	1.11	104.2	0.639
Station #1, L-2	N405006-08DUP3	052814_RP1581_AMH_008	32.90	16450						
Station #1, L-2	N405006-08DUP4	052814_RP1581_AMH_009	32.22	16110						

Spike Data

Station #	LIMS Tag #	Data File	$[MeOH]_{obs}$ (µg/mL)	[Spike]	$[Sample]_{obs}$ (µg/mL)	Spike Recovered (µg/mL)	% Recovery
Station #1, L-1	N405006-07MS1	052814_RP1581_AMH_006	41.20	25.00	17.26	23.94	95.8
Station #1, L-2	N405006-08MS2	052814_RP1581_AMH_010	55.11	25.00	32.77	22.34	89.4

Sample Preparations

Station #	NEIC Tag #	LIMS Tag #	Amt Internal Std (µL)	Amt of Sample (µL)	Amt of M.M.S. (µL)	Amt of Spike (µL)	Spike Conc (µg/mL)
Discharge Sample, L-2	H01499	N405006-02	20	20	1960	-	-
Station #1, L-2	-	N405006-08	20	4	1976	-	-
Station #1, L-2	-	N405006-08	20	4	1926	50	25
Station #2	-	N405006-09	20	5	1975	-	-
Station #3	-	N405006-10	20	10	1970	-	-
Station #4, L-2	-	N405006-12	20	5	1975	-	-
Storage Tank 393	H01500	N405006-03	20	20	1960	-	-
RDC-777 Tank 203, L-1	-	N405006-05	20	20	1960	-	-
Station #1, L-1	-	N405006-07	20	10	1970	-	-
Station #1, L-1	-	N405006-07	20	10	1920	50	25
Station #4, L-1	-	N405006-11	20	10	1970	-	-
Discharge Sample, L-1	H01499	N405006-01	20	10	1970	-	-
MCHM Tank	H01501	N405006-04	20	10	1970	-	-

Standard Calculations

Mid-Calibration Check		
Date	Known Conc (µg/mL)	Observed Conc (µg/mL)
5/22/2014	50.0	46.47
5/22/2014		50.39
5/28/2014		47.19
Average	-	**48.02**
% Difference from known value	-	-3.97

Second Source Check		
Date	Known Conc (µg/mL)	Observed Conc (µg/mL)
5/22/2014	50.0	45.86
% Difference from known value		**-8.28**

CombiPal Method

Alcohols in Water by Headspace GC/MS			
Headspace = COMBIPAL with 20-mL vials containing 2 mL total volume			
Incubate temp:	80C	Pullup Delay:	3 s
Incubate time:	6 min	Inject to:	GC Inj2
Inject volume:	500 uL	Inject Speed:	800 uL/s
Syringe Temp:	100C	Pre inj delay:	100 ms
Agi Speed:	250 rpm	Post inj delay:	500 ms
Agi on Time:	15 s	Syr Flushing:	0:03:00
Agi off Time:	5 s	GC Runtime:	0:15:33
Fill Speed:	50 uL/s		

METHOD CONTROL PARAMETERS

Method Information For: C:\MSDCHEM\1\METHODS\MeOH 052014.M
Method Sections To Run:

( ) Save Copy of Method With Data
( ) Instrument Control Pre-Run Cmd/Macro =
( ) Data Analysis Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Instrument Control Post-Run Cmd/Macro =
( ) Data Analysis Post-Run Cmd/Macro =
Method Comments:

END OF METHOD CONTROL PARAMETERS

INSTRUMENT CONTROL PARAMETERS: Intrument 1 [EQ]

C:\MSDCHEM\1\METHODS\MeOH 052014.M
Tue May 27 13:50:03 2014

Control Information

Sample Inlet : GC
Injection Source : External Device
Mass Spectrometer : Enabled

6890 GC METHOD

OVEN
Initial temp: 35 'C (On)
Initial time: 3.00 min
Ramps:

#	Rate	Final temp	Final time
1	15.00	220	0.00
2	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 15.33 min

Maximum temp: 260 'C
Equilibration time: 0.50 min

FRONT INLET (HP PTV)
Mode: Split
Initial temp: 50 'C (Off)
Pressure: 0.00 psi (Off)
Total flow: 45.0 mL/min
Gas saver: Off
Gas type: Helium

BACK INLET (SPLIT/SPLITLESS)
Mode: Split
Initial temp: 170 'C (On)
Pressure: 13.18 psi (On)
Split ratio: 30:1
Split flow: 24.0 mL/min
Total flow: 26.9 mL/min
Gas saver: Off
Gas type: Helium

COLUMN 1
Capillary Column
Model Number: J&W 128-1324
DB-624
Max temperature: 260 'C
Nominal length: 25.0 m
Nominal diameter: 200.00 um
Nominal film thickness: 1.12 um
Mode: constant flow
Initial flow: 0.8 mL/min
Nominal init pressure: 13.19 psi
Average velocity: 35 cm/sec
Inlet: Back Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2
(not installed)

FRONT DETECTOR (Signal-only)
Photo multiplier: Off

BACK DETECTOR (FPD)
Temperature: 250 'C (Off)
Hydrogen flow: 75.0 mL/min (Off)
Oxidizer flow: 100.0 mL/min (Off)
Oxidizer Gas Type: Air
Mode: Constant makeup flow
Makeup flow: 15.0 mL/min (Off)
Makeup Gas Type: Helium
Flame: Off
Lit offset: 2.00
Photo multiplier: Off

(b) (7)(C) [redacted] RP1581 06/16/2014

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 1
Use: MSD Transfer Line Heater
Description:
Initial temp: 250 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0(Off)		

THERMAL AUX 2
Unknown Thermal Aux Type

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

GC Injector

Front Injector:

Sample Washes	0
Sample Pumps	6
Injection Volume	1.00 microliters
Syringe Size	10.0 microliters
Nanoliter Adapter	Off
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

Back Injector:

Sample Washes	0
Sample Pumps	6
Injection Volume	1.00 microliters
Syringe Size	10.0 microliters
Nanoliter Adapter	Off
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

Column 1 Inventory Number : AB001
Column 2 Inventory Number :

MS ACQUISITION PARAMETERS

General Information
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Tune File : 11122013BFB.U
Acquistion Mode : Scan

(b) (7)(C) [redacted] P1581 06/16/2014

MS Information
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Solvent Delay : 1.50 min

EMV Mode : Absolute
Resulting EM Voltage : 1459

[Scan Parameters]

Low Mass : 28.5
High Mass : 220.0
Threshold : 100
Sample # : 3 A/D Samples 8
Plot 2 low mass : 29.0
Plot 2 high mass : 220.0

[MSZones]

MS Source : 230 C maximum 250 C
MS Quad : 150 C maximum 200 C

END OF MS ACQUISITION PARAMETERS

TUNE PARAMETERS for SN: US82321641

Trace Ion Detection is OFF.

EMISSION : 34.610
ENERGY : 69.922
REPELLER : 19.904
IONFOCUS : 73.075
ENTRANCE_LE : 0.000
EMVOLTS : 1811.765
Actual EMV : 1458.82
GAIN FACTOR : 0.43
AMUGAIN : 2128.000
AMUOFFSET : 131.000
FILAMENT : 2.000
DCPOLARITY : 0.000
ENTLENSOFFS : 13.302@ 3 13.302@ 50 9.537@ 69 11.043@131 11.294@219 18.824@ 414 23.090@502 23.090@799
MASSGAIN : -70.000
MASSOFFSET : -12.000

END OF TUNE PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

(b) (7)(C) [redacted] P1581 06/16/2014

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\MeOH 052014.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: No
Printer: Yes
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
C:\Database\NIST05a.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: epatemp.txt

Generate Report During Run Method: No

Methanol
Calibration Last Updated: Tue May 27 10:40:33 2014

Reference Window: 2.00 Minutes

Non-Reference Window: 1.00 Minutes
Correlation Window: 0.10 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

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1) Methanol-d3 (ISTD)

Ret. Time 1.903 min., Extract & Integrate from 1.403 to 2.403 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 35.10			*** METH DEFAULT ***
Q1 33.10	133.40	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/mL)	Response
1	50.000	28304
2	50.000	30352
3	50.000	28857
4	50.000	28840
5	50.000	29044
6	50.000	29170

Qualifier Peak Analysis ON ISTD conc: 50.000 ug/mL
Curve Fit: Linear

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2) Methanol ( )

Ret. Time 1.914 min., Extract & Integrate from 1.414 to 2.414 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 31.10			*** METH DEFAULT ***
Q1 29.10	60.00	20.0	*** METH DEFAULT ***
Q2 32.10	77.70	20.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/mL)	Response
1	5.000	6317
2	10.000	10608
3	25.000	19202
4	50.000	46008
5	75.000	68710
6	100.000	89457

Qualifier Peak Analysis ON
Curve Fit: Linear

END OF DATA ANALYSIS PARAMETERS

Tue May 27 13:50:04 2014

US EPA, National Enforcement Investigations Center Laboratory

SAMPLE RECEIPT REPORT

Work Order : N405006

Client: CID Region 3 - Philadelphia Area Office
Project: Freedom Industries
Project Manager: (b) (7)(C) [redacted]
Project Number: RP1581

Report To:
CID Region 3 - Philadelphia Area Office
(b) (7)(C) [redacted]
1650 Arch Street, 3CE00
Philadelphia, PA 19103-2029
Phone: 215-814-2381

Date Due:
02/06/17 00:00 (999 day Turn Around Time-TAT)

Received By: (b) (7)(C) [redacted]
Logged In By: [redacted]

Date Received: 05/14/14 08:42
Date Logged In: 05/14/14 08:42

Custody Seals : YES
Preservation Confirmed : NO
Containers Intact : NO
Received On Ice : NO
COC/Labels Agree : YES

Analysis	Assigned Analyst	Comments
N405006-01 Discharge Sample, L-1 [Liquid] Sampled 01/09/14 15:30-Eastern NEIC Tag: H01499		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Discharge Sample Collected on the Incident Date
EPA 8270	Angie Hunter	Discharge Sample Collected on the Incident Date
Water Content	Ben Burns	Discharge Sample Collected on the Incident Date
N405006-02 Discharge Sample, L-2 [Liquid] Sampled 01/09/14 15:30-Eastern NEIC Tag: H01499		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Discharge Sample Collected on the Incident Date
EPA 8270	Angie Hunter	Discharge Sample Collected on the Incident Date
Water Content	Ben Burns	Discharge Sample Collected on the Incident Date
N405006-03 Storage Tank 393 [Liquid] Sampled 03/05/14 10:44-Eastern NEIC Tag: H01500		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Content sample from storage tank
LCMS-RP1581	Daniel Hurlbut	Content sample from storage tank
Water Content	Ben Burns	Content sample from storage tank
N405006-04 MCHM Tank [Liquid] Sampled 01/09/14 20:00-Mountain NEIC Tag: H01501		
Alcohols by Headspace GC/MS ✓	Angie Hunter	MCHM product material
EPA 8270	Angie Hunter	MCHM product material
Water Content	Ben Burns	MCHM product material
N405006-05 RDC-777 Tank 203, L-1 [Liquid] Sampled 03/12/14 11:45-Eastern NEIC Tag: none		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample of tank contents, top liquid layer
LCMS-RP1581	Daniel Hurlbut	Sample of tank contents, top liquid layer
Water Content	Ben Burns	Sample of tank contents, top liquid layer
N405006-06 RDC-777 Tank 203, S-2 [Solid] Sampled 03/12/14 11:45-Eastern NEIC Tag: none		
LCMS-RP1581	Daniel Hurlbut	Sample of tank contents- bottom layer having sludge consistency
N405006-07 Station #1, L-1 [Liquid] Sampled 01/09/14 00:00-Eastern NEIC Tag: none Triplicate/Spike		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
EPA 8270	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
Water Content	Ben Burns	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer

Work Order : N405006

Analysis	Assigned Analyst	Comments
N405006-08 Station #1, L-2 [Liquid] Sampled 01/09/14 00:00-Eastern NEIC Tag: none Triplicate /Spike		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, lower liquid layer
EPA 8270	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, lower liquid layer
Water Content	Ben Burns	Sample submitted by Freedom Industries Legal Counsel, lower liquid layer
N405006-09 Station #2 [Liquid] Sampled 01/09/14 00:00-Eastern NEIC Tag: none		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel
EPA 8270	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel
Water Content	Ben Burns	Sample submitted by Freedom Industries Legal Counsel
N405006-10 Station #3 [Liquid] Sampled 01/09/14 00:00-Eastern NEIC Tag: none		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel
EPA 8270	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel
Water Content	Ben Burns	Sample submitted by Freedom Industries Legal Counsel
N405006-11 Station #4, L-1 [Liquid] Sampled 01/09/14 00:00-Eastern NEIC Tag: none		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
EPA 8270	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
Water Content	Ben Burns	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
N405006-12 Station #4, L-2 [Liquid] Sampled 01/09/14 00:00-Eastern NEIC Tag: none		
Alcohols by Headspace GC/MS ✓	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
EPA 8270	Angie Hunter	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer
Water Content	Ben Burns	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement and Compliance Assurance
Office of Criminal Enforcement, Forensics, and Training

Data Summary Report

Project Number: NEICRP1581

Project Name : Freedom Industries

Reported : August 07, 2014

Laboratory Project Leader : _____

(b) (7)(C) [redacted] Chemist_PM

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
PO Box 25227
Building 25, Denver Federal Center
Denver, Colorado 80225

NEIC Laboratory Report

ANALYTICAL REPORT FOR SAMPLES

Station ID	Station Comments	Laboratory ID	Matrix	Date Sampled	Date Received
Discharge Sample, 1	Discharge Sample Collected on the Incident Date	N405006-01	Liquid	01/09/14 15:30	03/12/14 10:15
Discharge Sample, 1	Discharge Sample Collected on the Incident Date	N405006-02	Liquid	01/09/14 15:30	03/12/14 10:15
Storage Tank 393	Content sample from storage tank	N405006-03	Liquid	03/05/14 10:44	03/12/14 10:15
MCHM Tank	MCHM product material	N405006-04	Liquid	01/09/14 20:00	05/14/14 10:15
RDC-777 Tank 203	Sample of tank contents, top liquid layer	N405006-05	Liquid	03/12/14 11:45	03/18/14 11:05
Station #1, L-1	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer	N405006-07	Liquid	01/09/14 00:00	03/31/14 10:30
Station #1, L-2	Sample submitted by Freedom Industries Legal Counsel, lower liquid layer	N405006-08	Liquid	01/09/14 00:00	03/31/14 10:30
Station #2	Sample submitted by Freedom Industries Legal Counsel	N405006-09	Liquid	01/09/14 00:00	03/31/14 10:30
Station #3	Sample submitted by Freedom Industries Legal Counsel	N405006-10	Liquid	01/09/14 00:00	03/31/14 10:30
Station #4, L-1	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer	N405006-11	Liquid	01/09/14 00:00	03/31/14 10:30
Station #4, L-2	Sample submitted by Freedom Industries Legal Counsel, upper liquid layer	N405006-12	Liquid	01/09/14 00:00	03/31/14 10:30

NEIC Laboratory Report

Volatile Organic Compounds

Station ID	Sample Tag	Lab Number	Analyte	Result	Report Limit	Units	Analyzed	Notes
Discharge Sample, L-1	H01499	N405006-01	Methanol	< 1000	1000	ug/mL	05/22/14	
Discharge Sample, L-2	H01499	N405006-02	Methanol	< 500	500	ug/mL	05/22/14	
MCHM Tank	H01501	N405006-04	**Methanol**	**2710**	1000	ug/mL	05/22/14	
RDC-777 Tank 203, L-1	none	N405006-05	Methanol	< 500	500	ug/mL	05/22/14	
Station #1, L-1	none	N405006-07	**Methanol**	**3660**	1000	ug/mL	05/28/14	
Station #1, L-2	none	N405006-08	**Methanol**	**16300**	2500	ug/mL	05/28/14	
Station #2	none	N405006-09	Methanol	< 2000	2000	ug/mL	05/22/14	
Station #3	none	N405006-10	Methanol	< 1000	1000	ug/mL	05/22/14	
Station #4, L-1	none	N405006-11	**Methanol**	**1140**	1000	ug/mL	05/22/14	
Station #4, L-2	none	N405006-12	**Methanol**	**7210**	2000	ug/mL	05/22/14	
Storage Tank 393	H01500	N405006-03	Methanol	< 500	500	ug/mL	05/22/14	

NEIC Laboratory Report

Volatile Organic Compounds - Quality Control

Analyte	Result	Report Limit	Units	Spike Level	Source Result	%REC	% Rec Limits	RPD	RPD Limit	Notes
Batch 4061201 - EPA 5021										
Blank (4061201-BLK1)				Prepared & Analyzed: 05/22/14						
Methanol	ND	5.00	ug/mL							
Blank (4061201-BLK2)				Prepared & Analyzed: 05/22/14						
Methanol	ND	5.00	ug/mL							
Calibration Check (4061201-CCV1)				Prepared & Analyzed: 05/22/14						
Methanol	46.5	5.00	ug/mL				0-200			
Calibration Check (4061201-CCV2)				Prepared & Analyzed: 05/22/14						
Methanol	50.4	5.00	ug/mL				0-200			
Batch 4061602 - EPA 5021										
Blank (4061602-BLK1)				Prepared & Analyzed: 05/28/14						
Methanol	ND	5.00	ug/mL							
Matrix Spike (4061602-MS1)		**Source: N405006-07**		Prepared & Analyzed: 05/28/14						
Methanol	41.2		ug/mL	25.000	17.3	95.8	5-100			
Matrix Spike (4061602-MS2)		**Source: N405006-08**		Prepared & Analyzed: 05/28/14						
Methanol	55.1		ug/mL	25.000	32.8	89.4	5-100			

Volatile Organic Compounds - Quality Control

Analyte	LabNumber	Result	Units	Avg	RPD	RPD Limit	RSD	RSD Limit
Batch ID : 4061602 Volatile Organic Compounds in Soils & Other Solids REPLICATE DATA								
Sample ID : N405006-07								
Methanol	N405006-07	3450	ug/mL					
	4061602-DUP2	3720	ug/mL	3660	7.47	200	4.98	
	4061602-DUP1	3800	ug/mL		9.60			
Sample ID : N405006-08								
Methanol	N405006-08	16400	ug/mL					
	4061602-DUP4	16100	ug/mL	16300	1.69	200	1.11	
	4061602-DUP3	16500	ug/mL		0.396			

Bench Sheet

Analysis : Alcohols by Headspace GC/MS Batch : 4061201

Matrix : Liquid Prep Method : EPA 5021

Lab Number	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike	Instrument File ID	Surr ID	ul Surr
4061201-BLK1	2.0000	2.0000				052214_RP1581_AMH_001.]		
4061201-BLK2	2.0000	2.0000				052214_RP1581_AMH_021.]		
4061201-CCV1	2.0000	2.0000				052214_RP1581_AMH_002.]		
4061201-CCV2	2.0000	2.0000				052214_RP1581_AMH_019.]		
4061601-SCV1	1.0000	1.0000				052214_RP1581_AMH_020.]		
4061602-BLK1	2.0000	2.0000				052814_RP1581_AMH_001.]		
4061602-DUP1	0.0100	2.0000		N405006-07		052814_RP1581_AMH_004.]		
4061602-DUP2	0.0100	2.0000		N405006-07		052814_RP1581_AMH_005.]		
4061602-DUP3	0.0040	2.0000		N405006-08		052814_RP1581_AMH_008.]		
4061602-DUP4	0.0040	2.0000		N405006-08		052814_RP1581_AMH_009.]		
4061602-MS1	0.0100	2.0000	N002199	N405006-07	50.0	052814_RP1581_AMH_006.]		
4061602-MS2	0.0040	2.0000	N002199	N405006-08	50.0	052814_RP1581_AMH_010.]		
4061603-CCV1	1.0000	1.0000				052814_RP1581_AMH_002.]		
N405006-01	0.0100	2.0000	Freedom Industries			052214_RP1581_AMH_013.]		
N405006-02	0.0200	2.0000	Freedom Industries			052214_RP1581_AMH_003.]		
N405006-03	0.0200	2.0000	Freedom Industries			052214_RP1581_AMH_017.]		
N405006-04	0.0100	2.0000	Freedom Industries			052214_RP1581_AMH_018.]		
N405006-05	0.0200	2.0000	Freedom Industries			052214_RP1581_AMH_012.]		
N405006-07	0.0100	2.0000	Freedom Industries			052814_RP1581_AMH_003.]		
N405006-08	0.0040	2.0000	Freedom Industries			052814_RP1581_AMH_007.]		
N405006-09	0.0050	2.0000	Freedom Industries			052214_RP1581_AMH_008.]		
N405006-10	0.0100	2.0000	Freedom Industries			052214_RP1581_AMH_009.]		
N405006-11	0.0100	2.0000	Freedom Industries			052214_RP1581_AMH_015.]		
N405006-12	0.0050	2.0000	Freedom Industries			052214_RP1581_AMH_010.]		

RP1581 06/19/2014 1852/21/06
(b) (6), (b) (7)
1

US EPA, National Enforcement Investigations Center Laboratory
PREPARATION BENCH SHEET
Alcohols by Headspace GC/MS Batch ID: 4061602

Matrix: Liquid **Prepared using: Volatiles - EPA 5021** **(No Surrogate)**

Lab Number		Prepared	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike	ul Surrogate	Sample Tag ID	Extraction Comments
4061602-BLK1	Blank	05/28/14 14:15	2	2						
4061602-DUP1	Duplicate	05/28/14 15:26	0.01	2		N405006-07				
4061602-DUP2	Duplicate	05/28/14 15:50	0.01	2		N405006-07				
4061602-DUP3	Duplicate	05/28/14 17:31	0.004	2		N405006-08				
4061602-DUP4	Duplicate	05/28/14 18:04	0.004	2		N405006-08				
4061602-MS1	Matrix Spike	05/28/14 16:23	0.01	2	N002199	N405006-07	50			
4061602-MS2	Matrix Spike	05/28/14 18:37	0.004	2	N002199	N405006-08	50			
N405006-07	Sample	05/28/14 15:02	0.01	2					none	
N405006-08	Sample	05/28/14 16:57	0.004	2					none	

US EPA, National Enforcement Investigations Center Laboratory
PREPARATION BENCH SHEET
Alcohols by Headspace GC/MS Batch ID: 4061201

Matrix: Liquid **Prepared using: Volatiles - EPA 5021** **(No Surrogate)**

Lab Number		Prepared	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike	ul Surrogate	Sample Tag ID	Extraction Comments
4061201-BLK1	Blank	05/22/14 10:33	2	2						
4061201-BLK2	Blank	05/22/14 19:00	2	2						
4061201-CCV1	Calibration Check	05/22/14 10:56	2	2						
4061201-CCV2	Calibration Check	05/22/14 18:10	2	2						
N405006-01	Sample	05/22/14 15:39	0.01	2					H01499	
N405006-02	Sample	05/22/14 11:21	0.02	2					H01499	
N405006-03	Sample	05/22/14 17:19	0.02	2					H01500	
N405006-04	Sample	05/22/14 17:45	0.01	2					H01501	
N405006-05	Sample	05/22/14 15:14	0.02	2					none	
N405006-09	Sample	05/22/14 13:27	0.005	2					none	
N405006-10	Sample	05/22/14 13:53	0.01	2					none	
N405006-11	Sample	05/22/14 16:29	0.01	2					none	
N405006-12	Sample	05/22/14 14:18	0.005	2					none	

CERTIFICATE OF ANALYSIS

PRODUCT NAME	Methyl-d_3 Alcohol
PRODUCT NO.	D-67
LOT NO.	X384P6
DATE:	October 2013

N.M.R.

Specification: 99.8 atom % D

Result: 99.9 atom % D

G.C.

Specification: 98% chemical purity

Result: 99.2% chemical purity

(b) (7)(C)

[redacted]

Quality Control Manager

Expiration: ***Stable for three years after receipt of order if stored under recommended conditions (see section 7 of MSDS). After three years, the compound should be re-checked for chemical purity before use.***

C/D/N ISOTOPES INC. • 88 Leacock Street • Pointe-Claire • Quebec • Canada • H9R 1H1

Tel. (514) 697-6254 (800) 565-4696 Fax (514) 697-6148 www.cdnisotopes.com sales@cdnisotopes.com

(b) (6), (b) (7)(C) RP1581 06/16/2014

Name	Project No.	Location	60281
Amy Hunter	RP1568		Date: 2/10/14

Prepping standards for headspace GC-MS (prepped in water)

Methanol-d_3

CDN Methyl-d_3 Alcohol
99.9% atom
Lot #: X384P6

balance id # 4338
verified balance w/ EPA K-5 weights

$$\frac{0.016g\ MeOH\text{-}d_3}{10mL} \times 0.999 \times \frac{1.0\times10^6\ \mu g}{1g} = 10{,}149.84 \rightarrow \underline{10{,}150\ \mu g/mL}$$

Methanol

B&J
Purge & Trap Grade 99.99%
Lot #: DH379

$$\frac{0.10760g\ MeOH}{10mL} \times 0.9999 \times \frac{1.0\times10^6\ \mu g}{1g} = 10{,}758.92 \rightarrow 10{,}760.\cancel{00}\ \mu g/mL$$ 2/10/14 AH

Storing in fridge [redacted] 2/10/14

2/11/14
Prepping diluted standards

Methanol-d_3 5000 µg/mL

$$(5mL)(5000\ \mu g/mL) = (10{,}150\ \mu g/mL)\,V \rightarrow V = \frac{(5mL)(5000\ \mu g/mL)}{10{,}150\ \mu g/mL} = 2.46\ mL$$

Methanol

$$(1000\ \mu g/mL)(5mL) = (10{,}760\ \mu g/mL)\,V \rightarrow V = \frac{(1000\ \mu g/mL)(5mL)}{10{,}760\ \mu g/mL} = 0.465mL \rightarrow 465\ \mu L$$

(~~Eppendorf~~ 0.5-10mL #219241.9, ~~100-1000~~ µL GRG4)

[redacted] 14

Name	Project No.	Location	60282
(b) (7)(C) [redacted]	RP1568		Date: 2/11/14

Calibration Curve for Methanol 5-100 ug/mL

Using 1000 ug/mL Methanol standard and Methanol-d4 5000 ug/mL as internal standard

Conc of Cal Pt (ug/mL)	Amt of Std (uL)	Amt of Int Std (uL)	Amt of Matrix M. (uL)
5	10	20	1970
10	20		1960
25	50		1930
50	100		AH 2/11/14 ~~1700~~ 1880
75	150		1830
100	200		1780

Methanol-d4 for all calibration & samples is @ 50 ug/mL

$$V = \frac{(50\ ug/mL)(2mL)}{5000\ ug/mL} = 0.02mL \rightarrow 20\ uL$$

Matrix Modifying solution was prepared 12/6/11 w/ 180g NaCl w/ 500mL H_2O. (M.M. was not required)

All standards & samples will be in a total volume of 2mL in headspace vial w/ screw cap

(b) (6), (b) (7) (C) 2/11/14

2/12/14

Processed calibration curve and all looked ok except the 5ug/mL. Noticed the incubation time was 12 minutes in headspace method, changed it to 10 minutes and analyzing a new 5 ug/mL to see if helps prior to analyzing a new calibration curve.

(b) (7) (C) [redacted]

Name	Project No.	Location	60283
(b) (7)(C) [redacted]	RP1568		Date: 2/13/14

Running reagent curve w/ complete incubation. See pg 66-82 for dilutions

Screening/Analyzing samples @ 20 uL w/ 20 uL Int. Std B MMS

[redacted]

Different project dtt 8/7/14 information.

dtt 2/13/14

[redacted]

dtt 2/13/14

Name	Project No.	Location	60290
(b) (7)(C) [redacted]	RP1581		Date: 5/12/14

Checking Methanol (MeOH) calibration set up on 2/13/14 (see pgs 60281 - 60283)
these pgs show cal ranges and set up, as well as standard prep

Using matrix modifying solution (MMS) prepared 12/6/11
(180g NaCl w/ 500 mL H_2O)

Using Methanol-d3 as internal standard for methanol analysis

Making standards: Using methylene Chloride as solvent BBJ lot# CG132
Using 6mL volumetric pipette & 100 uL Drummond pipette
Balance # ~~4575~~ 4338 unified
RH 5/12/14

4-methylcyclohexanemethanol (MCHM)
TCI
Lot # RCGNA-PT >98.0%

$$\frac{0.0971g}{6mL} \times 0.98 \times \frac{1.0 \times 10^6 ug}{1g} = 15,859.6 \rightarrow 15900 ug/mL$$

Dimethyl 1,4-cyclohexanedicarboxylate (DM-1,4DC)
Aldrich
Lot # MKBJ2148V 97%

$$\frac{0.1125g}{6mL} \times 0.97 \times \frac{1.0 \times 10^6 ug}{1g} = 18,187.5 \rightarrow 18200 ug/mL$$

1,4-cyclohexanedimethanol (1,4-CHDM)
Aldrich Solid used spatula
Lot # MKBP6651V 99%

$$\frac{0.0673g}{6mL} \times 0.99 \times \frac{1.0 \times 10^6 ug}{1g} = 11104.5 \rightarrow 11,100 ug/mL$$

Name	Project No.	Location	
(b) (7)(C) [redacted]	RP1581		Date: 5/19/14

60295

Screening samples for MeOH (headspace GC-MS)

10 µL of sample, 20 µL of I.S. (MeOH-d_3), and 1970 µL of MMS

Sample #
N405006-2 (L-2)
N40500608 (L-2)
-09
-10
-12
-03
-05

(b) (7)(C) [redacted] 5/19/14

5/20/14
Screening samples on direct inject GC-MS (10 µL sample, 50 µL surrogate, 940 µL $MeCl_2$)

Sample #
N405006-02
-08
-09
-10
-12
-03
-05

AH 5/20/14

Headspace & Direct inject
-01
-07
-11

(b) (7)(C) [redacted] 5/20/14

5/22/14
Analyzing samples for MeOH

Sample #	Amt I.S. (µL)	Amt of Sample (µL)	Amt M.M.S. (µL)	Spike Amt (µL)
N405006-02	20	20	1960	—
-08	↓	4	1976	50 (25 µg/mL spike) MMS = 1926
-09		5	1975	—
-10		10	1970	—
-12		5	1975	—
-03		20	1960	—
-05		20	1960	—

Name	Project No.	Location	60296
(b) (7)(C) [redacted]	RP1581		Date: 5/22/14

Sample Analysis (cont)

Sample #	Amt of I.S. (µL)	Amt Sample (µL)	Amt M.MS (µL)
N405006-07	20	10	1970
-11	20	10	1970
-01	20	10	1970

Making 2nd Source methanol standard

Honeywell
Lot #DH379

balance #4338 verified

Using 5mL volumetric pipette to add 5mL H_2O (Milli-Q H_2O 18.2Ω)

$$\frac{0.0270\ g}{5mL} \times \frac{1.0 \times 10^6\ \mu g}{1g} = 5400\ \mu g/mL$$

Diluting to 1000 µg/mL
V needed = 926 µL Total Volume = 5mL

Repreping 2 samples
N405006-08 (org) I forgot the internal std (4 µL of sample)
N405006-03 instrument's computer rebooted during this sample (20µL sample)
will place at end of analysis run.

[illegible] 5/22/14

Prepping
N405006-04 10µL of Sample, 20µL I.S. 1970 MMS
(b) (7)(C) [redacted] 5/22/14

Name	Project No.	Location	60297
(b) (7)(C) [redacted]	RP1581		Date: 5/28/14

Prepping 2 sets of triplicates & spikes

Sample #	Amt I.S. (µL)	Amt Sample (µL)	Amt Spike Std (µL)	Amt M.M.S. (µL)	
N405006-07	20	~~4~~ AH 5/28/14 10	50	1970/1920	(25 µg/mL MeOH)
N405006-08	20	4	50	1976/1926	

Direct Injection GC-MS

Diluting samples down
N405006-07, N405006-01, N405006-11 & N405006-04
10 µL of sample into 1mL of MeOH (total volume 1mL)
will then take ~~24~~ AH 5/28/14 25 µL of that solution into total volume of $MeCl_2$

Sample Analysis

Blank
DFTPP @ 50 ug/mL
CCV @ 50 ug/mL
AH 5/28/14 ~~N405~~
N405006-01
-07
-11
-04
original sample
10 µL sample, 50 µL cyclohexanol, 940 µL $MeCl_2$
spike & triplicate on -01 50 ug/mL spike
-01
-07
-11
-04
Diluted samples
25 µL sample, 50 µL cyclohexanol, 925 µL $MeCl_2$
CCV @ 50 ug/mL
Extraction Blank
-02A
-02B
-02C
-10
-12
-08
-09
-01 AH 5/28/14
200 µL of Extractions
-Extractions done by Dan Hulbert 5/28/14

(b) (7)(C) [redacted] 5/28/14