AR226-3787

Pace Analytical Services, Inc.
1700 Elm St. SE, Suite 200
Minneapolis, MN 55414

Phone: 612-607-1700
Fax: 612-607-6444

May 14, 2007

Mr. Kent Lindstrom
3M Environmental Laboratory
Building 260-5N-17
St. Paul, MN 55144

RE: Lab Project Number: 1050968
Client Project ID: E07-0296 3M Guin Water Special Study

Dear Mr. Lindstrom:

Enclosed are the analytical results for sample(s) received by the laboratory on May 3. 2007. Results reported herein conform to the most current NELAC standards, where applicable, unless otherwise narrated in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Diane Anderson

Diane Anderson

diane.anderson@pacelabs.com
Project Manager

enclosures

305036

Pace Analytical™
www.pacelabs.com

Pace Project Manager: Diane Anderson
Report Author: Brian Pike
Analytical Team: Semivolatile Organics Dept

Analytical Report
EPA 8270 GC/MS
Pace Workorder 1050968
May 8, 2007

1 Introduction

Gary Hohenstein of 3M submitted one sample for gas chromatography mass spectrometry (GC/MS) analysis. The sample was analyzed by GC/MS to verify the concentration of Diethylene Glycol Butyl Ether (CAS 112-34-5) using Method 3510C for the extraction and Method 8270 for the analysis. The results for the analysis for sample Distribution System Guin House yielded Diethylene Glycol Butyl Ether at a concentration of 541 ug/L. The sample duplicate yielded results of 508 ug/L.

2 Sample Receipt

The sample was received at Pace Analytical on May 3, 2007. Please see attached documentation for the sample condition upon receipt. The sample was described as follows:

Pace Analytical ID	Client ID
1050968001	Distribution System Guin House

3 Holding Times

The EPA 8270 method has a defined extraction holding time of 7 days from collection of sample with a subsequent 40 day analytical holding time. The sample was extracted and analyzed within this holding time.

4 Methods - Analytical and Preparatory

The sample was analyzed under guidelines set forth in the EPA 8270 method for analysis of semi-volatile analytes. The sample and all associated quality control samples were extracted by EPA 3510 (separatory funnel extraction). The sample was measured for volume and the pH was taken. The sample and the quality assurance samples were fortified with surrogates and matrix spikes and laboratory control spikes were fortified with matrix spike. The sample and quality samples were first acidified to a pH of <2 with 1:1 sulfuric acid and extracted using 3 separate aliquots of 60 mL of methylene chloride for 2 minutes each shake. The sample and quality samples were then brought to a pH >11 with 10N sodium hydroxide and extracted using 3 separate aliquots of 60 mL of methylene chloride for 2 minutes each shake. The extracts were dried by passing through baked and rinsed sodium sulfate prior to concentration. The sample and quality control samples were concentrated on a steam bath utilizing a KD/concentrator/snyder column set-up at 90 degrees C. The extract was concentrated to approximately 4-6 mL on the steam bath and then finally down to 1 mL by way of nitrogen blowdown. The semivolatile analytes were fortified with 8270 internal standard and directly injected onto the GC/MS analytical system for analysis.

The instrumentation used is listed below.

Gas Chromatograph: Hewlett Packard 5890
Column: J&W Scientific DB-5MS (30 m x 0.25 mm x 0.5 µm film thickness)

Detector: Hewlett Packard 5972 Mass Spectrometer
Carrier Gas: Helium

5 *Analysis*

5.1 Calibration

A neat standard of 2-(2-Butoxyethoxy)ethanol (also known as Diethylene Glycol Butyl Ether) was purchased from Chemservice and diluted into Methylene Chloride. The sample replicates were quantitated for Diethylene Glycol Butyl Ether using the average response factor generated from a six-point internal standard calibration curve per the EPA 8270 method. The analytes were calibrated from 10 to 200 ppb and the curve was not forced through zero. The quantitation was determined from the lowest standard level from the calibration curve. The limit of detection has not been statistically determined using a method detection limit study. The initial calibration was not verified by an external source.

5.2 Blanks

A method blank was analyzed along with the sample to determine system cleanliness. The method blank was prepared by adding 1000 mL of deionized water into a separatory funnel. The method blank was extracted the same as the samples (pH adjusted, etc.). Diethylene Glycol Butyl Ether was not detected in the method blank.

5.3 Surrogates

Surrogates were added to the sample, sample replicates, and all quality control samples before the extraction process. This was done by adding 100µL of 750-1000ug/mL surrogate standard. The recoveries for the surrogate standards may have been influenced by the concentration of target analytes and other non-target analytes present in the samples. The surrogate recoveries are summarized in a table later in this report.

5.4 Matrix Spikes

A matrix spike (MS) at a higher concentration (in upper range of the calibration) and matrix spike at a lower concentration (in lower range of the calibration) were prepared by spiking 300 µl and 50 µl of a 510 µg/mL solution respectively of Diethylene Glycol Butyl Ether. The laboratory matrix spikes were attempted but were not used for the accuracy assessment because the endogenous levels in the sample greatly exceeded the fortified spike amounts.

5.5 Laboratory Control Sample

The LCS and LCSD's were extracted the same as the samples. A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) at a higher concentration were prepared by spiking with 300 µL of 510 µg/mL of Diethylene Glycol Butyl Ether solution. The average recovery of Diethylene Glycol Butyl Ether was 77.4%. A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) at a lower concentration were prepared by spiking with 50 µL of 510 µg/mL of Diethylene Glycol Butyl Ether solution. The average recovery of Diethylene Glycol Butyl Ether was 57.2%.

5.6 Continuing Calibration Verification

Continuing calibration verification standards (CCV's) were analyzed during the analytical sequence to verify consistent system operation. The opening CCV for the dilutions performed had a recovery for Diethylene Glycol Butyl Ether of 101.6%.

6 Data Summary

Sample ID	On-column value (ug/mL)	Amount Extracted (mL)	Final Concentration	Comments
Distribution System Guin House	496.784	930	534 ug/L	Result is over the calibration range(Calibrated to 200 ug/L)
Distribution System Guin House 5X dilution	100.699	930	541 ug/L	
Distribution System Guin House Duplicate	477.071	930	513 ug/L	Result is over the calibration range(Calibrated to 200 ug/L)
Distribution System Guin House Duplicate 5X dilution	94.4645	930	508 ug/L	

Relative Percent Difference Sample to Sample Duplicate (diluted values) = 6.3 percent

Sample ID	On-column value (ug/mL)	Amount Extracted (mL)	Concentration of Matrix Spike added (ug/mL)	Percent recovery	Comments
Method Blank	No Peak Present	1000	Not Applicable	Not Applicable	
LCS Low Conc	14.5758	1000	25.5	57.16	
LCSD Low Conc	14.5966	1000	25.5	57.24	
LCS High Conc	117.231	1000	153	76.62	
LCSD High Conc	119.665	1000	153	78.21	
Distribution System Guin House MS Low Conc	500.152	930	25.5	1961.38	orig sample amount not subtracted out
Distribution System Guin House MS Low Conc 5X dilution	97.6686	930	25.5	1915.07	With sample subtracted is a negative value, detection too large in sample to achieve accurate recovery.
Distribution System Guin House MS High Conc	554.643	960	153	362.51	orig sample amount not subtracted out
Distribution System Guin House MS High Conc 5X dilution	111.676	960	153	364.95	Recovery with original sample subtracted (from 5X value) yields a 35.9% recovery. Recovery is possibly bias due to dilution and detection in the original sample.

Surrogate Recoveries (Percent Recovery):

	Method Blank	LCS Low Conc	LCSD Low Conc	LCS High Conc	LCSD High Conc	Distribution System Guin House MS Low Conc	Distribution System Guin House MS Low Conc 5X dilution	Distribution System Guin House MS High Conc	Distribution System Guin House MS High Conc 5X dilution	Example Recovery Limits[1]
2-Fluorphenol	41.4	35.5	34.3	35.1	38.1	28.0	23.2	29.3	24.5	19-100
Phenol-d6	24.2	21.0	19.9	21.0	22.9	0	0	0	0	11-100
Nitrobenzene-d5	77.1	74.3	74.3	77.4	78.7	76.5	76.8	79.6	76.9	51-100
2-Fluorobiphenyl	64.4	59.3	59.4	64.4	63.6	75.3	91.7	60.5	83.2	49-100
2,4,6-Tribromophenol	75.8	74.8	77.7	70.1	75.8	69.2	73.1	69.2	73.1	23-109
Terphenyl-d14	77.7	74.6	78.0	76.2	83.6	82.5	85.3	84.2	84.2	35-142
Diethylene Glycol Butyl Ether	ND	57.2	57.2	76.8	78.2	1961.4	1915.1	362.5	385	

*Note: Accuracy limits for Diethylene Glycol Butyl Ether could not be determined due to lack of data points.

[1] Recovery limits for seperatory funnel extractions derived from a combination of Pace Analytical Green Bay and Indianapolis recovery limits for EPA 8270

	Distribution System Guin House	Distribution System Guin House 5X	Distribution System Guin House Duplicate	Distribution System Guin House Dup 5X	Example Recovery Limits
2-Fluorphenol	31.7	28.5	29.6	27.1	19-100
Phenol-d6	0	0	0.5	0	11-100
Nitrobenzene-d5	82.0	81.7	75.0	74.9	51-100
2-Fluorobiphenyl	74.5	93.3	62.8	84.8	49-100
2,4,6-Tribromophenol	71.7	77.9	71.9	73.8	23-109
Terphenyl-d14	83.0	88.4	81.3	82.4	35-142

7 *Observation/Discussion*

MDL studies and initial demonstrations of capability were not performed for Diethylene Glycol Butyl Ether. The process outlined in this report is based on Pace Analytical's standard operating procedure for EPA 8270.

8 *Data / Sample Retention*

Hardcopies and electronic copies of the data are kept on file at the Pace Analytical.

8

Surrogate Recoveries

	Method Blank	LCS Low Conc	LCSD Low Conc	LCS High Conc	LCSD High Conc	Distribution System Guin House MS Low Conc	Distribution System Guin House MS Low Conc 5X dilution	Distribution System Guin House MS High Conc	Distribution System Guin House MS High Conc 5X dilution	Example Recovery Limits
2-Fluorphenol	41.4	35.5	34.3	35.1	38.1	28	23.2	29.3	24.5	19-100
Phenol-d6	24.2	21	19.9	21	22.9	0	0	0	0	11-100
Nitrobenzene-d5	77.1	74.3	74.3	77.4	78.7	76.5	76.8	79.6	76.9	51-100
2-Fluorobiphenyl	64.4	59.3	59.4	64.4	63.6	75.3	91.7	60.5	83.2	49-100
2,4,6-Tribromophenol	75.8	74.8	77.7	70.1	75.8	69.2	73.1	69.2	73.1	23-109
Terphenyl-d14	77.7	74.6	78	76.2	83.6	82.5	85.3	84.2	84.2	35-142
Diethylene Glycol Butyl E	ND	57.2	57.2	76.6	78.2	1961.4	1915.1	362.5	365	

	Distribution System Guin House	Distribution System Guin House 5X	Distribution System Guin House Duplicate	Distribution System Guin House Dup 5X	Example Recovery Limits
2-Fluorphenol	31.7	28.5	29.6	27.1	19-100
Phenol-d6	0	0	0.5	0	11-100
Nitrobenzene-d5	82	81.7	75	74.9	51-100
2-Fluorobiphenyl	74.5	93.3	62.8	84.8	49-100
2,4,6-Tribromophenol	71.7	77.9	71.9	73.6	23-109
Terphenyl-d14	83	88.4	81.3	82.4	35-142

Results for 3M sample for Diethylene Glycol Butyl Ether CAS#112-34-5

Sample ID	On-column value (ug/mL)	Amount Extracted (mL)	Final Concentration	Comments
Distribution System Guin House	496.784	930	534 ug/L	Result is over the calibration range(Calibrated to 200 ug/L)
Distribution System Guin House 5X dilution	100.699	930	541 ug/L	
Distribution System Guin House Duplicate	477.071	930	513 ug/L	Result is over the calibration range(Calibrated to 200 ug/L)
Distribution System Guin House Duplicate 5X dilution	94.4645	930	508 ug/L	

Relative Percent Difference Sample to Sample Duplicate (diluted values) = 6.3 percent

Quality Control Samples

Sample ID	On-column value	Amount Extracted (mL)	Concentration of Matrix Spike added	Percent recovery	Comments
Method Blank	No Peak Present	1000	None	Non-detect	
LCS Low Conc	14.5758	1000	25.5	57.16	
LCSD Low Conc	14.5966	1000	25.5	57.24	
LCS High Conc	117.231	1000	153	76.62	
LCSD High Conc	119.665	1000	153	78.21	
Distribution System Guin House MS Low Conc	500.152	930	25.5	1961.38	orig sample amount not subtracted out (pending dilutions for original sample)
Distribution System Guin House MS Low Conc 5X dilution	97.6686	930	25.5	1915.07	With sample subtracted is a negative value, detection too large in sample to achieve accurate recovery.
Distribution System Guin House MS High Conc	554.643	960	153	362.51	orig sample amount not subtracted out (pending dilutions for original sample)
Distribution System Guin House MS High Conc 5X dilution	111.676	960	153	364.95	Recovery with original sample subtracted (from 5X value) yields a 35.9% recovery. Recovery is possibly bias due to dilution and detection in the original sample.

***Calibration range for Diethylene Glycol Butyl Ether: 10 ppb (ug/mL) to 200 ppb (ug/mL)*

3M Environmental Laboratory

Chain of Custody / Request for Laboratory Analytical Services 1050968

Shipping Address:
3M - Bldg. 2-3E-05
935 Bush Avenue
St. Paul, MN 55106

Telephone:
Sample Receiving: (651) 778-6753
Alternate: (651) 778-4948
FAX: (651) 778-6176

Project ID/Project Name 3M Guin Volatiles Analysis
Template #
Project Lead Kent Lindstrom
Dept. # (main) 832202
Final Report Due Date
Internal Due Date
Class/Job/Project #

E07-0296

Contact Name Richard Wates
Company 3M
Mailing Address
City, State, Zip Guin, AL 35563
Telephone # 205-468-3315 FAX #

Date Available
Date Due
Contract Lab *Pace Analytical*

Special Instructions and/or Specific Regulatory Requirements:
(method, limit of detection, rep

Preservatives: HNO_3 | H_2SO_4 | VOCs | None | Other:

Total Number of Containers

Analysis Requested:
Complete below. Attach any associated information.

See Attached Lab Request

(Enter an 'X' in the box below to indicate request)

Item #	Client Sample Identification	3M LIMS #	Date Sampled	Time Sampled	Matrix/ media	Enter the number of containers of each	Total Number of Containers
1.	Distribution System Guin House		5/2/07	2:51 PM			20
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							

Collected by (print):
Collector's signature:

Sample Condition Upon Receipt: O Acceptable O Other:
Temperature: °C O Received on Ice
Other Associated CoCs:
Copies to:

Comments: This chain of custody prepared at 3M Environmental Lab. AC ibe 5/4/07.
Revised cofc rec'd via email 5/4/07.
See original for signatures.
Revision includes Client Sample Identification
Dian Anderson
Pace 5/4/07

Page ___ Of ___ Original - Accompanying Samples Last Page - Originator See Reverse Side for Instructions

1050968

3M Environmental Laboratory

Form 34773 - A - PWO

Shipping Address:
3M Center
Bldg. 260-5N-17
St. Paul, MN 55144

Telephone:
Sample Receiving: (651) 736-8598
Alternate: (651) 733-8873
FAX: (651) 733-8982

Chain of Custody /Request for Laboratory Analytical

13427

Project ID/Project Name	3M Guin Water / Volatiles Analysis
Template #	
Project Lead	Kent Lindstrom
Dept. # (main)	530711
Final Report Due Date	
Internal Due Date	
Class/Job/Project #	

3M Env. Lab Project # For Internal Use Only: E07-0296

Report Results to:

Contact Name	Richard Waters
Company	3M Guin
Mailing Address	6675 US Hwy 43
City, State, Zip	Guin, AL 35563
Telephone #	205-468-3315
FAX #	
Date Available	
Date Due	
Contract Lab	

Special Instructions and/or Specific Regulatory Requirements: (method, limit of detection, reporting units, etc.)

Send to Pace. Contact Gary H. for info. at 3M.

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/ Media	Preservatives: HNO_3	H_2SO_4	VOCs	None	Other	Total Number of Containers	Analysis Requested: (Volatiles)
1.	Distr. Sys. 3M Guin		5/2/07	2:51 pm	L						2	X
2.	(See corrected											
3.	Sample ID											
4.	on revised CofC.)											
5.												
6.												
7.												
8.												
9.												
10.												

Enter the number of containers of each. Complete below. Attach any associated information. (Enter an X in the box below to indicate request)

Chain of Custody

Collected by (print):

Collector's signature:

Item #	Relinquished By/Affiliation	Time	Date	Shipped Via	Received by/Affiliation	Time	Date
1	Richard Waters	5:00 pm	5/2/07	Fed Ex	J Richardson	12:02	5/3/07

Sample Condition Upon Receipt: ☒ Acceptable ○ Other:

Temperature: 4.5 °C ☒ Received on Ice

Other Associated CoCs:

Copies to:

Comments: Revised CofC rec'd via e-mail 5/4/07. (Client Sample Identification was updated.)
This form contains original signatures. Diane J. Anderson 5/4/07

Page ___ Of ___ Original - Accompanying Samples Last Page - Originator See Reverse Side for Instructions

T = 6.0

12

Sample Condition Upon Receipt

Pace Analytical

Client Name: 3M Guin Project # 1050968

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☒ Client ☐ Commercial ☐ Pace Other _____

Optional: Proj. Due Date: _____ Proj. Name: _____

Tracking #: _____

Custody Seal on Cooler/Box Present: ☐ yes ☒ no Seals intact: ☐ yes ☒ no

Packing Material: ☒ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other _____

Thermometer Used 230194010 Type of Ice: (Wet) Blue None ☒ Samples on ice, cooling process has begun

Cooler Temperature 4.5°/6.0° Biological Tissue is Frozen: Yes No

Date and Initials of person examining contents: CL 5-3-07

Temp should be above freezing to 6°C Comments:

Chain of Custody Present:	☐Yes ☒No ☐N/A	1.
Chain of Custody Filled Out:	☐Yes ☐No ☒N/A	2.
Chain of Custody Relinquished:	☐Yes ☐No ☒N/A	3.
Sampler Name & Signature on COC:	☐Yes ☐No ☒N/A	4.
Samples Arrived within Hold Time:	☒Yes ☐No ☐N/A	5.
Short Hold Time Analysis (<72hr):	☐Yes ☒No ☐N/A	6.
Rush Turn Around Time Requested:	☐Yes ☐No ☐N/A	7.
Sufficient Volume:	☒Yes ☐No ☐N/A	8.
Correct Containers Used:	☒Yes ☐No ☐N/A	9.
-Pace Containers Used:	☒Yes ☐No ☐N/A	
Containers Intact:	☐Yes ☒No ☐N/A	10. Cap cracked on 1 liter
Filtered volume received for Dissolved tests	☐Yes ☐No ☒N/A	11.
Sample Labels match COC:	☐Yes ☒No ☐N/A	12. no labels
-Includes date/time/ID/Analysis Matrix: WT		
All containers needing preservation have been checked.	☐Yes ☐No ☒N/A	13.
All containers needing preservation are found to be in compliance with EPA recommendation.	☐Yes ☐No ☒N/A	
exceptions: VOA, coliform, TOC, O&G, WI-DRO (water)	☒Yes ☐No	Initial when completed / Lot # of added preservative
Samples checked for dechlorination:	☐Yes ☐No ☐N/A	14.
Headspace in VOA Vials (>6mm):	☐Yes ☐No ☐N/A	15.
Trip Blank Present:	☐Yes ☐No ☐N/A	16.
Trip Blank Custody Seals Present	☐Yes ☐No ☐N/A	
Pace Trip Blank Lot # (if purchased):		

Client Notification/ Resolution: Field Data Required? Y / N

Person Contacted: _____ Date/Time: _____

Comments/ Resolution: Rec'd COC via e-mail on 5/4/07 DAI

Project Manager Review: Diane Anderson **Date:** 5/3/07

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

F-ALLC003rev.3, 11September2006