Quality Issue Final Report

2017-QI-0010

Section: ***Toxicology***

Associated: 206 *Initiated:* 9/7/2017 *TargetDate:* 12/7/2017

Associated II: 224 *External:* ☐ *Completed:* 10/25/2017

Opener: , *Class:* Class II *Confirm on:*

DR Number(s):

Description: Acetone was found in the nanonpure water used by Toxicology.

Criteria:

Notes:

Date	*Comments*
9/7/2017	Verbally notified by Tox TL John Musselman that the laboratory water supply for nanopure water contained unexpected levels of acetone. Acetone is a target analyte during routine Blood Alcohol analysis and can be reported as being present. Per TL Musselman the presence or lack of Acetone has no effect on blood alcohol results. Therefore no concern as to the accuracy of the reported BA results is present. TL Musselman was able to determine through research and interview that in the days prior a repair to piping in the system occurred. It lis likely that a PVC glue that contained acetone was used. ROOT CAUSE: Repair introduced level of acetone into system. CORRECTIVE ACTION PLAN: Cease reporting of Acetone pending resolution. Flush and refill water system. Test to confirm efficacy of plan.
9/19/2017	CAR review meeting held. Discussed root cause and corrective action plan. Root Cause: Maintanance introduced acetone into system. While QA data was reviewed for trends the QA practices were not sufficient to provide early notice. Corrective Action Plan: Review prior casework to determine if any reported results were inappropriately affected by presence of acetone, address on case by case basis. Document that water is clear of acetone based on a TBD detection limit. Develop a practice to prevent and detect a buildup of acetone in the water supply Develop a limit/threshold for reporting acetone
10/18/2017	Update meeting held. Preventative actions completed. Discussed providing amended reports and revision to relevant SOP. Notification completed. Asst Admin Jody Wolf notified City Prosecutor and County Attorney representatives on 9/19/2017. Remaining remediation includes: providing amended reports for applicable cases and updates to relevant SOP.
10/23/2017	Email update received from Tox Lead Swanson. Remaining components of correction action plan complete SOP revision complete (TOX-SOP-17 released 10-19-17) Amended reports complete Recommend closure.

18 total

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N/A ~~Analyst (if applicable)~~	Date
Supervisor or Technical Lead (if applicable)	Date 10/26/17
Assistant Administrator	Date 10/30/17
Assistant Quality Manager	Date 10-26-17
Quality Manager	Date 10-26-17
Lab Commander	Date 10/31/17

John E Knell

From: John J Musselman
Sent: Thursday, September 07, 2017 10:27 AM
To: John E Knell; Nancy Crump; Jody M Wolf
Subject: Acetone in the purified water system

Hi John and Nancy,
We have discovered a level of acetone in our nanopure water which may interfere with reporting acetone present during routine Blood alcohol analysis. I am requesting we ask Julian to drain the tank and regenerate fresh DI water, circulate, drain and regenerate again to flush out the system. The tank in the basement has an identifiable amount of acetone in it. I can test the water again once this has been completed. This has no effect on Blood alcohol measurement, but I would like this done ASAP.
thx,

John Musselman A4322
Forensic Scientist
Phoenix Police Department
602 534-8861 (Desk)
602 262-6197 (Lab)

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John E Knell

From: John J Musselman
Sent: Thursday, September 07, 2017 11:42 AM
To: Gayle M Swanson; Amanda B Gallegos; James S Hoban III; Donald J Stenberg; Richard M Bond; Natasha Imadiyi; Katherine E Kazanas
Cc: Jody M Wolf; John E Knell

All,
We are going to suspend the reporting of acetone identified in Blood alcohol analysis until further notice.
thank you,

John Musselman A4322
Forensic Scientist
Phoenix Police Department
602 534-8861 (Desk)
602 262-6197 (Lab)

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John E Knell

From: Gayle M Swanson
Sent: Monday, October 23, 2017 2:32 PM
To: John E Knell; Jody M Wolf
Cc: John J Musselman; Amanda B Gallegos; Natasha Imadiyi; James S Hoban III; Katherine E Kazanas; Richard M Bond; Donald J Stenberg
Subject: 2017-QI-0010 Toxicology CAR

Summation of 2017-QI-0010 in Toxicology:

There was a noticeable bump at the time of acetone in a HSGC internal Standard made on 8/30/17. Trace levels of Acetone were present in the 800 gallon RO (reverse osmosis) water tank located in the basement of the Phoenix Crime Laboratory building when tested on 9/7/17. This reserve feeds into the 18 MΩ-cm water dispensed by the Barnstead Nanopure water system in the Toxicology section, which is used as the source of the water to make an Internal Standard (IS) solution for Alcohol Analysis. Acetone was also present when tested on 9/5/17, albeit at even lower amounts, in this water. Acetone reporting was suspended on September 6th, 2017 for Alcohol Analysis reports.

Root Cause: In speaking with our Building Equipment Operator, there was a leak repaired in DNA on 7/21/17. There was also a replacement of the recirculation pump for the RO system on 4/18/17. Both repairs would have been performed with PVC cement, which contains and potentially liberated acetone into the water supply. This trace amount of acetone may have affected the reporting of acetone in case samples analyzed using IS solutions made after the initial repair on 4/18/17. There was no ethanol detected in the RO or Nanopure water at any time.

Corrective Action: A work order was placed to drain the 800 gallon tank, refill, rinse/recirculate, drain and refill. This was completed on 9/11/17. Tests of the 800 gallon tank water on 09/12/17 showed a >90% reduction in acetone and were deemed acceptable. In addition, the filter in the Nanopure water dispenser unit in toxicology was replaced on 9/15/17. Tests from water samples on 9/15/17 were acceptable, with no detectable levels of acetone present.

Preventative measures recommended: 1) Annually test, and if applicable drain and refill, the 800 gallon tank. 2) Test the water for volatiles prior to preparing Internal Standard solutions.

Evaluation of case reports affected: Acetone is completely resolved from ethanol on both analytical columns; therefore, this trace amount of acetone in no way impacted the measured and reported ethanol concentrations. Normal integration parameters result in acetone measurements at 0.0009 g/100mL and above. Due to the fact that this trace amount of acetone was not integrated on Internal Standard checks or negative controls, the integration threshold had to be lowered in order to evaluate the contribution. The greatest contribution of acetone in the checks of IS solutions made after 4/18/17 was determined to be 0.0004 g/100mL. Although this potentially caused more acetone positives to be reported per TOX-SOP-17 Rev.10, the trace level of acetone was not the sole contribution of a positive finding of acetone in a blood (urine) sample. Meaning acetone was present at some level in the samples, but it may not have been reported without the presence of acetone in the water supply. An evaluation of all the cases where acetone was reported using IS solutions made after 4/18/17 (IS lot #s: 042517JM, 053117AJM, 053117BJM, 070617JM) was conducted and 53 amended reports have been authored for those cases where the lowest acetone concentration was detected <0.0015 g/100mL.

Endogenous acetone levels in healthy adults have been measured up to 0.001 g/100mL [1]. This incident prompted an evaluation of the relevance of reporting acetone at low levels that are consistent with endogenous levels of acetone. Additional testing was performed to determine the limits of detection (LOD) of acetone, methanol, and isopropanol. Although the LOD for the instrumentation has been shown to be extremely low for acetone (0.001 g/100mL) and other volatiles, the Phoenix Crime Laboratory has elected to set higher administrative cutoffs for the reporting of volatiles other than ethanol consistent with the beginning of industrial exposure as well as the LOD for ethanol. Administrative cutoffs for volatiles other than ethanol (acetone, methanol, and isopropanol) have been amended in TOX-SOP-17 Rev.11 to ≥0.005 g/100mL.

Implementation of Preventative Measures: 1) Testing and possible draining of the tank added to the annual checklist TOX-WS-31D Toxicology Lab Maintenance Annual Checklist Policy #7538. 2) TOX-SOP-17 Protocol for the Analysis of Ethanol, updated in Revision 11 to include the testing of the water from the Nanopure system in the Toxicology section before making an Internal Standard solution, including the acceptance criteria for this check.

1. *Disposition of Toxic Drugs and Chemicals in Man,* 10th Edition. 2014. Randall C. Baselt. Biomedical Publications.

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Gayle M. Swanson
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Amended Reports

IR	analyst	analysis date	IS lot#	Instrument	Reported BAC	Lowest ACT	Acetone 1	Acetone 2
201700001110984	abg	6/28/2017	053117AJM	Jack	0.272	0.0011	0.0011	0.0012
201700001113513	abg	6/28/2017	053117AJM	Jack	0.000	0.0011	0.0011	0.0012
201700001396493	abg	8/14/2017	070617JM	Jack	0.000	0.0012	0.0012	0.0012
201700001406100	abg	8/15/2017	070617JM	Jack	0.304	0.0012	0.0012	0.0012
201700001149490	abg	7/6/2017	053117AJM	Jill	0.000	0.0013	0.0013	0.0013
201700000836445	djs	5/18/2017	042517JM	Jack	0.000	0.0012	0.0012	0.0012
201700001361378	djs	8/9/2017	053117BJM	Jack	0.165	0.0012	0.0012	0.0012
201700000127509	djs	5/16/2017	042517JM	Jack	0.106	0.0013	0.0013	0.0013
201700001038226	djs	6/20/2017	042517JM	Jack	0.000	0.0014	0.0014	0.0014
201700001285538	djs	8/9/2017	053117BJM	Jack	0.216	0.0014	0.0014	0.0014
201700001371709	djs	8/9/2017	053117BJM	Jack	0.289	0.0014	0.0014	0.0014
201700001463095	gms	8/28/2017	070617JM	Jack	0.248	0.0011	0.0011	0.0011
201700001274089	gms	7/25/2017	070617JM	Jack	0.184	0.0012	0.0012	0.0012
201700001278543	gms	7/25/2017	070617JM	Jack	0.277	0.0012	0.0012	0.0012
201700001467954	gms	8/28/2017	070617JM	Jack	0.000	0.0012	0.0012	0.0012
201700001486360	gms	8/28/2017	070617JM	Jack	0.197	0.0012	0.0012	0.0012
201700001278836	gms	7/25/2017	070617JM	Jack	0.159	0.0013	0.0013	0.0013
201700000976965	jh	6/9/2017	053117BJM	Jill	0.058	0.0009	0.0010	0.0009
201700001184102	jh	7/12/2017	053117AJM	Jill	0.217	0.0010	0.0010	0.0010
201700000930122	jh	6/9/2017	053117BJM	Jill	0.000	0.0010	0.0010	0.0010
201700000865647	jh	6/1/2017	042517JM	Jill	0.000	0.0011	0.0011	0.0011
201700001448382	jh	8/23/2017	053117AJM	Jack	0.000	0.0012	0.0012	0.0012
201700001190549	jh	7/12/2017	053117AJM	Jill	0.000	0.0013	0.0013	0.0013
201700001519814	jh	9/1/2017	053117AJM	Jack	0.143	0.0013	0.0013	0.0013
201700001456794	jh	8/23/2017	053117AJM	Jack	0.107	0.0014	0.0014	0.0014
201700001506563	jh	9/1/2017	053117AJM	Jack	0.232	0.0014	0.0014	0.0014
201700000979814	jh	6/9/2017	053117BJM	Jill	0.112	0.0014	0.0014	0.0014
201700000814729	jm	5/12/2017	042517JM	Jill	0.227	0.0009	0.0009	0.0009
201700000709641	jm	5/10/2017	042517JM	Jill	0.000	0.0010	0.0010	0.0010
201700000618267	jm	5/12/2017	042517JM	Jill	0.000	0.0011	0.0011	0.0011
201700000783753	jm	5/10/2017	042517JM	Jill	0.229	0.0011	0.0011	0.0011
201700000807369	jm	5/12/2017	042517JM	Jill	0.297	0.0011	0.0011	0.0011
201700001100859	jm	6/28/2017	053117BJM	Jill	0.140	0.0014	0.0014	0.0014
201700000954654	kk	6/6/2017	053117BJM	Jack	0.127	0.0014	0.0014	0.0014
201700000824655	ni	5/17/2017	042517JM	Jill	0.000	0.0009	0.0009	0.0009
201700000935390	ni	6/2/2017	042517JM	Jack	0.169	0.0012	0.0012	0.0013
201700000941608	ni	6/2/2017	042517JM	Jack	0.315	0.0014	0.0014	0.0014
201700000737114	rb	5/3/2017	042517JM	Jill	0.000	0.0009	0.0009	0.0009
201700001015599	rb	6/13/2017	053117AJM	Jill	0.184	0.0009	0.0009	0.0010
201700001072289	rb	6/22/2017	053117AJM	Jill	0.261	0.0009	0.0009	0.0009
201700001265710	rb	7/24/2017	070617JM	Jill	0.052	0.0009	0.0009	0.0009
201700001280354	rb	7/28/2017	070617JM	Jill	0.074	0.0009	0.0009	0.0009
201700001285683	rb	7/28/2017	070617JM	Jill	0.248	0.0009	0.0009	0.0009
201700001297539	rb	7/28/2017	070617JM	Jill	0.344	0.0009	0.0010	0.0009
201700000708911	rb	5/3/2017	042517JM	Jill	0.231	0.0010	0.0010	0.0010
201700001061721	rb	6/22/2017	053117AJM	Jill	0.329	0.0010	0.0010	0.0010
201700001142964	rb	7/24/2017	070617JM	Jill	0.000	0.0010	0.0010	0.0010
201700001266957	rb	7/24/2017	070617JM	Jill	0.086	0.0010	0.0010	0.0010
201700001292387	rb	7/28/2017	070617JM	Jill	0.000	0.0010	0.0010	0.0010
201700001292387	rb	7/28/2017	070617JM	Jill	0.000	0.0011	0.0011	0.0011
201700001335501	rb	8/4/2017	070617JM	Jill	<0.025	0.0012	0.0012	0.0012
201700001292387	rb	7/28/2017	070617JM	Jill	0.000	0.0014	0.0014	0.0014
201700001296858	rb	7/28/2017	070617JM	Jill	0.000	0.0014	0.0014	0.0014

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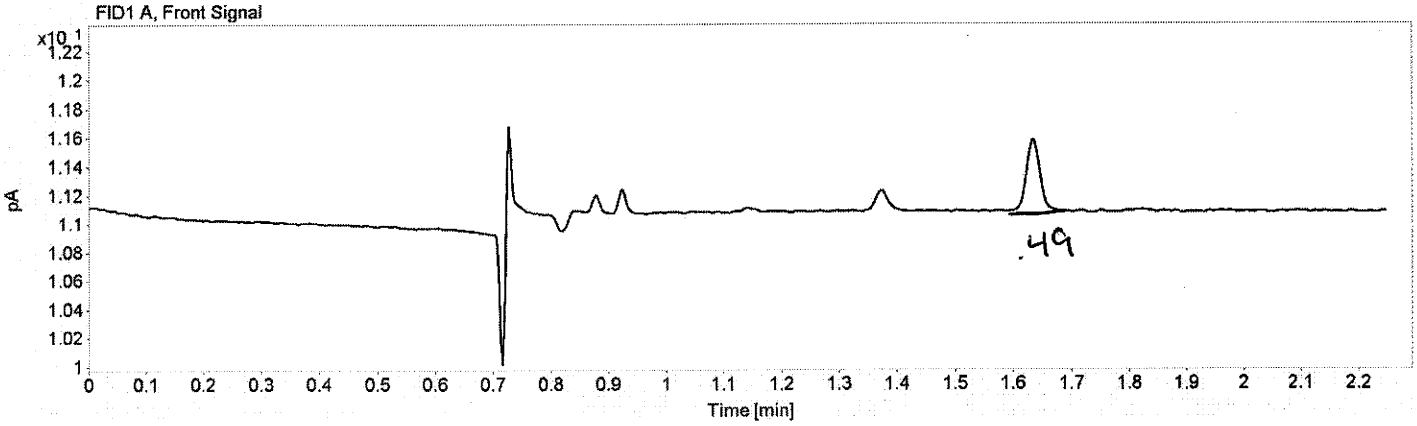
Phoenix Crime Lab: Alcohol Report

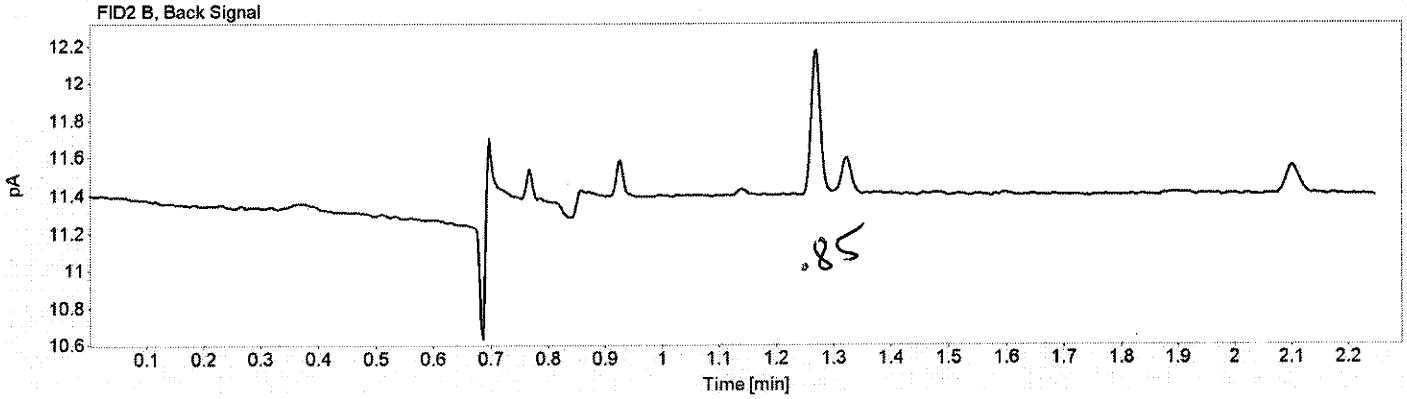
Sample name: H2O - from 18 MΩ dispenser
Location: 3
Sequence: JM090517 2017-09-05 09-57-37
Acq. method: ALCOHOL.M
Data file: D:\Data\KK\JM090517 2017-09-05 09-57-37\17.D

Item Number:
Injection date: 9/5/2017 10:21:57 AM
Instrument: Jack
Analyst: KK



A. Quantitation

Compound	Time (min)	Peak Area	Amount (g/100ml)



B. Confirmation

Compound	Time (min)	Peak Area

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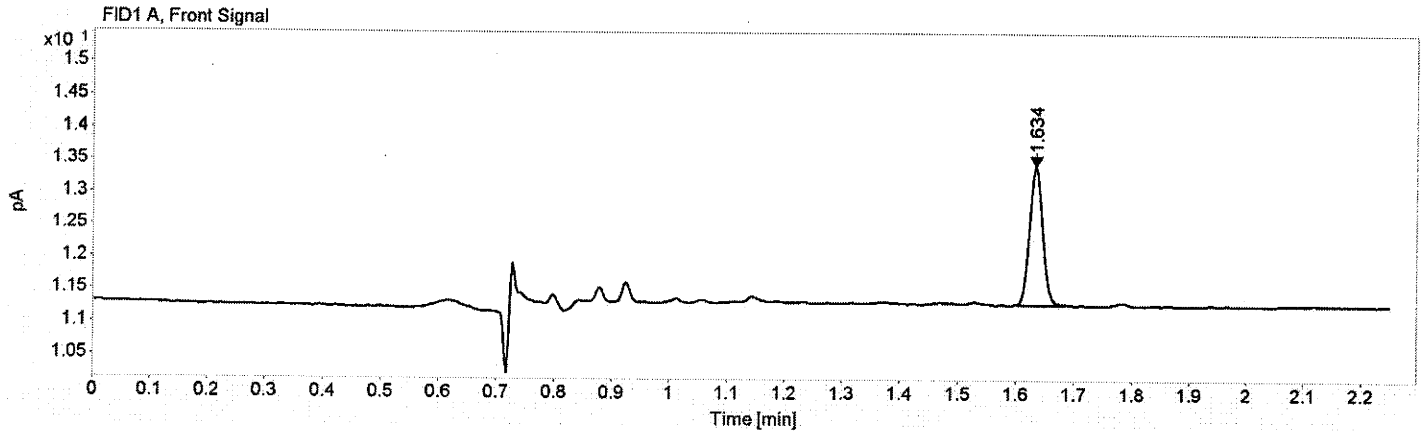
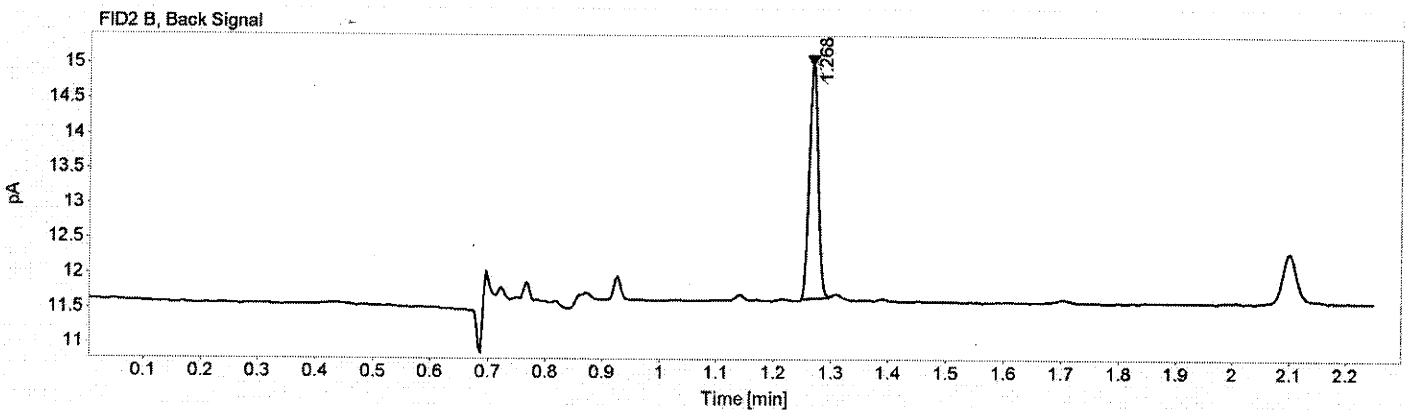
Phoenix Crime Lab: Alcohol Report

Sample name:	Tank water	**Item Number:**	
Location:	20	**Injection date:**	9/7/2017 9:16:09 AM
Sequence:	JM090517 2017-09-07 08-55-20	**Instrument:**	Jack
Acq. method:	ALCOHOL.M	**Analyst:**	KK
Data file:	D:\Data\KK\JM090517 2017-09-07 08-55-20\020F2001.D		



A. Quantitation

Compound	Time (min)	Peak Area	Amount (g/100ml)
Acetone #1	1.634	3.3255	0.0000



B. Confirmation

Compound	Time (min)	Peak Area
Acetone #2	1.268	3.5646

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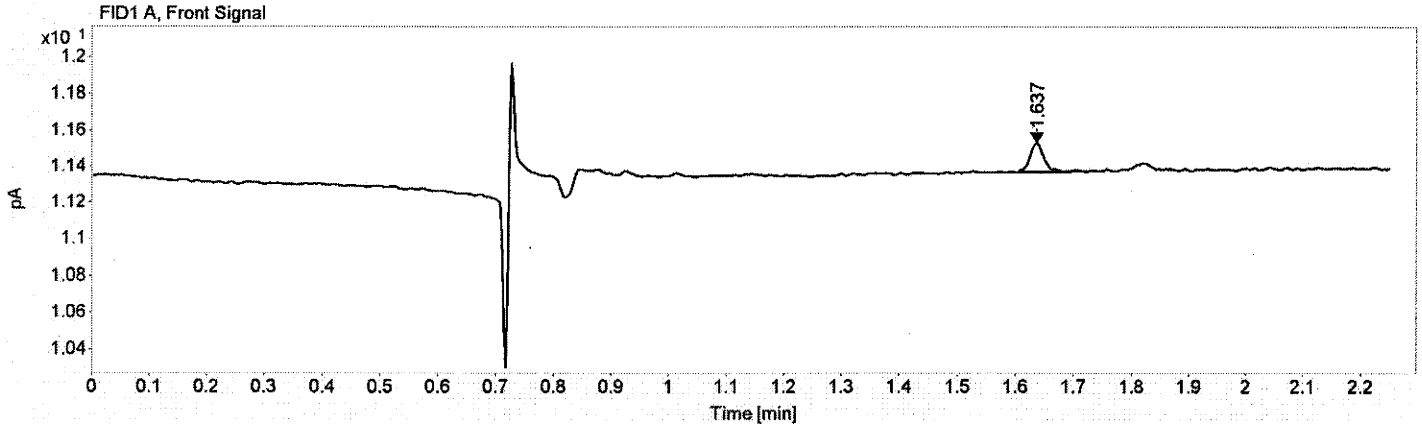
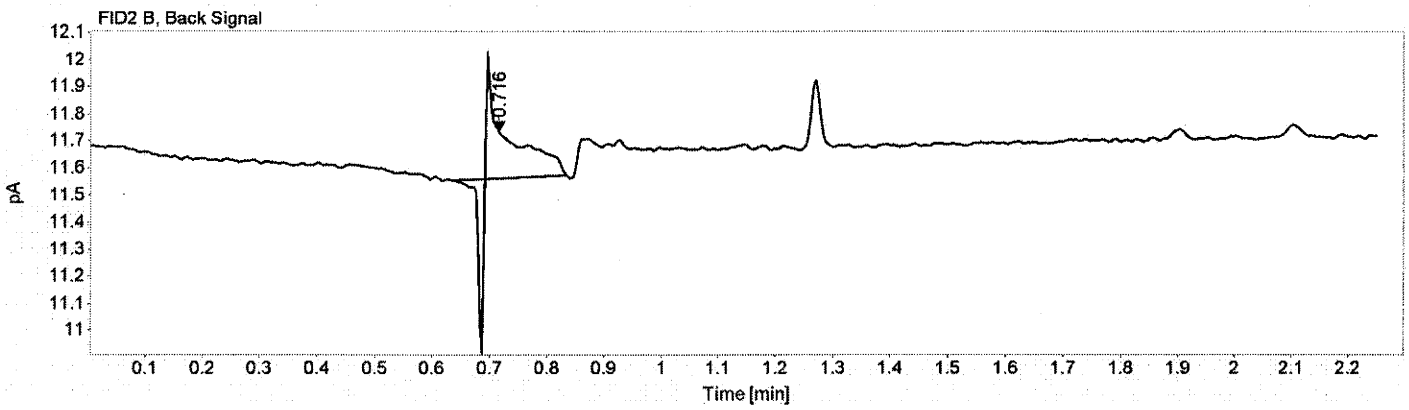
Phoenix Crime Lab: Alcohol Report

Sample name:	Tank water 091217	**Item Number:**	
Location:	25	**Injection date:**	9/12/2017 9:35:14 AM
Sequence:	JM090517 2017-09-12 09-17-54	**Instrument:**	Jack
Acq. method:	ALCOHOL.M	**Analyst:**	KK
Data file:	D:\Data\KK\JM090517 2017-09-12 09-17-54\025F2501.D		



A. Quantitation

Compound	Time (min)	Peak Area	Amount (g/100ml)
Acetone #1	1.637	0.2554	0.0000



B. Confirmation

Compound	Time (min)	Peak Area

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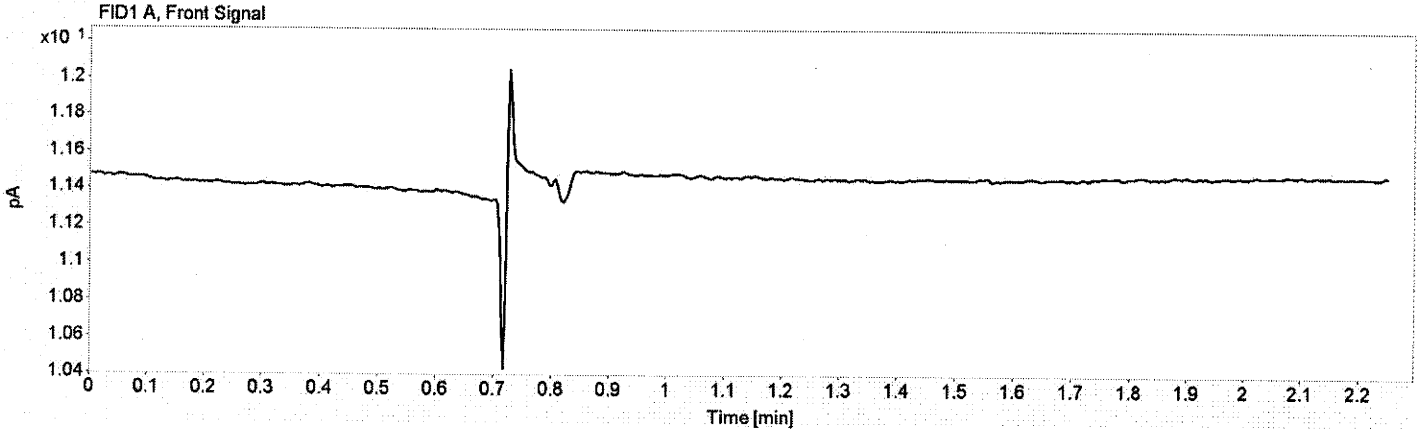
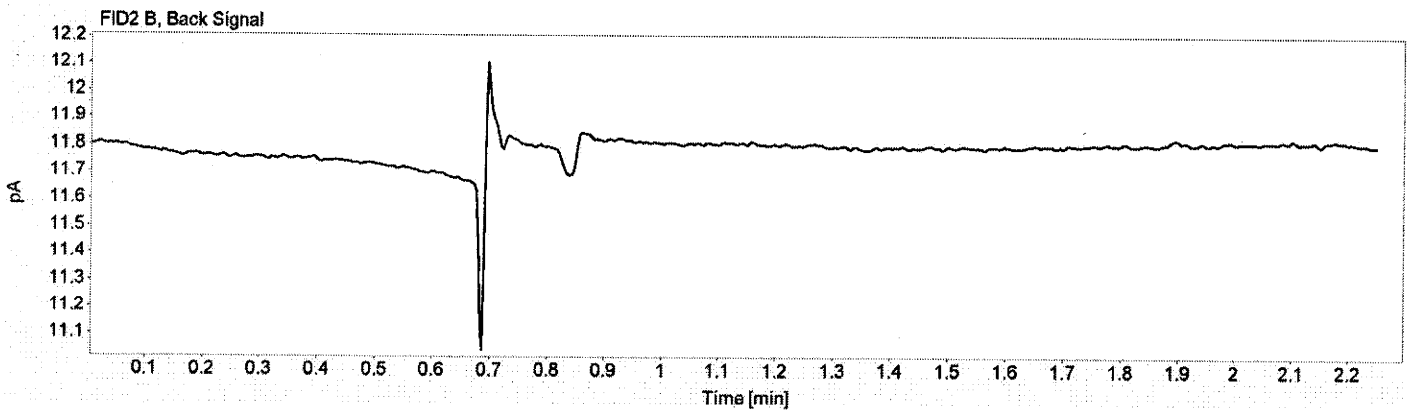
Phoenix Crime Lab: Alcohol Report

Sample name:	18 M ohm water new filter	**Item Number:**	
Location:	3	**Injection date:**	9/15/2017 1:39:50 PM
Sequence:	JM091517 2017-09-15 13-15-33	**Instrument:**	Jack
Acq. method:	ALCOHOL.M	**Analyst:**	JM
Data file:	D:\Data\JM\JM091517 2017-09-15 13-15-33\003F0301.D		



A. Quantitation

Compound	Time (min)	Peak Area	Amount (g/100ml)



B. Confirmation

Compound	Time (min)	Peak Area

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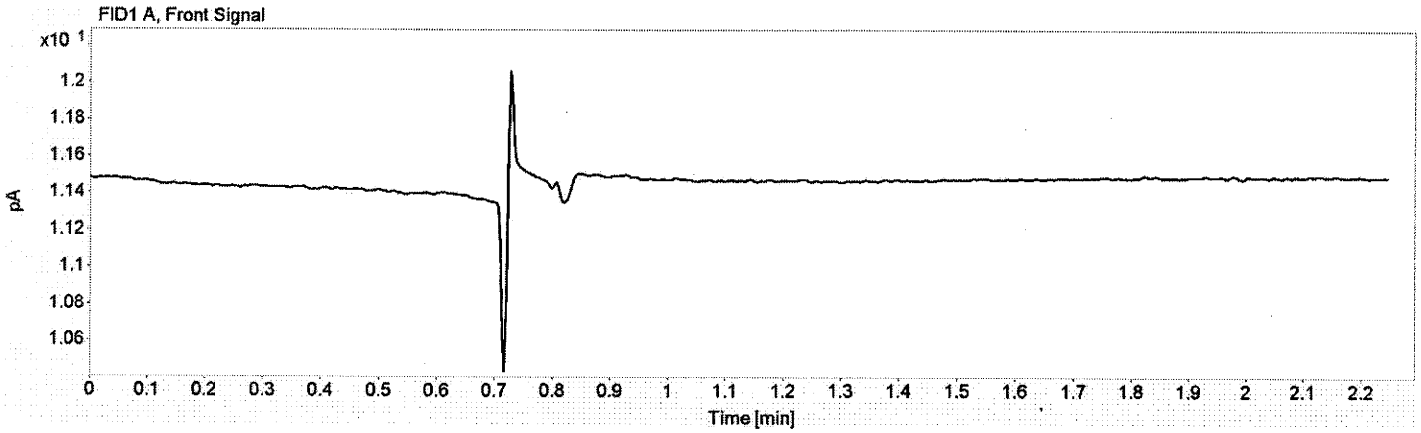
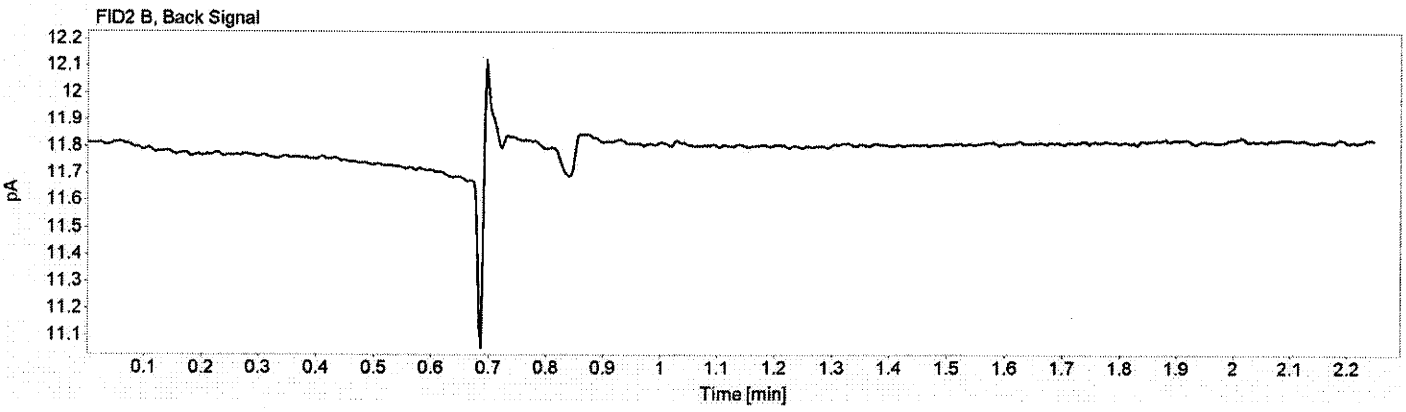
Phoenix Crime Lab: Alcohol Report

Sample name:	18 M ohm water new filter	**Item Number:**	
Location:	4	**Injection date:**	9/15/2017 1:43:20 PM
Sequence:	JM091517 2017-09-15 13-15-33	**Instrument:**	Jack
Acq. method:	ALCOHOL.M	**Analyst:**	JM
Data file:	D:\Data\JM\JM091517 2017-09-15 13-15-33\004F0401.D		



A. Quantitation

Compound	Time (min)	Peak Area	Amount (g/100ml)



B. Confirmation

Compound	Time (min)	Peak Area

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1. PROTOCOL FOR THE ANALYSIS OF ETHANOL

PURPOSE

The Toxicology section is routinely asked to analyze evidence for the presence and quantity of ethanol. This protocol outlines the procedure to follow when performing this type of analysis.

PLAN

A. Instrumentation

(1) Perkin-Elmer Gas Chromatograph equipped with:

(a) Totalchrom Software

(b) Dual capillary columns – Restek Corp.

- BAC 1-WCOT fused silica, 30m length, .32 mm i.d. stationary phase, 1.8 µm film thickness
- BAC 2-WCOT fused silica, 30m length, .32 mm i.d. stationary phase, 1.2 µm film thickness

(2) TurboMatrix 110 Headspace Autosampler

(3) Agilent Gas Chromatograph equipped with:

(a) OpenLAB Software

(b) Dual capillary columns – Agilent J&W

- DB-ALC1 fused silica, 30m length, .32 mm i.d. stationary phase, 1.8 µm film thickness
- DB-ALC2 fused silica, 30m length, .32 mm i.d. stationary phase, 1.2 µm film thickness

(4) Agilent 7697A Headspace Autosampler

B. Solutions, Standards, Calibrators and Controls
~~Note: these should be checked by HSGC prior to use.~~

(1) **HSGC Internal Standard Solution**
This standard consists of 0.015% v/v n-propanol and 0.5M ammonium sulfate in deionized water.

(a) Check water before use, verify no peaks present (acceptable peak area ≤0.25)

(b) Fill a ~~volumetric flask~~ 2L container half full of deionized water.

(c) Add 300 µl of n-propanol to the ~~flask~~ container.

(d) Add 132.08g ammonium sulfate to ~~flask~~ the container. Mix thoroughly until dissolved.

(e) Dilute to volume (2000ml) with deionized water. Invert and mix thoroughly.

(f) ~~Transfer to working containers~~ Check by HSGC prior to use, verify n-propanol peak area comparable to previous lot of IS (guideline: 100 to 150) and no other volatiles present.

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(g) Record preparation and checks in HSGC Reagent Log Book

(h) Stability 1 year

(2) **Preparation of mixed volatiles standard**
Note: acetaldehyde must be pipetted cold. Boiling point is 20.8°C.

(a) Pipette into a 100ml volumetric flask:

- 2~~0~~5µl acetaldehyde
- ~~80~~75µl methanol
- 2~~0~~5µl acetone
- ~~80~~75µl ethanol
- 50µl isopropyl alcohol

(b) Add deionized water to 100ml mark.

(c) Transfer to amber glass bottles and cap

(d) Record preparation in HSGC Reagent Log Book

(e) Stability 1 year

(3) **Calibrators**

(a) 0.025%, 0.050%, 0.100%, 0.200%, 0.400% aqueous ethanol calibrators purchased from an independent vendor

(4) **Controls**

(a) Aqueous ethanol controls purchased from an independent vendor (different than the vendor for the calibrators).

(b) Whole blood control at a mid range concentration purchased from an independent vendor or prepared in house (stability 1 year) as follows:

- Negative whole blood is prepared as follows: Add 900mg of sodium chloride to a 100 mL volumetric flask. Dilute to volume (100mL) with deionized water. In a 200 mL volumetric add this 100mL 0.9% NaCl solution and 100 mL of red blood cells. Mix well gently by inversion for 10 minutes.

- Negative whole blood is tested by HSGC prior to addition of ethanol to demonstrate the absence of ethanol. If ethanol or another volatile is detected, prepare a new batch of negative whole blood.

- An interim 2% aqueous ethanol stock solution is prepared as follows: Fill a 100 mL volumetric flask approximately 1/3 full with deionized water. Place on analytical balance, tare, and gravimetrically add 2.00 grams of ethanol from an ethanol source with >99% purity (i.e. >198 proof). Record the weight. Dilute to volume with deionized water. Discard after use.

- Addition of preservatives, anticoagulants, and inhibitors: Fill a 200 ml volumetric flask 2/3 full with negative whole blood. Add 0.4 grams of Potassium Oxalate (KOX), 2.0 grams of Sodium Fluoride (NaF), 4.0 grams of

Sodium Hydrosulfite (dithionite) ($Na_2S_2O_4$), and 10.0 mg of Pyrazole ($C_3H_4N_2$). Mix well by inversion, over a two hour period.

- Addition of ethanol: Volumetrically add 20.0 mL of the 2% aqueous ethanol stock to the blood prepared in the step above. Dilute to volume with negative whole blood. Mix thoroughly by inversion for 10 minutes.
- Propagation of aliquots: Pipette 1.1 mL of the blood control into 2.0 mL amber crimp cap vials. Crimp tightly and place in refrigerator.
- Assignment of value: Analyze at least 40 replicates on at least 2 different instruments and determine the average value. This will establish the target value for that batch of whole blood control.

(c) Prepare a negative control using DI water and internal standard.

C. Priming of Hamilton Microlab® ~~500A Series~~ Dispenser Diluters

Microlab 500A Series

(1) Verify that the pipettor is set up correctly. The left syringe should be set to a speed of 4, delivering 1000 µl of internal standard. The right syringe should be set to a speed of 2, delivering 100 µl of sample.

(2) Prime the instrument with internal standard solution for at least three cycles by depressing toggle switch labeled "step/prime" down into the "prime" position.

Microlab 600 Series

(1) Verify that the pipettor is set up correctly. Press Quick Start on screen, the left syringe should be set to deliver 1000 µl of internal standard. The right syringe should be set to deliver 100 µl of sample.

(2) Prime the instrument with internal standard solution for at least three cycles by pressing the 'prime' button, to stop priming press 'prime' button again. Before use, reset pipettor by pressing button twice on hand probe.

D. Sample Preparation

(1) Allow all calibrators, controls and samples to come to room temperature before starting.

(2) Whole blood samples should be mixed thoroughly before pipetting. Plasma/serum and urine samples require no preparation. In the case of samples in Serum Separation tubes (SST's) or samples for toxic vapor analysis an additional vial, with internal standard and DI water only, should follow each of the case sample vials to ensure no possible carryover from toluene. Clotted samples must be THOROUGHLY homogenized before pipetting or alternatively centrifuged and reported as plasma/serum.

(3) Prepare the following: calibrators in duplicate; a mixed volatile standard (without toluene); a negative control; enough ethanol whole blood and aqueous controls to bracket up to five samples in duplicate; and subject samples in duplicate with the following procedure:

(a) Insert probe tip into the sample vial and push the button on the probe tip holder once.

(b) Remove probe tip from sample container and extend tip into the headspace vial designated for the sample.

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(c) Push the button on the probe tip once to automatically dispense the preset volumes of sample and internal standard into the headspace vial.

(d) Tightly crimp cap onto the vial.

(e) Rinse pipette tip with DI water between each sample.

(f) Create and print sequence.

(g) Load samples into autosampler according to sequence and have it verified by another analyst prior to unloading.

(h) Setup and start specimen analysis.

(i) Complete Blood Alcohol Analysis Face Sheet.

(4) Preparation of Liquor Samples; Prior to pipetting, samples will be diluted as follows:

(a) Beer should be diluted 1:20 with DI water.

(b) Wine should be diluted to 1:50 with DI water.

(c) Spirits and other specimens should be diluted 1:100 (or other appropriate dilution) with DI water.

(5) Urine samples may be analyzed for ethanol if no blood sample is available. If positive for ethanol report qualitatively and test the urine sample for ~~ketones and~~ glucose using a "Keto-Diastix" or equivalent. Test both positive and negative controls, recording the lot number of the controls as well as the "Keto-Diastix."

E. Quality Assurance Checks for analytical result acceptability:

(1) Check linearity of calibration. Calibration curve must have $R^2 \geq 0.99$

(2) Check standards and controls for accuracy and precision. Standards and controls must be within ±5% of the target values.

(3) Ensure ethanol is identified on both channels

(4) Check duplicates for precision. The difference between duplicate samples must be within ± 5% of the average of the two results (or ± 0.005 grams per 100mL for results where the average is <0.025 grams per 100mL).

(5) Check resolution of the mixed volatile standard.

(6) Check for the absence of ethanol in negative control.

(7) File Face Sheet, printouts for calibration and controls in appropriate electronic file/file cabinet

F. Instrument and Quality Control Corrective Actions

The following actions are not intended to cover all possible scenarios, but are provided to address some infrequently experienced occurrences. Unforeseen future issues will be addressed as they arise.

(1) **Issue:** Calibration failure due to any of the following; any individual calibrator value exceeding ± 5%; linear regression analysis where R^2<0.99; or ethanol not present in any of the calibration chromatograms. **Correction:** Re-pipette the calibrators and/or entire batch if applicable.

(2) **Issue:** Negative control contains ethanol or any other volatile, properly identified per reporting guidelines. **Correction:** Prepare a valid negative control and re-pipette and analyze entire batch.

(3) **Issue:** Not all components of the mixed volatile standard are properly identified on one or both of the GC columns. **Correction:** Update the retention times in the method and reprocess the entire batch, or update the retention times and re-start the entire batch.

(4) **Issue:** A quality control sample, either blood or aqueous control has exceeded ± 5%. **Correction:** Re-pipette and analyze any case samples bracketed by the out of tolerance control. Note: These may be analyzed at the end of a completed batch with bracketing controls, provided the calibration is within 24 hours. Applies to Perkin Elmer HSGC

(5) **Issue:** An individual case sample has duplicates which exceed the ± 5% or ± 0.005 grams per 100mL requirement. **Correction:** Re-pipette and analyze the individual case sample. Note: These may be analyzed at the end of a completed batch with bracketing controls, provided the calibration is within 24 hours. Applies to Perkin Elmer HSGC

(6) **Issue:** Crane failure error during the batch. **Correction:** Visually evaluate and document what caused the failure if possible, press the stop button on the autosampler display. When the vials have been unloaded from the oven and the autosampler is in standby, restart the batch from the computer with the vial # the batch had stopped on. Samples which were thermostatted in the oven for an extended period of time (i.e. overnight) will be re-pipetted and analyzed. Note: These may be analyzed at the end of a completed batch with bracketing controls, provided the calibration is within 24 hours. Applies to Perkin Elmer HSGC

(7) **Issue:** If on an individual result or multiple samples in a batch any of the following are observed: ethanol and/or internal standard retention time shift, or area counts are abnormally high or low (i.e. exceeds by 50% or more the average area count observed throughout the batch). **Correction:** Visually inspect the vials associated with the affected chromatogram for abnormalities, e.g. loose vial cap, etc. Re-pipette and analyze the affected case samples. Note: These may be analyzed at the end of a completed batch with bracketing controls, provided the calibration is within 24 hours. Applies to Perkin Elmer HSGC

G. Conclusions

(1) Any average result between 0.025 and 0.400 grams per 100mL report the result truncated to the third digit and the measurement uncertainty associated with the result.

(2) If either result is less than 0.005 grams per 100mL it will be reported as "no ethyl alcohol detected."

(3) Any average result between 0.005 and 0.025 grams per 100mL will be reported as "ethyl alcohol detected <0.025 grams per 100mL".

(4) Any result greater than 0.400 grams per 100mL will be reported as "ethyl alcohol detected > 0.400 grams per 100mL".

(5) For liquor cases report results qualitatively but convert w/v to v/v. (v/v = (w/v)/0.789 * dilution amount) on worksheet.

(6) Qualitatively report volatile compounds other than ethanol and acetaldehyde, only if identified on all four chromatograms, ~~and~~ the retention time matches the mixed volatile standard within ± 3%, and the concentration is ≥0.005 g/100mL. Do not report toluene in samples from Serum Separation tubes (SST's) since they contain toluene.