The ODS Renovation Project & Elk River Spill 2014

(or how much time do I have before the "It" hits the fan?)

ORSANCO Technical Committee Meeting
Jerry Schulte, Lila X. Ziolkowski & Travis Luncan
February 12-13, 2014

Preliminary Spill Event Data for Elk River

- First NRC spill report generated about the Elk River spill was from private citizen as “a strong chemical smell in the air” at 1500 in the afternoon.
- A second NRC report was generated from the NRC at 1900 indicating an incident on the Elk river occurred around 1045 that morning.
- Elk River velocity was estimated at between 0.5-0.8 mph. NOAA’s National Weather Service estimated the velocity for Kanawha 2.3 mph.
- Time of travel estimates to other water utilities were made using “back of the envelope” calculations and hydrologic modeling software

Incident Report # 1070606

INCIDENT DESCRIPTION

*Report taken by: CIV NYDIA RAWLS at 15:45 on 09-JAN-14
Incident Type: FIXED
Incident Cause: UNKNOWN
Affected Area:
Incident occurred on 09-JAN-14 at 15:00 local incident time.
Affected Medium: AIR ATMOSPHERE

INCIDENT LOCATION
OHIO STREET County: KANAWHA
City: CHARLESTON State: WV
IN THE VICINITY OF LOCATION BELOW

RELEASED MATERIAL(S)
CHRIS Code: UNK Official Material Name: UNKNOWN MATERIAL
Also Known As:
Qty Released: 0 UNKNOWN AMOUNT

DESCRIPTION OF INCIDENT
CALLER IS REPORTING A STRONG CHEMICAL SMELL IN THE AIR FROM AN UNKNOWN SOURCE.

INCIDENT DETAILS
Package: N/A
Building ID:
Type of Fixed Object: PRIVATE RESIDENCE
Power Generating Facility: UNKNOWN
Generating Capacity:
Type of Fuel:
NPDES:
NPDES Compliance: UNKNOWN

REMEDIAL ACTIONS
MAKING NOTIFICATION
Release Secured: UNKNOWN
Release Rate:
Estimated Release Duration:

Incident Report # 1070627

INCIDENT DESCRIPTION

*Report taken by: CIV KEVIN WILLIAMS at 19:16 on 09-JAN-14
Incident Type: STORAGE TANK
Incident Cause: UNKNOWN
Affected Area: ELK RIVER
Incident occurred on 09-JAN-14 at 10:47 local incident time.
Affected Medium: WATER ELK RIVER

REPORTING PARTY
Organization: NATIONAL RESPONSE CENTER
WASHINGTON, DC

PRIMARY Phone: (800)4248802
Type of Organization: FEDERAL GOVERNMENT

SUSPECTED RESPONSIBLE PARTY
Name: UNKNOWN
Organization: WEST VIRGINIA AMERICAN WATER

INCIDENT LOCATION
ELK RIVER County: KANAWHA
1015 BARLOW DRIVE
State: WV

RELEASED MATERIAL(S)
CHRIS Code: MCY Official Material Name: METHYLCYCLOHEXANE
Also Known As:
Qty Released: 0 UNKNOWN AMOUNT Qty in Water: 0 UNKNOWN AMOUNT

INCIDENT DETAILS
Description of Tank:
Tank Above/Below Ground: ABOVE
Transportable Container: UNKNOWN
Tank Regulated: UNKNOWN
Tank Regulated By:
Tank ID:
Capacity of Tank:
Actual Amount:
---WATER INFORMATION---
Body of Water: ELK RIVER
Tributary of:
Nearest River Mile Marker:_____
Water Supply Contaminated: UNKNOWN

DESCRIPTION OF INCIDENT
RESIDENTS IN CHARLESTON AND SURROUNDING COUNTIES WERE TOLD THURSDAY EVENING NOT TO DRINK, COOK WITH OR WASH WITH WATER SUPPLIED BY WEST VIRGINIA AMERICAN WATER, FOLLOWING A LEAK EARLIER IN THE DAY AT A CHEMICAL FACILITY ALONG THE ELK RIVER. ANY WATER SUPPLIED BY WEST VIRGINIA AMERICAN WATER IN KANAWHA, PUTNAM, BOONE, JACKSON AND LINCOLN COUNTIES WAS TO BE USED ONLY FOR FLUSHING TOILETS AND PUTTING OUT FIRES. THE STATE OF EMERGENCY INCLUDES WEST VIRGINIA AMERICAN WATER CUSTOMERS IN BOONE, LINCOLN, KANAWHA, JACKSON AND PUTNAM COUNTIES, ACCORDING TO NBC STATION WSAZ. RESIDENTS WERE TOLD NOT TO DRINK THE WATER, BATHE IN IT OR COOK WITH THE WATER AND ONLY USE IT FOR FLUSHING AND FIRE EMERGENCIES. BOILING IT WILL NOT REMOVE THE HEMICALS. THE DECLARATION IS BELIEVED TO IMPACT 100,000 CUSTOMERS, WSAZ REPORTED. THE STATION SAID THE CHEMICAL LEAKED FROM A TANK AT FREEDOM INDUSTRIES IN CHARLESTON. THE LEAKED PRODUCT IS 4-METHYLCYCLOHEXANE METHANOL, WHICH IS USED IN THE FROTH FLOTATION PROCESS OF COALWASHING AND PREPARATION. ACCORDING TO WEST VIRGINIA AMERICAN WATER, DUE TO A CHEMICAL LEAK THAT WENT INTO THE ELK RIVER THURSDAY, A DO NOT USE WATER ORDER HAS BEEN ISSUED FOR THE COUNTIES OF BOONE, JACKSON, KANAWHA, LINCOLN, AND PUTNAM IN WV. RESIDENTS LIVING IN THESE COUNTIES WHO USE WEST VIRGINIA AMERICAN WATER ARE BEING ASKED TO NOT USE TAP WATER FOR DRINKING, COOKING,WASHING, OR BATHING UNTIL FURTHER NOTICE. THE PUBLIC SERVICE DISTRICT SAYS THIS ONLY AFFECTS CUSTOMERS WHO USE WEST VIRGINIA AMERICAN WATER. CUSTOMERS IN ST. ALBANS AND CEDAR GROVE ARE NOT AFFECTED BY THIS. IT IS BELIEVED THAT THE MATERIAL MAY BE HAZARDOUS AND MAY HAVE ENTERED THE WATER SYSTEM. THE GOVERNOR OF WV HAS DECLARED A STATE OF EMERGENCY. A SPOKESPERSON FOR WEST VIRGINIA AMERICAN WATER SAYS IF YOU CAME INTO CONTACT WITH THE WATER AND FEEL ILL, CALL THE POISON CONTROL HOTLINE AT 1-800-222-1222.

Environmental Impact: UNKNOWN
Media Interest: HIGH Community Impact due to Material:

REMEDIAL ACTIONS

Release Secured: UNKNOWN
Release Rate:
Estimated Release Duration:

NOAA NATIONAL WEATHER SERVICE FLOW DATA

River Flow and Velocity Forecasts
NWS Ohio River Forecast Center, Wilmington, OH
10:34am EST Thursday, January 9, 2014

Note: All forecasts include expected precipitation through the first 48 hours.
Flows are units of kCFS. Velocity units are MPH. * = HEC-RAS estimation

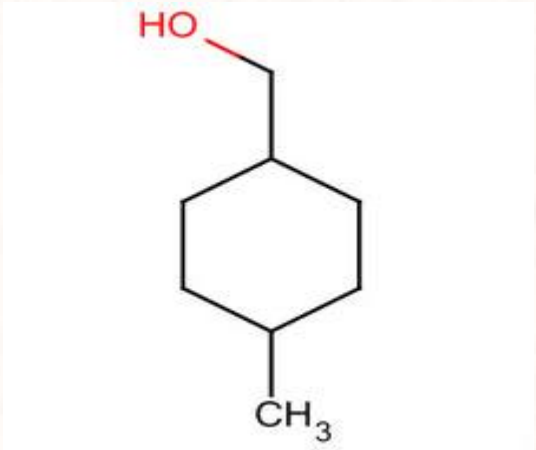
Forecast Point...	Jan 09 Flow	Jan 09 Vel	Jan 10 Flow	Jan 10 Vel	Jan 11 Flow	Jan 11 Vel	Jan 12 Flow	Jan 12 Vel
Allegheny River...								
C.W. Bill Young L and D	20.1	0.9	24.4	1.1	21.6	1.0	25.1	1.1
Monongahela River...								
Braddock	19.3	1.2	16.5	1.0	17.0	1.1	25.7	1.5
Beaver River...								
Beaver Falls	6.0	0.8	5.1	0.7	5.4	0.7	9.1	1.2
Ohio River...								
Dashields Lock and Dam	40.3	1.1	41.5	1.1	39.5	1.0	51.7	1.3
Montgomery Lock and Dam	46.4	1.2	44.7	1.1	43.6	1.1	58.8	1.5
Wheeling	48.8	2.0	46.2	1.9	45.8	1.8	63.0	2.5
Moundsville	49.5	1.2	46.8	1.1	46.8	1.1	64.8	1.5
Willow Island L and D	51.6	1.7	48.4	1.7	49.7	1.7	69.0	2.1
Parkersburg	64.3	1.4	61.3	1.3	66.2	1.4	91.1	1.8
Racine Lock and Dam	67.8	1.4	64.8	1.4	71.8	1.5	98.4	2.0
R C Byrd Dam	90.9	1.6	87.5	1.6	91.0	1.6	121.2	2.1
Huntington	96.9	1.7	91.5	1.7	92.4	1.7	119.6	2.1
Lloyd Greenup Dam	109.9	1.6	99.5	1.5	100.4	1.5	121.1	1.8
Meldahl Dam	124.7	2.5	107.9	2.4	106.6	2.4	119.9	2.5
Cincinnati	139.3	2.4	119.3	2.2	112.9	2.1	126.0	2.3
Markland Dam	145.2	2.5	134.4	2.4	124.4	2.3	152.4	2.5
McAlpine Upper	154.5	1.6	154.0	1.6	139.4	1.5	153.9	1.6
McAlpine Lower	154.0	2.5	153.6	2.5	139.1	2.4	153.4	2.5
Cannelton Dam	156.7	2.5	158.4	2.5	151.3	2.5	155.7	2.5
Evansville	180.3	2.1	177.1	2.1	171.8	2.1	174.7	2.1
J T Myers Dam	238.3	2.1	219.4	1.9	215.0	1.9	208.5	1.8
Shawneetown	248.1	2.3	223.9	2.2	218.9	2.2	213.0	2.1
Golconda	275.4	1.8	240.7	1.6	226.4	1.5	225.3	1.5
Smithland Lock and Dam	285.6	1.9	249.3	1.7	230.0	1.5	227.6	1.5
Muskingum River...								
McConnelsville	6.9	0.7	7.6	0.7	8.3	0.8	9.7	0.9
Kanawha River...								
Kanawha Falls	12.5	2.3	8.0	1.9	9.2	2.0	16.5	2.6

Composition 2	
75%	4-methyl cyclohexane methanol
11.9%	4-(methoxymethyl) cyclohexane methanol, a monoether
8.9%	4-carbomethoxy cyclohexane methanol, a monoether
3.1%	water
0.9%	a monoaldehyde of 4-methyl cyclohexane methanol
0.1%	cyclohexane dimethanol

SYNONYMS OF 4-MCHM

4-Methylcyclohexanemethanol
(4-Methylcyclohexyl)methanol
4-Methyl-1-cyclohexanemethanol
Cyclohexanemethanol, 4-methyl-
1-(Hydroxymethyl)-4-methylcyclohexane
34885-03-5
Cyclohexanemethanol, 4-methyl-, trans-
ACMC-20amee
AC1L3MEZ
SureCN155951
AC1Q7C7Z
SureCN3485266
SureCN8216437
DSSTox_CID_21813
DSSTox_RID_79850
DSSTox_GSID_41813
cis-4-Methylcyclohexanemethanol
CTK1B4020
trans-4-Methylcyclohexanemethanol
Tox21_301528
AR-1G3608
AKOS009158915
Cyclohexanemethanol, 4-methyl-, cis-
NCGC00255649-01
AI3-28423
CAS-34885-03-5
FT-0692545
M1412
M112072
3937-49-3

4-Methylcylcohexane Methanol (4-MCHM)

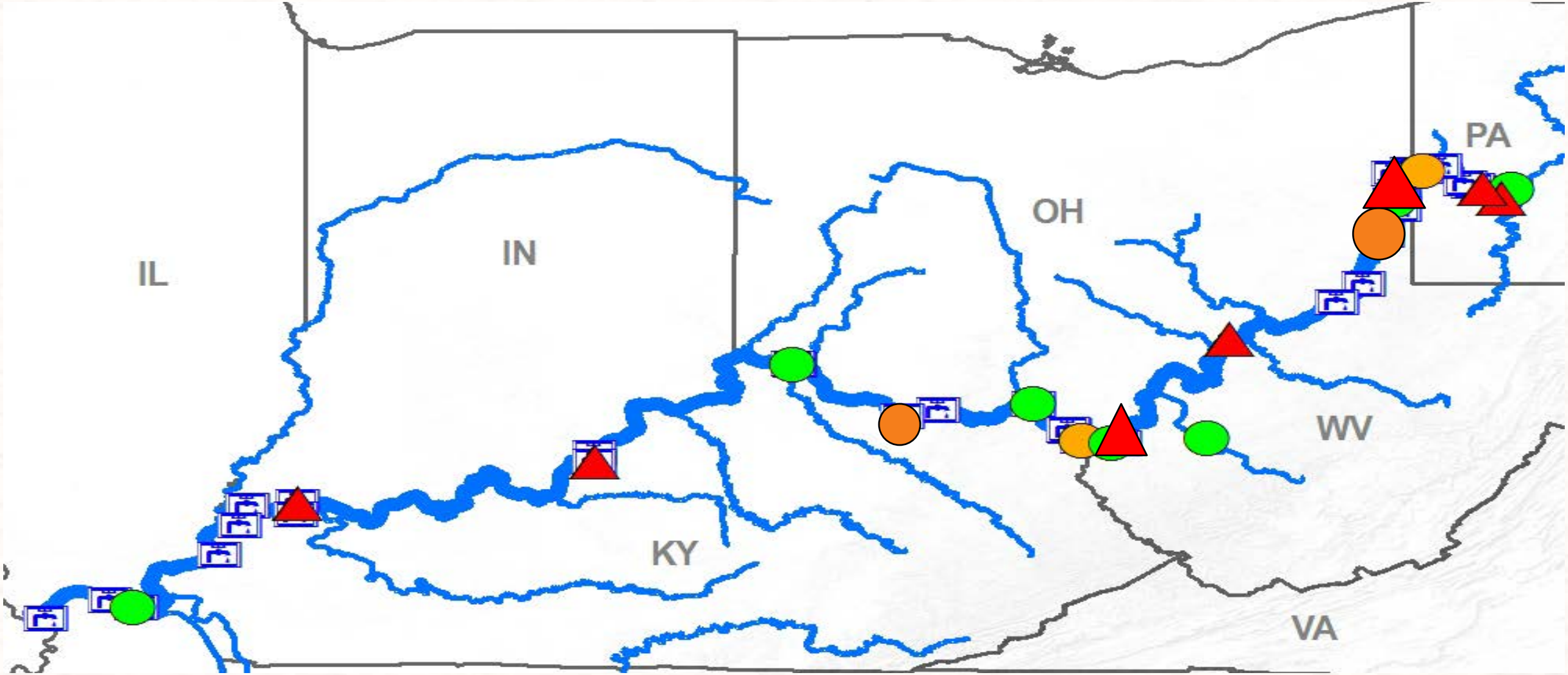


9.1 Information on basic physical and chemical properties

Appearance Physical State: Liquid
Form: Liquid
Color: Colorless
Odor: Alcohol
Odor Threshold: No data available.
pH: No data available.
Freezing Point: 0 °C
Boiling Point: 180 °C
Flash Point: 112.8 °C (Setaflash Closed Cup)
Evaporation Rate: No data available.
Flammability (solid, gas): No data available.
Flammability Limit - Upper (%)–: No data available.
Flammability Limit - Lower (%)–: No data available.
Vapor pressure: No data available.
Vapor density (air=1): No data available.
Relative density: < 1 (estimated)
Solubility(ies)
Solubility in Water: Appreciable
Solubility (other): No data available.
Partition coefficient (n-octanol/water): No data available.
Autoignition Temperature: No data available.
Decomposition Temperature: Thermal stability not tested. Low stability hazard expected at normal operating temperatures.
Viscosity: No data available.
Explosive properties: No data available.
Oxidizing properties: No data available.

"…The sad truth is this chemical is one of *tens of thousands* of chemicals on the market today with little or no safety data. MCHM is one of the 62,000 chemicals that were already in use when TSCA, our nation's main chemical safety law, was passed in 1976. All of these chemicals were grandfathered by TSCA: That means they were simply presumed to be safe, and EPA was given no mandate to determine whether they are actually safe. Even to require testing of these chemicals under TSCA, EPA must first provide evidence that the chemical may pose a risk – a toxic *Catch-22*. " Richard Dennison, PHD, *EDF Health*

ODS MONITORING LOCATIONS



Water Utility

GC/MS technology

Early 90's technology

CMS5000 process GC

Closed Networked System

- We were able to access analytical instrumentation while at other locations to verify that systems were operational and make some adjustments as necessary.
  - –System wide monitoring at our fingertips!

So what's the operator (and ORSANCO) to do?

- Round the clock sample collecting procedures at the Charleston Water Plant started the evening of January 9th.
- Which means round the clock analysis…
- Which means round the clock operation of instrumentation…
- Which means round the clock ODS personnel…
- And frequent communication…

(and keep lots of Coffee, Mountain Dew, and Red Bull nearby…)

Only problem is….

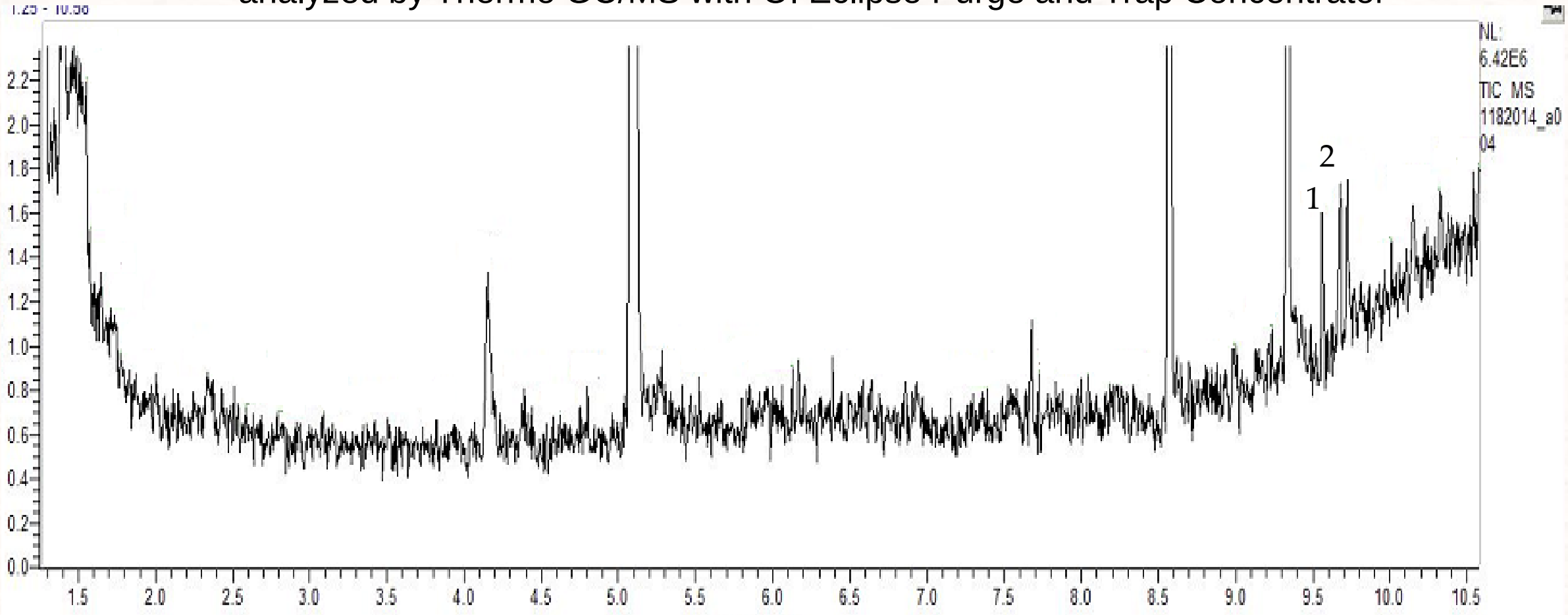
- 4-MCHM is not regulated under the Clean Water Act (CWA or SDWA) so there is no USEPA method…
- Literature review had very few studies done on this compound, and analyses that were performed, used instrumentation unavailable in the ODS system.
- However, based on information from chemical manufacturer's SDS information, it appeared that our volatiles instrumentation may be able to detect this (with fingers crossed)
- We hypothesized that it would be a later eluting compound because of boiling point and structure but had no idea of what peak response would be like
- Where do we find pure reference standard?

We hit the ground running…

Using our routine “daily” method, we were able to detect 4-MCHM…in raw (crude) material product obtained from the spiller

The Autosampler carousel allows for ~70 samples to be preloaded and analyzed

4 ppb standard 4-MCHM
analyzed by Thermo GC/MS with OI Eclipse Purge and Trap Concentrator



Chromatogram of Crude MCHM spiked in deionized water. Column DB-624, 30 m, 0.18 mm id, 1.0 µm
Purge 11 minutes
Temperature Profile: 40 °C (hold 4.0 min.) to 100 °C at 18 °C/min, to 210 °C at 40 °C/min (hold 4.0 min)
Peaks: 1 = cis 4methylcyclohexanemethanol (4 µg/L), 2 = trans 4methylcyclohexanemethanol (4 µg/L)

Source: TML Huntington 2014

How about St. Albans…

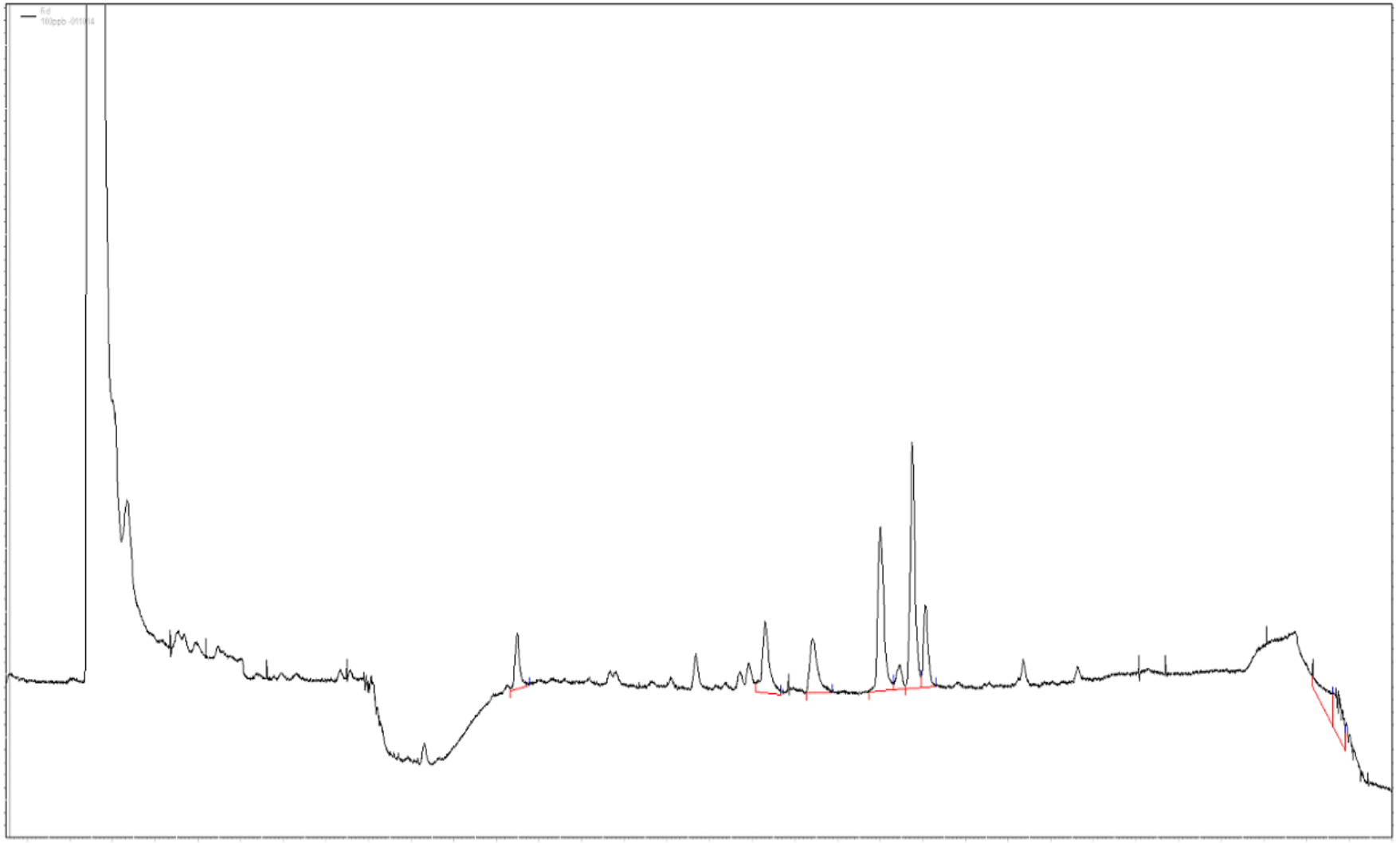
- This AEP facility was one of the first ODS stations. They are not a water utility, but are in a key location on the Kanawha river, ~40 miles upstream of the confluence with the Ohio.
  - They have limited staffing availability and are working with '90's technology'.
    - Manual injection Purge and Trap with GC
- We took a gamble with this site and also ran under our regular daily method, with no modifications other than initially extending the run time and keeping our fingers crossed.
  - It takes about 50 minutes for a sample run.

We were able to detect this compound…barely

This chromatogram is a Kanawha River sample collected on January 9, 2014.

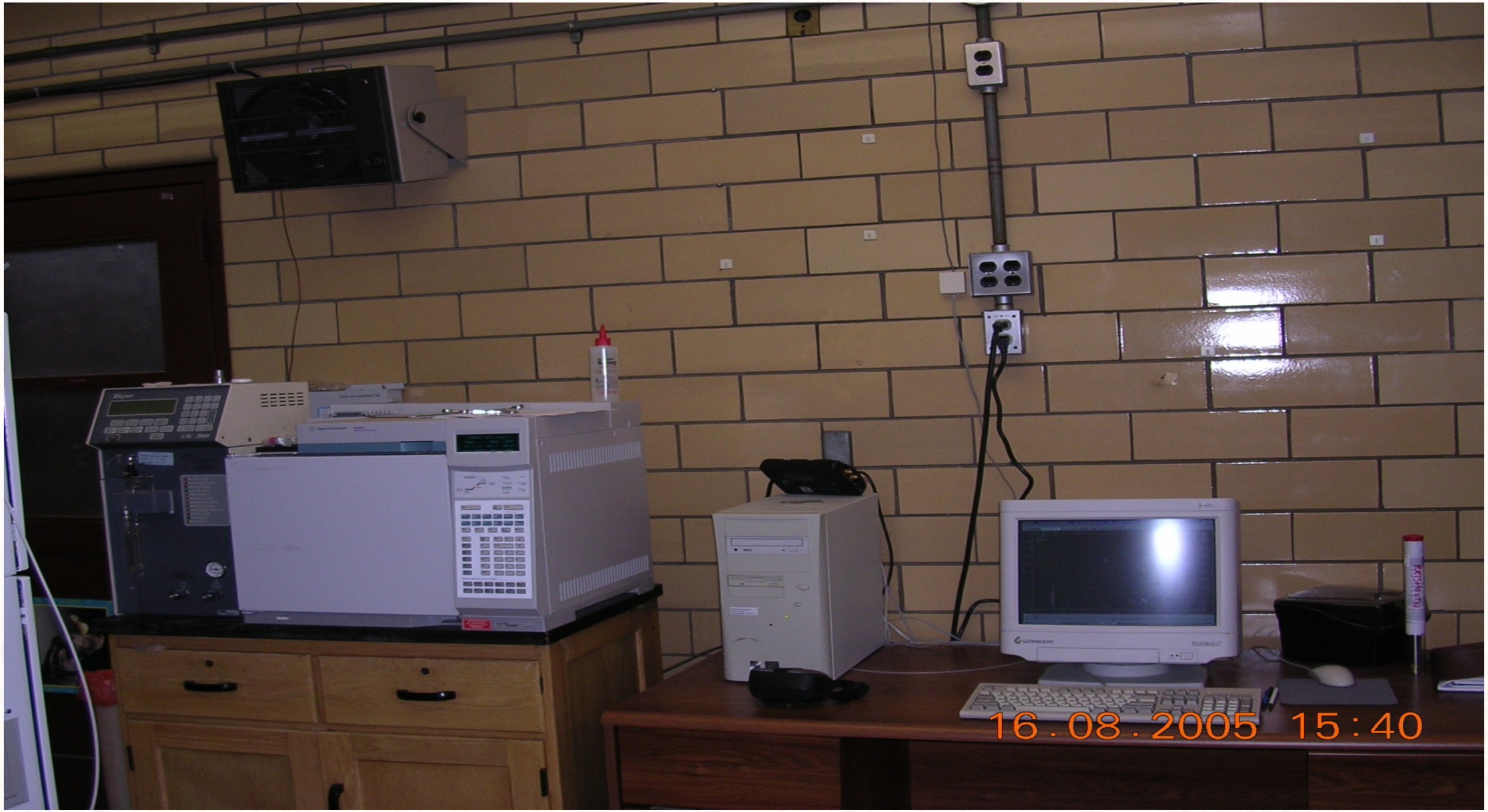
The estimated concentration of crude 4-MCHM in this sample was ~100 ppb.

100 ppb standard 4-MCHM
Analyzed on Agilent 6890 GC and OI 4560 P&T at AEP St. Albans



And then there's Portsmouth…

- Predicted arrival time 01/13/14. Around 1300 on 1/13/14, operators at Portsmouth reported smelling 4-MCHM (licorice like smell) in the plant.
- Because concentrations were expected to be well below the CDC's threshold of 1ppm for human health treatment facility treated raw water influent with carbon until spilled had passed through the area. Implemented sampling protocol of every 2-4 hours until spill passed.
  - Portsmouth ODS system is one of the oldest in system. Manual injection of sample into Purge and Trap unit and gas chromatograph.
    - Used same operating parameters as for routine daily analysis.
    - Added crude 4-MCHM to existing calibration curve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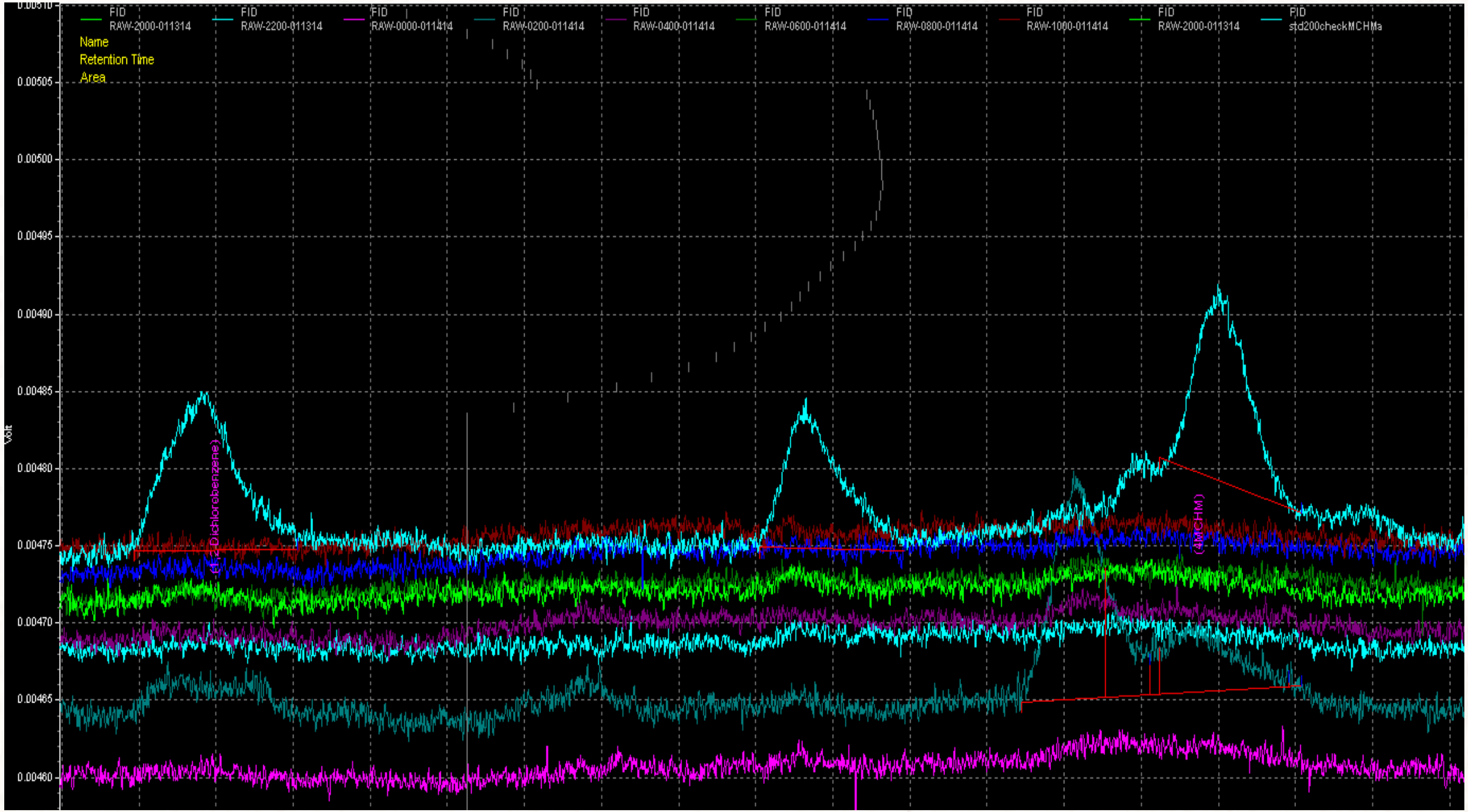
FID
RAW-2000-011314
FID
RAW-2200-011314
FID
RAW-0000-011414
FID
RAW-0200-011414
FID
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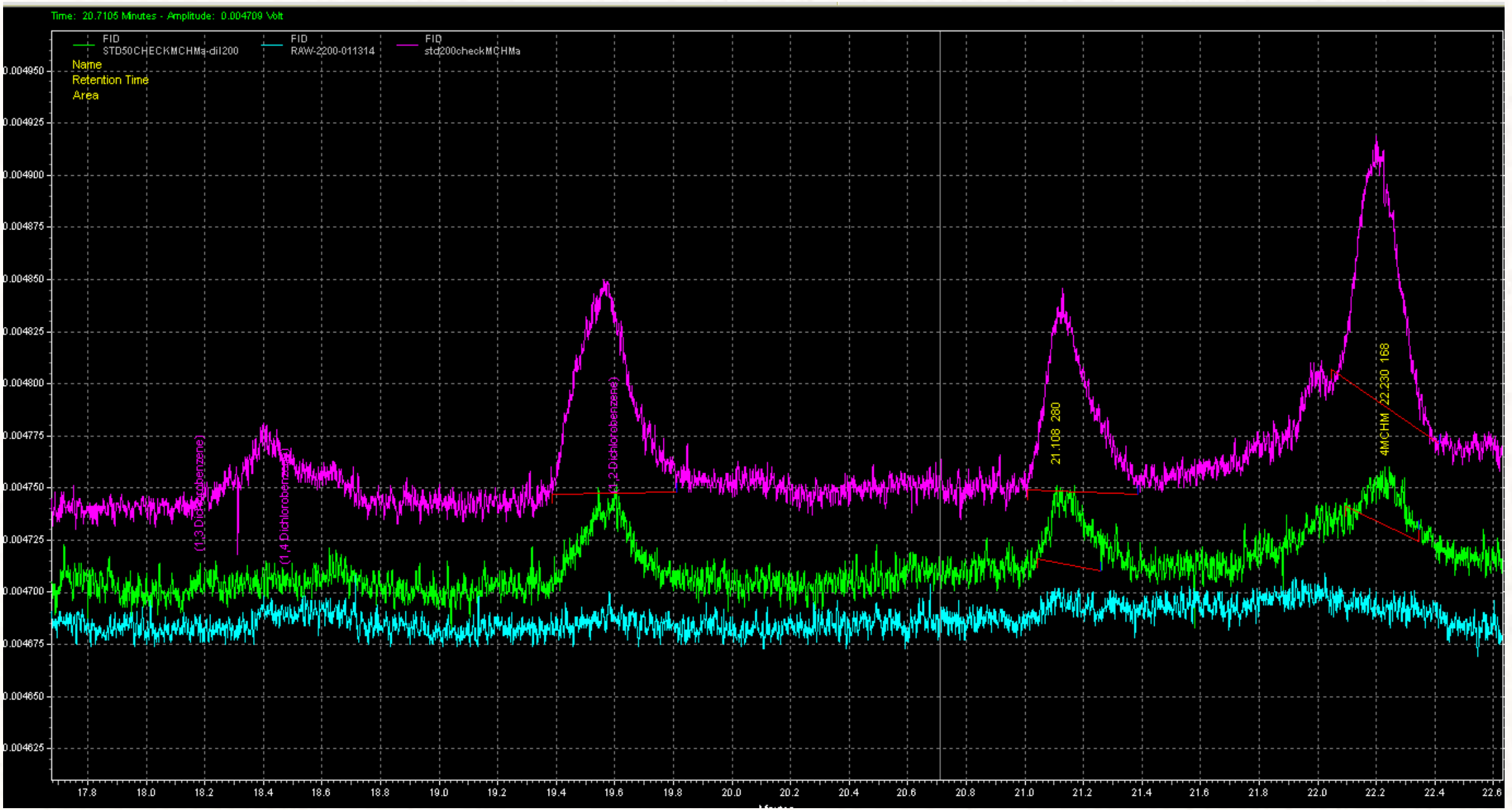
FID
RAW-0600-011414
FID
RAW-0800-011414
FID
RAW-1000-011414
FID
RAW-2000-011314
FID
std200checkMCHMa
Name
Retention Time
Area
Volt
0.00505
0.00500
0.00495
0.00490
0.00485
0.00480
0.00475
0.00470
0.00465
0.00460
(1,2-Dichlorobenzene)
(4MCHM)


Time: 20.7105 Minutes - Amplitude: 0.004709 Volt
FID
STD50CHECKMCHMa-dil200
FID
RAW-2200-011314
FID
std200checkMCHMa
Name
Retention Time
Area
(1,3 Dichlorobenzene)
(1,4 Dichlorobenzene)
(1,2-Dichlorobenzene)
21.108 280
22.230 168
4MCHM
0.004950
0.004925
0.004900
0.004875
0.004850
0.004825
0.004800
0.004775
0.004750
0.004725
0.004700
0.004675
0.004650
0.004625
17.8
18.0
18.2
18.4
18.6
18.8
19.0
19.2
19.4
19.6
19.8
20.0
20.2
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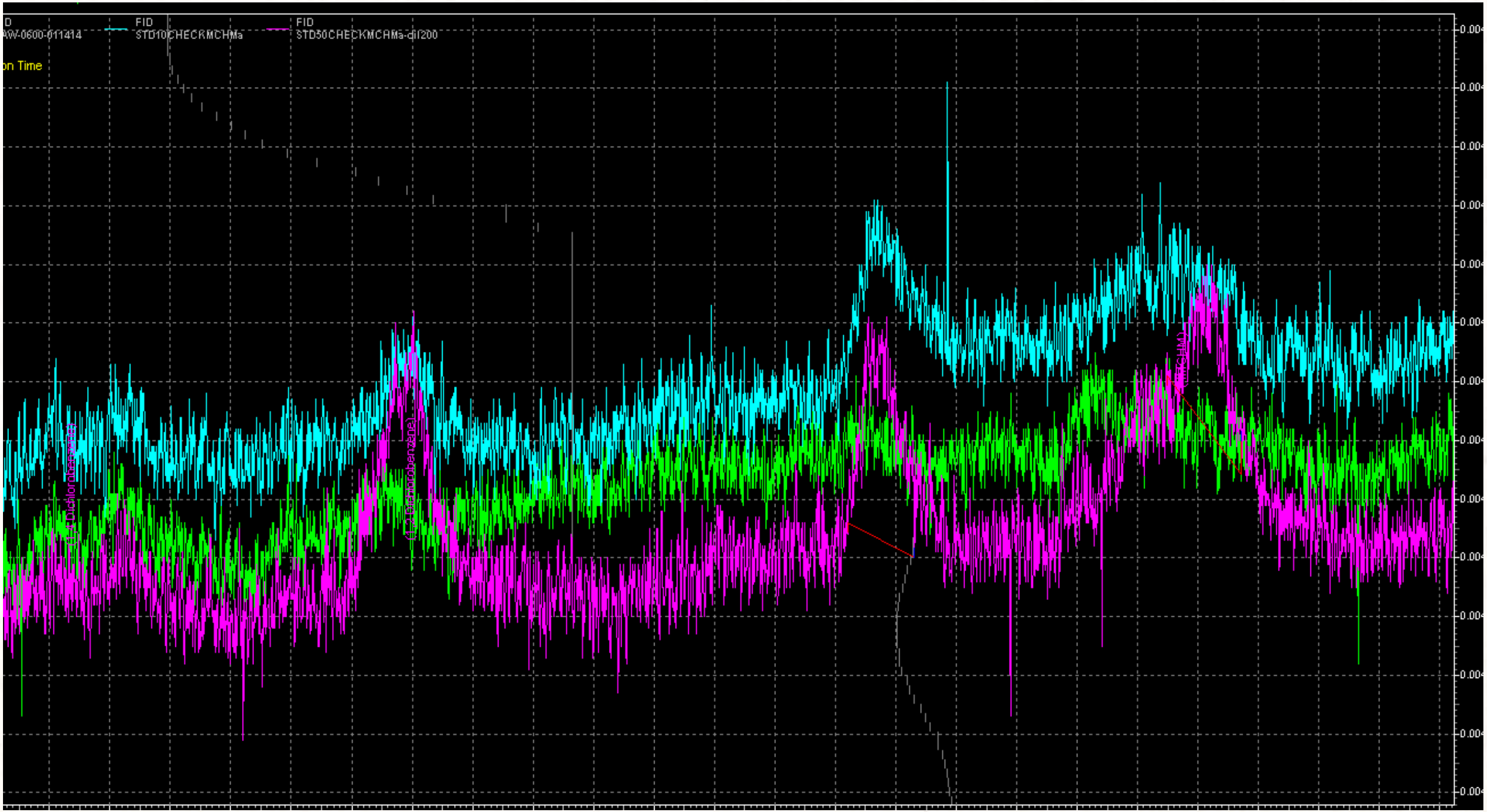
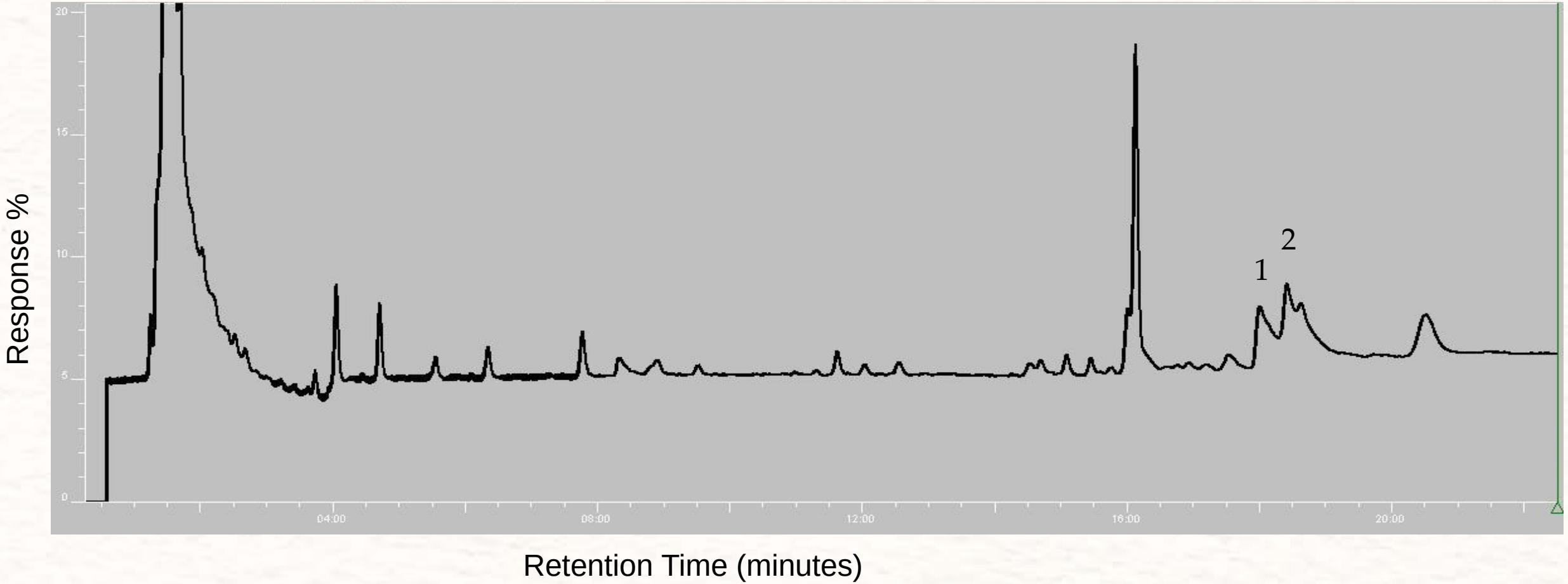
22.6


D
WW-0600-011414
FID
STD10CHECKMCHMa
FID
STD50CHECKMCHMa-dil200
on Time
0.00

Ashland and Maysville were on deck…

Travis headed to both these sites to after working with the CMS5000 that is located at GCWW we have been pilot testing during the renovation project.

80 ppb standard 4-MCHM
Analyzed on CMS 5000 at GCWW



Chromatogram of Crude MCHM spiked in deionized water. Column DB-624, 30 m, 0.53 mm id, 3.0 µm
Conc. Fill: 12 min.
Temperature Profile: 50 °C (hold 0.5 min.) to 85 °C at 4 °C/min, to 180 °C at 8 °C/min (hold 0.5 min), to 150 °C at 20°C/min.
Peaks: 1 = cis 4methylcyclohexanemethanol (80 µg/L), 2 = trans 4methylcyclohexanemethanol (80 µg/L)

The CMS5000 proved more challenging…

- While the Sample delivery system and constant flow through process offer round the clock analysis in near real time, some functionality and sensitivity is lost with this instrument
  - Cannot heat sample before transfer to GC component
  - Limited oven temperature programming , works best when in isothermal mode
  - Has very short sparging cycle (1 minute, cannot be changed)
  - The work around to increase sensitivity was to increase the pretreatment loop fill time (we went from 1 minute to 8 minutes)
    - Using these adjustments to method, could see response around 40 ppb, and lower for qualitative ID

Bring on the heat!

Heating a sample to > 40 degrees allows for volatization to occur more rapidly.

Winding down to Cincinnati, Louisville, and Evansville…

- These facilities had the luxury of time and feedback from Huntington and Dupont allowed them to refine method protocols, optimize detection limits and experiment with other methodologies.
  - The detection limit dropped to 3 ppb from around 40 ppb intially.
    - Based on the efforts of Cincinnati and Louisville ODS stations, Huntington was able to lower their detection limits

    Evansville's GCMS system was also geared up to detect this compound at very low levels.

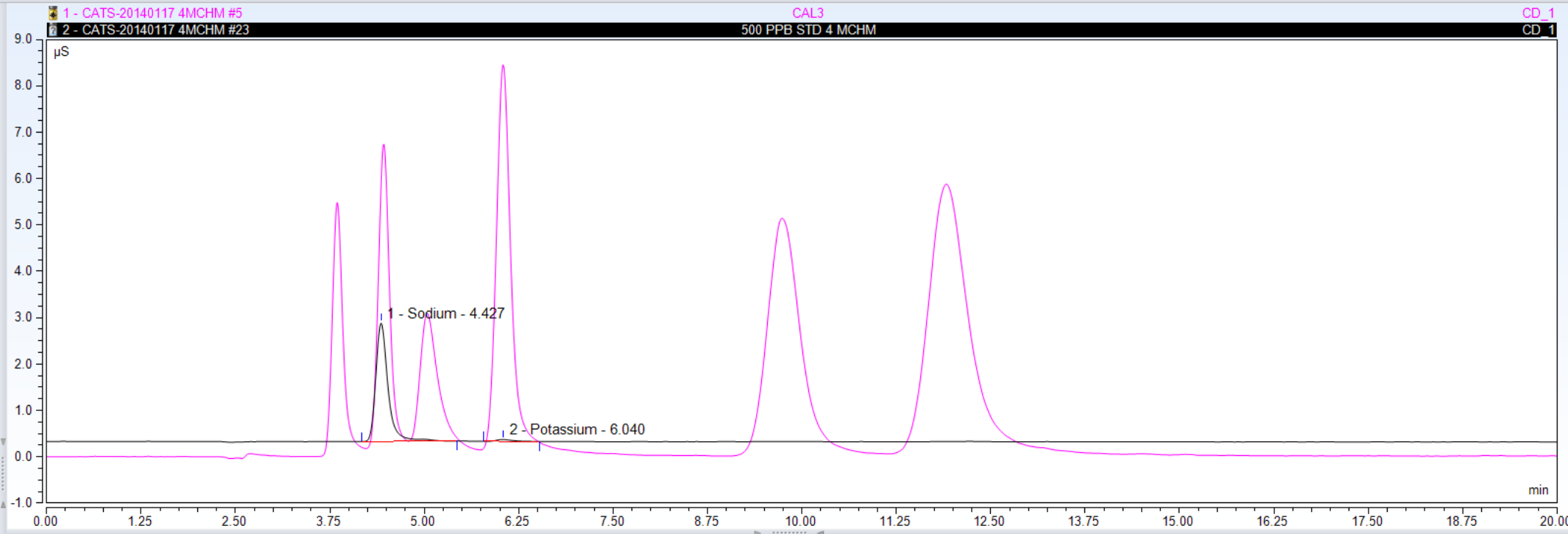
And because I could..

- I screened the crude 4-MHCM on the Ion Chromatograph (IC)
  - The IC is designed to look for inorganic, water soluble cations and anions in a mixture

Data Processing

- Injections
  - INST BLANK
  - MQ
  - CAL1
  - CAL2
  - CAL3
  - CAL4
  - CAL5
  - CAL6
  - CCV-C1 50UL/100ML
  - MQ
  - INST BLANK
  - MQ
  - CAL1
  - MQ
  - INST BLANK
  - MQ
  - CCV-C1
  - INST. BLANK
  - MQ
  - 300 PPB STD 4MCHM CRUDE
  - 300 PPB STD 4MCHM CRUDE
  - MQ
  - 500 PPB STD 4 MCHM
  - MQ
  - SHUTDOWN
- Channels
  - CD_1
  - CD_1_Total
  - Pump_2_Pressure
- Processing Methods
  - AS19 procesing
  - cations

Injection List
Instrument Method
Data Processing
Report Designer
Electronic Report
Spectral Library



	A	B	C	D	E	F	G	H	I	J	K	L
1	Peak No.	Peak Name	Ret.Time min	Amount	Rel.Area %	Area µS*min	Height µS	Type	Width (50%) min	Asym. EP	Resol. EP	Plates EP
4	1	Sodium	4.427	1.3963	97.92	0.4830	2.55	BMB	0.161	1.39	5.00	4188
5	2	Potassium	6.040	0.0384	2.08	0.0103	0.04	BMB	0.219	1.59	n.a.	4197
6	Maximum			1.3963	97.92	0.4830	2.55		0.219	1.59	5.00	4197
7	Minimum			0.0384	2.08	0.0103	0.04		0.161	1.39	5.00	4188
8	Sum			1.4347	100.00	0.4933	2.59					

Summary | Peak Results | Peak Purity | Library Search | System Suitability Test | Calibration | Audit Trail

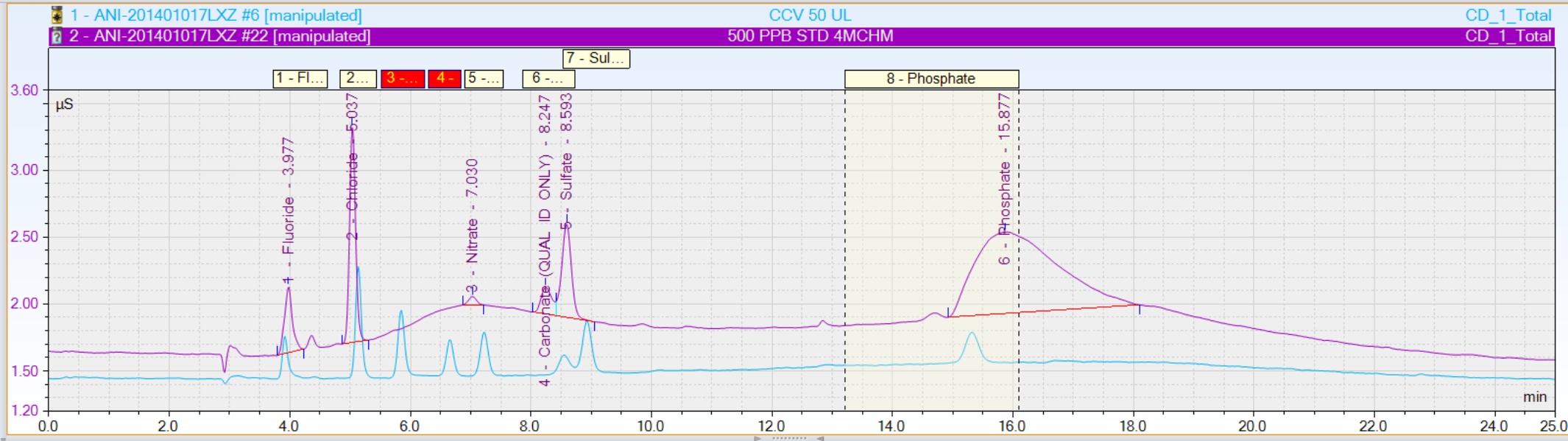
Data Processing

- CCV 7.5 UL
- CCV 15 UL
- CCV 25 UL
- CCV 50 UL
- CCV 2.5ML/25ML
- CCV 1.5ML/5ML
- CCV 3ML/5ML
- CCV 4.5ML/5ML
- CCV FULL STR
- MQ
- MQ
- INST. BLANK
- SHUTDOWN
- INST. BLANK
- MQ
- CCV-A
- MQ
- 300 PPB STD 4MCHM CRUDE
- MQ
- 500 PPB STD 4MCHM
- MQ
- INST. BLANK
- SHUTDOWN
- INST. BLANK
- MQ
- CCV-A1
- MQ
- 500 PPB STD
- MQ
- SHUTDOWN

Channels

- CD_1
- CD_1_Total
- Pump_1_Pressure

- Injection List
- Instrument Method
- Data Processing
- Report Designer
- Electronic Report
- Spectral Library



	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
1	Peak No.	Peak Name	Ret.Time min	Amount ug/mL	Rel.Area %	Area µS*min	Height µS	Type	Width (50%) min	Asym. EP	Resol. EP	Plates EP				
3																
4	1	Fluoride	3.977	0.1098	5.30	0.0753	0.49	BMB	0.134	1.14	5.20	4905				
5	2	Chloride	5.037	0.3750	13.18	0.1872	1.61	BMB	0.107	1.12	9.30	12297				
6	3	Nitrate	7.030	0.0334	0.66	0.0094	0.06	BMB*	0.146	0.99	n.a.	12837				
7	4	Carbonate (QUAL ID ONL	8.247	0.3998	2.97	0.0421	0.20	BM *	n.a.	n.a.	n.a.	n.a.				
8	5	Sulfate	8.593	0.3573	9.22	0.1310	0.70	MB*	0.170	n.a.	4.96	14138				
9	*6*	*Phosphate*	*15.877*	*6.0439*	*68.67*	*0.9750*	*0.61*	*BMB**	*1.564*	*1.66*	*n.a.*	*571*				
10	Maximum			6.0439	68.67	0.9750	1.61		1.564	1.66	9.30	14138				
11	Minimum			0.0334	0.66	0.0094	0.06		0.107	0.99	4.96	571				
12	Sum			7.3192	100.00	1.4199	3.67									
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Summary | Peak Results | Peak Purity | Library Search | System Suitability Test | Calibration | Audit Trail

What Renovation has done

- Broader analytical capability
  - NIST library Confirmatory ID, even in screening mode, allows for qualitative identification of hundreds of chemicals. NIST v.11 contains over 212,961 unique chemicals.
  - Increase throughput-automated samplers allowed samples to be preloaded into carousel; instrument has been run continuously at Huntington (and is still being run).
  - The sample design system on the CMS5000 allows for continuous ambient monitoring on a day to day basis, even though some sensitivity may be lost. We are looking at ways to improve sensitivity.
  - Sample pretreatment includes ability to heat samples, which allows for greater efficiency in stripping volatile organics from the water into carrier gas phase (this is needed for analysis by GC or GCMS
  - All systems of similar type and kind use the same operating parameters, methods, and column configurations.
  - We have remote access into ODS stations wherever we have a functioning internet connection.

What the spill has done for us...

- Allows us to use this to educate ourselves on how we can better detect and track spills to provide our drinking water utilities with the most comprehensive and accurate information so they can protect their public.