EAST BAY MUNICIPAL UTILITY DISTRICT

Gas Chromatography/Mass Spectrometry (GC/MS) in Wastewater Analysis

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BACWA Laboratory Committee Meeting
April 13, 2016

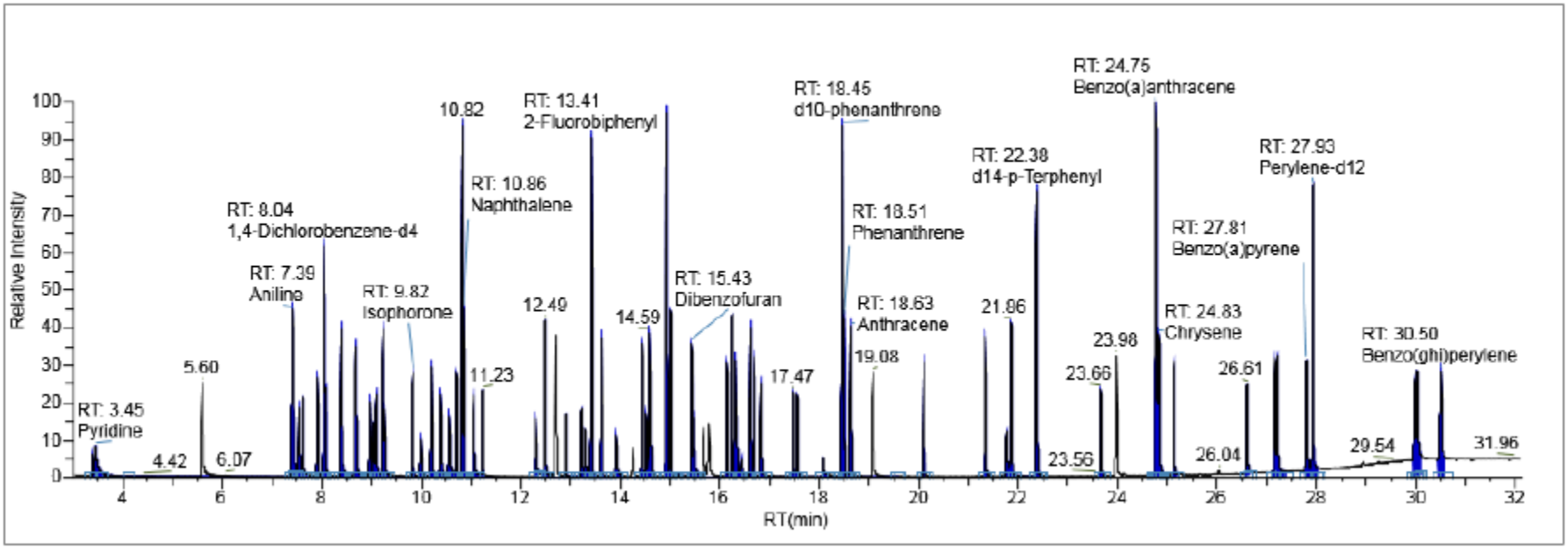
Overview

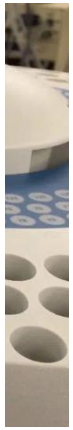
- GC/MS based methods
- Basic principles of GC/MS
- Instrument selection process
- New instrument validation
- Sample preparation techniques
- GC/MS/MS

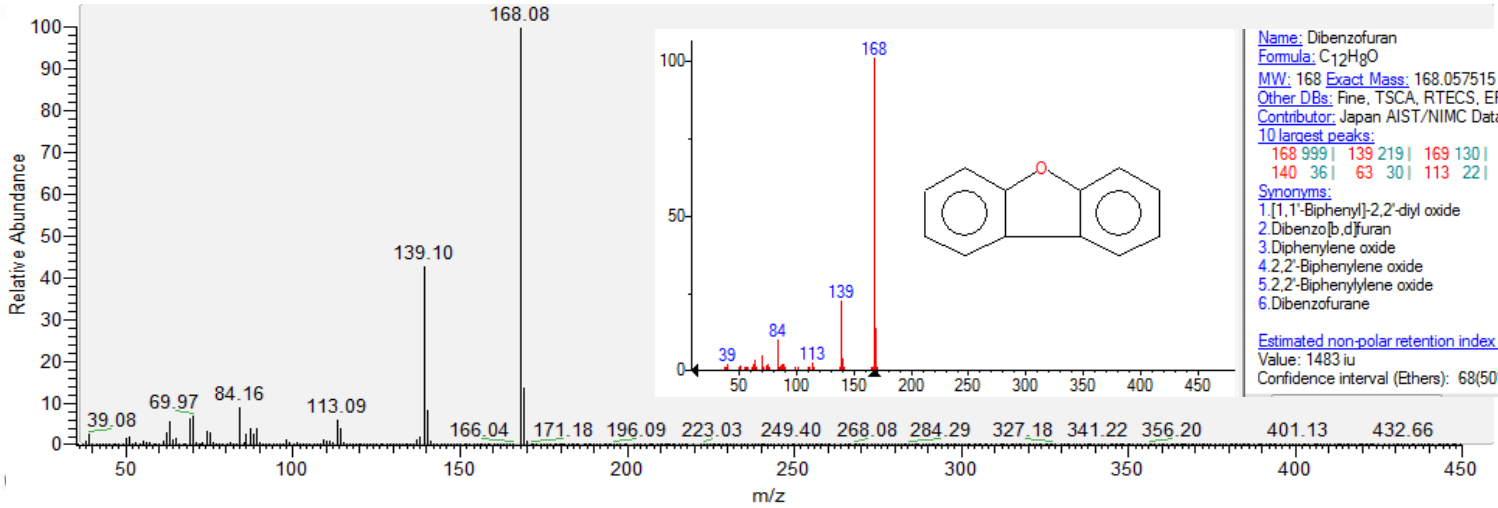
GC/MS Wastewater and Biosolids methods

- EPA 625: “Base/Neutrals and Acids”
- EPA 8270: “Semivolatile Organic Compounds by GC/MS”
- EPA 624: “Purgeables”
- EPA 8260B: “Volatile Organic Compounds by GC/MS”
- EPA 8260B: “Gasoline”
- EPA 8015M: “Diesel And Motor Oil”
- SM6710: “Tributyl Tin”

GC/MS Principle







Name: Dibenzofuran
Formula: $C_{12}H_8O$
MW: 168 Exact Mass: 168.057515 CAS#: 132-64-9 NIST#: 228192 ID#: 138909 DI
Other DBs: Fine, TSCA, RTECS, EPA, HODOC, NIH, EINECS, IRDB
Contributor: Japan AIST/NIMC Database- Spectrum MS-NW- 354
10 largest peaks:
168 999 | 139 219 | 169 130 | 84 94 | 69 44 |
140 36 | 63 30 | 113 22 | 114 22 | 70 21 |
Synonyms:
1.[1,1'-Biphenyl]-2,2'-diyl oxide
2.Dibenzo[b,d]furan
3.Diphenylene oxide
4.2,2'-Biphenylene oxide
5.2,2'-Biphenylylene oxide
6.Dibenzofurane

Estimated non-polar retention index (n-alkane scale):
Value: 1483 iu
Confidence interval (Ethers): 68(50%) 293(95%) iu

GC/MS Instrument Specifications:
Gas Chromatograph

- Temperature programmable split/splitless (SSL) injector
- GC oven operating temperature 3-450°C
- Autosampler capacity – 100 samples
- Programmable flow and pressure controllers
- Helium as carrier gas with capability of converting to Hydrogen

GC/MS Instrument Specifications :
Mass Spectrometer

- Mass range 2-1100 amu in 0.1 amu steps
- Programmable scan range throughout analytical run
- Electron Impact Ionization source (inert)
- Mass scan speed up to 11,000 amu/s
- Ion source temperature up to 350°C
- Simultaneous Selected Ion Monitoring (SIM) and Scan acquisition
- SIM acquisition up to 240 scans/s
- Transfer line temperature up to 400°C
- High capacity turbomolecular vacuum pump (300 L/s)
- Sensitivity requirement: Using a 30m x 0.25mm x 0.25 um 5%-phenyl column in EI Full Scan mode S/N ≥ 600:1 for 1 pg Octafluoronaphthalene (OFN)
- Linear range of at least 7 orders of magnitude
- Must meet Decafluorotriphenylphosphine (DFTPP) tuning criteria per EPA Method 625

GC/MS Instrument Specifications: *Software*

- Comprehensive software package:
  - Instrument control
  - Tuning
  - Data acquisition
  - Data analysis and reporting
- Qualitative and quantitative analysis
- Report files in a format compatible with LIMS (e.g. CSV, XLS, txt, etc.)
- Allow reprocessing of acquired data including use of historical calibration data
- Process data files for unknown compounds using up-to-date searchable NIST library for tentatively Identified compounds (TIC)
- User-defined Quality Control parameters
- Report format must provide sample name, method name, acquisition date and time of injection, sample identifier, file name, etc.
- Software must perform all necessary calculations, such as dilution/weight and Internal standard corrections

Vendors

- Agilent Technologies
- Thermo Scientific
- Perkin Elmer
- Shimadzu

GC/MS Instrument Purchase:
Additional Considerations

- Support: availability of local service engineers and typical lead times
- Application support
- Ease of maintenance: e.g. cleaning ion source without breaking the vacuum
- Second SSL injector and/or PTV injector with Large Volume Injection (LVI) capability
- Cost of annual service contracts beyond standard warranty
- Review manufacturer's application notes and other published materials
- Contacting 2-3 references

New Setup: Thermo ISQ

New Setup: Thermo ISQ

- Two injectors: suitable for water and wastewater
- Large capacity vacuum pump (reduced downtime)
- MS maintenance does not require venting
- Improved sensitivity for PAHs
- New powerful software – steep initial learning curve for staff

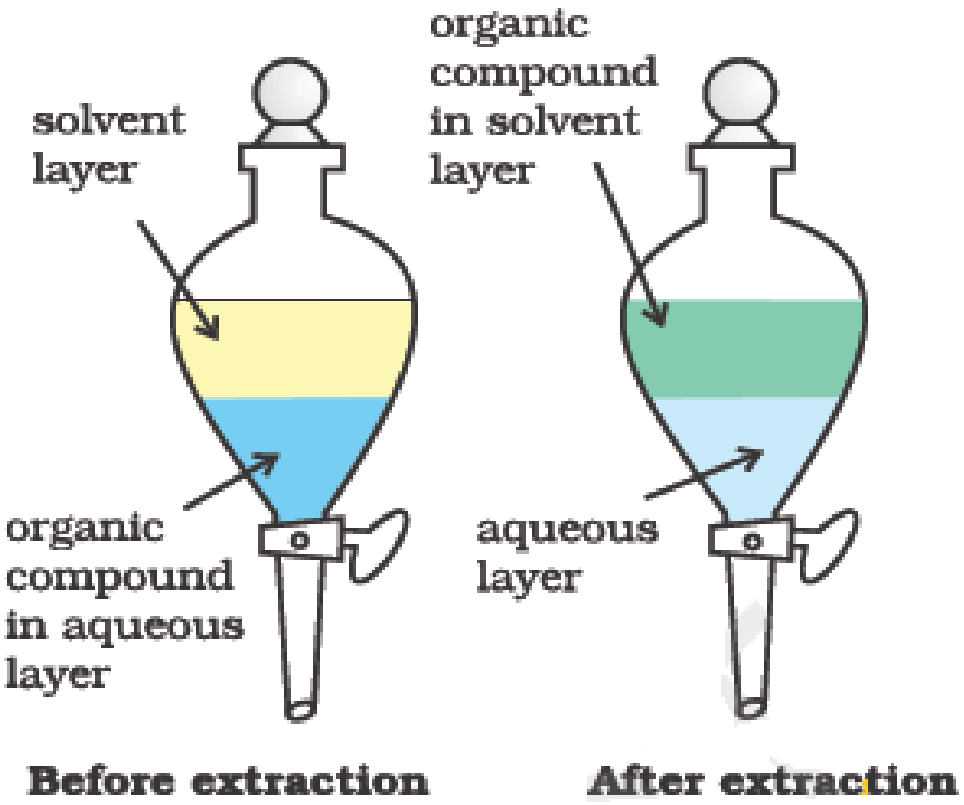
EPA Method 625

- Samples are collected in 1-liter bottles, typically pre-preserved with sodium thiosulfate
- Surrogates are added prior to extraction
- Analytes are serially extracted by Liquid-Liquid Extraction (LLE) at pH>11 and pH<2
- Extracts are dried with sodium sulfate and concentrated under stream of N_2 (RapidVap)
- Internal standards are added prior to analysis
- Samples are analyzed by GC/MS

Typical Extraction Techniques

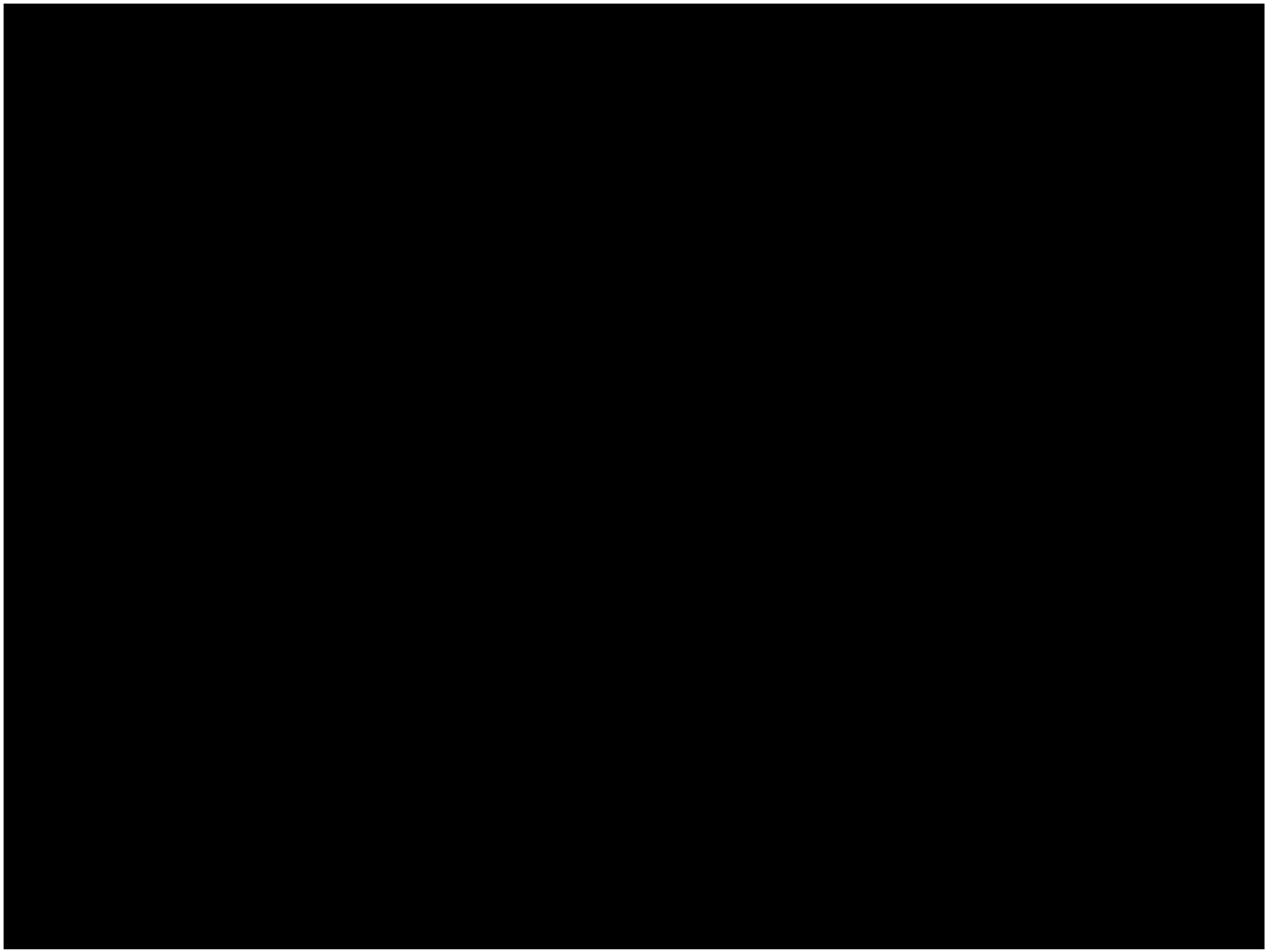
- Liquid-Liquid Extraction (LLE)
- Solid Phase Extraction (SPE)
- Pressurized Extraction (ASE)

Liquid-Liquid Extraction

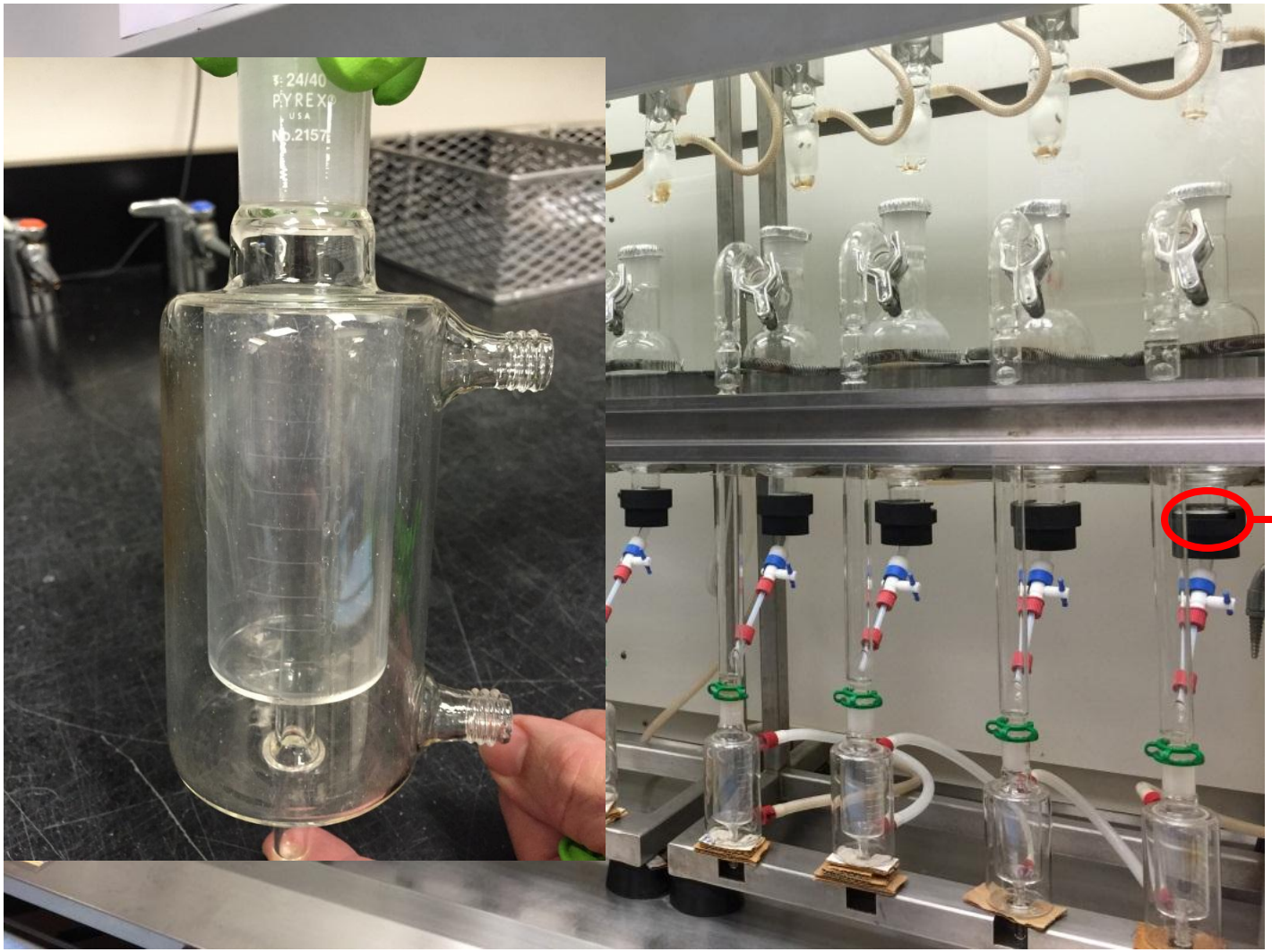


Wikipedia

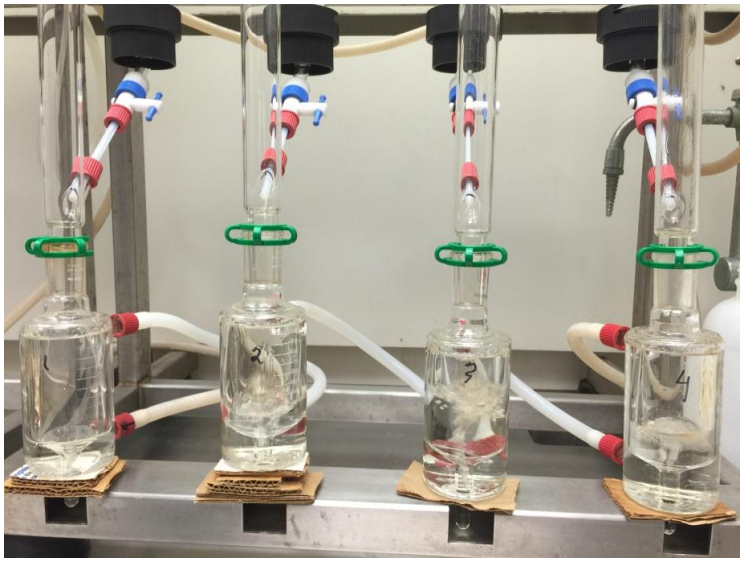
Continuous LLE “One-Step”

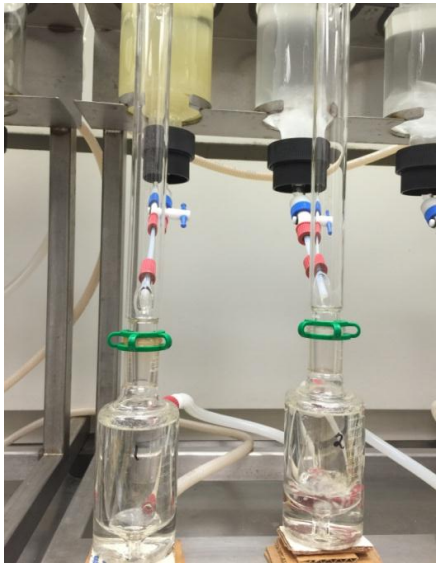


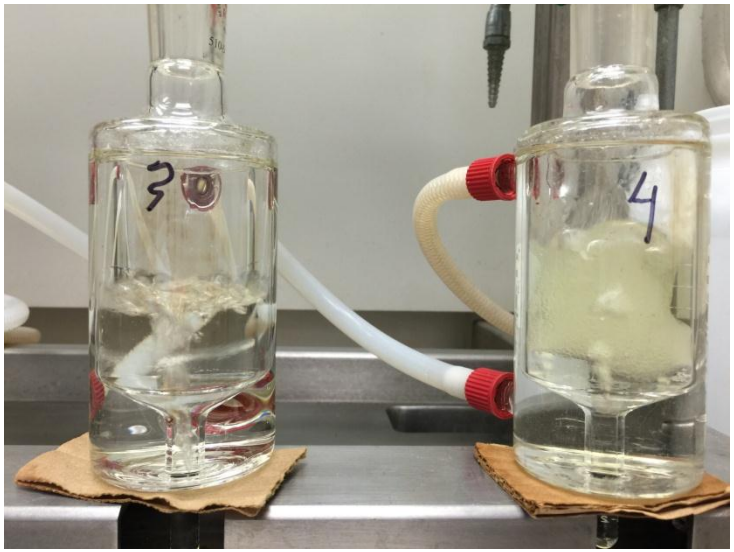
Continuous LLE "One-Step"

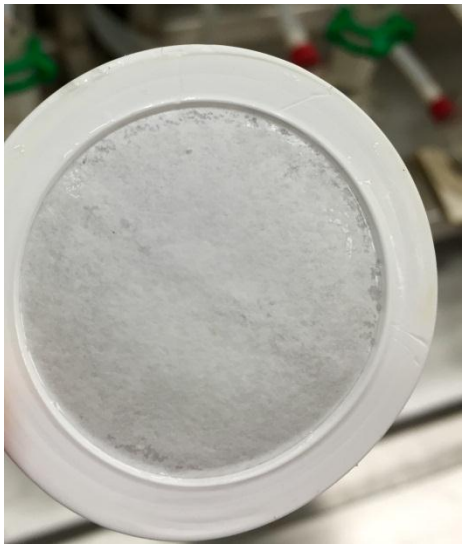


LLE One-Step









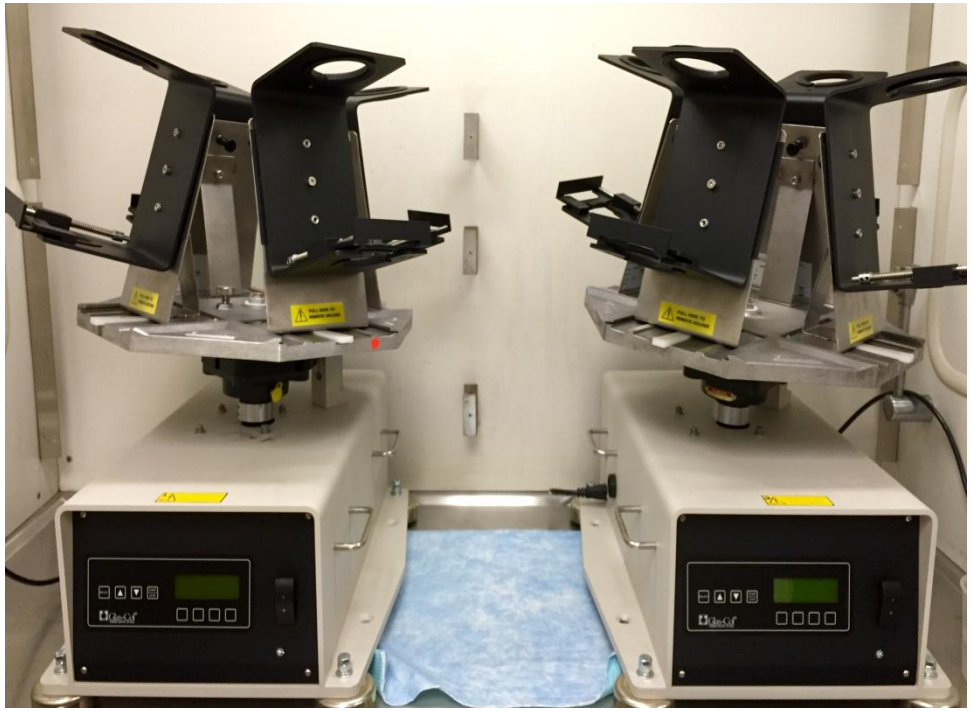
Glas-Col Shaker

Glas-Col Shaker

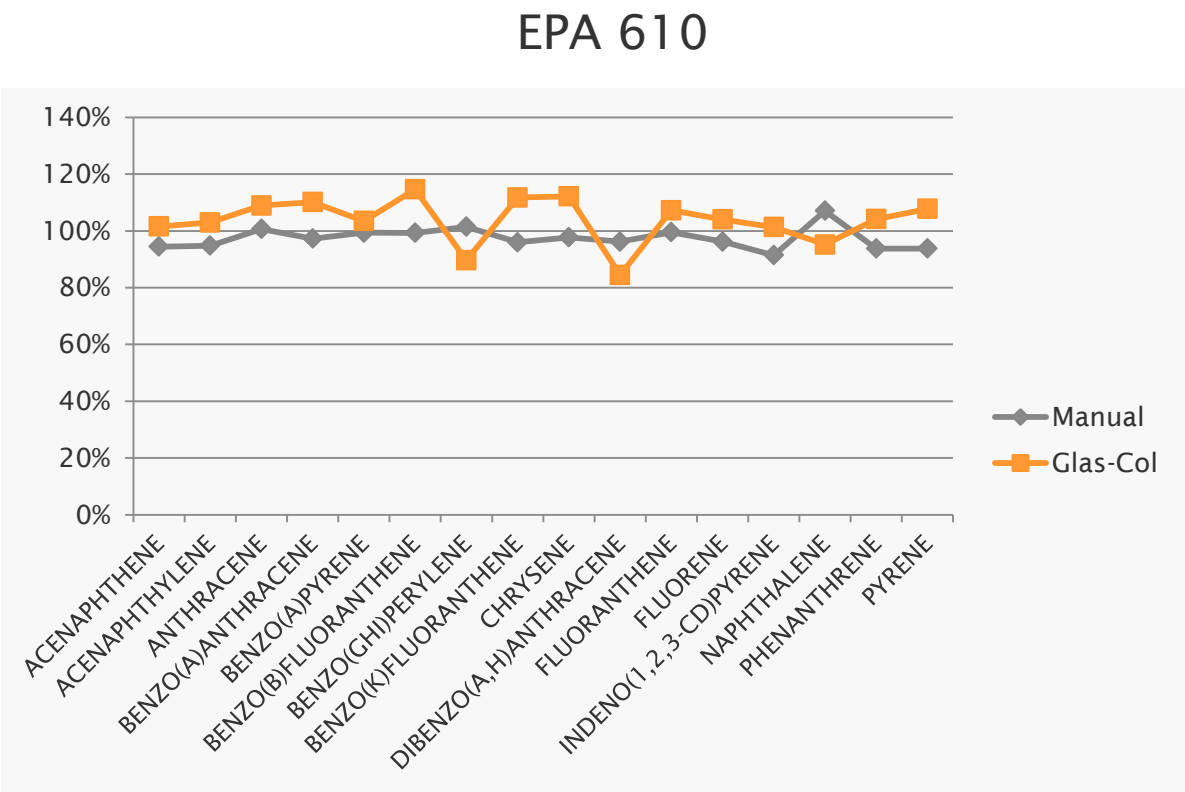
- 8 samples simultaneously
- Variable speed and duration
- Programmable methods

Optimization parameters:

- Solvents
- RPM
- Shakeout time

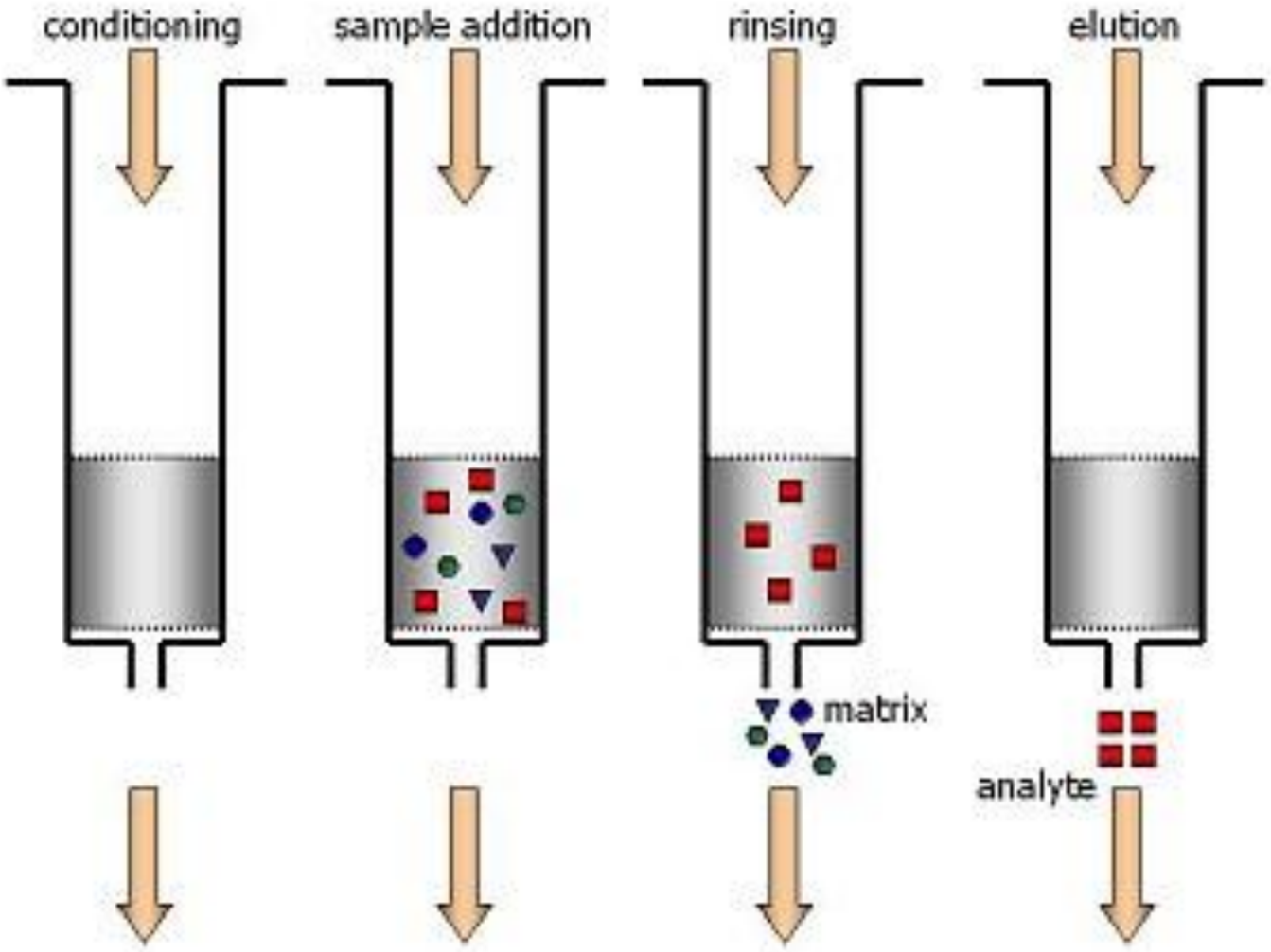


Manual Shakeout vs. Glas-Col



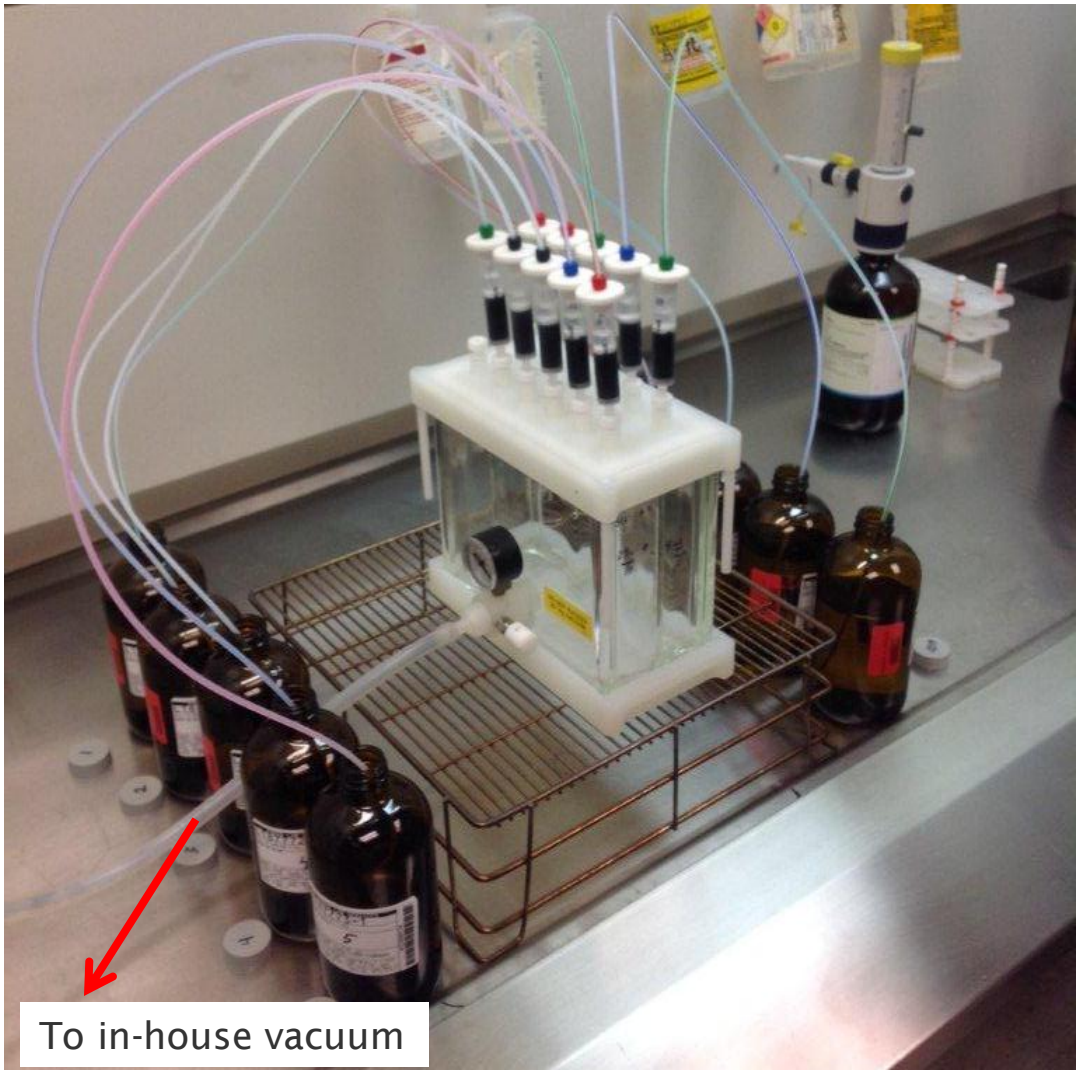
NAPHTHALENE	107%	2%		95%	2%
PHENANTHRENE	94%	2%		104%	2%
PYRENE	94%	2%		108%	1%
AVERAGE	97%			104%	

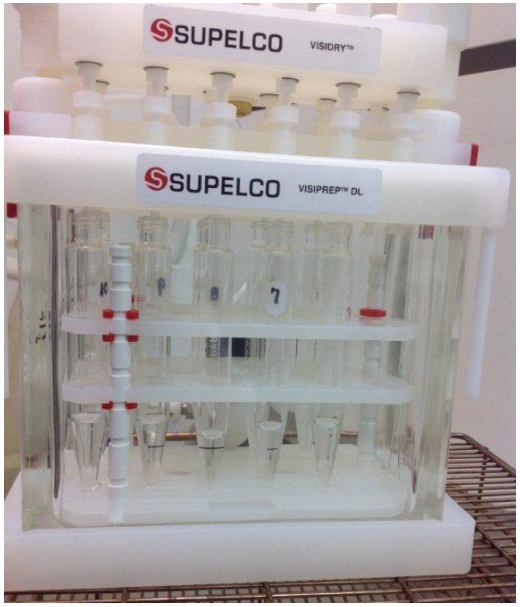
Solid Phase Extraction (SPE)



Guide to Solid Phase Extraction, Supelco Bulletin 910 (1998) 1-12.

Manifold





Horizon SPE-DEX 4790

- 4 samples simultaneously
- Steps programmed via controller

  Optimization parameters:

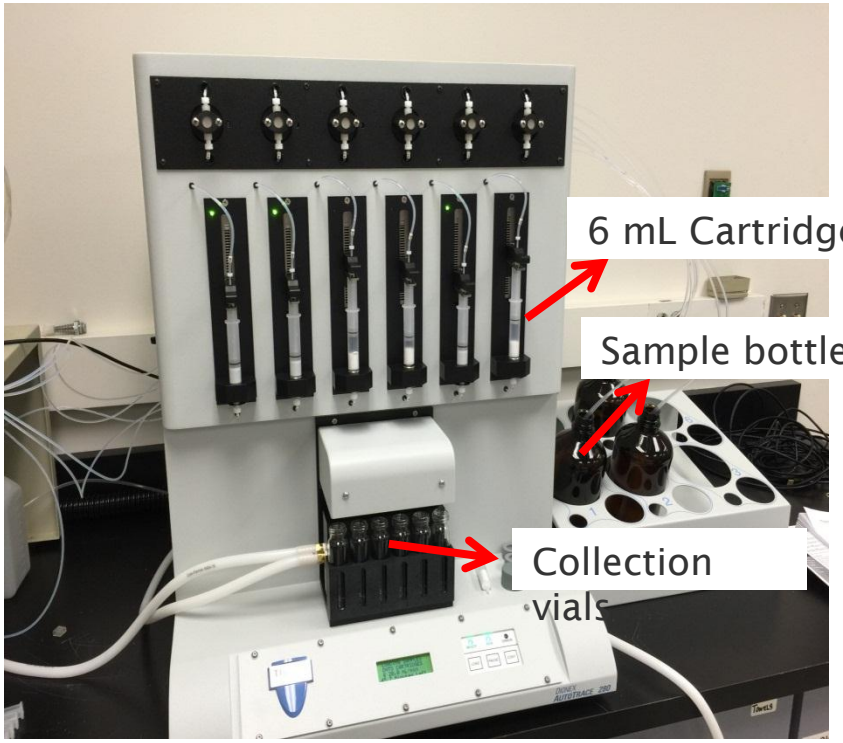
  - Solvents
  - Sorbent type
  - Pressure
  - Air Dry Time
  - Soak time
  - Conditioning time
  - Elution time

AutoTrace 280

- Purchased in 2014
- Suitable for drinking water
- Steps programmed via software
- 50% increase in capacity

  (6 samples vs. 4)
- Reduced solvent use

  (6 mL cartridge capacity vs. 86 mL/disks)
- Less moisture = consistent recovery



Pressurized Solvent Extraction (ASE)

Soil

Sludge

Biosolids

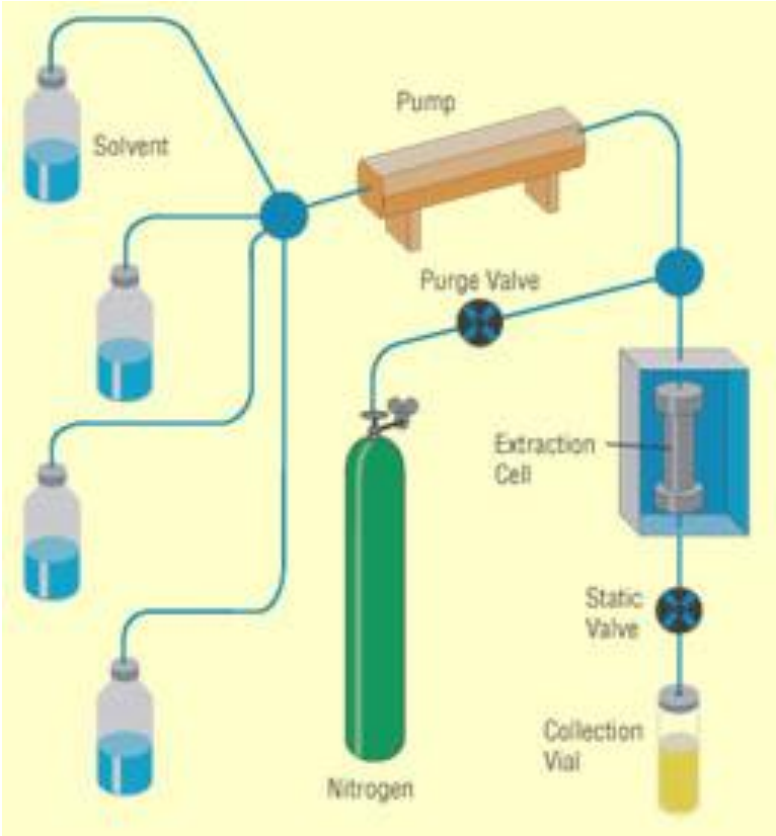
Truck waste

EPA 8270 (GC/MS)

EPA 8081 (GC/ECD)

Optimization parameters:

- Solvents
- Number of cycles
- Temperature
- Sample amount



Instrument Method Optimization

GC:

- GC oven ramp
- Inlet temperature
- injection volume
- GC column
- Inlet liner
- Split or Splitless injection
- Tailing factor: Benzidine < 3.0
  Pentachlorophenol < 5.0

MS:

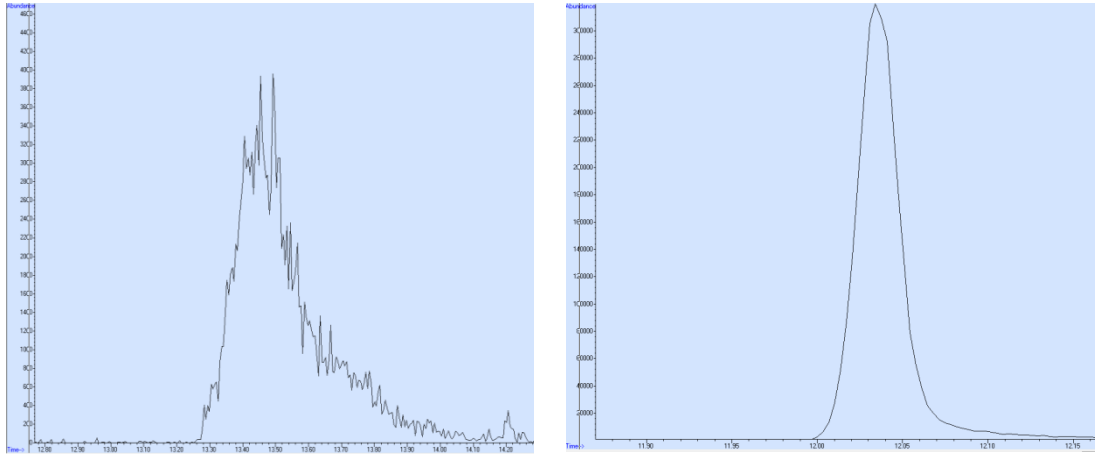
- Emission current
- Detector gain
- Ion Source temperature
- Must meet EPA 625 tuning (DFTPP) criteria
- Must meet EPA 625 calibration criteria (RSD ≤ 35%)

Software:

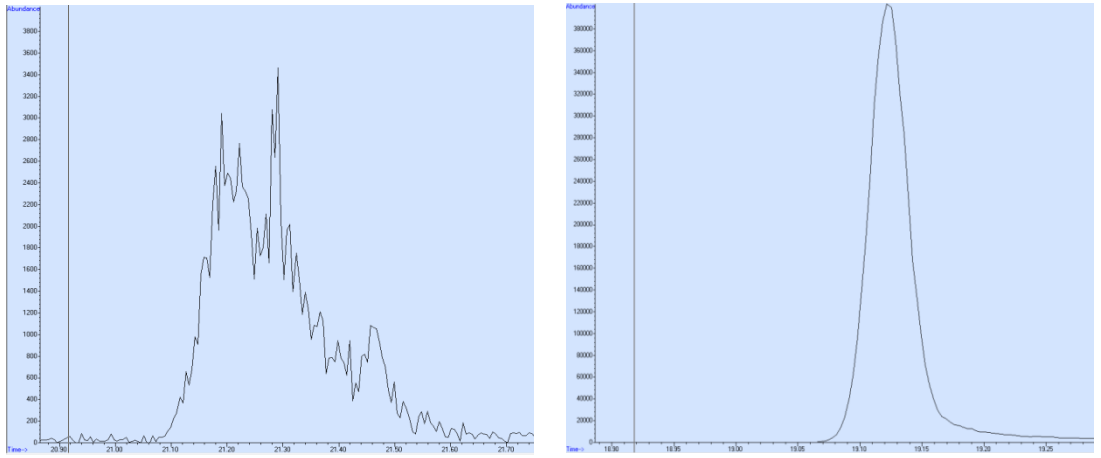
- Verify software calculations
- Customize reports

GC/MS Method Optimization

GC: Siltek-deactivated sample pathway (liner, seal, column) show increased resistivity to acidic extracts resulting in durable chromatography for pentachlorophenol and chlorobenzilate

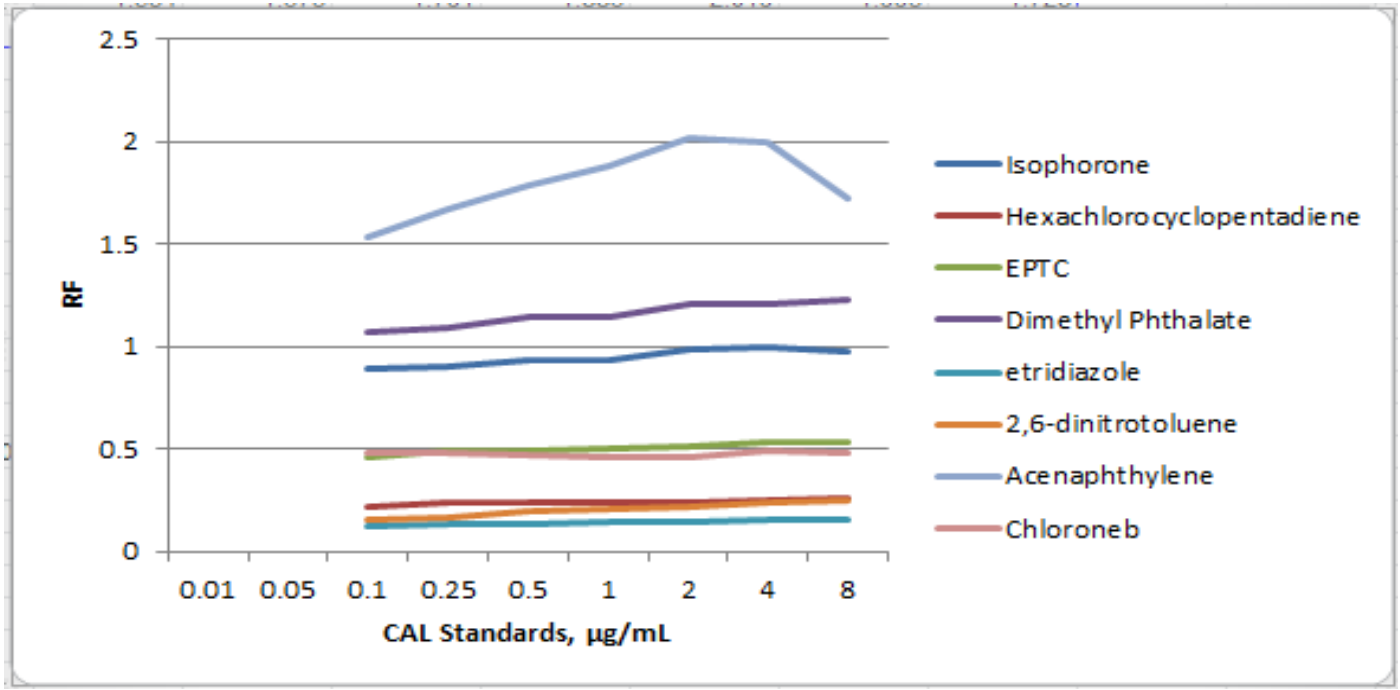


Pentachlorophenol (Spiked Blank)



Chlorobenzilate (Spiked Blank)

GC/MS Method Optimization



- MS parameters provided by the vendor produced limited linear range for a number of target analytes in EPA 525.2
- Calibration range and MS emission current must be optimized for linearity and sensitivity

DFTPP tuning

Table 9—DFTPP Key Masses and Abundance Criteria

Mass	m/z Abundance criteria
51	30-60 percent of Mass 198.
68	Less than 2 percent of Mass 69.
70	Less than 2 percent of Mass 69.
127	40-60 percent of Mass 198.
197	Less than 1 percent of Mass 198.
198	Base peak, 100 percent relative abundance.
199	5-9 percent of Mass 198.
275	10-30 percent of Mass 198.
365	Greater than 1 percent of Mass 198.
441	Present but less than Mass 443.
442	Greater than 40 percent of Mass 198.
443	17-23 percent of Mass 442.

New Instrument Validation Challenges

- Instrument range: 67 analytes in the method – varied GC/MS response
- GC setup: Splitless injection results in enhanced Internal standard recovery phenomenon. Split injection provides more consistent result when Methylene Chloride is used as a solvent
- MS setup: Linear range required adjustment to emission current settings
- Software:
  - Factory report format required customization
  - New Software - learning curve for staff

Table 6—QC Acceptance Criteria—Method 625

Parameter	Test conclusion (µg/L)	Limits for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (Percent)
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4,-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dhlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26-155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120

IDOC

EBMUD

Parameter	Unit	Spike	% Rec #1	% Rec # 2	% Rec #3	% Rec # 4	% Rec Mean	Acceptable Range[a]		STD % Rec	%RSD	Max RSD[b]
Surrogates												
2-Fluorobiphenyl	ug/L	20	86%	83%	95%	84%	87%	42	109	5%	6%	30.0%
2,4,6-Tribromophenol	ug/L	20	100%	100%	100%	94%	99%	54	113	3%	3%	30.0%
d14-p-Terphenyl	ug/L	20	99%	110%	100%	100%	102%	72	149	5%	5%	30.0%
Target Analytes												
ACENAPHTHENE	ug/L	10	86%	83%	80%	85%	83%	60.1	132.3	3%	3%	27.6%
ACENAPHTHYLENE	ug/L	10	87%	84%	83%	86%	85%	53.5	126	2%	2%	40.2%
ANILINE	ug/L	10	70%	64%	67%	69%	68%	46	134	3%	4%	94.0%
ANTHRACENE	ug/L	10	89%	89%	87%	89%	88%	43.4	118	1%	1%	32.0%
AZOBENZENE	ug/L	10	93%	93%	89%	93%	92%	28	132	2%	2%	87.0%
BENZIDINE	ug/L	10	67%	71%	50%	63%	63%	D	200	9%	15%	200.0%
BENZO(A)ANTHRACENE	ug/L	10	98%	100%	100%	99%	99%	41.8	133	1%	1%	27.6%
BENZO(B)FLUORANTHENE	ug/L	10	140%	140%	140%	140%	140%	42	140.4	0%	0%	38.8%
BENZO(K)FLUORANTHENE	ug/L	10	120%	120%	110%	115%	116%	25.2	145.7	5%	4%	32.3%
BENZOIC ACID	ug/L	10	69%	53%	76%	65%	66%	4	165	10%	15%	192.0%
BENZO(GHI)PERYLENE	ug/L	10	140%	130%	140%	135%	136%	D	195	5%	4%	58.9%
BENZO(A)PYRENE	ug/L	10	140%	140%	130%	138%	137%	31.7	148	5%	3%	39.0%
BENZYLALCOHOL	ug/L	10	85%	81%	75%	84%	81%	54	150	5%	6%	58.0%
BENZYLBUTYLPHTHALATE	ug/L	10	100%	110%	120%	110%	110%	D	139	8%	7%	23.4%
BIS(2-CHLOROETHOXY)METHANE	ug/L	10	82%	76%	73%	80%	78%	49.2	164.7	4%	5%	34.5%
BIS(2-CHLOROETHYL)ETHER	ug/L	10	78%	75%	75%	82%	78%	42.9	126	3%	4%	55.0%
BIS(2-CHLOROISOPROPYL)ETHER	ug/L	10	85%	78%	73%	83%	80%	62.8	138.6	5%	7%	46.3%
BIS(2-ETHYLHEXYL)PHTHALATE	ug/L	10	96%	100%	100%	102%	99%	28.9	136.8	2%	2%	41.1%
4-BROMOPHENYL PHENYL ETHER	ug/L	10	99%	99%	92%	98%	97%	64.9	114.4	3%	3%	23.0%
4-CHLOROANILINE	ug/L	10	77%	77%	71%	75%	75%	32	98	3%	4%	52.0%
P-CHLORO-M-CRESOL	ug/L	10	97%	96%	93%	97%	96%	40.8	127.9	2%	2%	37.2%
2-CHLORONAPHTHALENE	ug/L	10	100%	98%	99%	102%	100%	64.5	113.5	2%	2%	13.0%
2-CHLOROPHENOL	ug/L	10	72%	67%	64%	72%	69%	36.2	120.4	4%	6%	28.7%
4-CHLOROPHENYL PHENYL ETHER	ug/L	10	79%	76%	73%	77%	76%	38.4	144.7	2%	3%	33.4%
CHRYSENE	ug/L	10	98%	110%	100%	102%	103%	44.1	139.9	5%	5%	48.3%
DIBENZO(A,H)ANTHRACENE	ug/L	10	140%	140%	140%	140%	140%	D	199.7	0%	0%	70.0%
DIBENZOFURAN	ug/L	10	92%	85%	86%	89%	88%	38	127	3%	4%	63.0%

16.1 The method detection [illegible] centration of a substance that can be r[illegible] that the value is above zero.[1] The MDL [illegible]e obtained using reagent water.[13] The M[illegible] ill vary depending on instrument sensitivi[illegible]

- MDL Study is comp[illegible]ndix B Part 136
- Extract and analyze[illegible] samples (LCS)
- MDL results must b[illegible]S concentration)
- Must satisfy NPDES [illegible]
- Four (4) concentrat[illegible]ed to complete MDL Study

1,2,4-TRICHLOROBENZENE
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE
2,6-DINITROTOLUENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-CRESOL
2-METHYLNAPHTHALENE
2-NITROANILINE
2-NITROPHENOL
3,3'-DICHLOROBENZIDINE
3-,4-METHYLPHENOL
3-NITROANILINE
4,6-DINITRO-O-CRESOL
4-BROMOPHENYL PHENYL ETHER
4-CHLOROANILINE
4-CHLOROPHENYL PHENYL ETHER
4-NITROANILINE
4-NITROPHENOL
ACENAPHTHENE
ACENAPHTHYLENE
ANILINE
ANTHRACENE
AZOBENZENE
BENZIDINE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(GHI)PERYLENE
BENZO(K)FLUORANTHENE
BENZOIC ACID
BENZYL ALCOHOL
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-CHLOROISOPROPYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
BUTYLBENZYL PHTHALATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIBENZO(A,H)ANTHRACENE
DIBENZOFURAN
DIETHYL PHTHALATE
DIMETHYL PHTHALATE
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISOPHORONE
N-NITROSODI-N-PROPYLAMINE
N-NITROSODIMETHYLAMINE
N-NITROSODIPHENYLAMINE
NAPHTHALENE
NITROBENZENE
P-CHLORO-M-CRESOL
PENTACHLOROPHENOL
PHENANTHRENE
PHENOL
PYRENE
PYRIDINE

MDL Study

- EBMUD's EPA 625 MDL range: 0.0063-3.5 µg/L
  - Reporting limits range: 1-5 µg/L
- EBMUD's Low-level PAHs MDL range: 0.015 – 0.071 µg/L
  - Reporting limits range: 0.05 – 10 µg/L

Low-Level PAHs

Parameter	Unit	LIMS HPLC MDL	SIP LC ML	New 625 MDL	SIP GC/MS ML	GO*
ACENAPHTHENE	ug/L	0.052	0.5	0.015	1	2700
ACENAPHTHYLENE	ug/L	0.039	0.2	0.015	10	N/A
ANTHRACENE	ug/L	0.064	2	0.010	10	110,000
BENZO(A)ANTHRACENE	ug/L	0.02	N/A	0.0076	5	0.049
BENZO(A)PYRENE	ug/L	0.044	2	0.011	10	0.049
BENZO(B)FLUORANTHENE	ug/L	0.031	10	0.0090	10	0.049
BENZO(GHI)PERYLENE	ug/L	0.023	0.1	0.0078	5	N/A
BENZO(K)FLUORANTHENE	ug/L	0.015	2	0.0063	10	0.049
CHRYSENE	ug/L	0.017	5	0.0080	10	0.049
DIBENZO(A,H)ANTHRACENE	ug/L	0.016	0.1	0.0090	10	0.049
FLUORANTHENE	ug/L	0.027	0.05	0.012	1	370
FLUORENE	ug/L	0.049	0.1	0.015	10	14000
INDENO(1,2,3-CD)PYRENE	ug/L	0.016	0.05	0.0084	10	0.049
NAPHTHALENE	ug/L	0.071	0.2	0.017	1	N/A
PHENANTHRENE	ug/L	0.042	0.05	0.014	5	N/A
PYRENE	ug/L	0.029	0.05	0.010	10	11000

** Table F-6 NPDES Permit*

EPA 625 Validation Summary

- IDOC meets EPA 625 criteria
- New MDLs < EPA 625 published MDLs, and typically < our previous MDLs
- MDLs < reporting limits for all analytes
- MDLs satisfy NPDES governing criteria (Table F-6)
- MDLs for PAHs meet reporting limits for low level PAHs in Full Scan Mode and generally lower than EPA 610 MDLs
- Streamlined sample preparation and analysis: 2-in-one EPA Methods (625/610) with single calibration/single extraction/single sampling/single analysis
- Default factory report templates were customized
- Existing LIMS upload script was modified for the new software

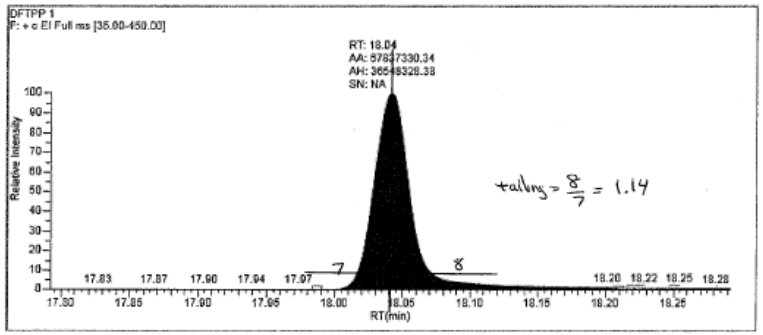
EPA 625 Data Packet

- DFTPP Tuning (beginning of the run)
- Tailing factors (Benzidine < 3.0, Pentachlorophenol < 5.0 – beginning of the run)
- Calibration (Average RF, RSD ≤ 35%; Linear or Quadratic)
- Continuing calibration verification ± 20% recovery: Beginning of the run, Every 12 hours and end of the run
- Method Blank : Target analytes < MDLs
- Spiked Blank: Recoveries within control limits (Table 6, EPA Method)
- Surrogates: Recoveries within control limits (Table 6, EPA Method)
- MS/MSD: Recoveries within control limits (Table 6, EPA Method)
- Internal Standard: 50-200% recovery

EPA 625 Data Packet

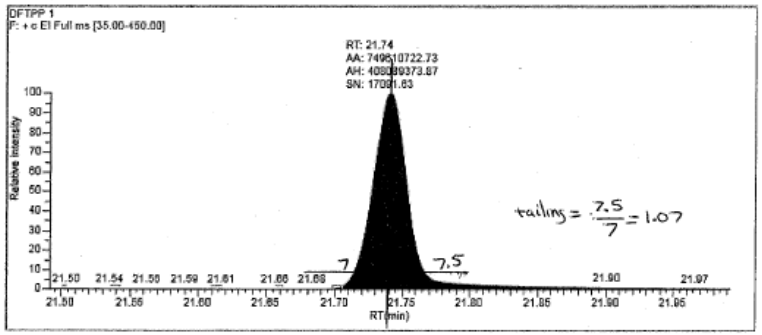
Tailing Report

Last Acquisition Date	:	3/15/2016 12:11
Vial Position	:	1
Batch Folder Path	:	C:\TraceFinderData\32\Projects\625\2016\625 2016Mar14
User Name	:	tc
Instrument Name	:	Thermo Scientific Instrument



Compunnd Name : Pentachlorophenol

Sample Raw File Name : DFTPP 1



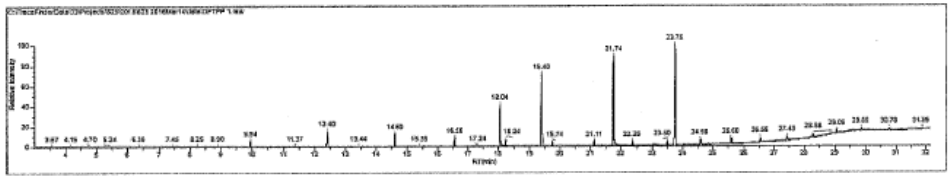
Compunnd Name : Benzidine

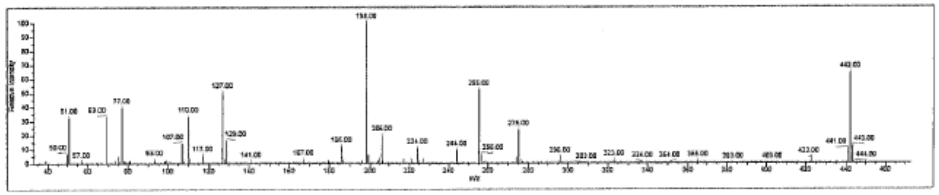
Sample Raw File Name : DFTPP 1

Tune Report

Lab Name:	EBMUD
Instrument:	Thermo Scientific Instrument
User:	tc
Batch:	625 2016Mar14
EPA Method:	8270C
Data File:	C:\TraceFinderData\32\Projects\625\2016\625 2016Mar14\Data\DFTPP 1.raw
Acquisition Date:	3/14/2016 2:42:22 PM
Mass Spectrum Data:	Scan#: 9458 - 9460 RT: 19.40 - 19.41 SB: Scan 9434 @ 19.36 min

Method: Hexachlorobenzene625_031416sip



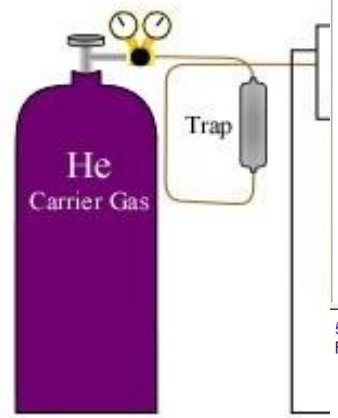


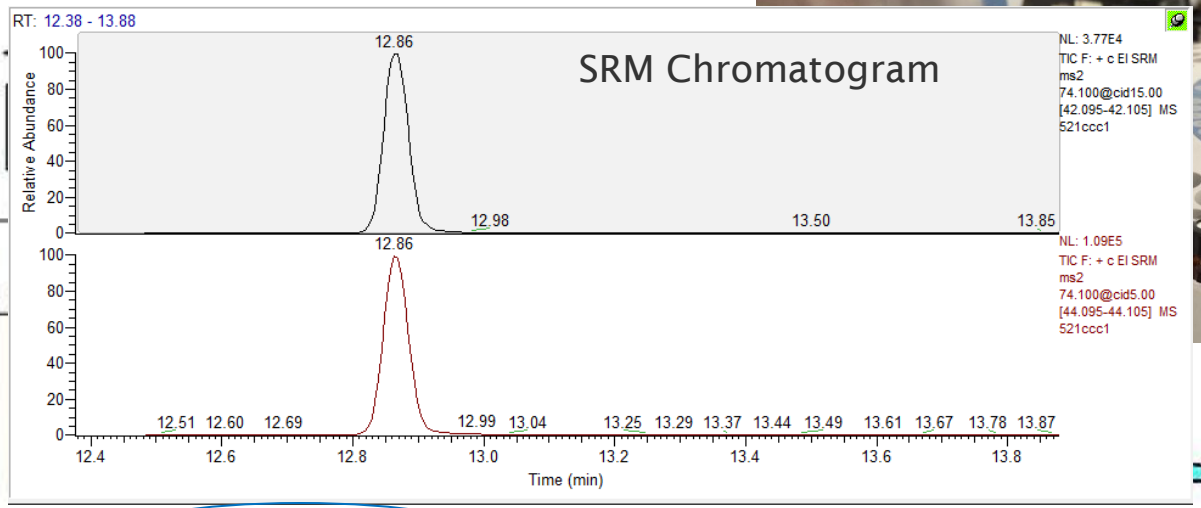
m/z	Ion Abundance Criteria	% Relative Abundance	Pass/Fail
51	greater than or equal to 30% AND less than or equal to 60% of m/z 198	33.3	Pass
68	less than 2% of m/z 69	0	Pass
70	less than 2% of m/z 69	0.5	Pass
127	greater than or equal to 40% AND less than or equal to 60% of m/z 198	50.8	Pass
197	less than 1% of m/z 198	0	Pass
198	equals 100% of Base Peak	100	Pass
199	greater than or equal to 5% AND less than or equal to 9% of m/z 198	6.5	Pass
275	greater than or equal to 10% AND less than or equal to 30% of m/z 198	24.1	Pass
365	greater than 1% of m/z 198	1.6	Pass
441	greater than 0% AND less than 100% of m/z 443	85.8	Pass
442	greater than 40% of m/z 198	64.4	Pass
443	greater than or equal to 17% AND less than or equal to 23% of m/z 442	19.4	Pass

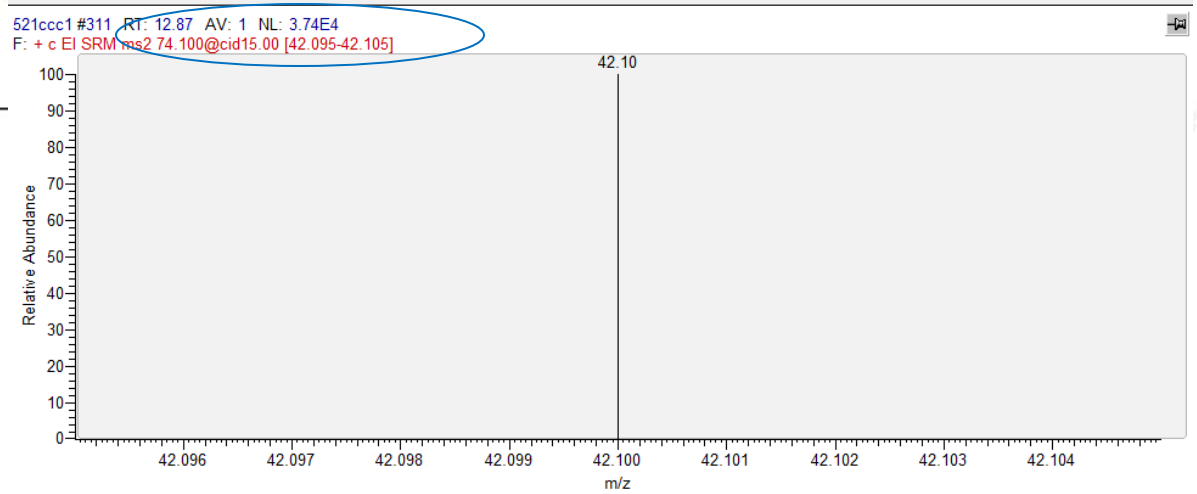
Determined base peak: 198 m/z

8270C passed

Triple Quad GC/MS/MS

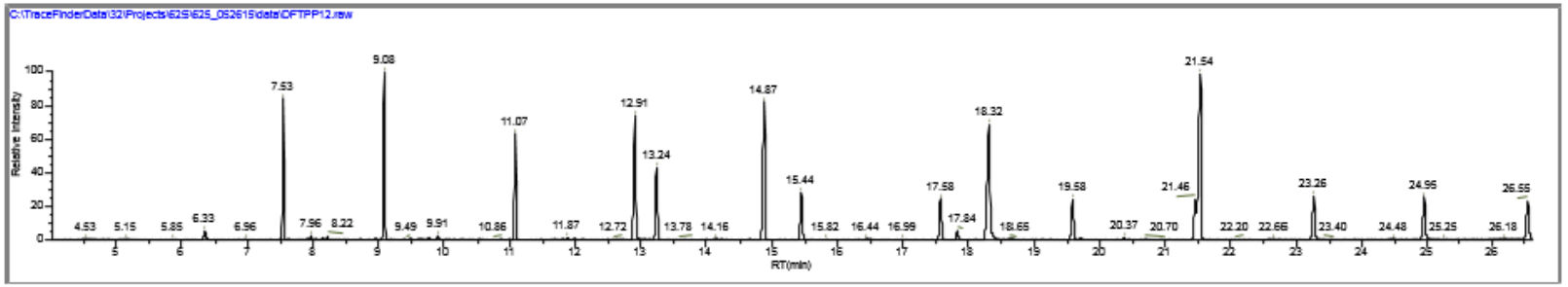


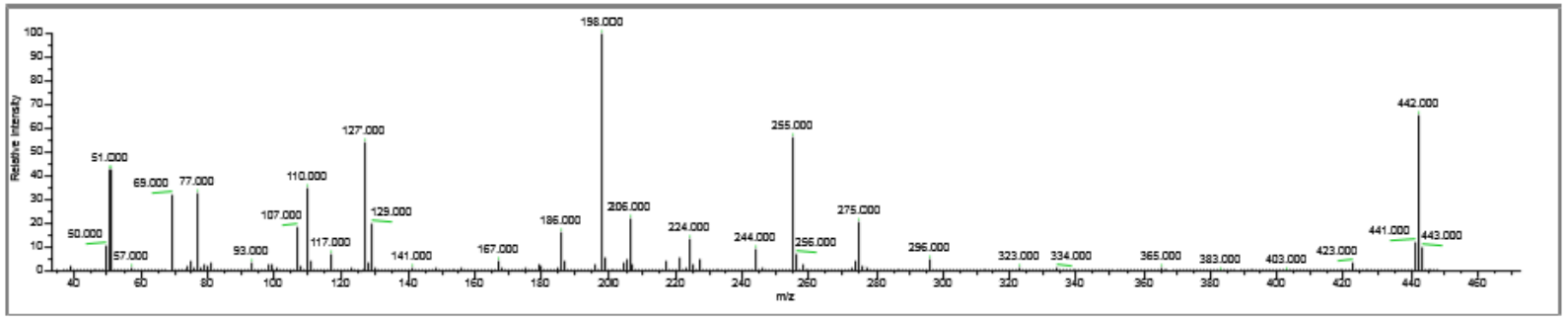




GC/MS/MS

TSQ DFTPP Tune

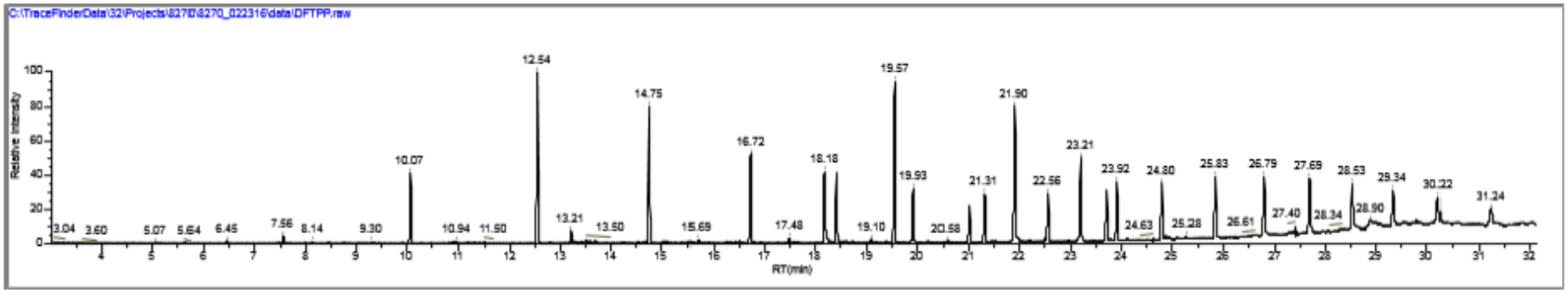


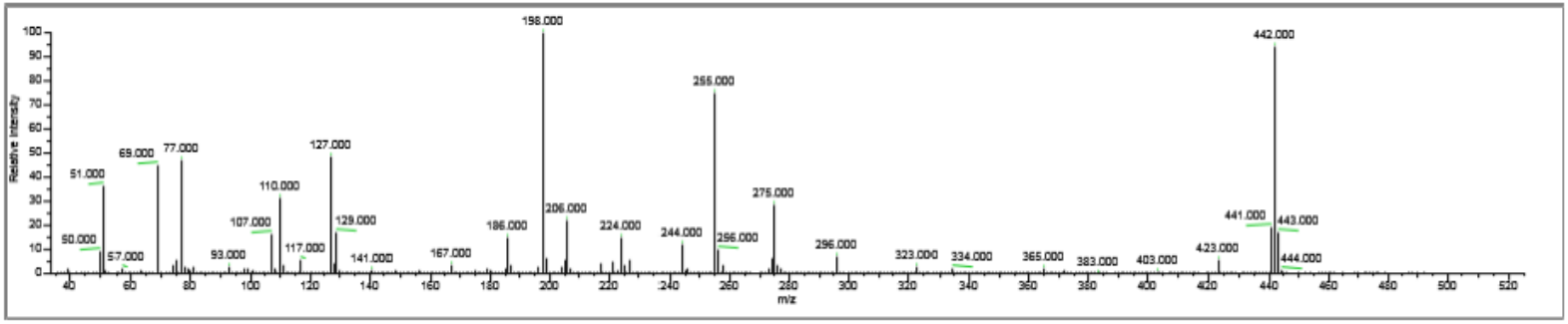


m/z	Ion Abundance Criteria	% Relative Abundance	Pass/Fail
51	greater than or equal to 30% AND less than or equal to 60% of m/z 198	43.5	Pass
68	less than 2% of m/z 69	0	Pass
70	less than 2% of m/z 69	0.5	Pass
127	greater than or equal to 40% AND less than or equal to 60% of m/z 198	54.6	Pass
197	less than 1% of m/z 198	0.5	Pass
198	equals 100% of Base Peak	100	Pass
199	greater than or equal to 5% AND less than or equal to 9% of m/z 198	6	Pass
275	greater than or equal to 10% AND less than or equal to 30% of m/z 198	20.8	Pass
365	greater than 1% of m/z 198	1.9	Pass
441	greater than 0% AND less than 100% of m/z 443	115.9	Fail
442	greater than 40% of m/z 198	65.8	Pass
443	greater than or equal to 17% AND less than or equal to 23% of m/z 442	16.2	Fail

Determined base peak: 198 m/z

TSQ DFTPP Tune





m/z	Ion Abundance Criteria	% Relative Abundance	Pass/Fail
51	greater than or equal to 10% AND less than or equal to 80% of Base Peak	36.7	Pass
68	less than 2% of m/z 69	0	Pass
70	less than 2% of m/z 69	0.3	Pass
127	greater than or equal to 10% AND less than or equal to 80% of Base Peak	49	Pass
197	less than 2% of m/z 198	0	Pass
198	greater than 50% AND less than or equal to 100% of Base Peak	100	Pass
199	greater than or equal to 5% AND less than or equal to 9% of m/z 198	6.5	Pass
275	greater than or equal to 10% AND less than or equal to 60% of Base Peak	28.7	Pass
365	greater than 1% of m/z 198	2.7	Pass
441	greater than 0% AND less than 24% of m/z 442	21	Pass
442	greater than 50% AND less than or equal to 100% of Base Peak	94.9	Pass
443	greater than or equal to 15% AND less than or equal to 24% of m/z 442	18.7	Pass

Determined base peak: 198 m/z

DFTPP

EPA 8270C

TABLE 3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a,b]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

[a] Data taken from Reference 3.
[b] Alternate tuning criteria may be used, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

EPA 8270D

TABLE 3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a,b]

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

[a] Data taken from Reference 3.
[b] Alternate tuning criteria may be employed, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

DFTPP

EPA 625.1

Table 9A – DFTPP Key m/z's and Abundance Criteria for Quadrupole Instruments[1]	
m/z	**Abundance criteria**
51	30 - 60 percent of m/z 198
68	Less than 2 percent of m/z 69
70	Less than 2 percent of m/z 69
127	40 - 60 percent of base peak m/z 198
197	Less than 1 percent of m/z 198
198	Base peak, 100 percent relative abundance
199	5 - 9 percent of m/z 198
275	10 - 30 percent of m/z 198
365	Greater than 1 percent of m/z 198
441	Present but less than m/z 443
442	40 - 100 percent of m/z 198
443	17 - 23 percent of m/z 442

[1] Criteria in these tables are for quadrupole and time-of-flight instruments. Alternative tuning criteria may be used for other instruments, provided method performance is not adversely affected.

Questions?

Contact Artem Dyachenko at adyachen@ebmud.com

Disclaimer: Information presented is not an endorsement of any particular vendor.