



# **The Application of Solid Phase Extraction in EPA Method 625 for Synthetic Wastewater and TCLP Leachate Samples**

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# Target analytes of EPA method 625

> 200 compounds with varied physicochemical properties:

Acids



Bases

Neutrals

Polar (NDMA:  $\text{LogP} = -0.496$ )

Non-polar (DDT:  $\text{LogP} = 6.46$ )

# UCT SPE method: extraction setup

## Simultaneous extraction of 12 samples



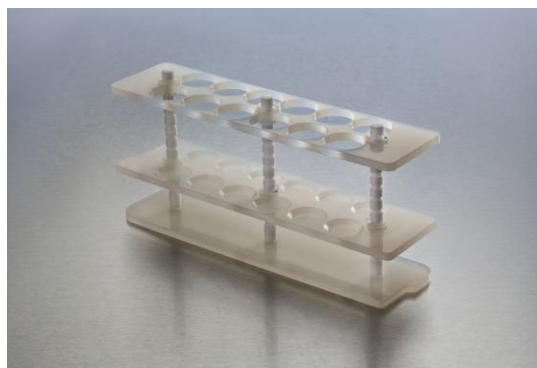
# UCT SPE method: elution setup



**Elution of EC8270 cartridges**



**Elution of carbon cartridges**



**Newly designed collection rack  
for 40, 50, and 60 mL vials**

# Can be automated too!



**FMS TurboTrace ABN SPE system**

# Phase I study

## ASTM D5905 synthetic wastewater

0.4 g flour

2 g ocean salts

0.08 g Kaolin (China clay)

0.024 g Triton™ X-100 surfactant

120 mL beer

Diluted to 2 L with reagent water

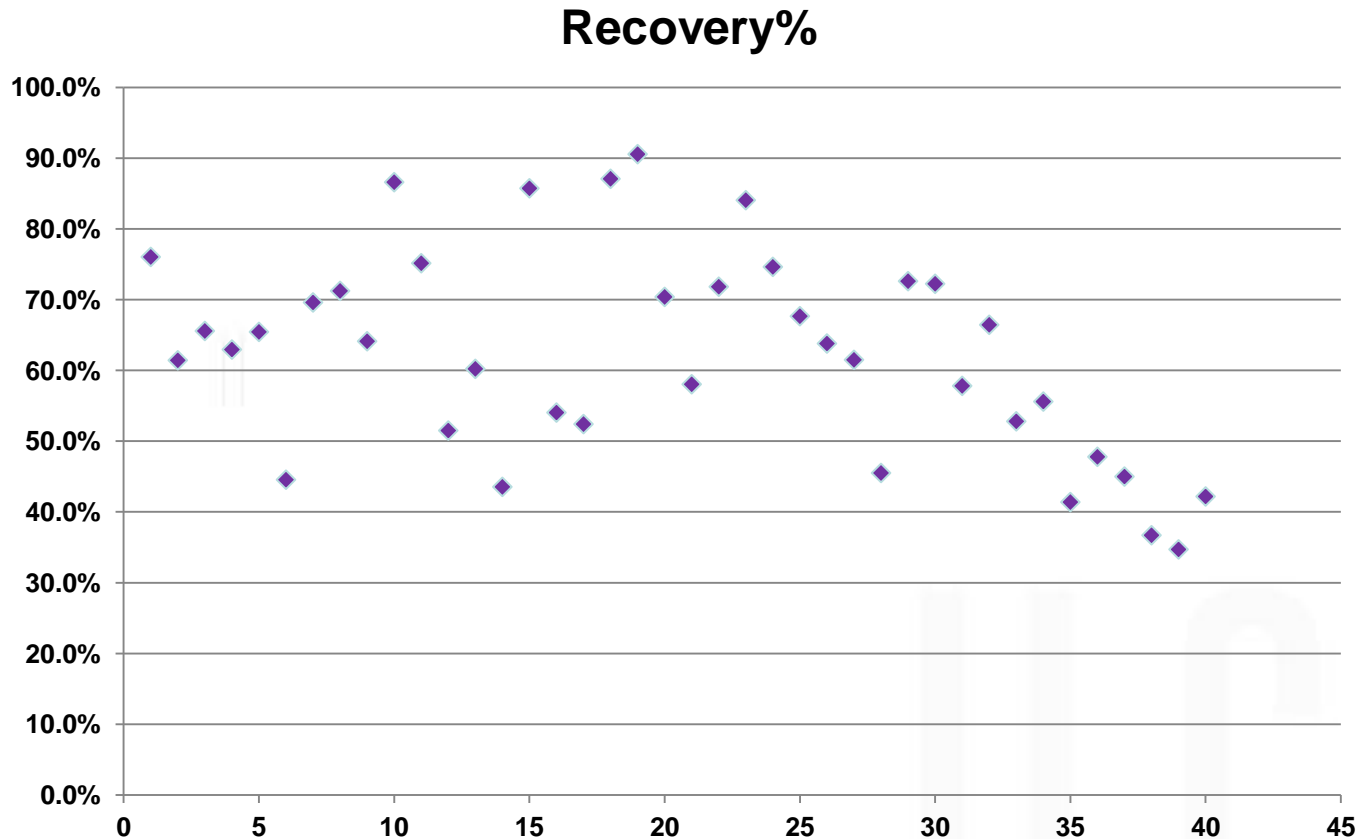
UCT SPE product: **p**



# Synthetic wastewater results

## Lab 5: Los Angeles County Sanitation District

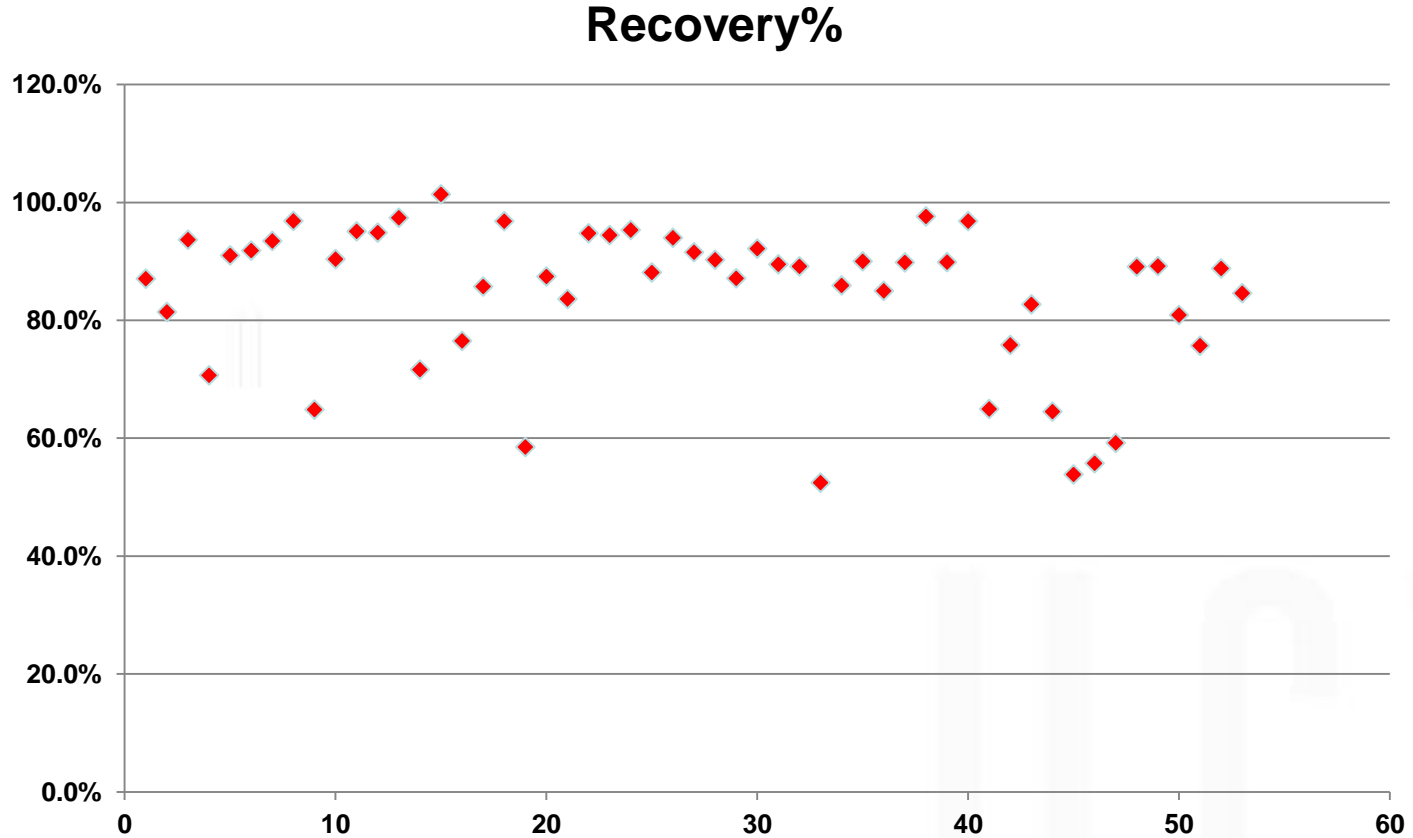
Credit to Henry Liu



All compounds passed the acceptance limits.

# Synthetic wastewater results

## Lab 10: UCT R&D Lab



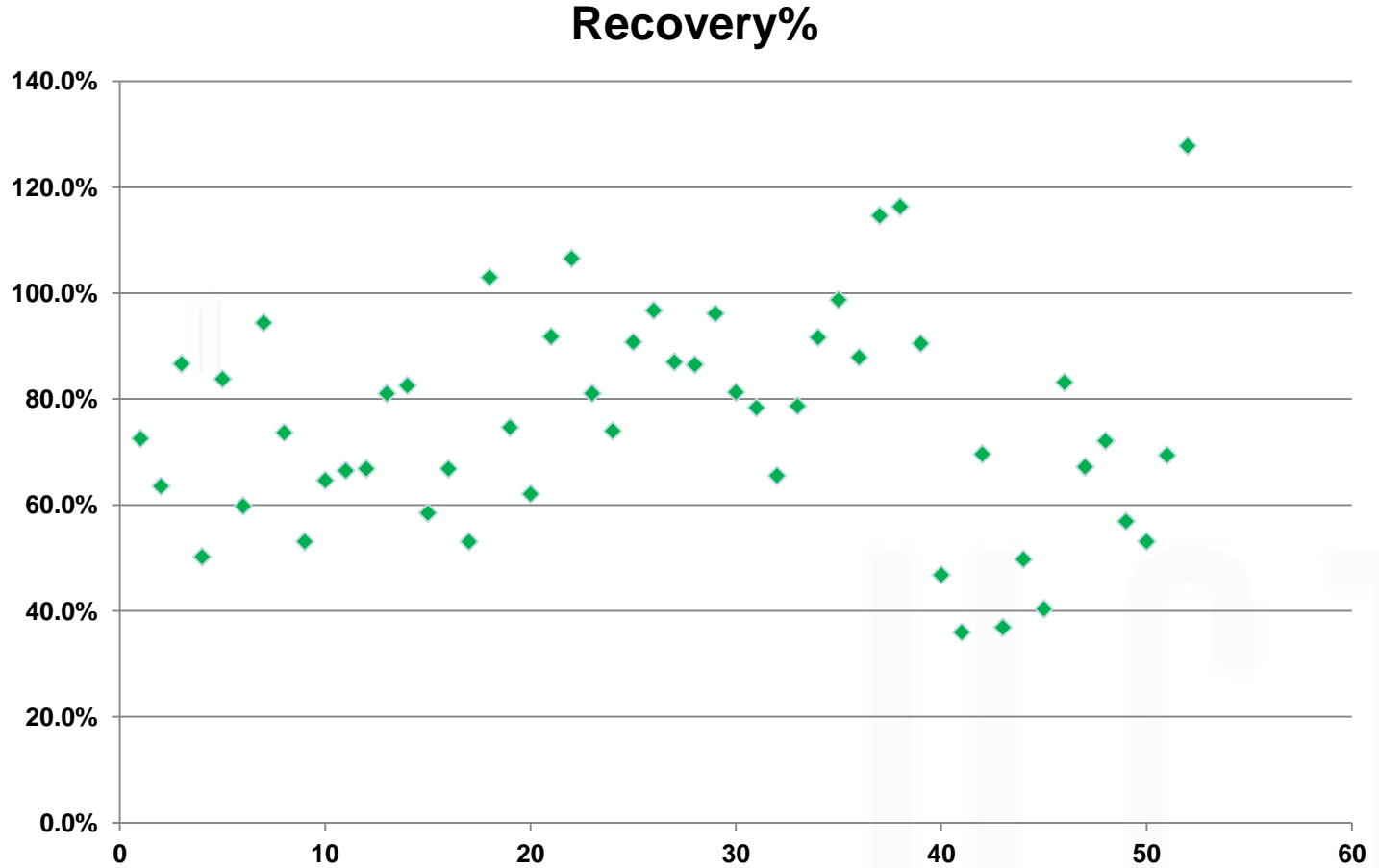
All compounds passed the acceptance limits.



# Synthetic wastewater results

## Lab 11: FMS (automated SPE)

Credit to Phil Bassignani



All compounds passed the acceptance limits.

# Phase I synthetic wastewater UCT SPE vs CLLE

Bases and Neutrals	Recovery%	
	UCT	CLLE
Acenaphthene	85.06%	79.66%
Anthracene	83.32%	76.18%
Benzo(a)anthracene	66.70%	98.17%
Benzo(k)fluoranthene	51.06%	70.97%
Benzo(g,h,i)perylene	61.52%	59.97%
Benzo(a)pyrene	48.80%	62.86%
4-Bromophenyl-phenylether	81.83%	75.17%
Butyl benzyl phthalate	80.04%	65.01%
bis(2-Chloroethyl)ether	68.80%	62.36%
bis(2-Chloroisopropyl) ether	71.53%	81.86%
4-Chlorophenyl-phenylether	78.40%	74.04%
Dibenzo(a,h)anthracene	43.61%	55.64%
Dibenzofuran	94.14%	75.98%
Di-n-butylphthalate	90.02%	81.21%
1,3-Dichlorobenzene	60.44%	70.15%
Diethyl phthalate	90.79%	77.45%
Dimethyl phthalate	87.82%	77.82%
2,6-Dinitrotoluene	84.23%	88.41%
bis(2-ethylhexyl) phthalate	55.78%	103.64%
Fluoranthene	90.89%	76.63%
Fluorene	81.81%	79.58%
Hexachlorobenzene	75.37%	76.49%
Hexachlorobutadiene	51.71%	68.56%
Hexachloroethane	54.16%	64.28%
Indeno(1,2,3-cd)pyrene	46.77%	55.53%
Isophorone	77.61%	84.50%
1-Methylnaphthalene	74.66%	62.05%
2-Methylnaphthalene	72.85%	68.42%
Naphthalene	70.93%	74.66%
Nitrobenzene	74.88%	75.95%
N-Nitroso-di-n-propylamine	78.66%	79.92%
Phenanthrene	85.45%	74.31%
1,2,4-Trichlorobenzene	62.16%	71.81%
<b>Overall Mean</b>	<b>72.18%</b>	<b>74.22%</b>

Acids	Recovery%	
	UCT	CLLE
Benzoic acid	71.64%	114.27%
4-Chloro-3-methylphenol	92.05%	99.13%
2-Chlorophenol	81.97%	82.53%
2,4-Dichlorophenol	86.36%	94.77%
2,6-Dichlorophenol	96.82%	85.65%
2,4-Dimethylphenol	88.36%	99.15%
2-Methyl-4,6-Dinitrophenol	60.60%	48.85%
2-Methylphenol	87.40%	70.21%
4-Methylphenol (and/or 3-Methylphenol)	93.94%	70.28%
2-Nitrophenol	75.28%	86.21%
4-Nitrophenol	78.44%	48.61%
Phenol	78.55%	45.55%
Pentachlorophenol	75.72%	71.81%
2,4,5-Trichlorophenol	84.47%	85.24%
2,4,6-Trichlorophenol	80.21%	99.25%
<b>Overall Mean</b>	<b>74.17%</b>	<b>74.67%</b>

# Phase I DI water UCT SPE vs CLLE

Bases and Neutrals	Recovery%	
	UCT	CLLE
Acenaphthene	68.79%	69.07%
Anthracene	80.62%	72.06%
Benzo(a)anthracene	76.66%	77.06%
Benzo(k)fluoranthene	75.42%	75.09%
Benzo(g,h,i)perylene	91.35%	86.25%
Benzo(a)pyrene	78.85%	85.25%
4-Bromophenyl-phenylether	67.80%	71.07%
Butyl benzyl phthalate	73.57%	105.34%
bis(2-Chloroethyl)ether	67.90%	96.51%
bis(2-Chloroisopropyl) ether	67.41%	71.69%
4-Chlorophenyl-phenylether	63.46%	69.79%
Dibenzo(a,h)anthracene	84.46%	83.22%
Dibenzofuran	74.17%	54.23%
Di-n-butylphthalate	83.79%	79.08%
1,3-Dichlorobenzene	55.19%	65.16%
Diethyl phthalate	79.85%	62.21%
Dimethyl phthalate	76.64%	71.77%
2,6-Dinitrotoluene	69.28%	76.09%
bis(2-ethylhexyl) phthalate	71.50%	76.95%
Fluoranthene	83.19%	79.58%
Fluorene	67.29%	68.78%
Hexachlorobenzene	71.27%	79.55%
Hexachlorobutadiene	44.26%	85.46%
Hexachloroethane	48.38%	98.53%
Indeno(1,2,3-cd)pyrene	80.30%	80.56%
Isophorone	71.26%	66.92%
1-Methylnaphthalene	62.15%	8.43%
2-Methylnaphthalene	63.39%	45.91%
Naphthalene	65.92%	64.96%
Nitrobenzene	69.45%	66.67%
N-Nitroso-di-n-propylamine	72.61%	86.23%
Phenanthrene	74.97%	69.21%
1,2,4-Trichlorobenzene	56.97%	69.32%
<b>Overall Mean</b>	<b>70.85%</b>	<b>73.27%</b>

Acids	Recovery%	
	UCT	CLLE
Benzoic acid	94.00%	25.36%
4-Chloro-3-methylphenol	72.97%	78.17%
2-Chlorophenol	76.34%	74.95%
2,4-Dichlorophenol	72.17%	79.52%
2,6-Dichlorophenol	86.50%	
2,4-Dimethylphenol	74.88%	74.18%
2-Methyl-4,6-Dinitrophenol	43.65%	41.67%
2-Methylphenol	79.11%	87.52%
4-Methylphenol (and/or 3-Methylphenol)	83.93%	87.42%
2-Nitrophenol	67.08%	72.45%
4-Nitrophenol	59.40%	51.50%
Phenol	70.85%	47.65%
Pentachlorophenol	58.28%	49.56%
2,4,5-Trichlorophenol	69.54%	57.50%
2,4,6-Trichlorophenol	61.32%	65.35%
<b>Overall Mean</b>	<b>76.29%</b>	<b>69.84%</b>

# Phase II study

## TCLP Leachate Samples

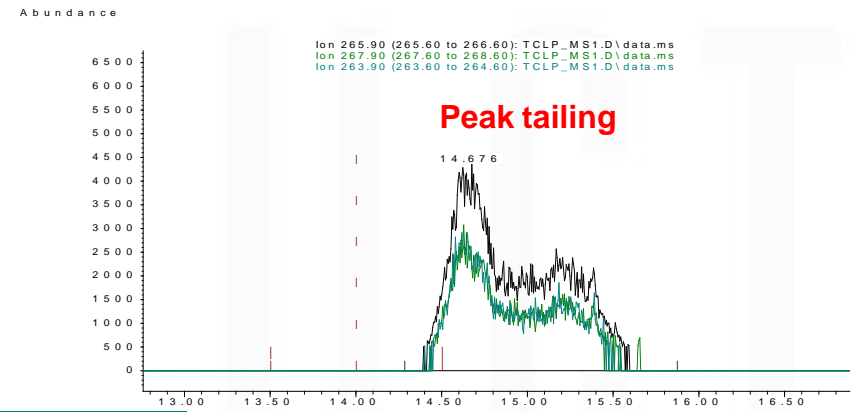
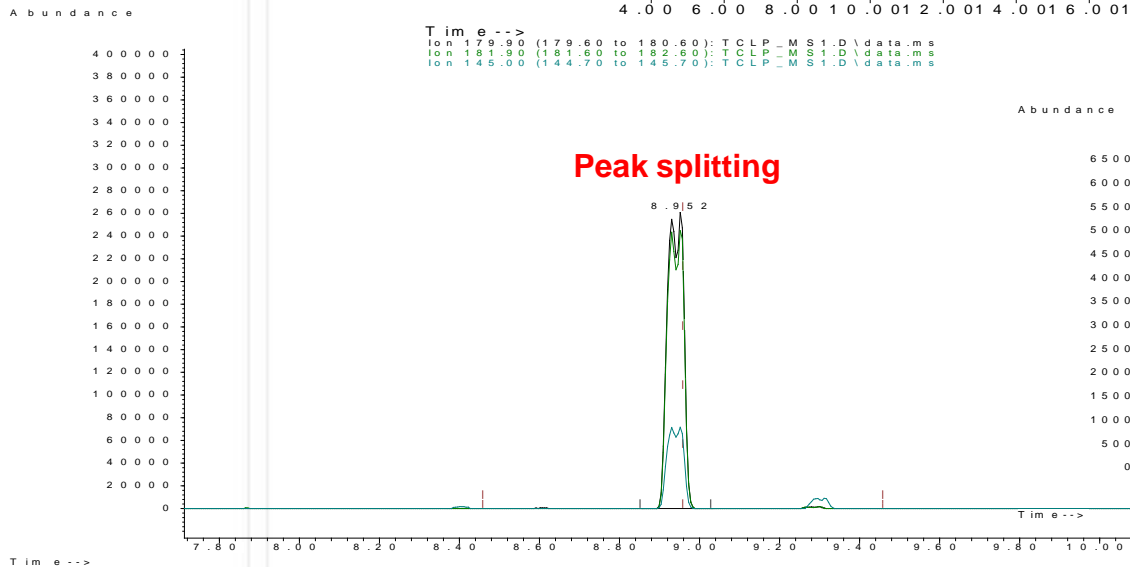
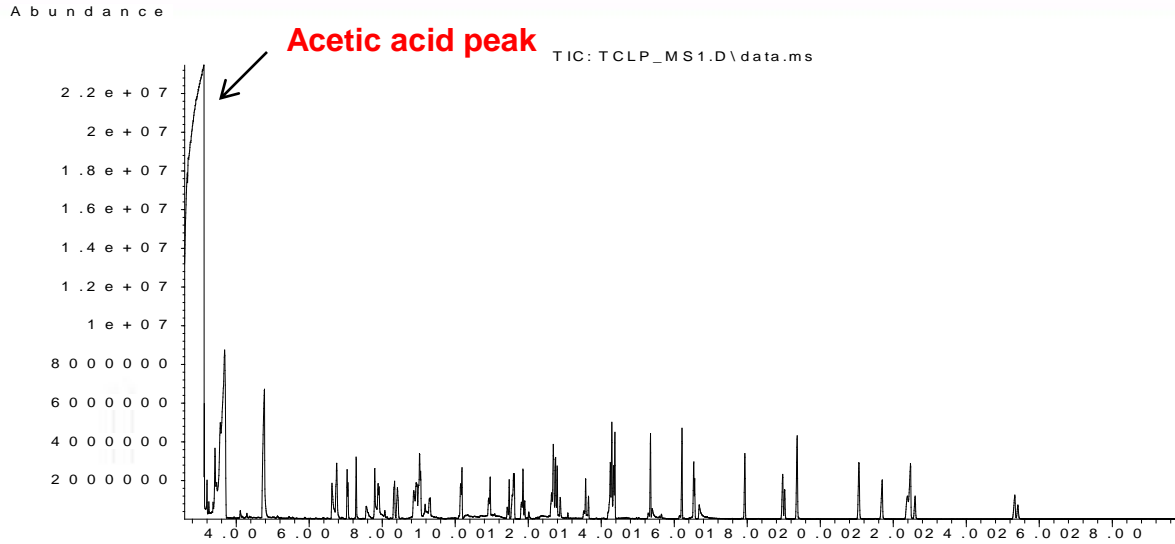
5.7 mL glacial acetic acid

64.3 mL 1 N sodium hydroxide

Diluted to 1 L with reagent water

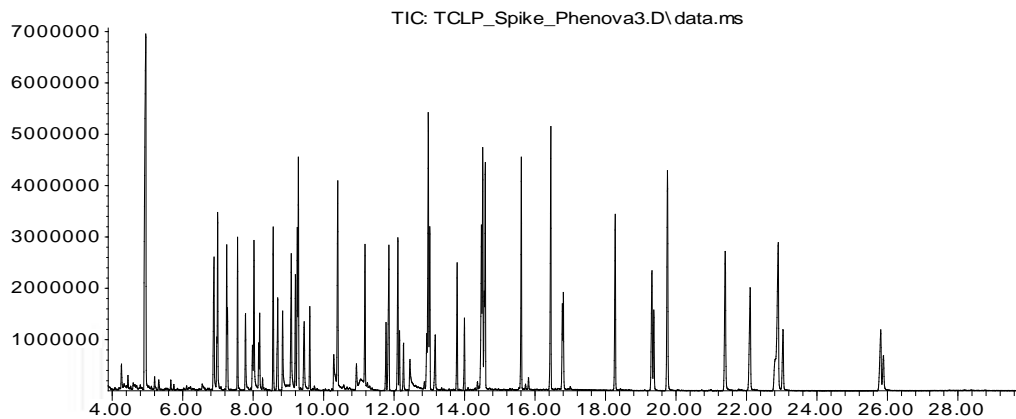
UCT SPE Product: **P6**

# Deteriorated chromatogram for TCLP matrix



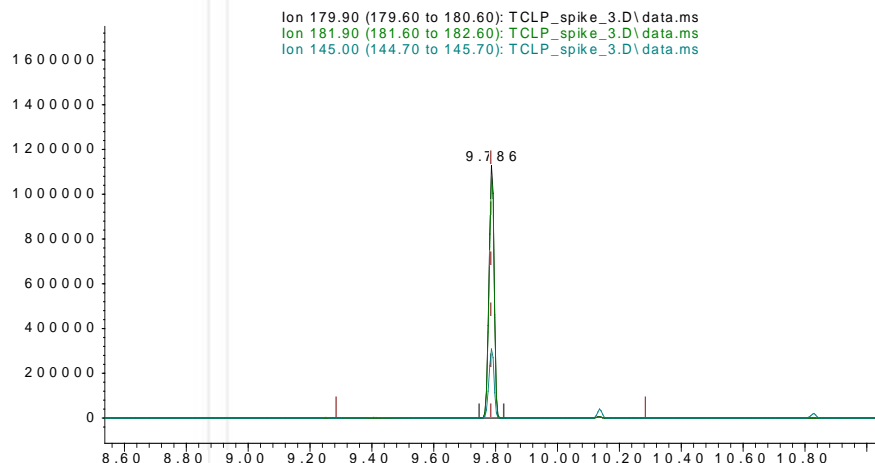
# With optimized wash

Abundance



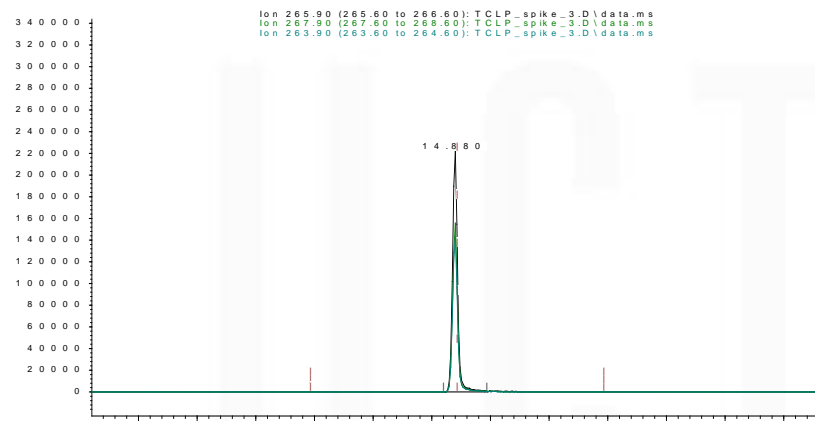
Time-->

Abundance



Time-->

Abundance



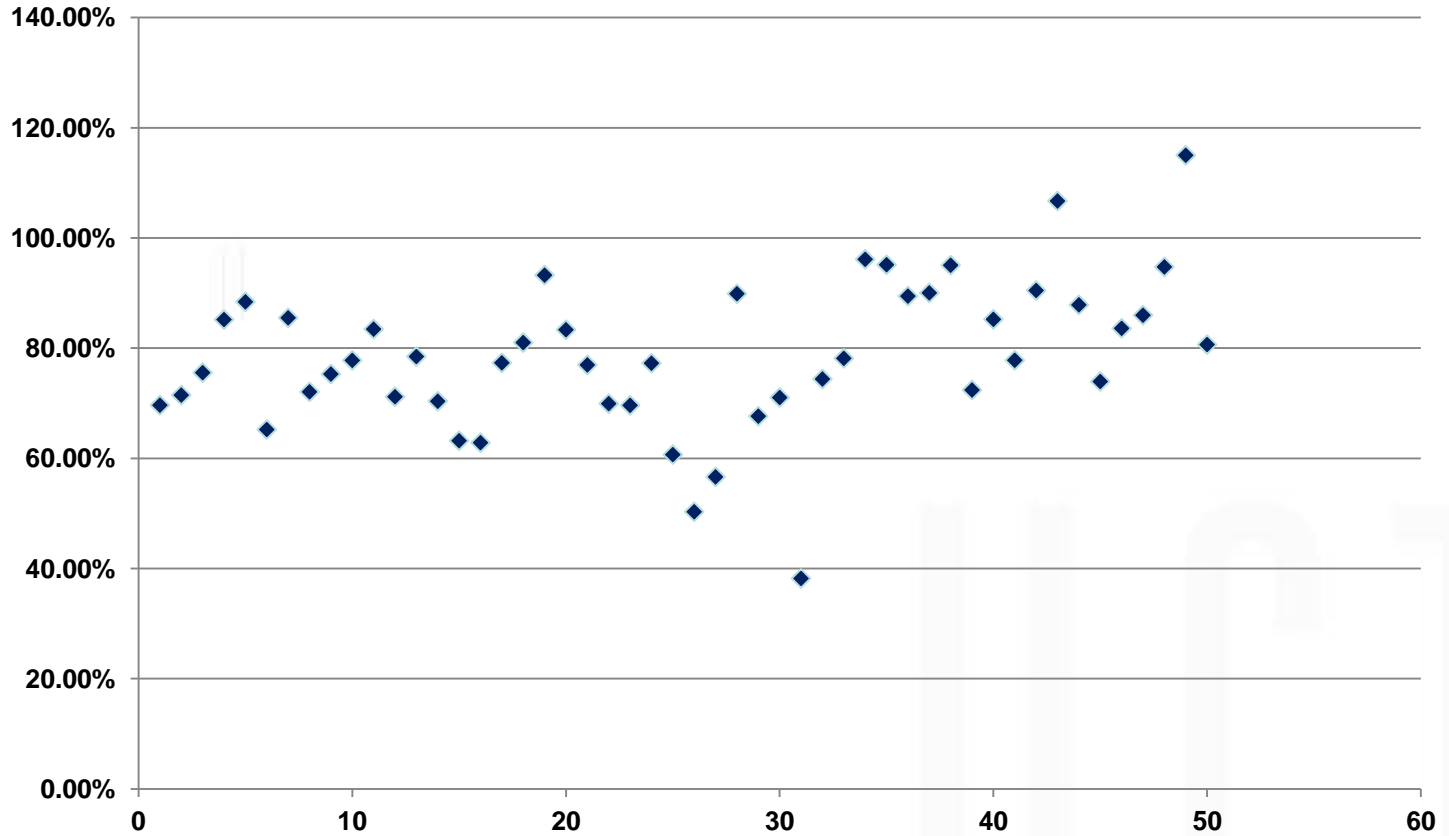
Time-->



# Phase II TCLP results: Lab18 (LACSD)

Credit to Henry Liu

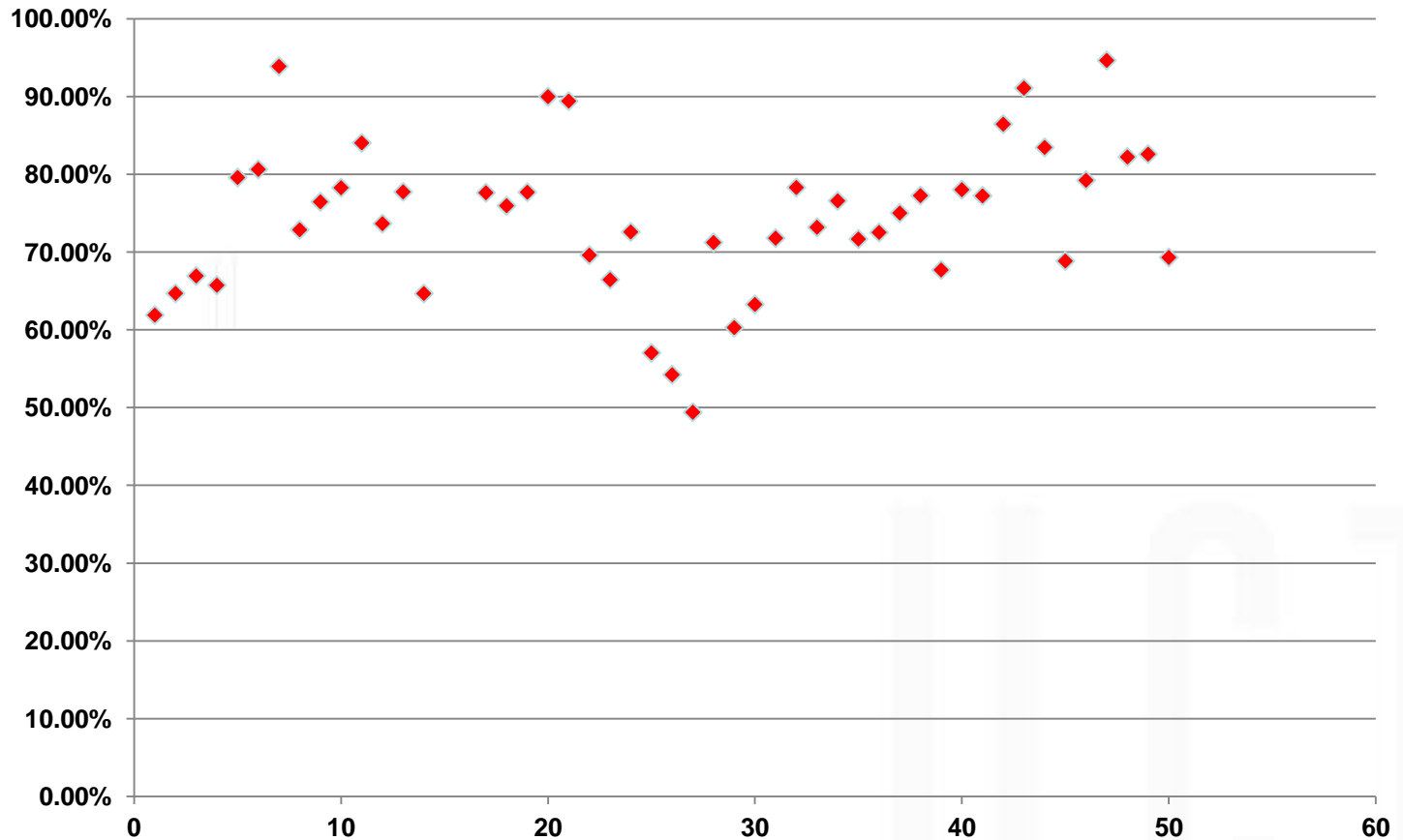
## Lab18 TCLP Recovery



# Phase II TCLP results: Lab19 (FLDEP)

Credit to Mohammad Ghaffari

## Lab19 TCLP Recovery

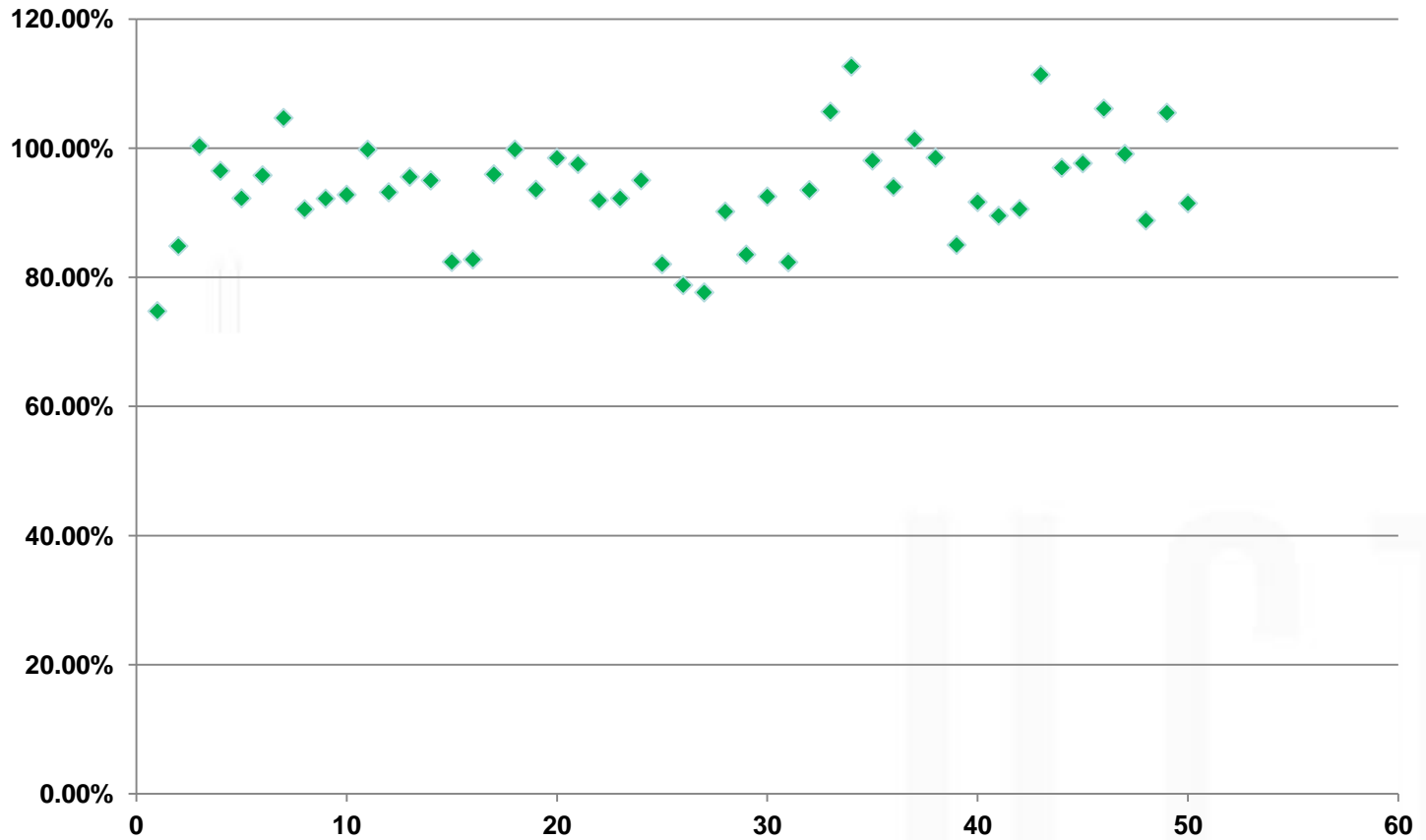






# Phase II TCLP results: Lab20 (UCT)

## Lab20 TCLP Recovery

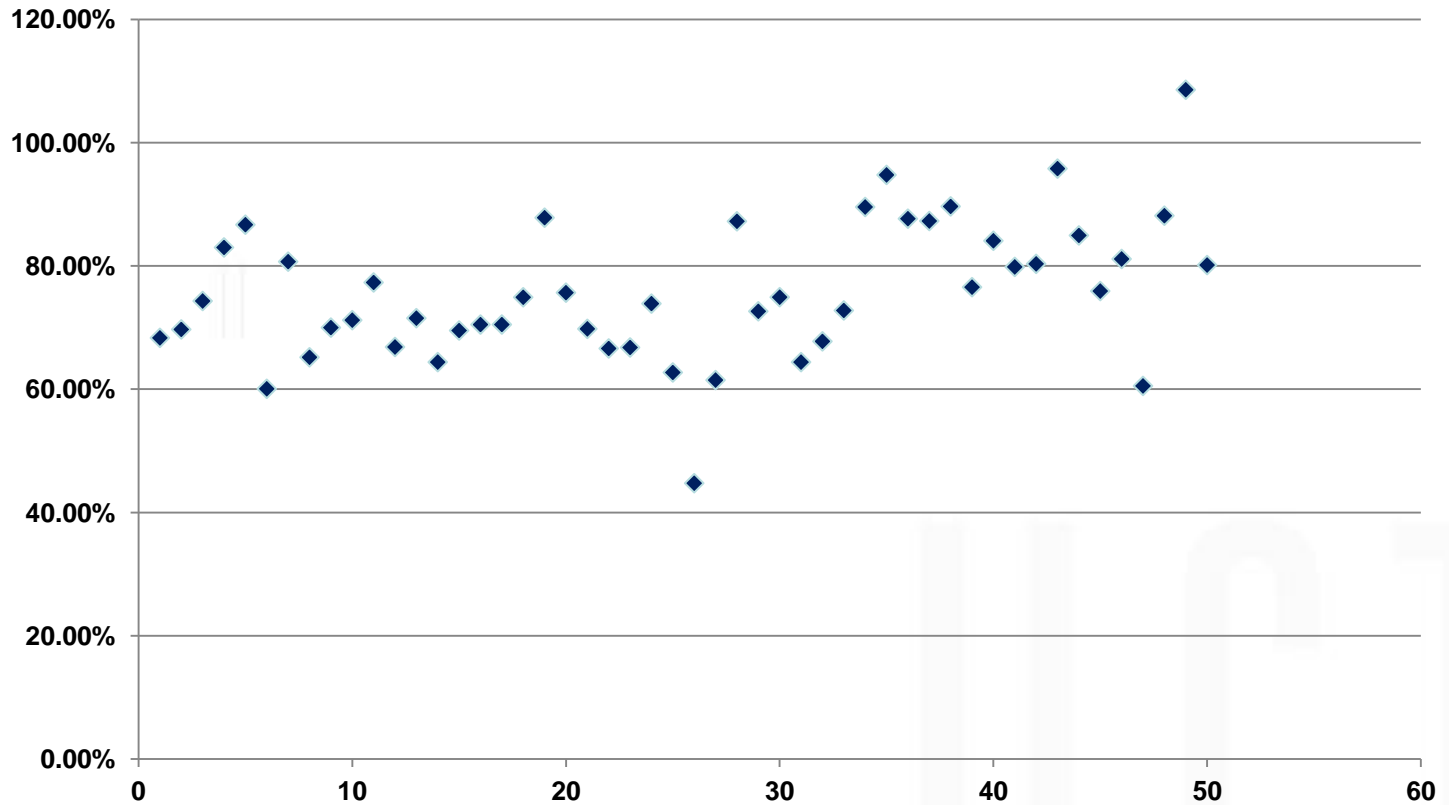




# Phase II DI water results: Lab18 (LACSD)

Credit to Henry Liu

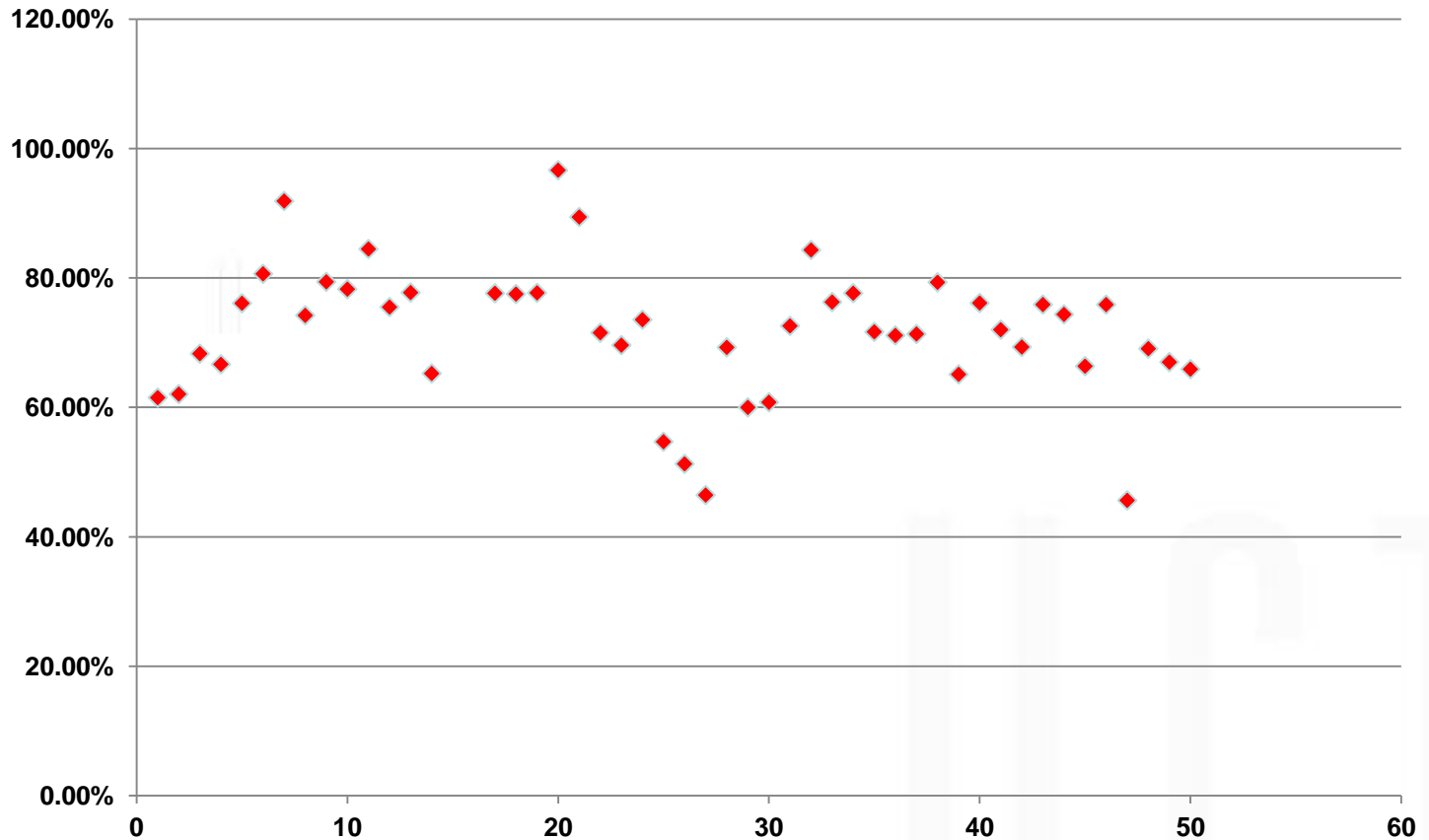
## Lab18 DI Water Recovery



# Phase II DI water results: Lab19 (FLDEP)

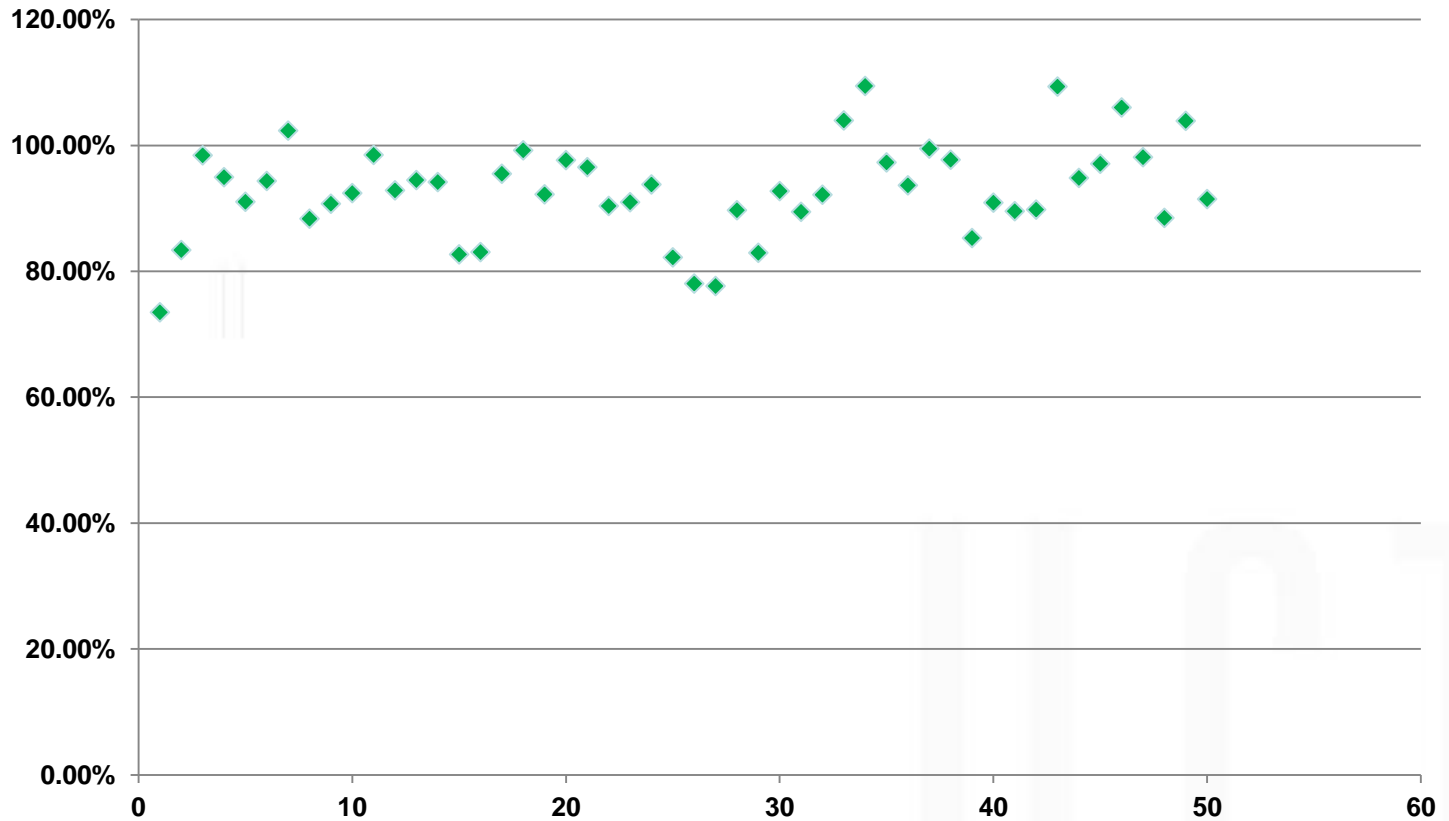
Credit to Mohammad Ghaffari

## Lab19 DI Water Recovery



# Phase II DI water results: Lab20 (UCT)

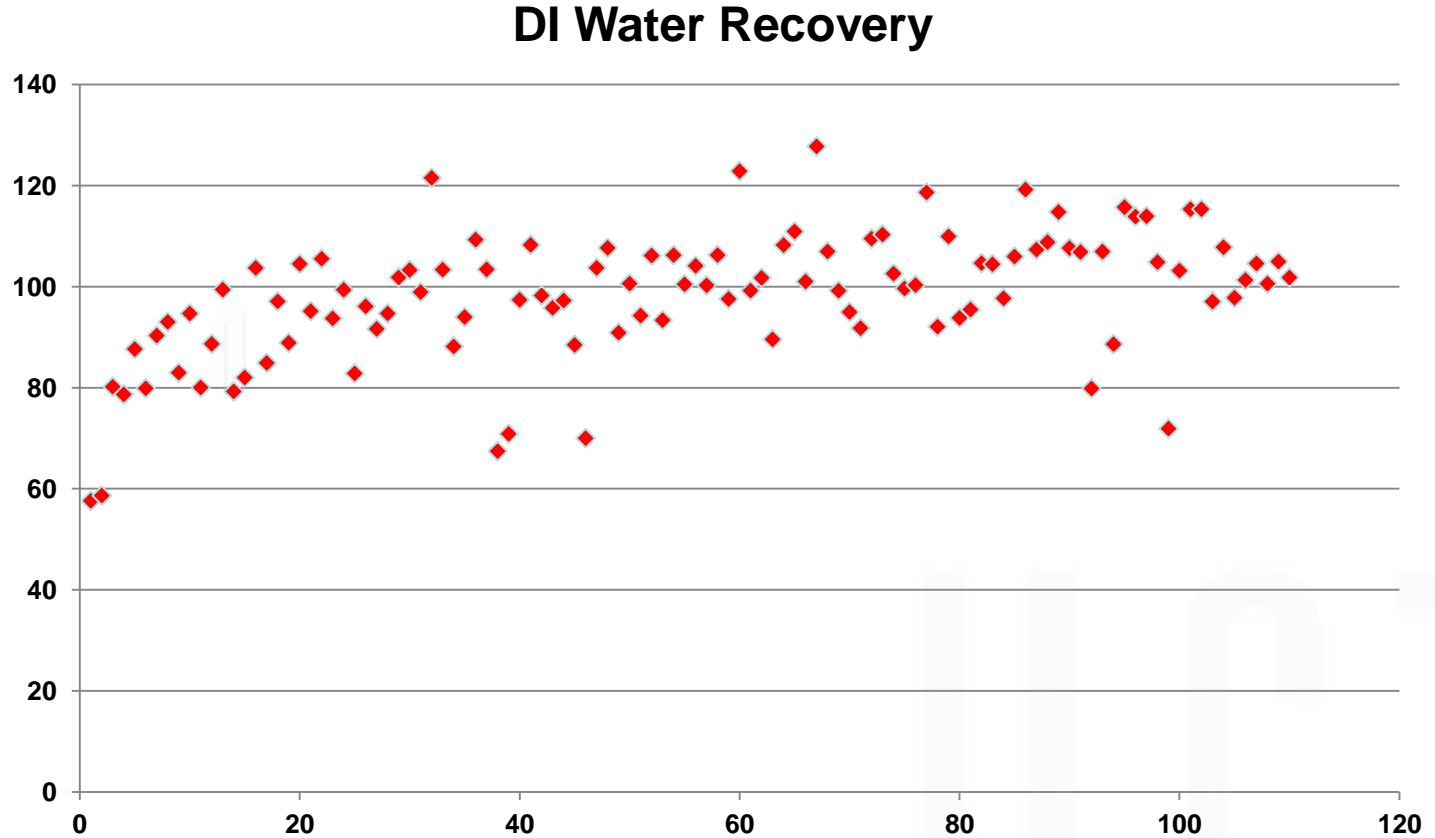
## Lab20 DI Water Recovery



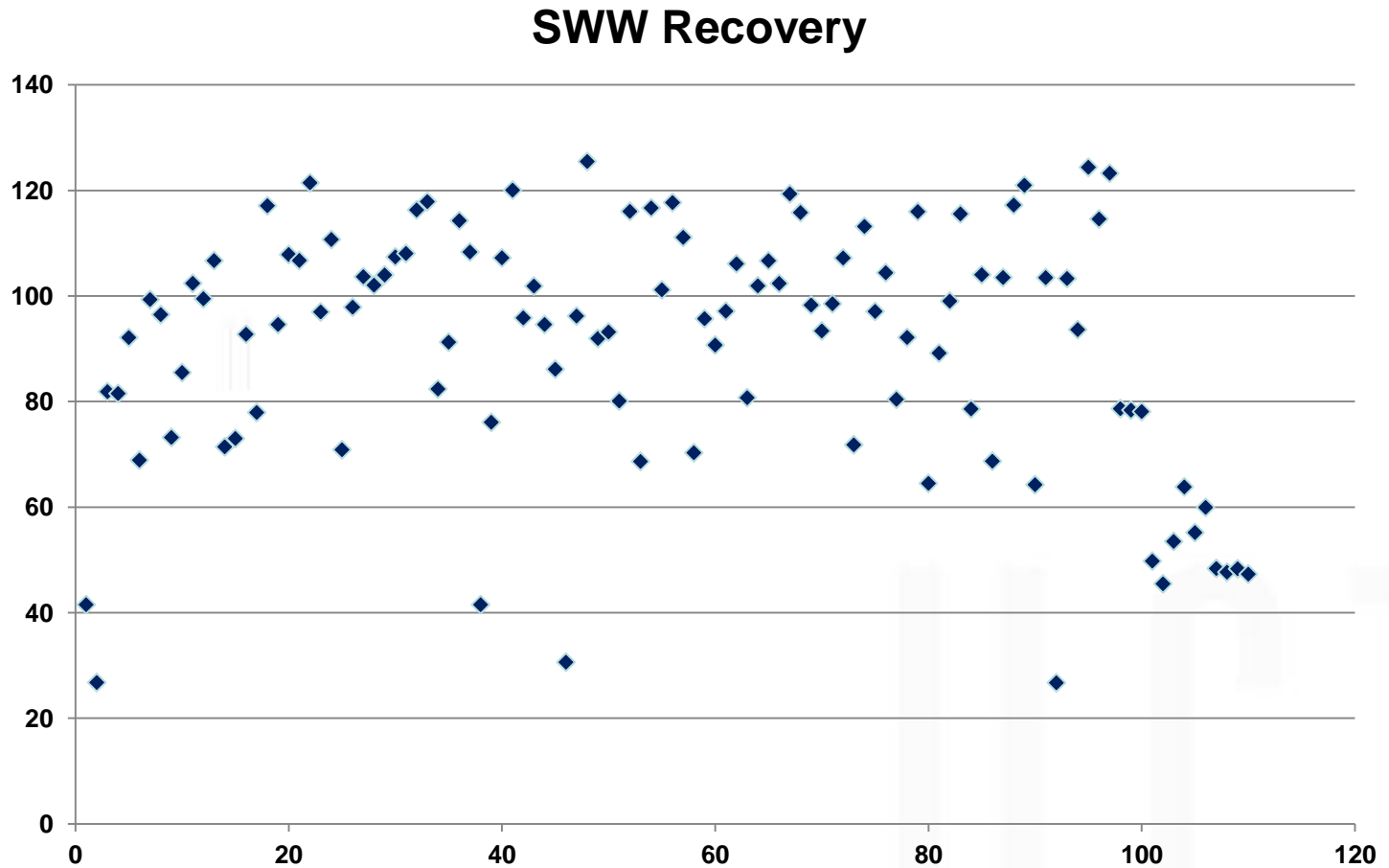
# What about other compounds NOT included in the PT samples?

**110 known compounds spiked in  
3 different sample matrices  
using UCT SPE method**

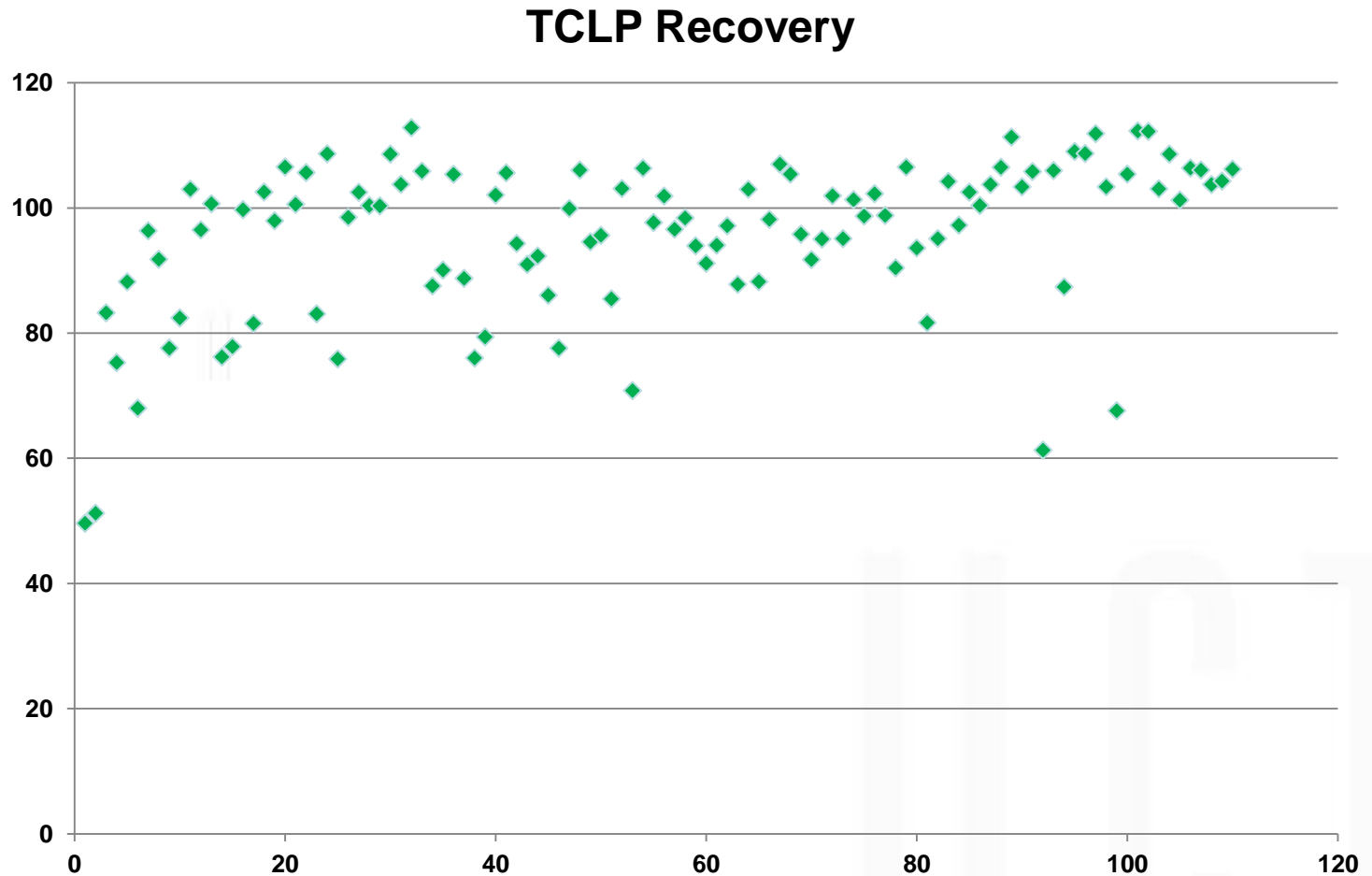
# Recovery of 110 target analytes in DI water



# Recovery of 110 target analytes in synthetic wastewater



# Recovery of 110 target analytes in TCLP sample

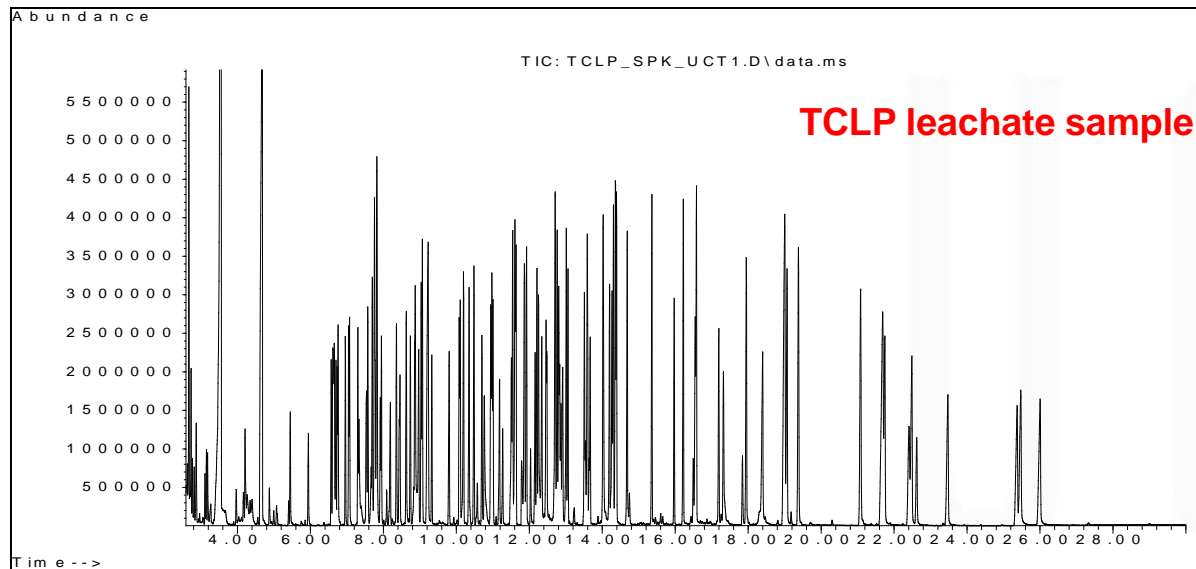
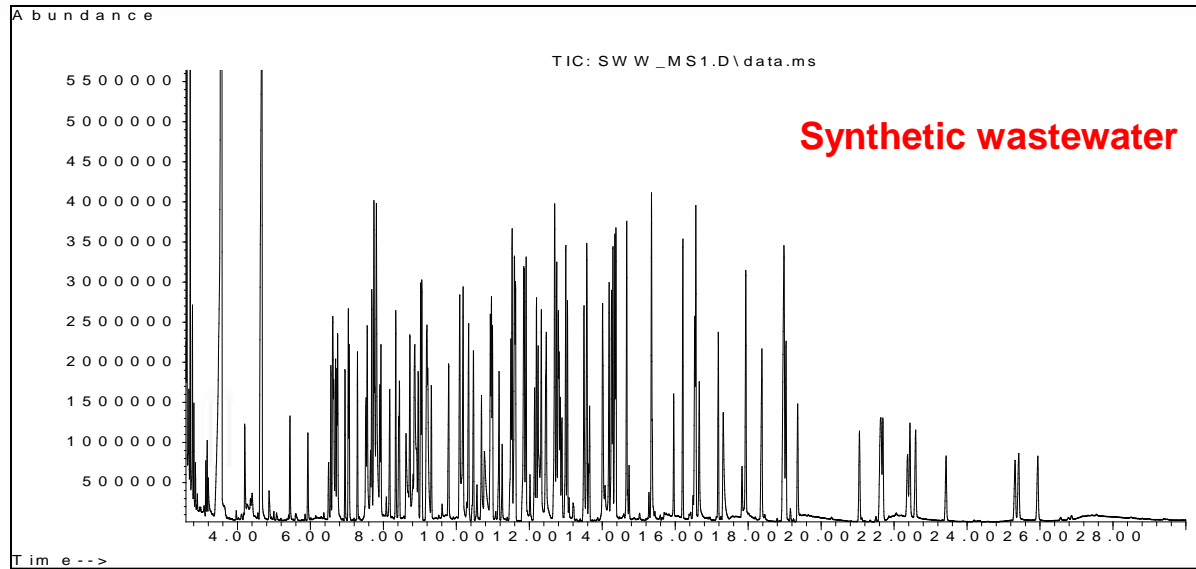




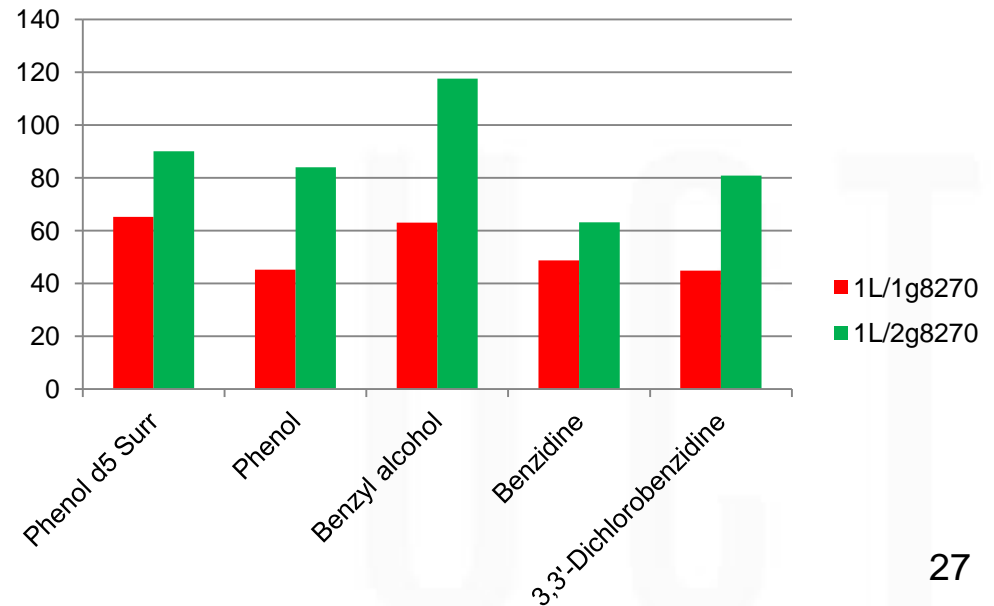
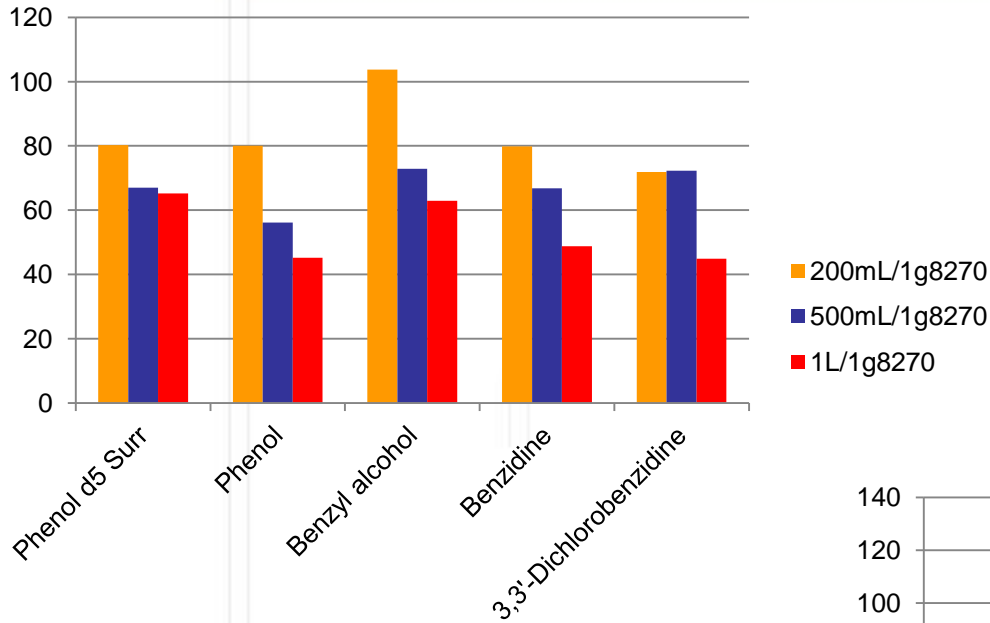
# Recovery of 17 surrogates in 3 sample matrices

Surrogate	Average Recovery%		
	DI water	SWW	TCLP
N-nitrosodimethylamine d6	77	69	81
Phenol d5	80	78	92
Bis(2-chloroethyl) ether d8	89	82	94
2-Chlorophenol d4	95	86	92
4-Methylphenol d8	99	98	98
Nitrobenzene d5	91	81	91
2-Nitrophenol d4	100	87	93
2,4-Dichlorophenol d3	98	95	96
4-Chloroaniline d4	94	85	79
Dimethylphthalate d6	99	95	94
Acenaphthylene d8	93	81	86
4-Nitrophenol d4	111	88	86
Fluorene d10	92	85	89
4,6-Dintro-2-methylphenol d2	119	69	96
Anthracene d10	92	84	90
Pyrene d10	95	86	93
Benzo(a)pyrene d12	95	50	98
<b>Overall mean</b>	<b>95</b>	<b>82</b>	<b>91</b>

# Chromatograms



# Does sample volume matter?



# Conclusions

- One sample pass ( $\text{pH} < 2$ )
- No emulsion formed
- Much less organic solvent used
- Shorter sample preparation time
- Cleaner extracts
- Results comparable to CLLE
- Higher throughput: simultaneous extraction of 12 samples
- Can be automated: FMS TurboTrace ABN SPE system

# Acknowledgement

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Henry Liu @ Los Angeles County Sanitation District

Mohammad Ghaffari @ Florida Department of  
Environmental Protection

625 SPE working group

# Questions?

**UCT Booth #31**

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