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Environmental & Process Monitoring

Sorbent Tubes for the Measurement of Metals in Gaseous Fuels

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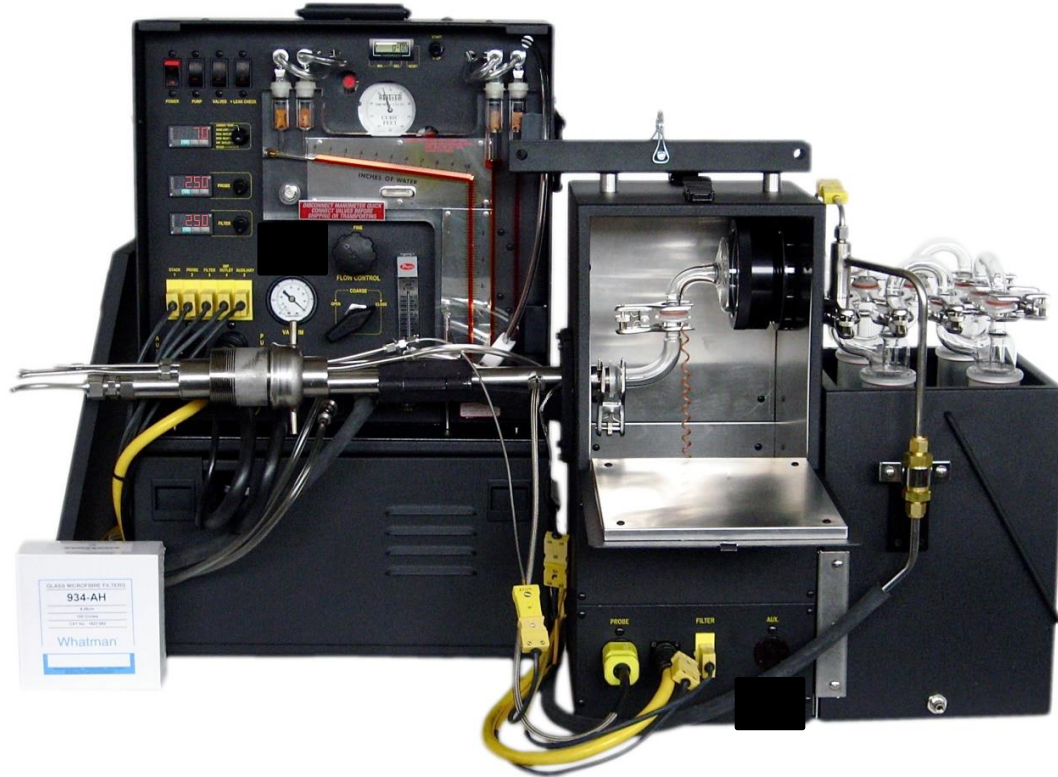
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Context

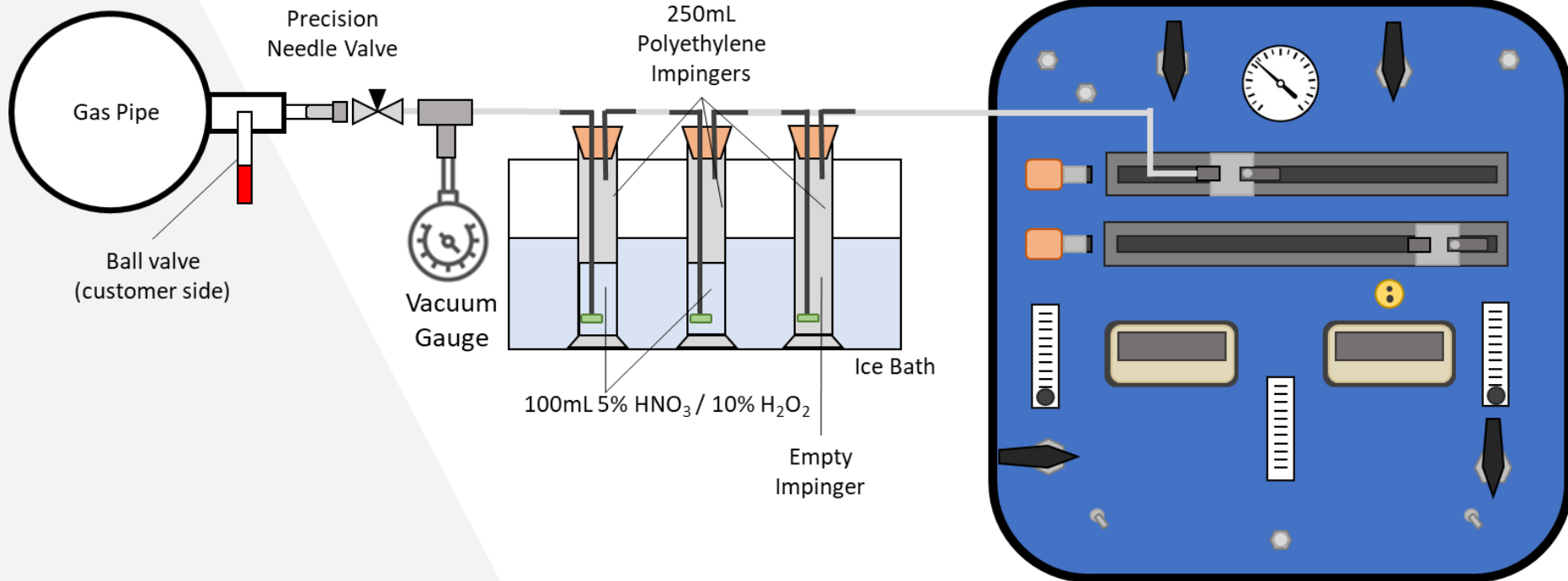
- ▶ **Why are we measuring metals in the first place?**
 - Utility tariffs and gas quality specifications
 - Carcinogenic and non-carcinogenic Health Protective Constituents (these metals are toxic)
 - Renewable Natural Gas (RNG) producers must demonstrate compliance
 - Currently includes Copper (Cu), Arsenic (As), Antimony (Sb), and Lead (Pb)

- ▶ **What is the purpose of this method development?**
 - Replace EPA Method 29 with sorbent tubes

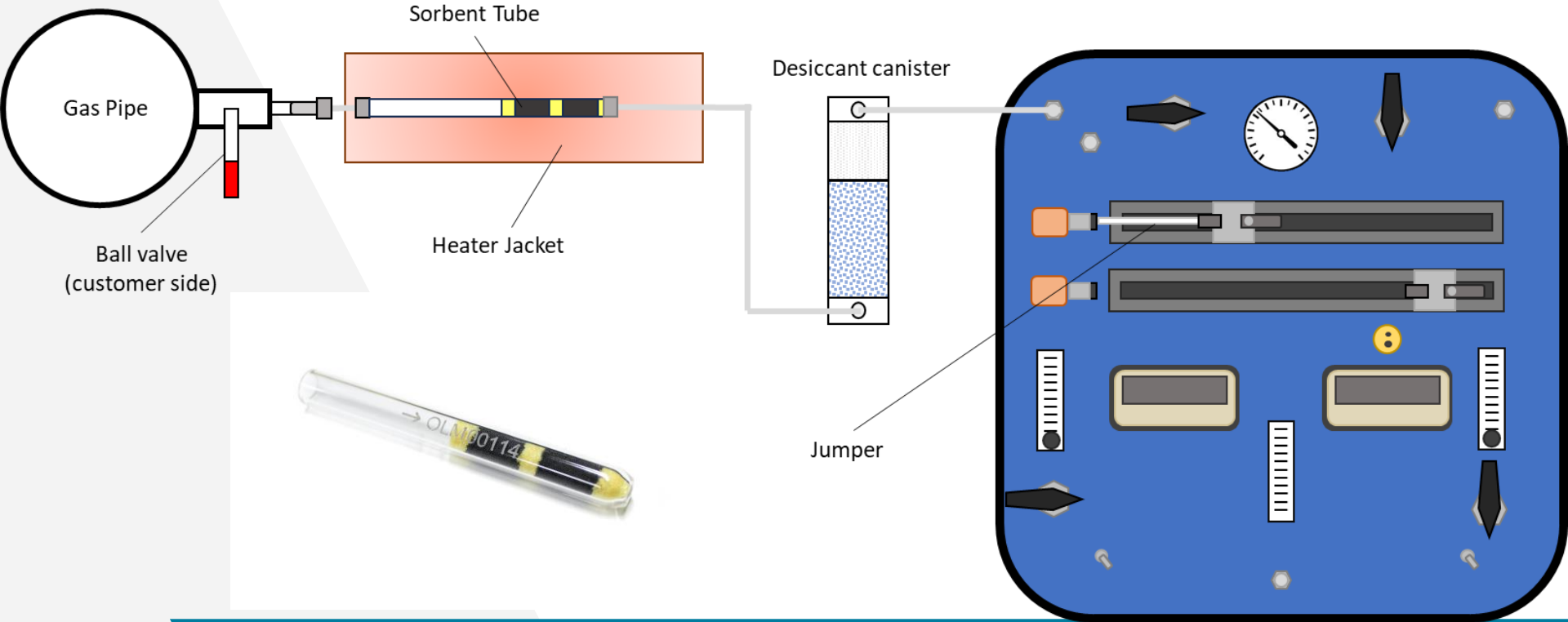
EPA Method 29



Modified Method 29



Sorbent Tubes

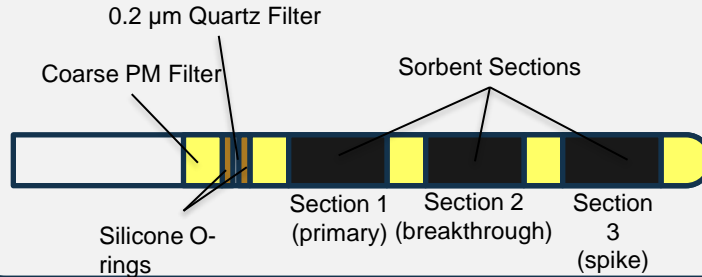


Background

EPA SBIR
Research
Project

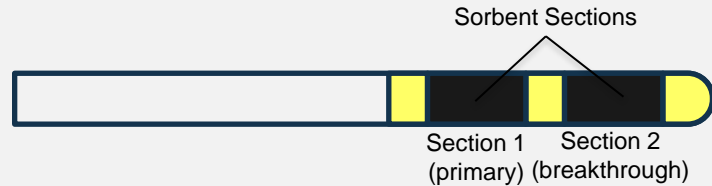
Sorbent
Material
Development

Emissions Sorbent Tube



- Coal-fired power plants, waste incinerators, cement, etc.
- Be, Cr, Mn, Co, Ni, Zn, As, Se, Cd, Cu, Sb, Pb, others
- Continuous compliance (7-day runs) and alternative to Method 29
- Modeled after EPA PS 12B

Gaseous Fuels Sorbent Tube

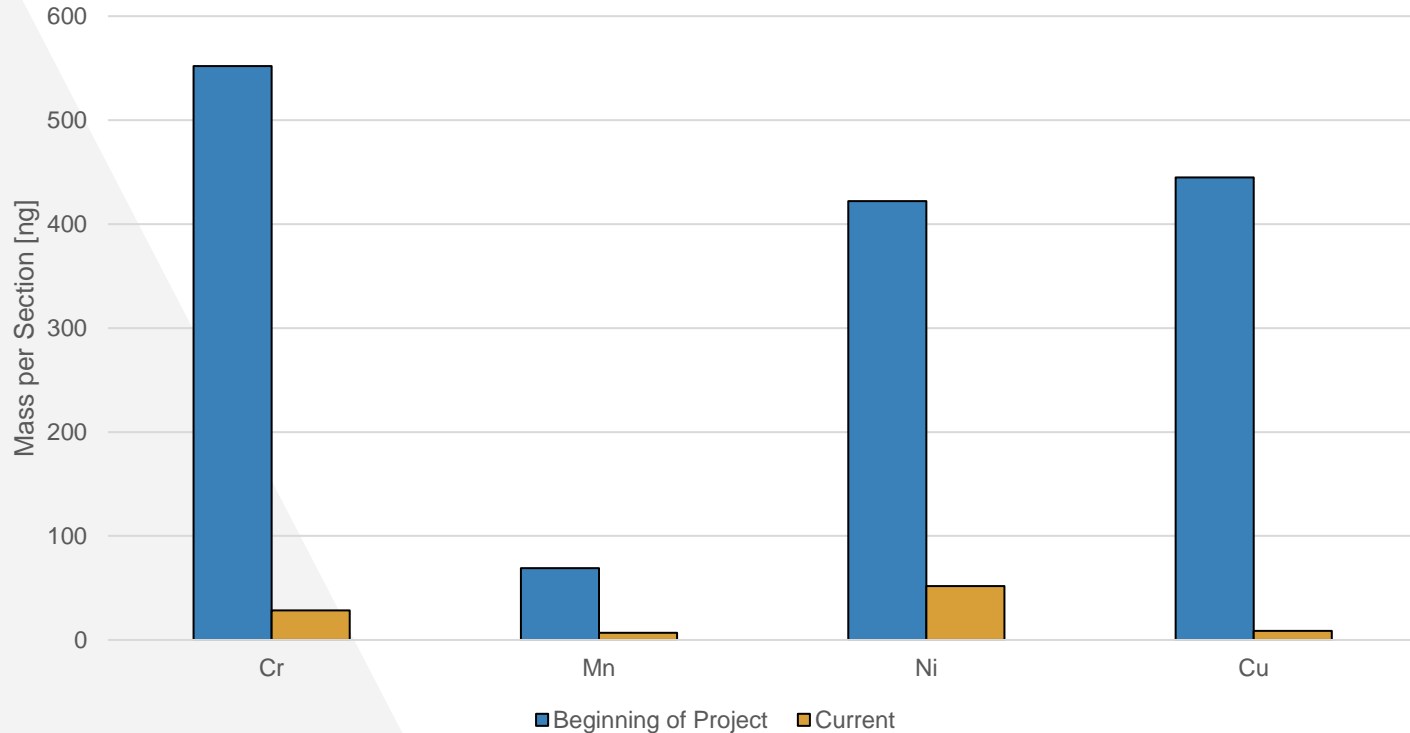


- Biogas and Renewable Natural Gas
- Primary focused on As, Sb, Cu, and Pb
- 1 hour runs

Objective #1 – Reduce Background Metals

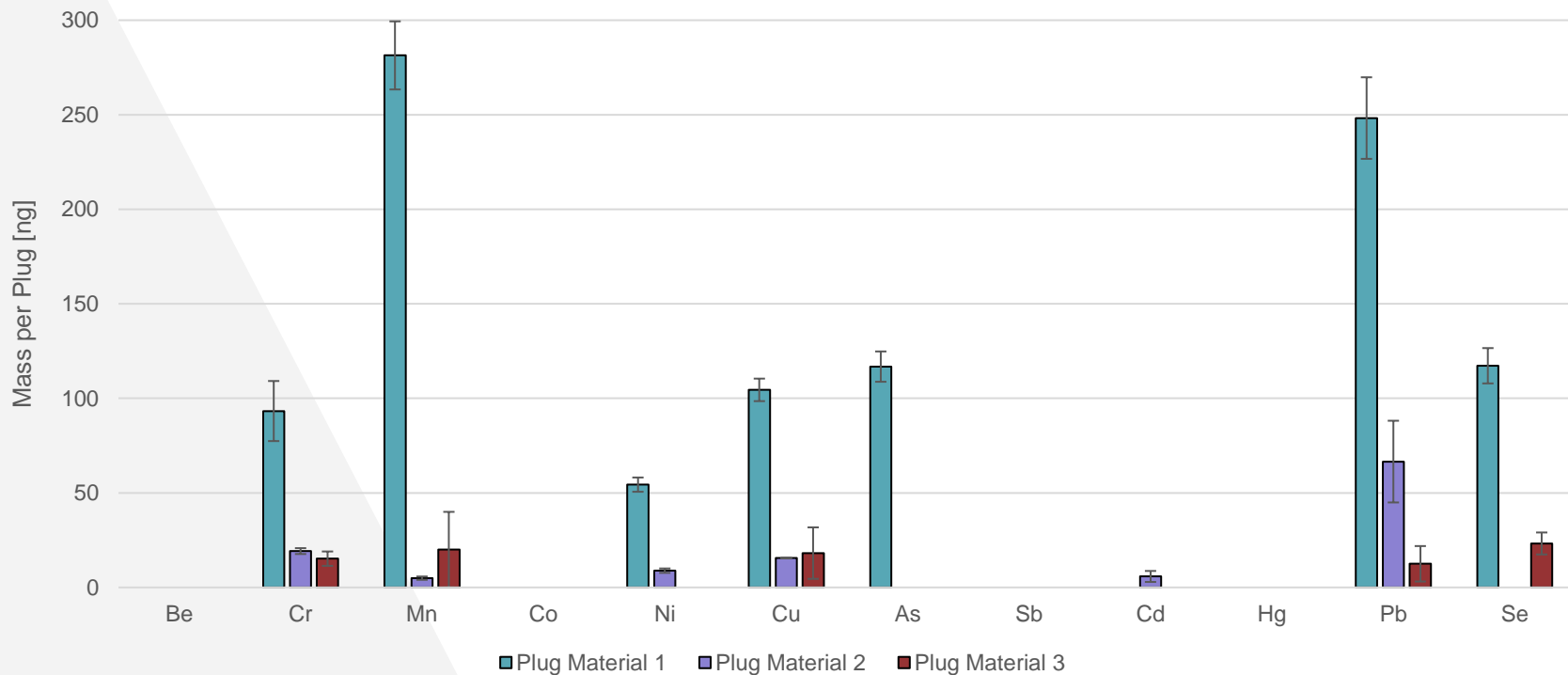
Background – Sorbent Material

Improvement in Background Metals



Background – Plug Material

Plug Material Background Comparison

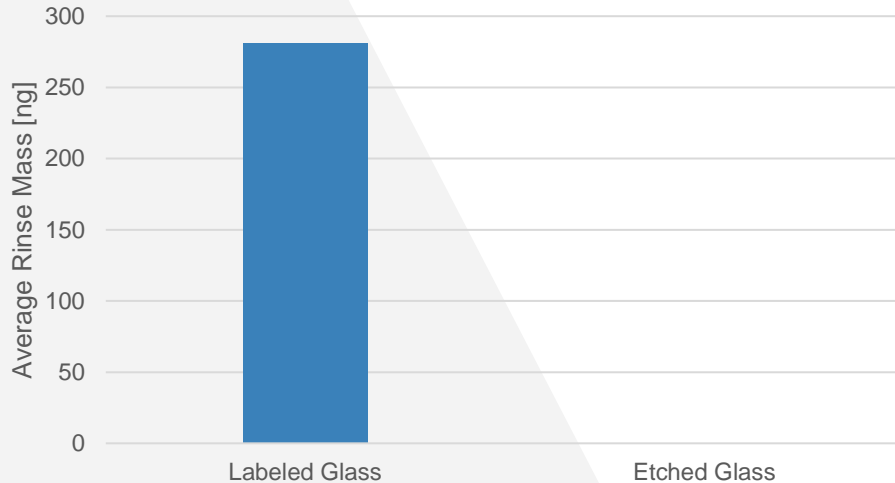


Background – Glass Tubes

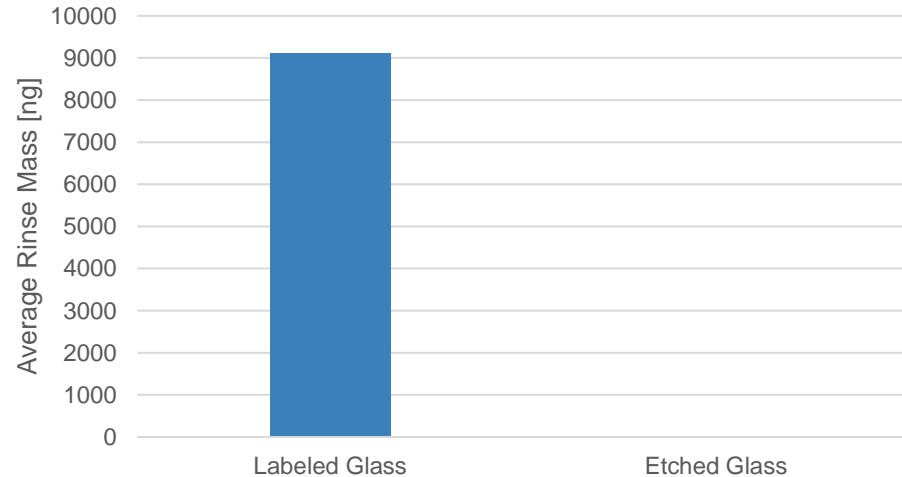
	Average Rinse Mass [ng]	
	Cd	Pb
Labeled Glass	280.8	9115.0
Etched Glass	0	0



Cd in Glass Rinse

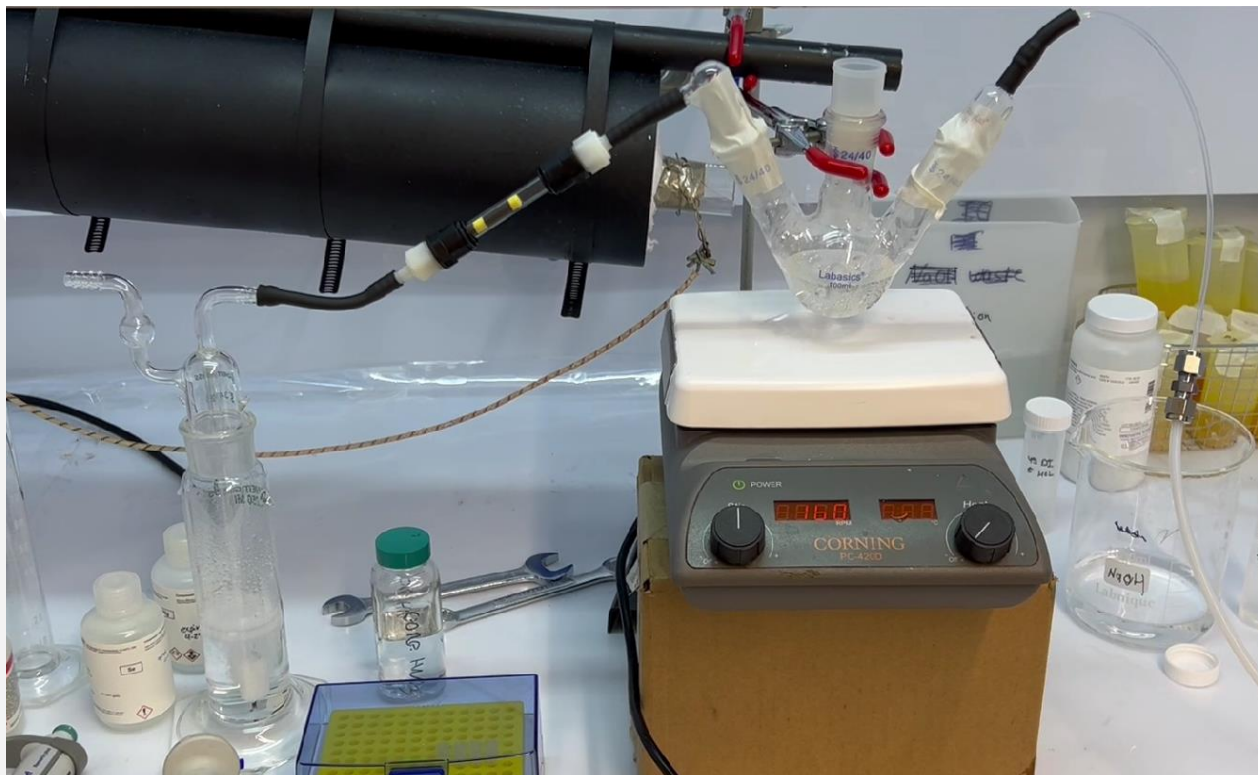


Pb in Glass Rinse



Objective #2 – Optimize Capture Efficiency

Hydride Generation Experiment



Hydride Generation Experiment

Sample ID		Measured Mass (ng)			
		Metal Hydride			
		As	Sb	Hg	Se
TYPICAL BACKGROUND					
Blank	OLM0005 - S1	1.6	2.4	0.0	2.6
	OLM0005 - S2	2.5	3.2	0.0	0.8
Run 1	OLM0004 - S1	10968.9	17778.7	1755.2	5042.5
	OLM0004 - S2	1874.8	124.7	268.6	0.0
	Breakthrough	17.1%	0.7%	15.3%	0.0%
Run 2	OLM0002 - S1	10165.8	15436.3	2388.5	4189.7
	OLM0002 - S2	1802.0	64.4	272.3	0.1
	Breakthrough	17.7%	0.4%	11.4%	0.0%
Run 3	OLM0003 - S1	8063.2	16396.8	2566.4	3511.4
	OLM0003 - S2	1745.2	113.4	155.0	0.0
	Breakthrough	21.6%	0.7%	6.0%	0.0%
Run 4	OLM0001 - S1 (Arsenic only)	13445.7			
	OLM0001 - S2 (Arsenic only)	2446.4			
	Breakthrough	18.2%			
AVERAGE BREAKTHROUGH:		18.7%	0.6%	10.9%	0.0%

Field Validation Test 1 – No treatment

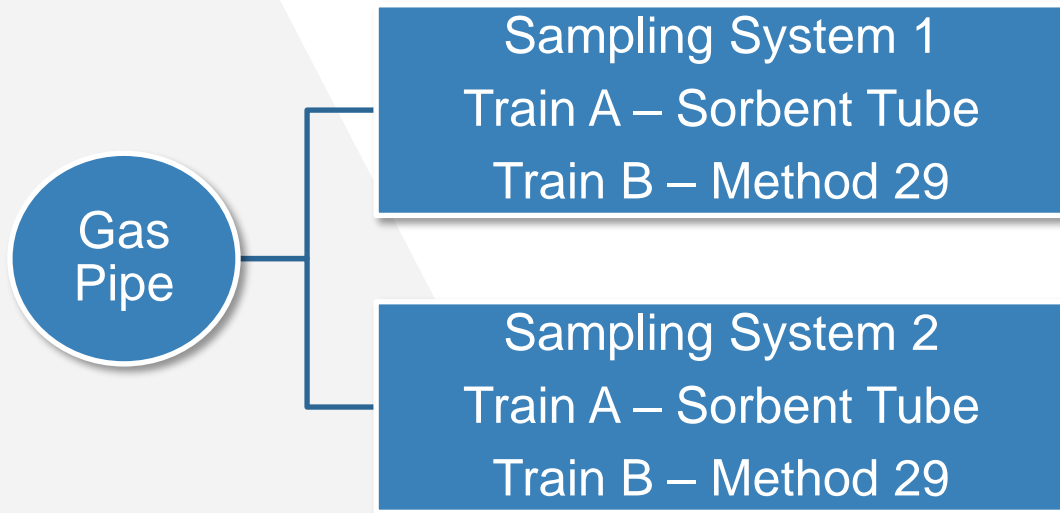
Sample ID	Gas Volume [L]	Concentration [ng/L]				Total [ng/L]				Breakthrough [%]			
		Cu	As	Sb	Pb	Cu	As	Sb	Pb	Cu	As	Sb	Pb
OLM0018-S1	18.83	4.6	161.8	147.3	1.0	7.3	274.3	176.2	2.8	58.6%	69.5%	19.6%	187.4%
OLM0018-S2		2.7	112.5	28.9	1.8								
OL720094-S1	49.00	1.9	50.1	75.0	69.1	8.0	138.1	167.3	78.3	312.7%	175.5%	123.2%	13.3%
OL720094-S2		6.0	88.0	92.4	9.2								
OL720089-S1	22.60	2.0	108.2	138.0	34.9	7.1	289.7	277.3	184.5	260.9%	167.8%	100.9%	429.5%
OL720089-S2		5.1	181.5	139.2	149.7								

Field Validation Test 3

Sample ID	Gas Volume [L]	Concentration [ng/L]		Total [ng/L]		Breakthrough [%]		Notes
		As	Sb	As	Sb	As	Sb	
OLM00133-F	12.08	1.8	3.8	474.6	424.3	0.3%	0.1%	Flow rate = 0.2lpm
OLM00133-S1		471.4	420.0					
OLM00133-S2		1.3	0.4					
OLM00107-F	11.56	2.1	5.1	488.7	425.0	0.3%	0%	Flow rate = 0.2lpm
OLM00107-S1		485.2	419.9					
OLM00107-S2		1.3	ND					
OLM00106-F	35.78	0.8	2.7	470.1	413.3	0.3%	0.1%	Flow rate = 0.5lpm
OLM00106-S1		467.7	410.3					
OLM00106-S2		1.5	0.3					
OLM00108-F	34.56	0.6	1.9	448.3	392.2	0.4%	0.5%	Flow rate = 0.5lpm
OLM00108-S1		445.7	388.3					
OLM00108-S2		2.0	2.1					
OLM00130-F	58.31	0.2	2.2	450.5	421.6	6.9%	0.1%	Flow rate = 1.0lpm
OLM00130-S1		421.3	419.2					
OLM00130-S2		29.0	0.2					
OLM00134-F	58.65	0.3	1.9	431.9	408.0	6.6%	0%	Flow rate = 1.0lpm
OLM00134-S1		404.8	406.1					
OLM00134-S2		26.8	ND					
OLM00102-S1	28.63	400.9	398.2	402.4	398.2	0.4%	0%	No Filter. Flow rate = 0.5lpm
OLM00102-S2		1.5	ND					
OLM00103-S1	30.54	413.0	408.4	414.8	409.9	0.5%	0.4%	No Filter. Flow rate = 0.5lpm
OLM00103-S2		1.9	1.4					
Avg. Relative Deviation						Avg. Breakthrough		
1.9%						1.4%		
						2.0%		0.2%

Objective #3 – Compare with Method 29

Method 29 Comparison - Design



- Analyze all M29 breakthrough impingers
- Minimize M29 dilution
- Sample each quad for same duration
- Pair sorbent traps directly against M29 on each console (eliminate possibility of systematic bias between consoles)

x6

Method 29 Comparison - Data

Sampling Interval	Trap Total [ng/L]		Trap BT [%]		Trap RD [%]		M29 Total [ng/L]		M29 BT [%]		M29 RD [%]		RPD%	
	As	Sb	As	Sb	As	Sb	As	Sb	As	Sb	As	Sb	As	Sb
09/10/2024 11:39-12:39	385.0	359.6	1.5%	3.8%	6.1%	3.6%	316.5	335.8	70.8%	59.7%	4.1%	3.0%	19.5%	6.9%
09/10/2024 11:39-12:39	434.8	386.8	1.1%	1.5%			291.8	316.4	60.3%	47.4%			39.4%	20.0%
09/10/2024 13:40-14:40	369.0	349.9	1.8%	1.6%	11.4%	10.4%	314.5	320.3	60.7%	47.1%	1.3%	1.5%	15.9%	8.8%
09/10/2024 13:40-14:40	463.7	431.3	1.7%	2.4%			306.4	330.2	55.9%	49.1%			40.9%	26.5%
09/10/2024 15:37-16:37	398.0	361.7	2.5%	1.7%	2.9%	3.7%	337.7	331.0	72.2%	61.4%	4.1%	2.5%	16.4%	8.8%
09/10/2024 15:37-16:37	421.5	389.1	1.6%	2.1%			311.4	315.0	59.3%	40.3%			30.1%	21.1%
09/10/2024 17:31-18:31	394.8	372.2	1.4%	4.8%	1.0%	1.7%	319.5	326.5	51.6%	42.4%	5.1%	3.4%	21.1%	13.1%
09/10/2024 17:31-18:31	402.4	384.8	3.3%	2.9%			354.1	349.7	63.7%	61.3%			12.8%	9.6%
09/10/2024 19:26-20:26	413.3	372.8	2.9%	4.0%	2.7%	2.6%	313.2	309.5	73.2%	61.3%	3.0%	1.7%	27.6%	18.6%
09/10/2024 19:26-20:26	436.4	392.4	3.0%	1.3%			332.5	320.4	46.1%	57.0%			27.0%	20.2%
09/10/2024 21:18-22:18	427.5	387.5	5.2%	0.7%	0.9%	2.9%	304.3	298.8	67.5%	52.5%	5.6%	3.4%	33.7%	25.8%
09/10/2024 21:18-22:18	420.1	365.8	4.4%	3.2%			340.4	320.1	66.6%	63.3%			21.0%	13.3%
AVERAGE	413.9	379.5	2.5%	2.5%	4.1%	4.1%	320.2	322.8	62.3%	53.6%	3.9%	2.6%	25.4%	16.1%

Extrapolation Assuming Breakthrough through Impinger 2

Impinger 1 Avg:	197.3	210.2
Impinger 2 Avg:	122.9	130.9
Lost to Atmosphere:	76.6	81.6
Total Conc:	396.8	422.7
RPD%	2.1%	5.4%

Method Status

▷ **Available for Use Now**

▷ **ASTM Standard**

- WK91049 has been drafted and is currently going through balloting/revision process
- Includes sampling and analysis criteria
 - Analysis is done by ICP-MS following microwave assisted digestion
- Includes criteria for developing an alternative to Ohio Lumex sorbent material
- Expect publication some time in 2025

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Q&A



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Questions