ITEX

Ultra-Trace Volatile Organic Compounds (VOCs) Analysis Methodology to Achieve Low Level Regulatory Requirements

RemTech 2012

Overview

1. Drivers for lower VOC DLs
2. Static Headspace Theory
3. ITEX Solution

Detection Limit Trend

Analytical Pricing Trend

Regulatory Environment

Why are VOCs Regulated?

- Protection of human health from subsurface VOCs

VOC Regulations:

- CCME, AB Tier 1, BCMOE, Risk Based

What is the problem?

- Ability to achieve low DLs (e.g. AB Tier 1) for some VOCs using conventional equipment and techniques
- Data is being reported **at** the lower limit of detection
- Method modifications can "*theoretically*" achieve DLs but not always the best science for all samples. (i.e. direct soil purge)

Regulatory Environment

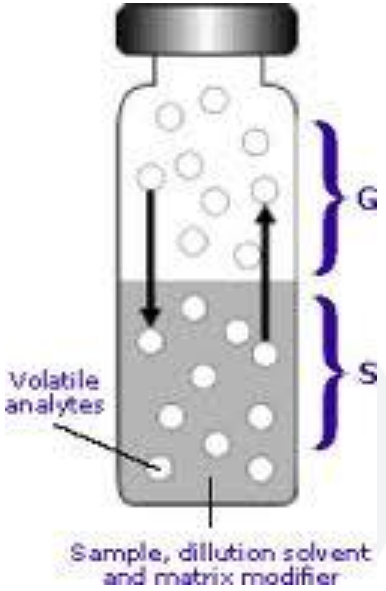
Analyte	Lowest ABT1 Reg (mg/kg)	Lowest CCME Reg (mg/kg)	Lowest BCMOE (mg/Kg)	MeOH RDL (mg/kg)	Direct RDL (mg/kg)
1,1-Dichloroethene	0.021	0.1	-	0.05	0.005
1,2-Dichlorobenzene	0.097	0.1	0.1	0.05	0.005
1,2-Dichloroethane	0.0027	0.1	-	0.05	0.005
1,4-Dichlorobenzene	0.051	0.1	0.1	0.05	0.005
Benzene	0.046	0.0068	0.04	0.03	0.002
Carbon Tetrachloride	0.00056	0.1	0.1	0.05	0.005
Chlorobenzene	0.018	0.1	0.1	0.05	0.005
Chloroform	0.001	0.1	0.1	0.07	0.005
Dichloromethane	0.048	0.1	0.1	0.50	0.02
Ethylbenzene	0.11	0.018	1	0.05	0.005
Tetrachloroethene	0.16	0.1	0.1	0.05	0.005
Toluene	0.49	0.08	1.5	0.20	0.005
Trichloroethene	0.012	0.01	0.015	0.01	0.005
Vinyl Chloride	0.00034	-	0.79	0.10	0.005

Headspace Theory

- **Instrumentation:**
  - Headspace with GC/MS
  - At the limit of conventional technology.

- **Sample Introduction:**
  - Purge and Trap
  - Static Headspace

- **Important Variables:**
  - Soil Partitioning (getting VOC out of soil)
    - MeOH Extaction vs. Direct. Analysis
  - Partition Coefficient (K) = C_s/C_g
    - Low K = easier partitioning
    - Heat & matrix modifiers
  - Phase Ratio (β) = V_g/V_s



Headspace VOC Analysis

What can be done to achieve lower DLs?

1. Improve GC/MS Instrument Sensitivity

- Limited by current technology; cost; contamination; carry-over

2. Increase Sample Volume

- MeOH extraction
- Direct purge – extraction efficiency questionable

3. Increase Headspace Aliquot to GC/MS

- Too much volume onto GC/MS can degrade chromatography.

ITEX Approach (#3) - concentrates headspace before injecting to GC/MS

Headspace VOC Analysis

ITEX Apparatus

ITEX Trap &
Syringe Unit

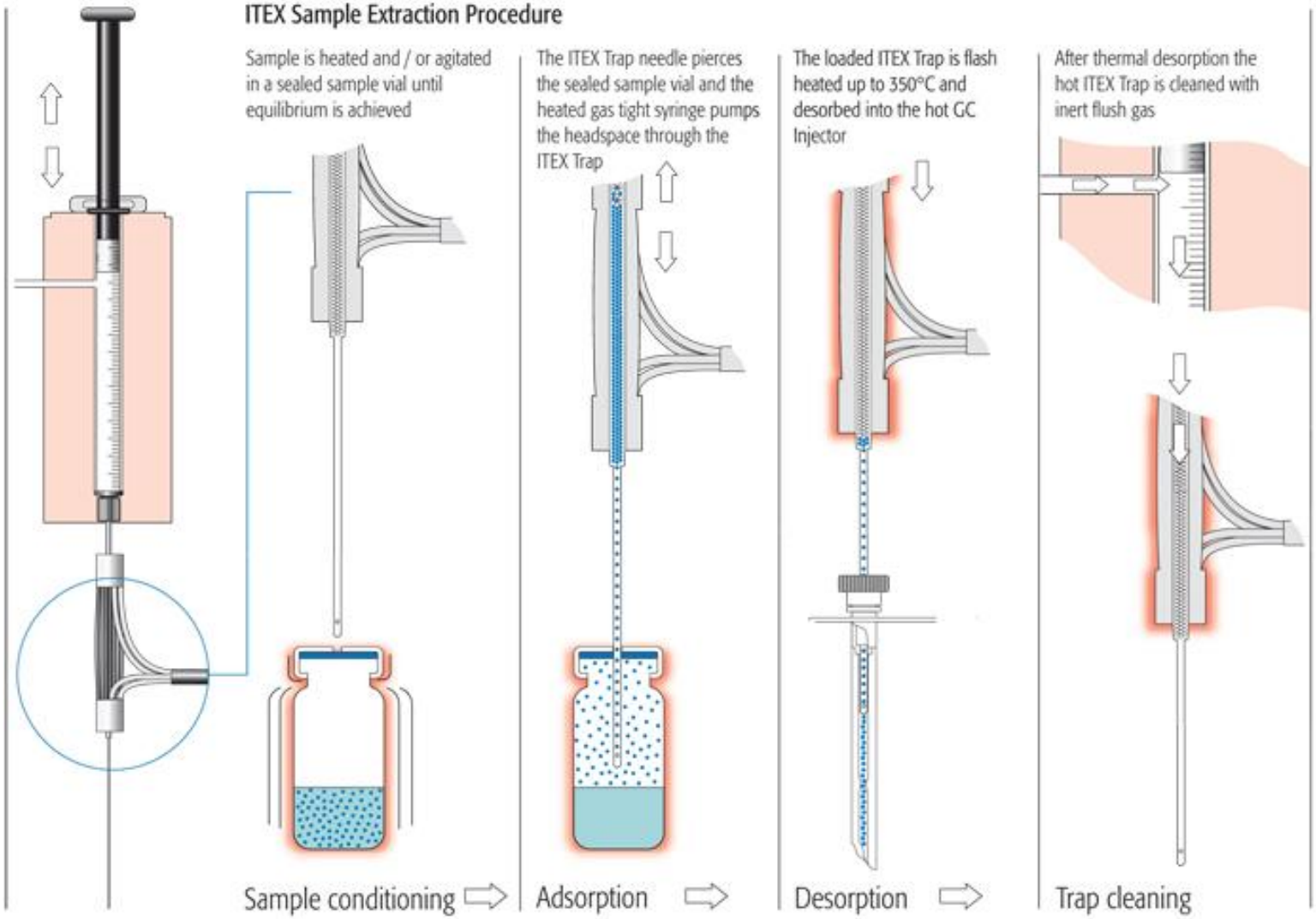
Sample Agitator
& Heater

Heated Trap

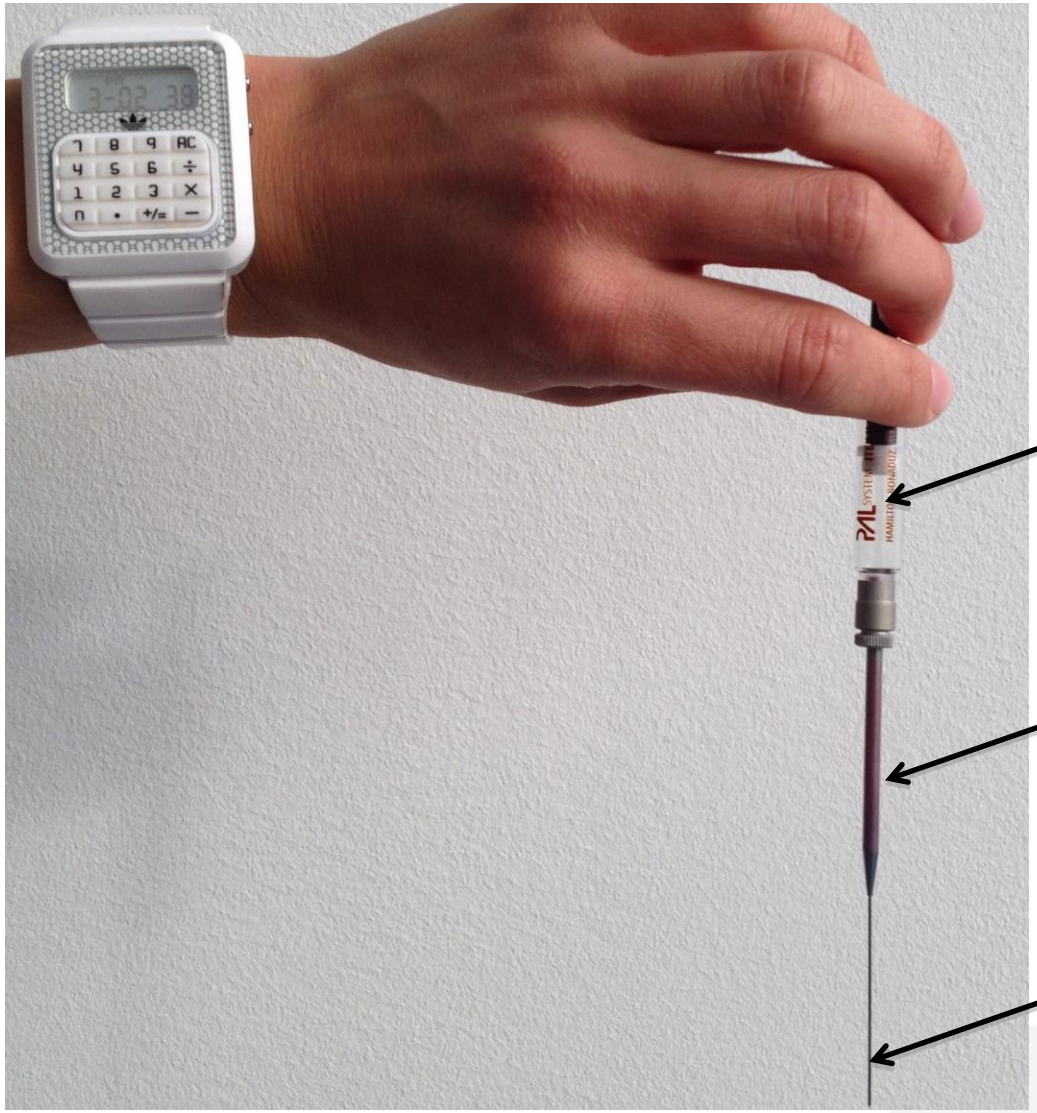
Heated GC Injector

Headspace VOC Analysis

How does ITEX work?



ITEX Syringe



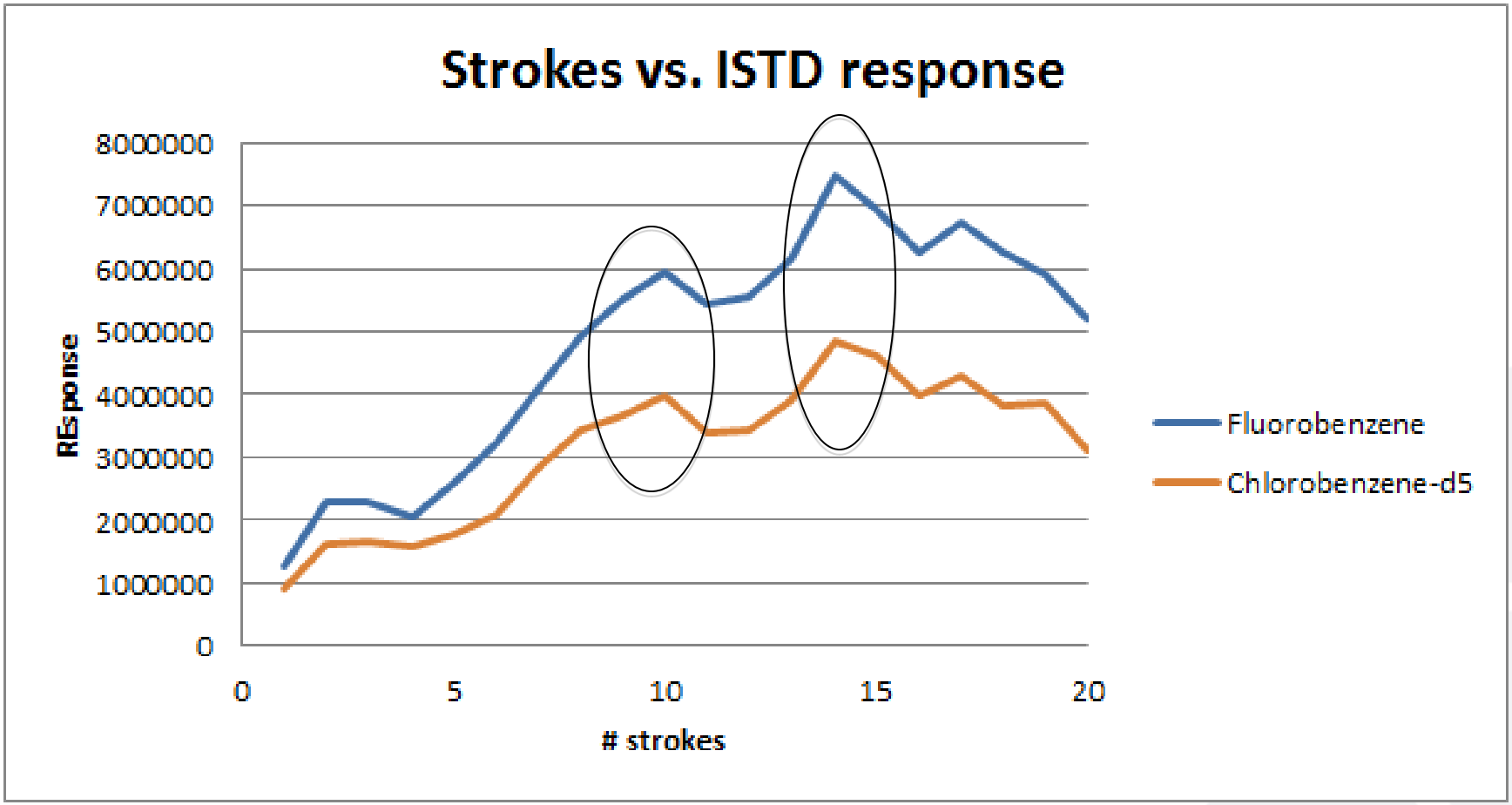
ITEX VOC Analysis

1. Validation Approach

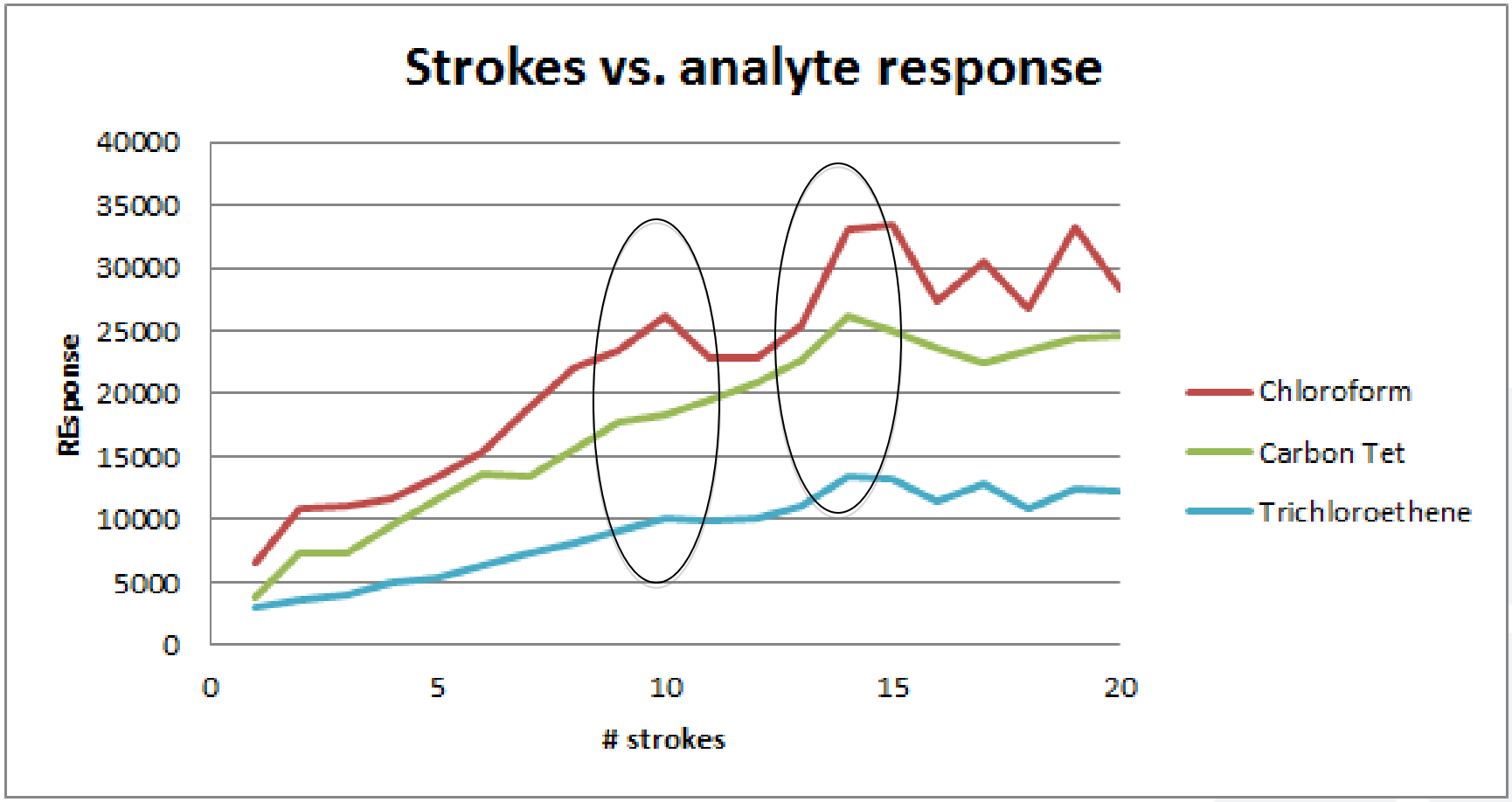
1. Method/Parameter Optimization
2. MDL Study
3. Precision Study
4. Accuracy Study
5. Carry over study

1. Findings

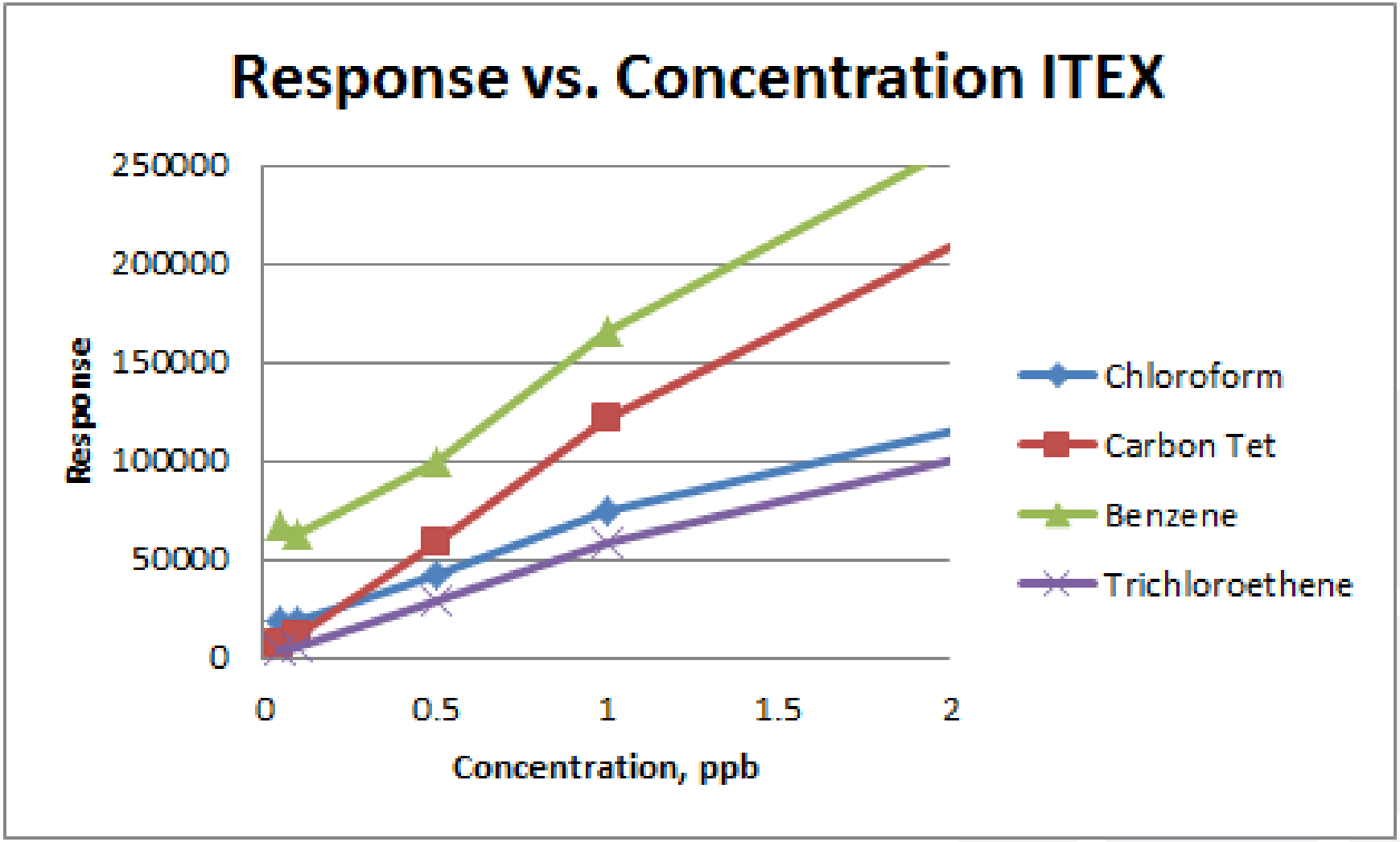
Method Optimization



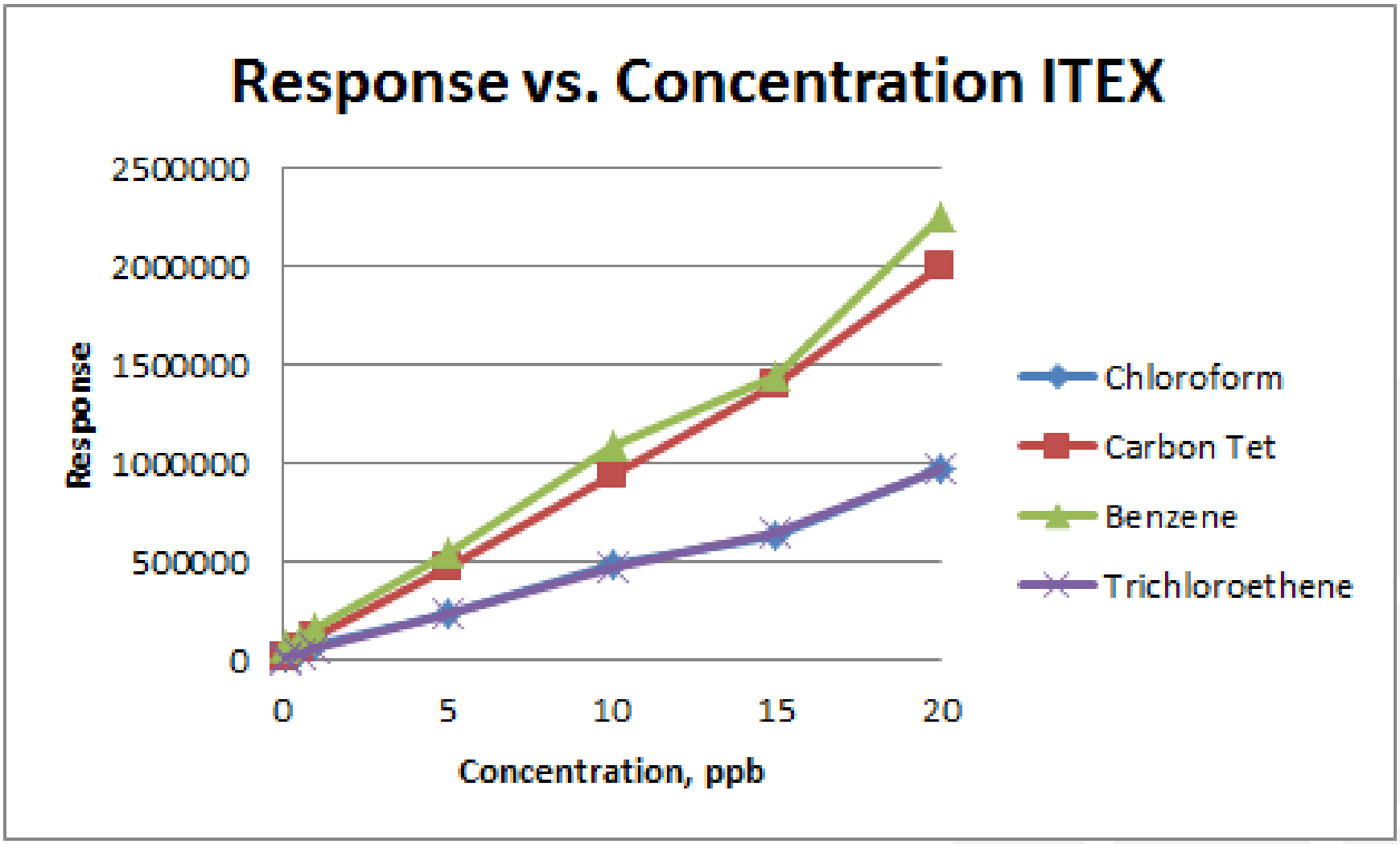
Method Optimization



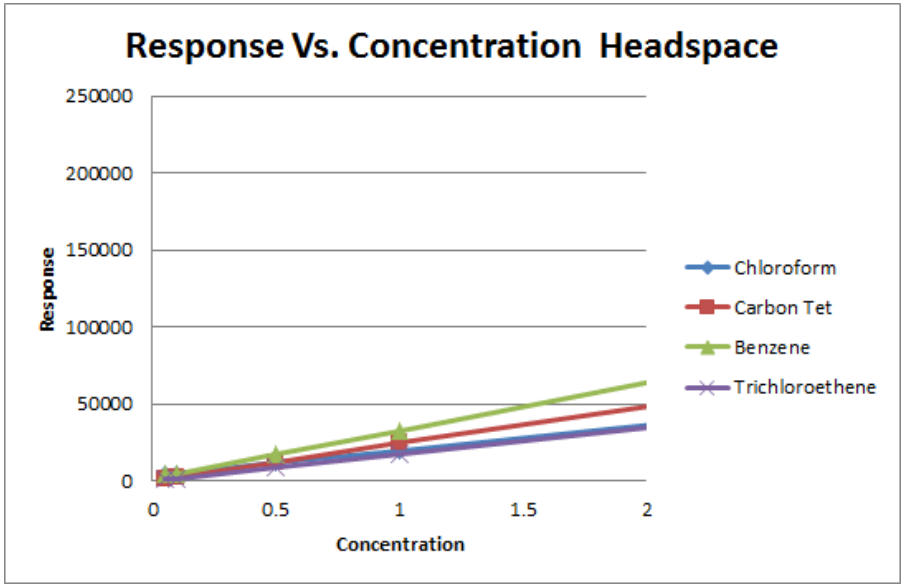
ITEX Linearity – Low Level

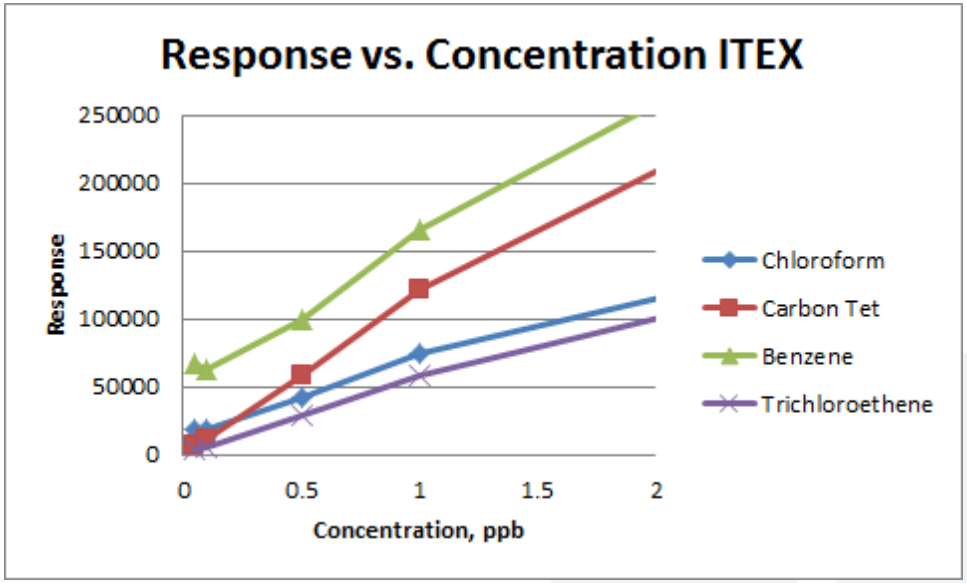


ITEX Linearity– Broad Range



Response Comparison– Low Level





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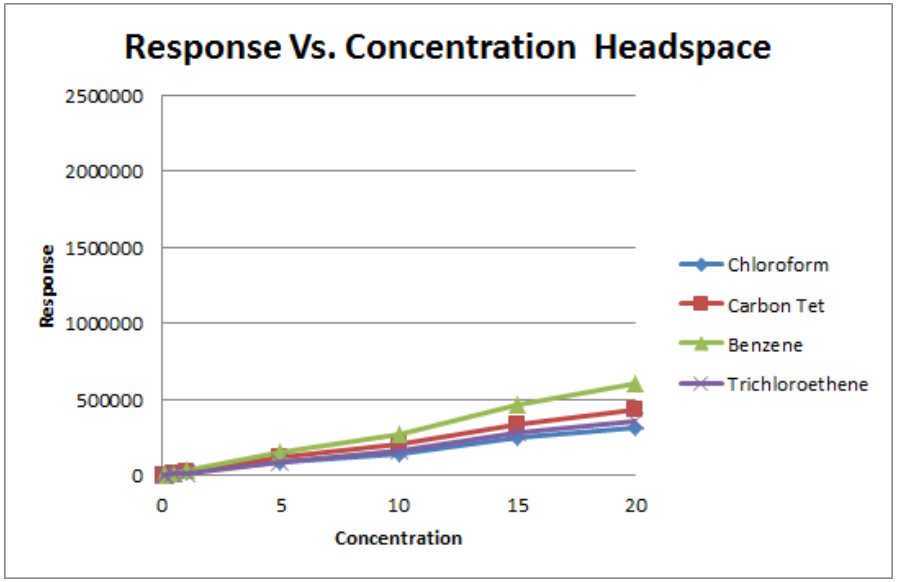

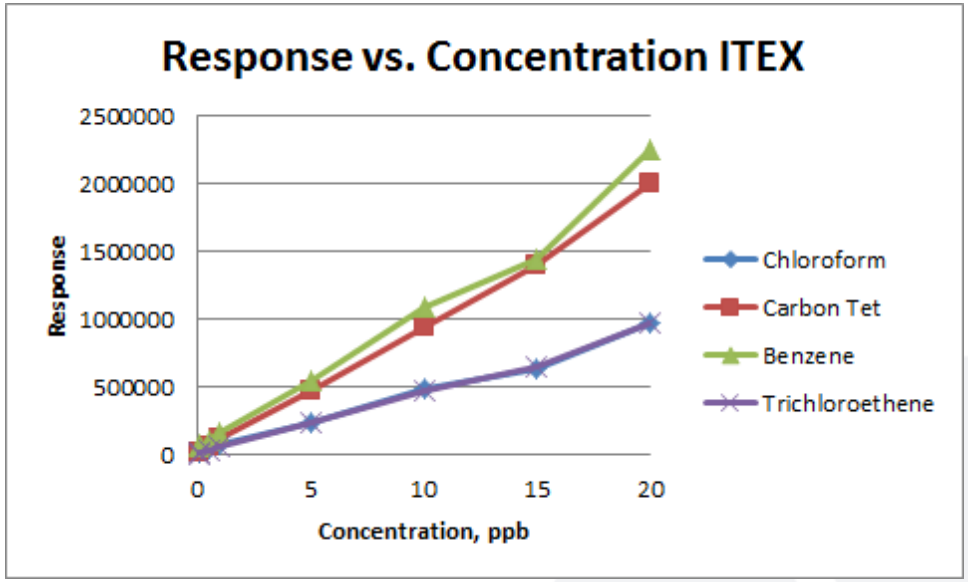

Findings

ITEX is able to increase VOC sensitivity 4-7 times that of conventional headspace analysis.

Challenges:

1.Low MW VOCs (gasses)

- Poor chromatography – peak splitting, broad peaks, etc.
- Possible Solutions:
  - Cryo-cooling – to improve sample introduction focus
  - Trap Optimization - single vs. multi-bed; adsorbents selection

2.Contamination Amplification

- ITEX concentration “amplifies” contamination affects
- Ultra-clean conditions - prep, environment, reagents

3.Partitioning Consistency Vs. # of Strokes

Next Steps

1. Asses optimum trap design
2. Assess cryo-cooling
3. Can we load more onto trap consistently?

Heading

“*Back To The Future*”

Conclusion

The low level VOC regulations forced comprehensive understanding of science, regulatory requirements, client needs.

We are making meaningful and practical improvements to the state of the art**.**

…. focusing on the science to support our industry.

Thanks

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