EPA Method 625 SPE Validation Study – A New Approach

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Study Participants

Under a program organized and supported by the Independent Laboratory Institute (ILI), a broad coalition representing government, the commercial analytical laboratory community, the technology innovation community and academia worked together to develop a generic protocol for the use of Solid Phase Extraction (SPE) as a technique for concentrating chemical contaminants in aqueous samples for organic chemical analysis.

Study Objectives

- Establish a generic SPE protocol for the validation of Solid Phase Extraction for test methods
- Have embed into the protocol, the proper QC elements necessary to flag any individual sample or product failings.
- Apply that protocol in a blind feasibility study involving multiple segments of the laboratory and vendor community.
- Evaluate that data suitability and study parameters for usage to validate Solid Phase Extraction in test methods.
- Develop a fluid protocol to be used as a template in the application of future methods.

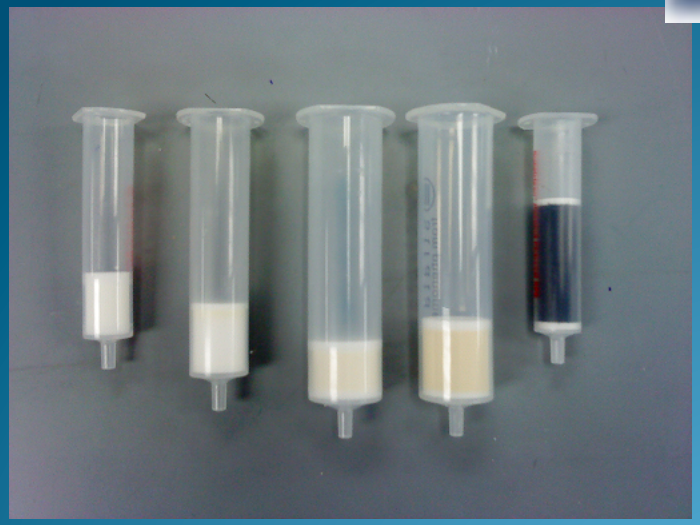
Study History

- Coalition began meeting in 2012
- Began a comprehensive review of existing Vendor SPE applications and EPA method procedures
- Examined the analyte lists found in EPA 625 and cross referenced those individual analytes with optimal sorbent types, pH requirements and other extraction requirements.
- Examined the various different SPE platforms and technologies available on the market.

Study Overview

- The complete study was comprised of two different Phases.
- Each Phase was designed to improve efficiency and the performance for EPA Method 625.

SPE Product Types



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Study Participant Contribution

- Over 18 individual products/techniques tested
- 27 Contributing Labs
- Over 100 different extractions and analyses completed
- Hours of data analysis and review

Study Protocol

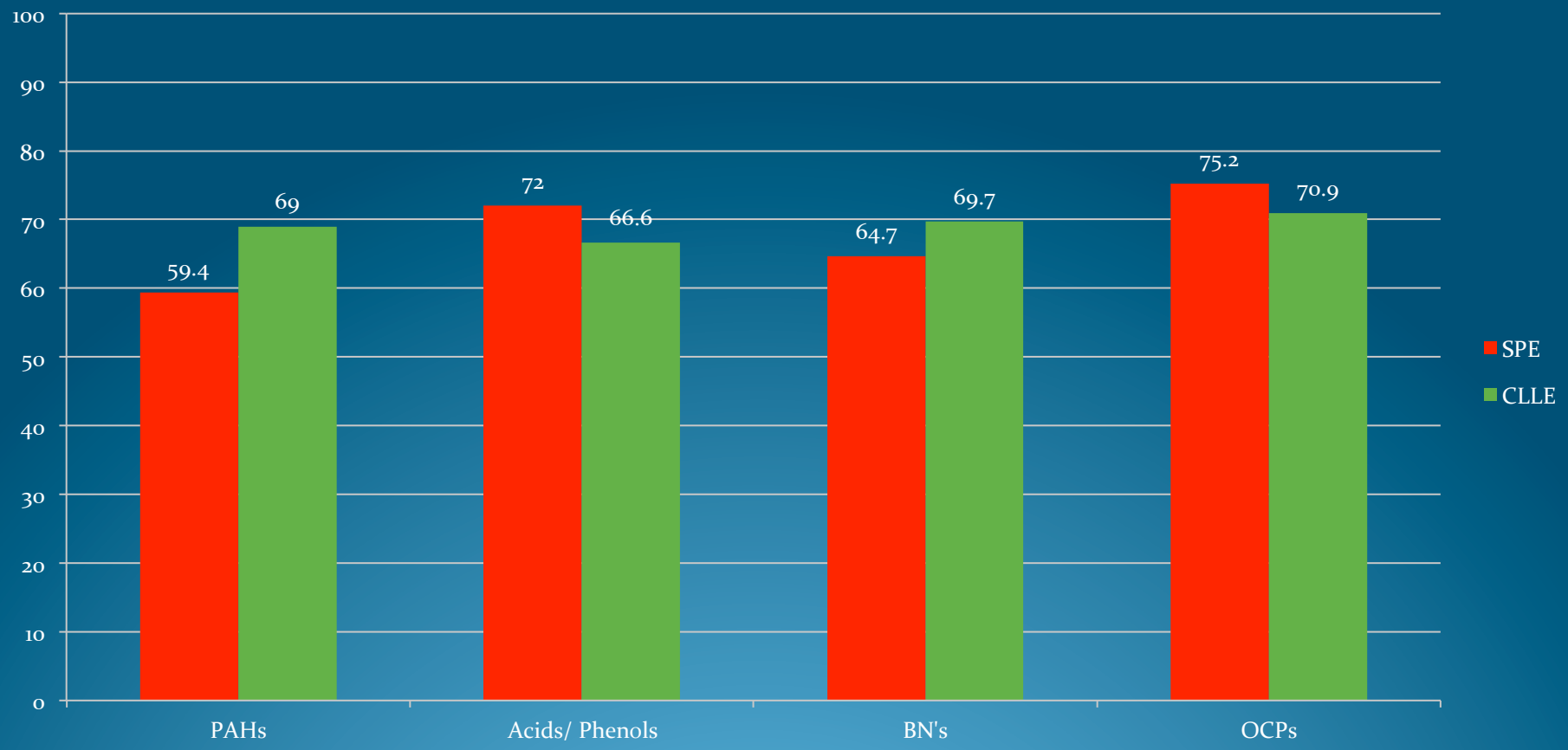
- Focusing on the analytes from Tables #1 and #2 from EPA 625
  - Additionally OCPs were an optional add on
- Establish a blind Round Robin study
- Require 3 participating labs per product tested
- RR samples to be analyzed in both a clean matrix (DI), TCLP Extraction Fluid # 1 and a synthetic waste water matrix (ASTM D5905)
- Surrogate spikes provided (P2)

Phase 1

Phase One Objectives

- Determine the performance of a board spectrum of market available SPE products in a standardized waste water matrix.
- Compare data from SPE products to current Liquid-Liquid Extraction (LLE) performance.
- Evaluate the data – Does SPE work as well as LLE

Phase 1 Analyte Category Data



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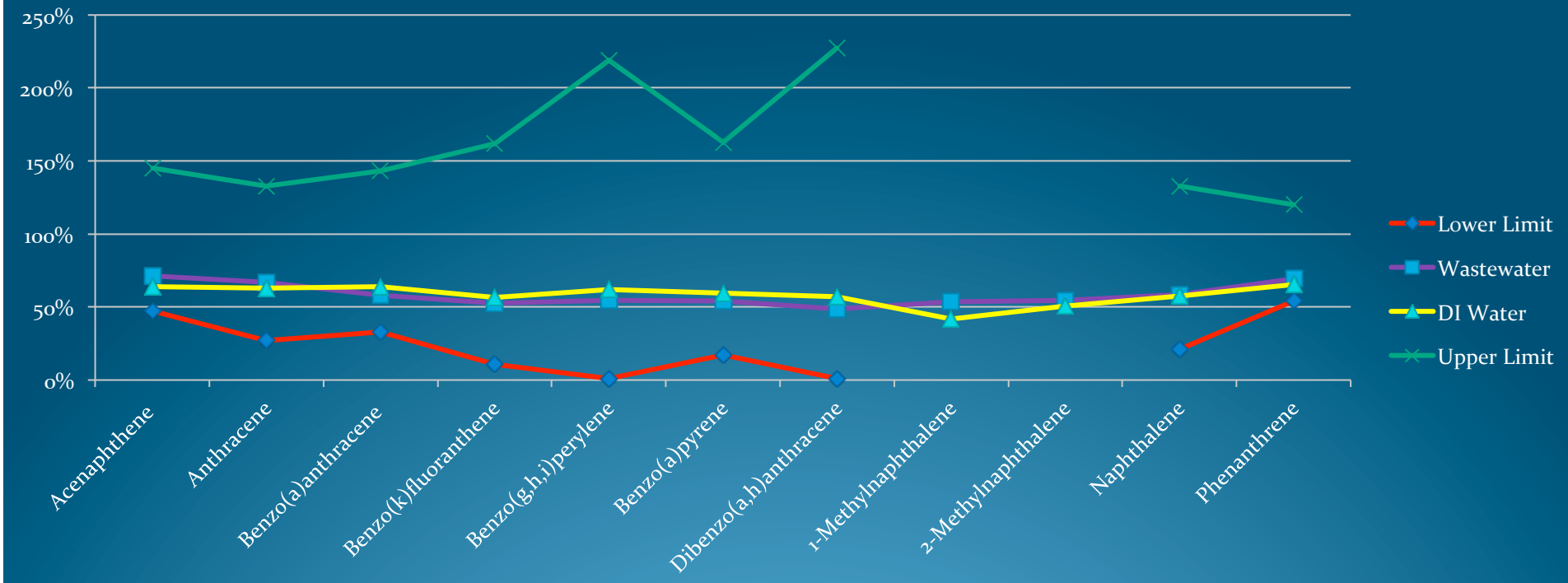
PAHs Waste Water vs. DI Water



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Phenol/Acids
Waste Water vs. DI Water

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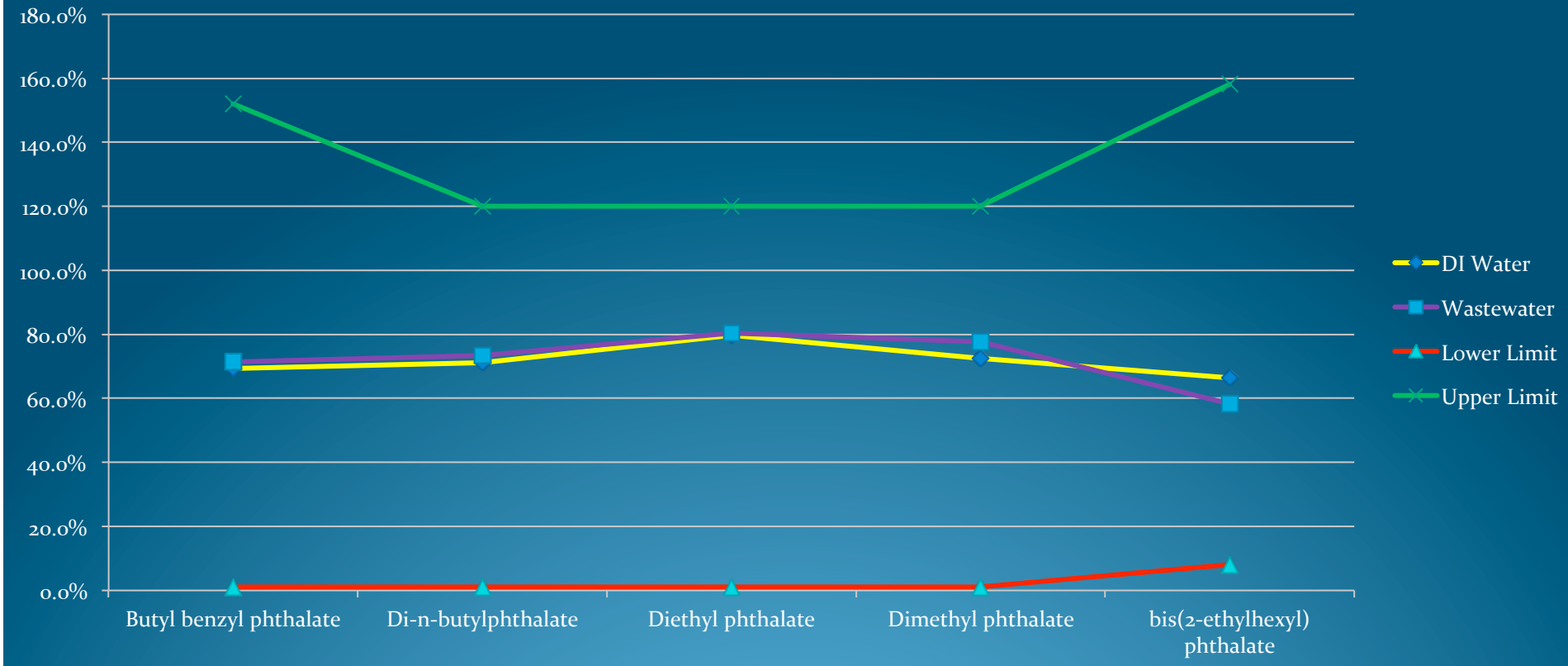
Phthalates
Waste Water vs. DI Water



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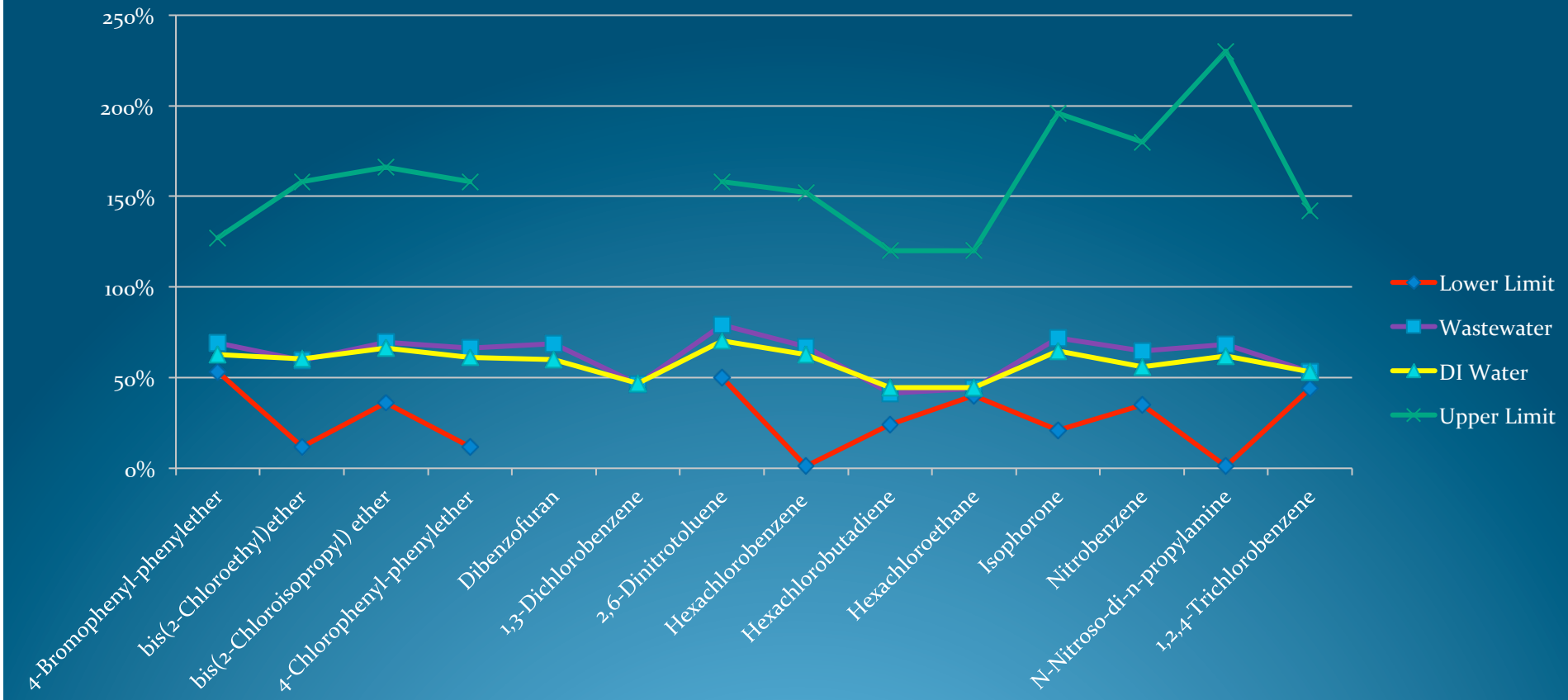
Base/Neutrals
Waste Water vs. DI Water



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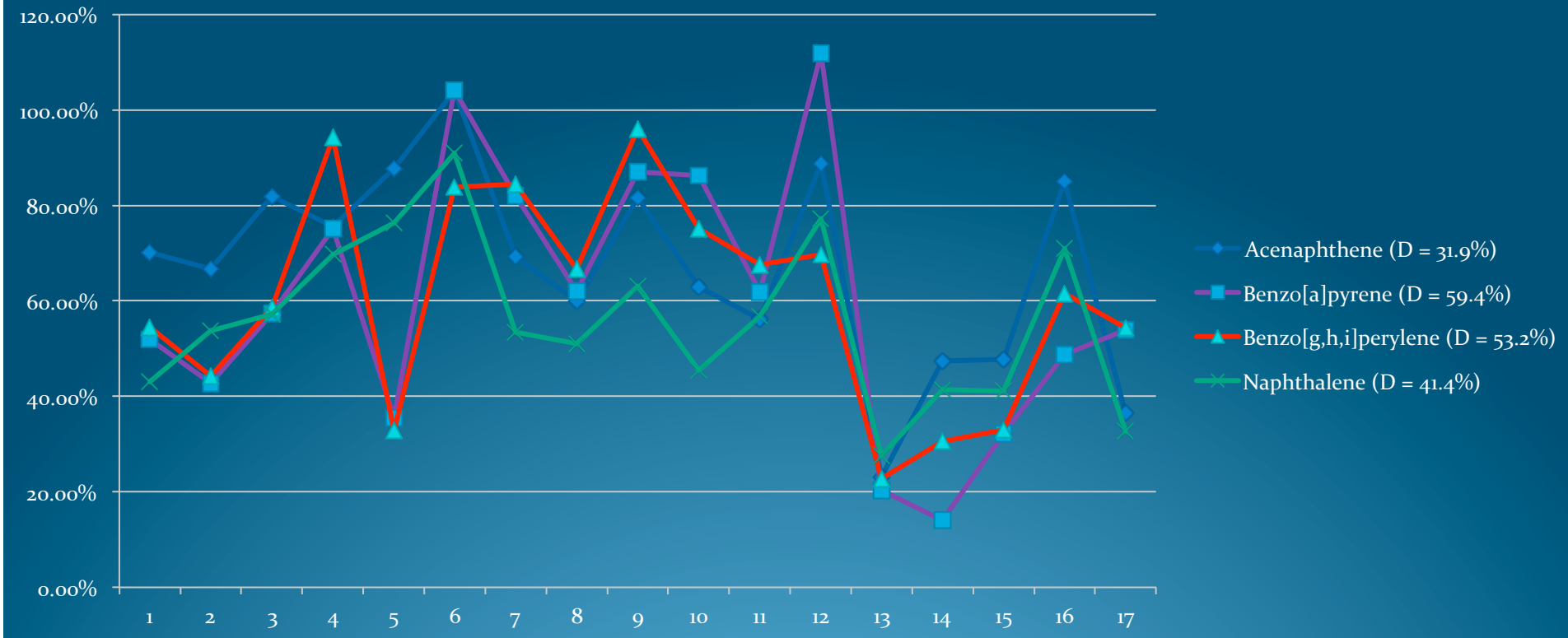
PAH Variability



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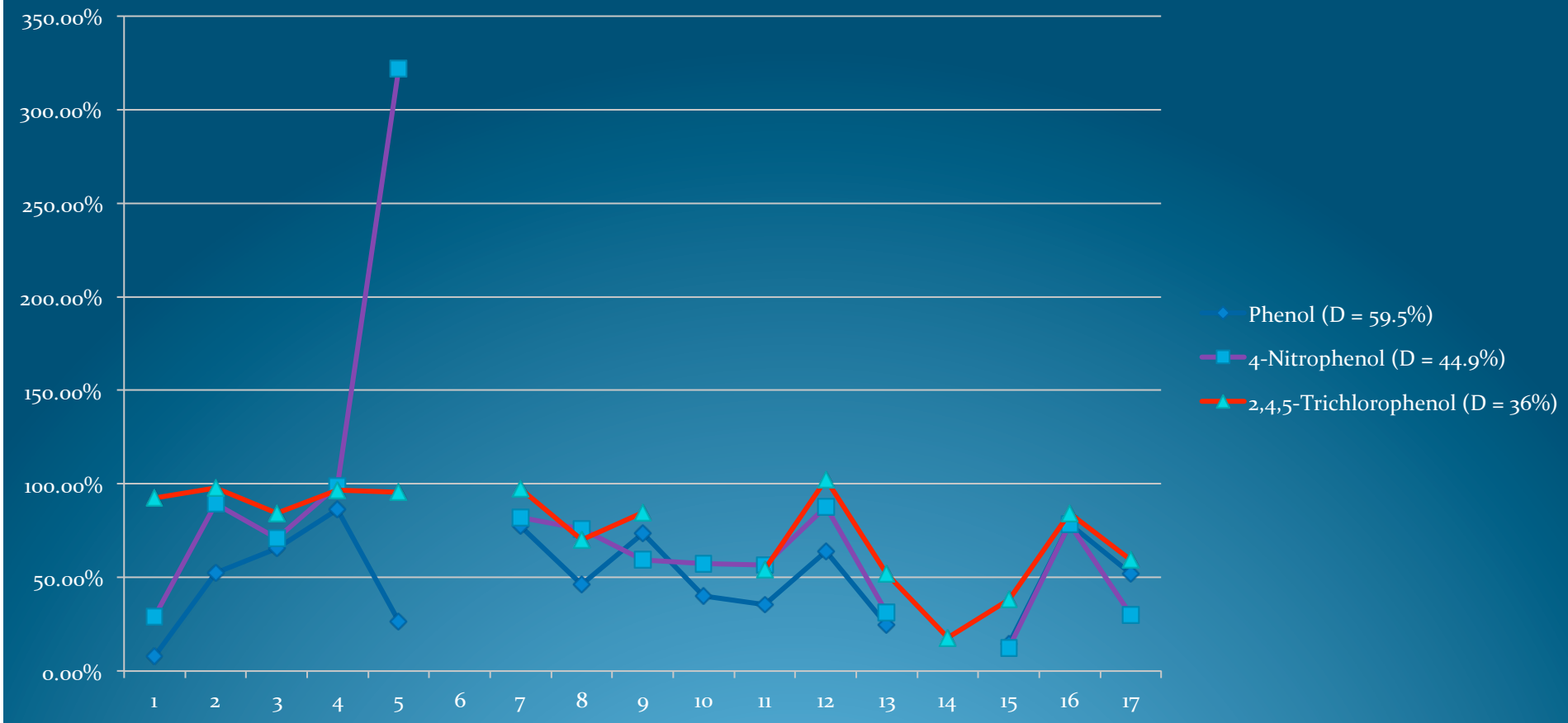
Phenol Variability



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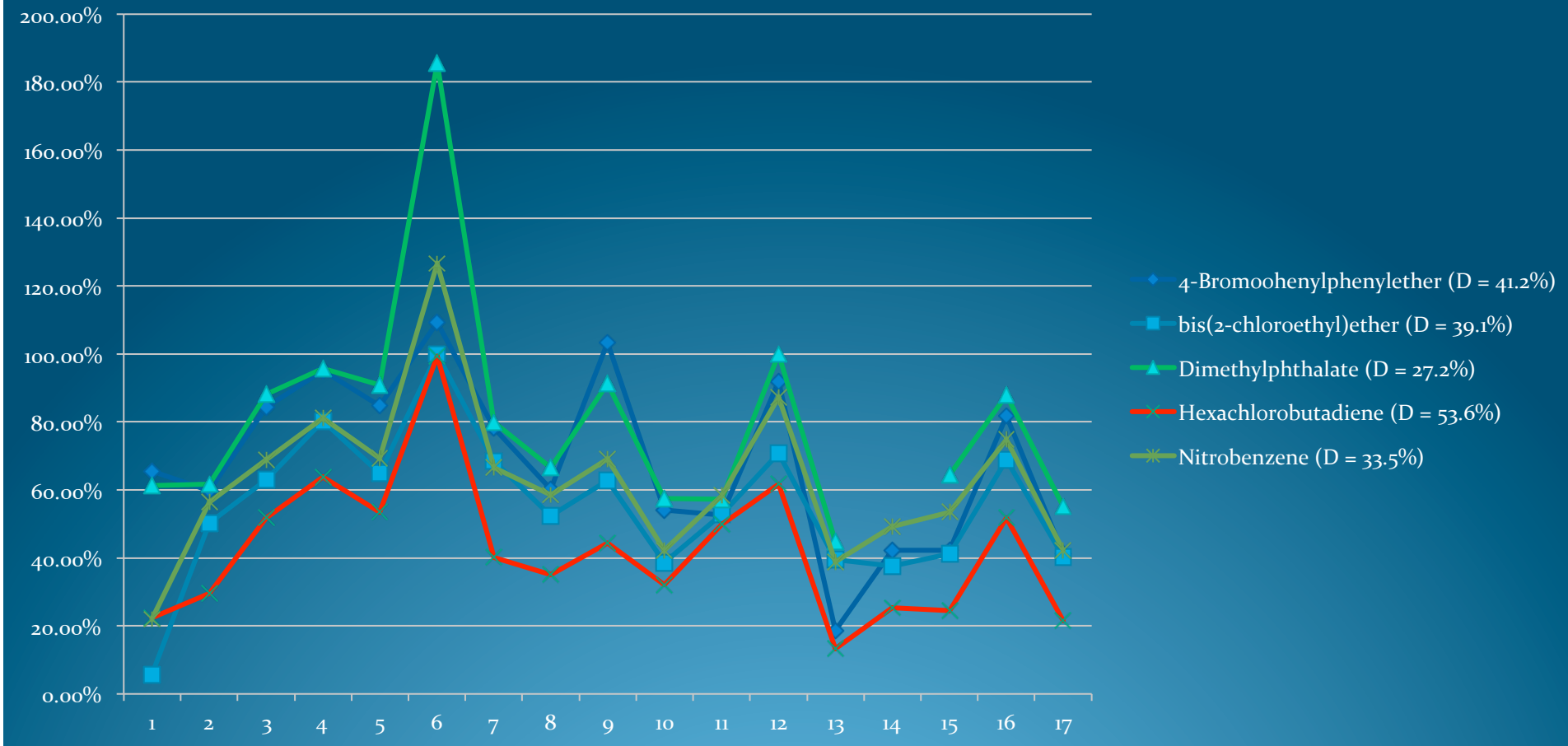
Base/Neutral Variability



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Phase 1 Conclusions

- Data demonstrates the across the wide variety of analytes SPE products tested are as accurate as traditional LLE.

- Study results were within the current method criteria for EPA 625 and within the acceptance limits in the TNI FoPT tables.

HOWEVER….

- Issues were noted with the surrogates that did not demonstrate the failure of an extraction or product.

- Rigorous quality control to allow laboratories to know of a potential issue was need provided with the current surrogate list.

- Answer – Phase 2

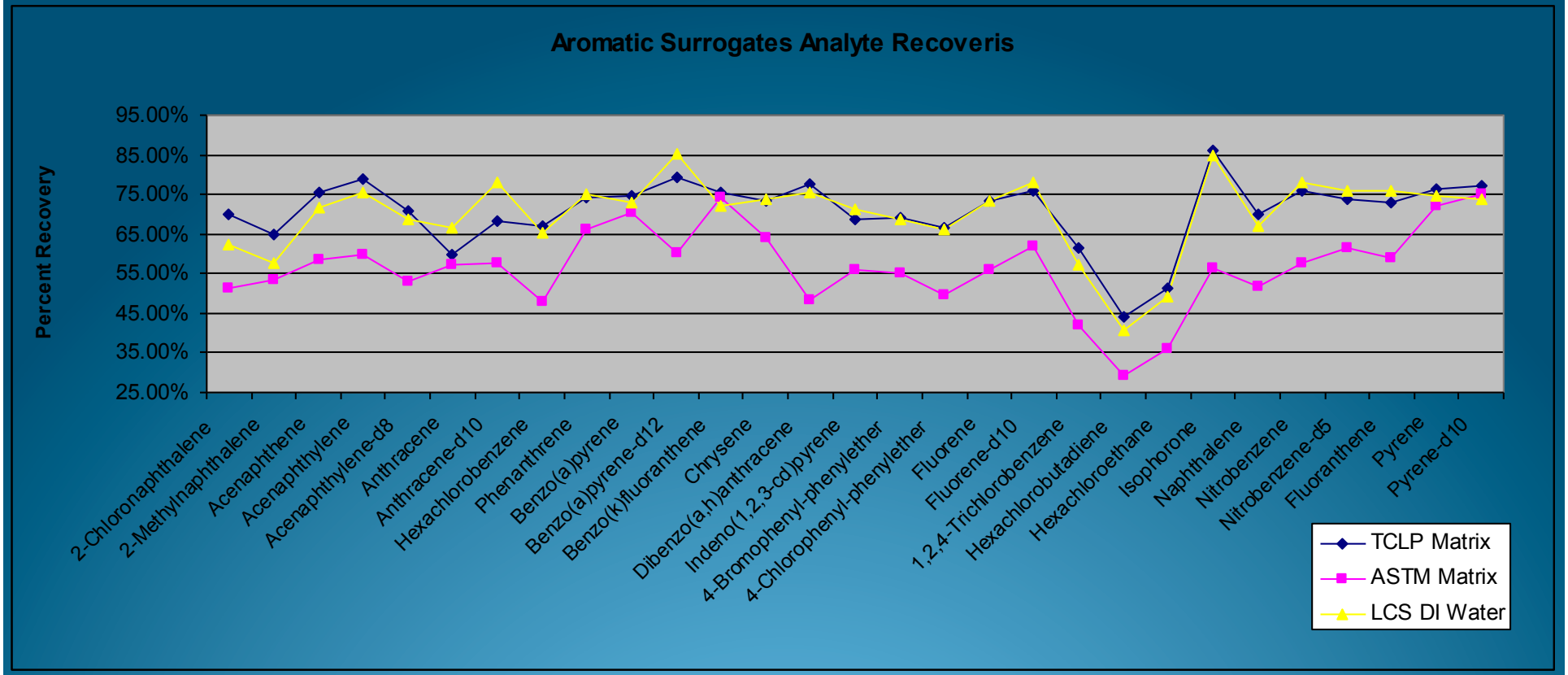
Phase 2

Phase 2 Objectives

- Provide more vendors to participate in the study.
  - ASTM Waste Water matrix provided again.
- Provide a second challenge matrix
  - EPA Method 1311 (Toxicity Characteristic Leaching Procedure or TCLP)
  - Evaluate the results of the new challenge matrix
- Provide a new set of surrogate compounds for evaluation.
  - Evaluate the new surrogate list to analyte recovery
  - Do the surrogates provide the intended quality assurance?

Surrogate Analyte Recoveries Matrix Comparison

Aromatic Surrogate Analyte Recoveries Matrix Comparison



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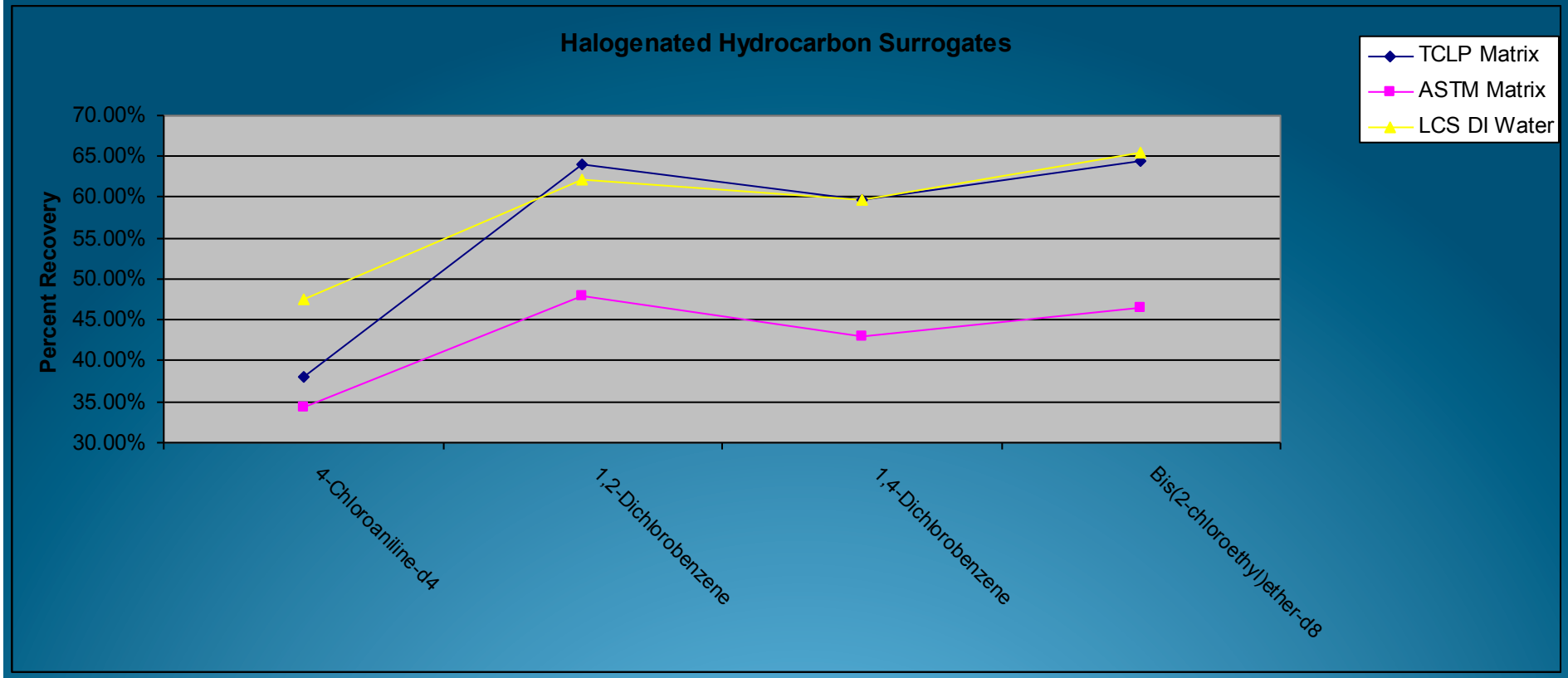
Halogenated Hydrocarbon Surrogate Analyte Recoveries Matrix Comparison



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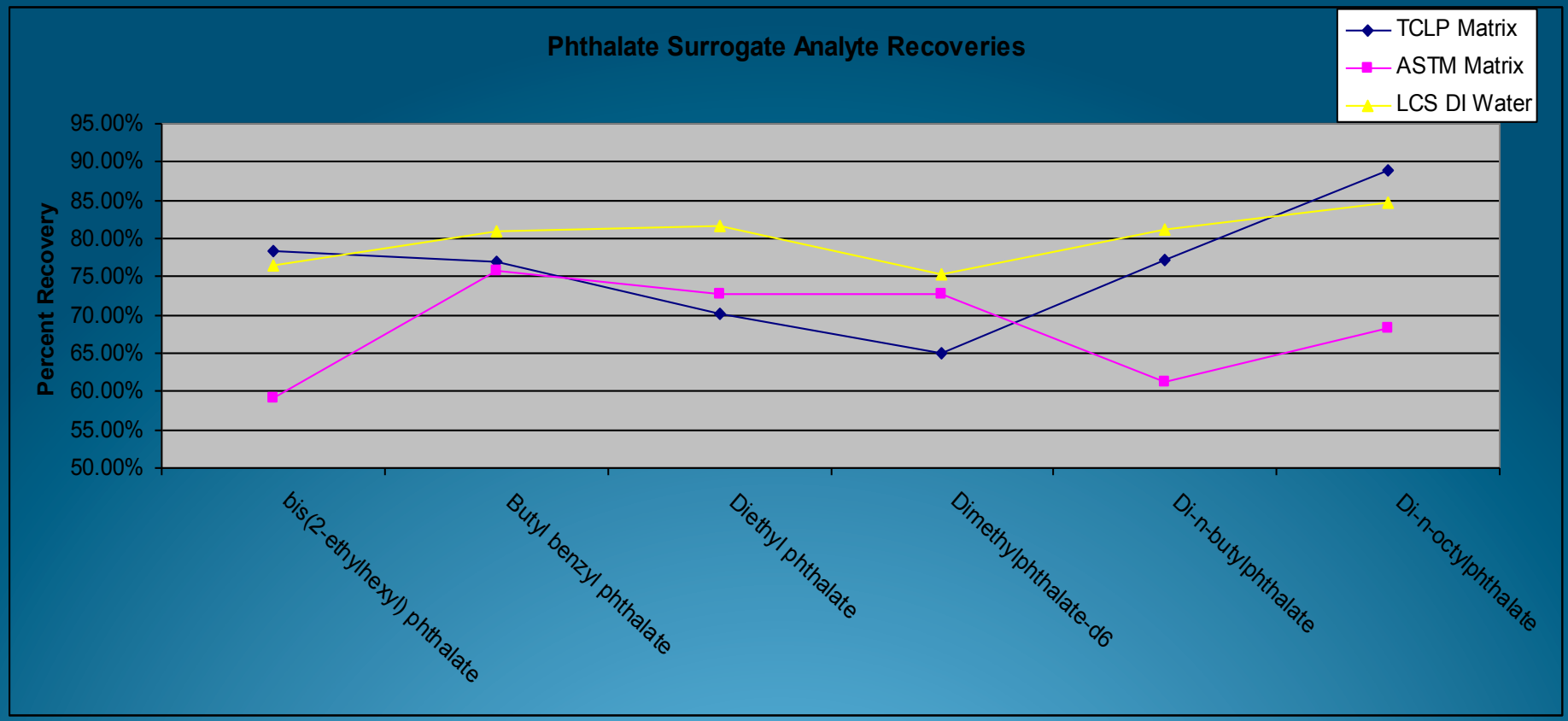
Phthalate Surrogate Analyte Recoveries Matrix Comparison



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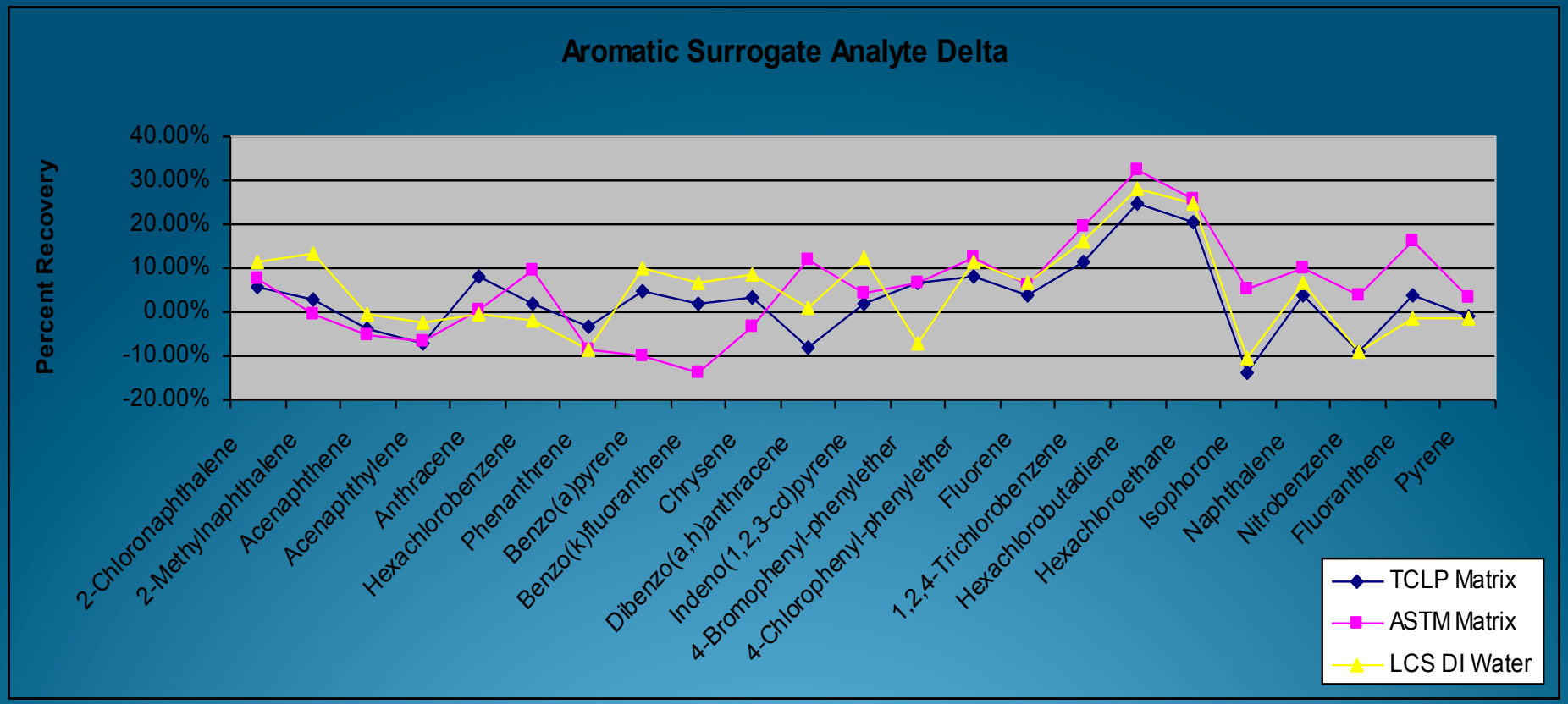
Surrogate Analyte Recoveries Delta Across Matrix Comparison

Aromatic Surrogate Analyte Recovery Delta



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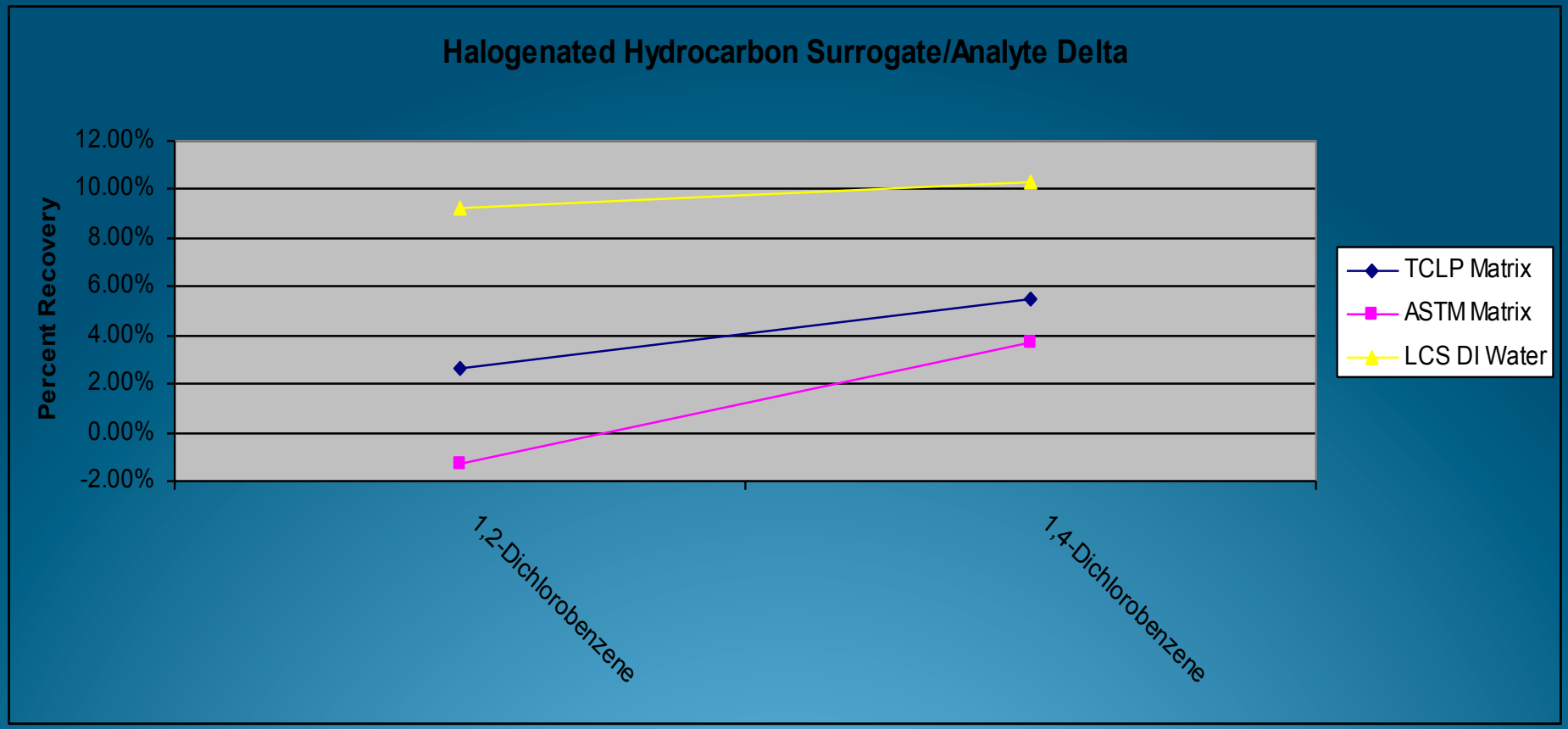
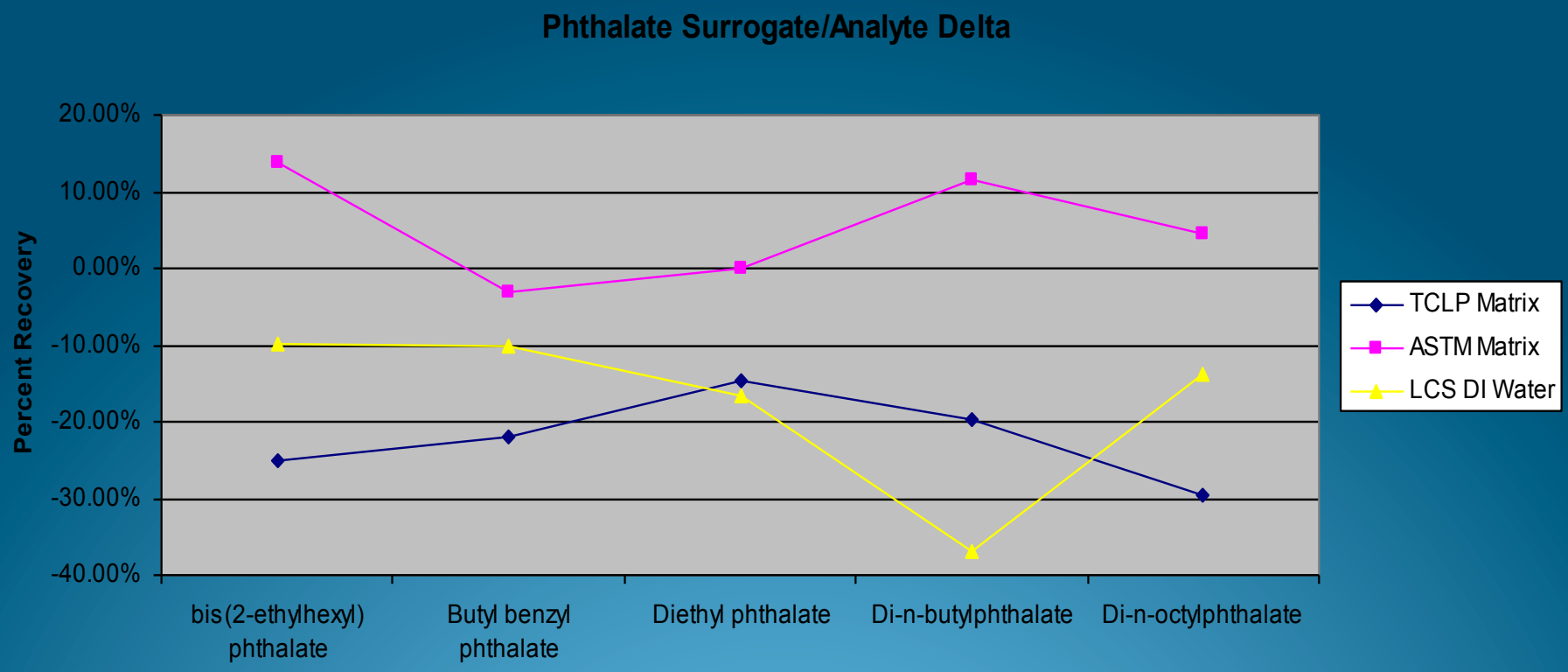
Halogenated Hydrocarbon Surrogate Analyte Recovery Delta



Phthalate Surrogate Analyte Recovery Delta



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Surrogates Selected

- Acenaphthylene-d8
- Anthracene-d10
- Benzo(a)pyrene-d12
- Bis(2-chloroethyl)ether-d8
- 4-Chloroaniline
- Dimethylphthalate-d6
- Fluorene-d10
- Nitrobenzene-d5
- N-Nitrosodimethylamine-d6
- Pyrene-d10
- 2,4-Dichlorophenol-d3
- 2-Chlorophenol-d4
- 2-Nitrophenol-d4
- 4,6-Dinitro-2-methylphenol-d2
- 4-Methylphenol-d_8
- 4-Nitrophenol-d4
- Phenol-d5

Phase 2 Conclusions

- Data demonstrates the across the wide variety of analytes SPE products tested are as accurate as traditional LLE.
- Study results were within the current method criteria for EPA 625 and within the acceptance limits in the TNI FoPT tables.
- With the exception of Phthalate Surrogates the new batch of analytes were a significant improvement.
- SPE Products perform equivalent to LLE performance across difficult matrices.

Acknowledgements

- SPE Vendors
- Laboratories
- Phenova
- ILI
- EPA
- ACIL
- Restek

Questions?

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