
Chromatographic Analysis of Bisphenol A: A Comparative Study

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Presentation Outline

- BPA Background
- Study Purpose/Experimental Design
- Method Comparison Results
- Suggestions for Future Studies



What is Bisphenol A?

- Bisphenol A has been in commerce for more than 50 years
- It is used in the synthesis of polyesters, polysulfones, and polyether ketones; as an antioxidant in some plasticizers, and as a polymerization inhibitor in PVC.
- It is a key monomer in production of polycarbonate plastic and epoxy resins.
 - Baby and water bottles
 - Sports equipment
 - Medical and dental devices
 - Lenses, and household electronics
 - CD's and DVD's
 - Food and beverage can coatings
 - Precursor to the flame retardant tetrabromobisphenol A
 - Fungicide



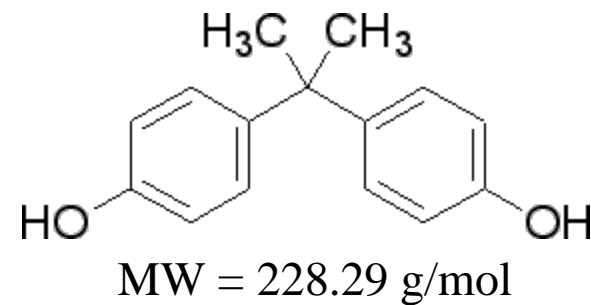
Where to Find BPA

- There are Seven Classes of Plastics
 - Type 7 is the catch-all "other" class, and some type 7 plastics, such as polycarbonate (sometimes identified with the letters "PC" near the recycling symbol) and epoxy resins, are made from bisphenol A monomer
 - Type 3 (PVC) can also contain bisphenol A as antioxidant in plasticizers Types 1 (PET), 2 (HDPE), 4 (LDPE), 5 (polypropylene), and 6 (polystyrene) do not use bisphenol A during polymerization or package forming.
- When such plastics are exposed to hot liquids, bisphenol A leaches out 55 times faster than it does under normal conditions, at up to 32 ng/hour.



Bisphenol A Interest

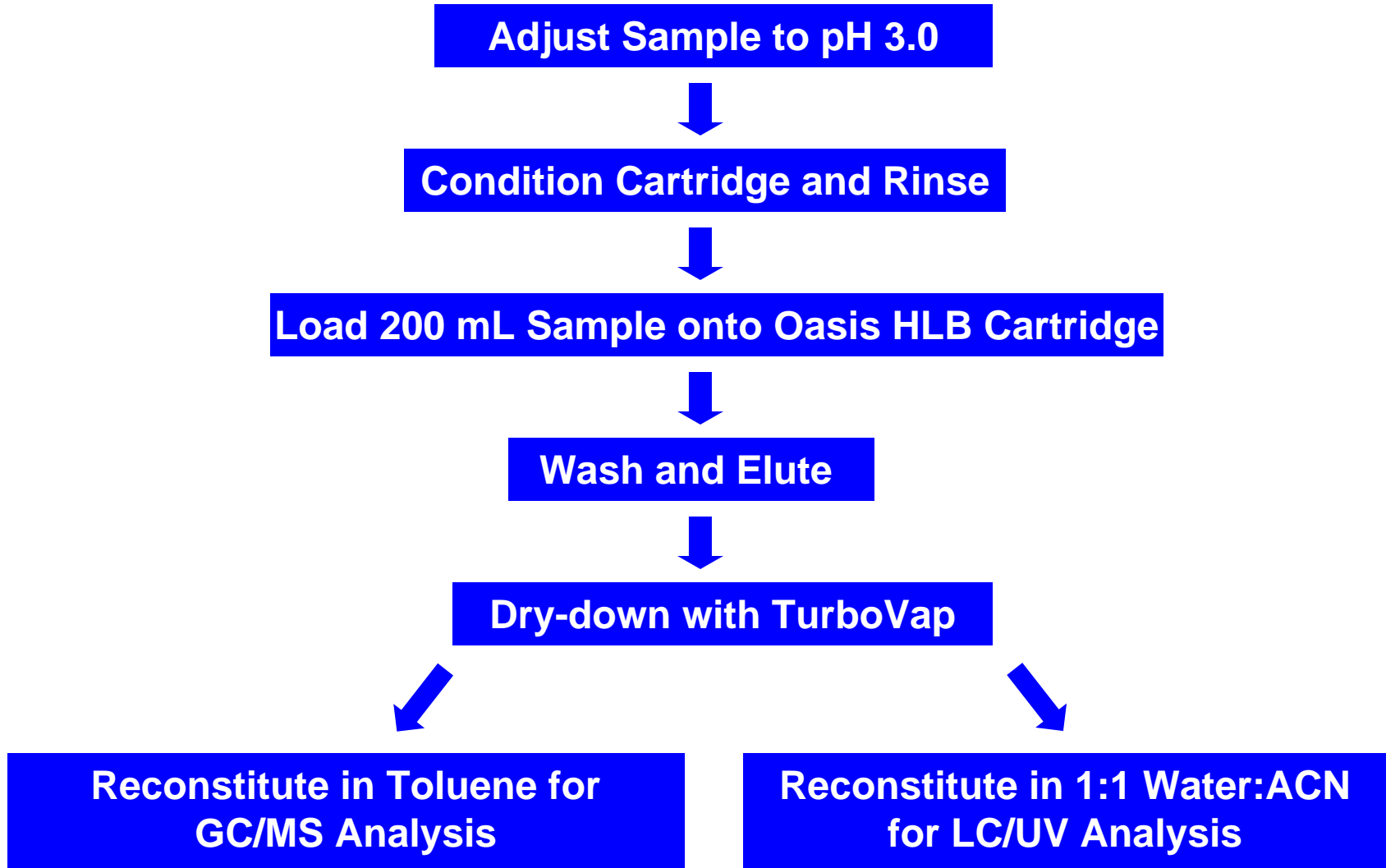
- Suspected as Hazardous Since 1930's
 - Possible endocrine disruptor that mimics estrogen; linked to cancer, infertility
- Lots of Media Attention Lately
 - ABC News, Aug'07: Possibly Unsafe Chemical Found in Baby Bottles: Bisphenol-A, or BPA, Is Found in Products Ranging From CDs to Baby Bottles
 - CEN Oct'08: Canada bans baby bottles containing BPA, to regulate in can liners and wastewater
- Many States Have Pending Legislation on its Use
 - WalMart, Nalgene, Toys-R-Us have Banned Products Containing BPA



Study Purpose/Experimental Design

- Evaluate Sample Preparation Method for both HPLC and GC
- Compare HPLC Method to GC Method
 - Linearity
 - Accuracy
 - Precision
 - Recovery
 - LOQ
 - Ease of Use
 - Throughput
- Evaluate Both Methods for Sample Analysis
- Identify Improvements and/or Future Work

Sample Prep Method



Reconstitute in Toluene for
GC/MS Analysis

Reconstitute in 1:1 Water:ACN
for LC/UV Analysis

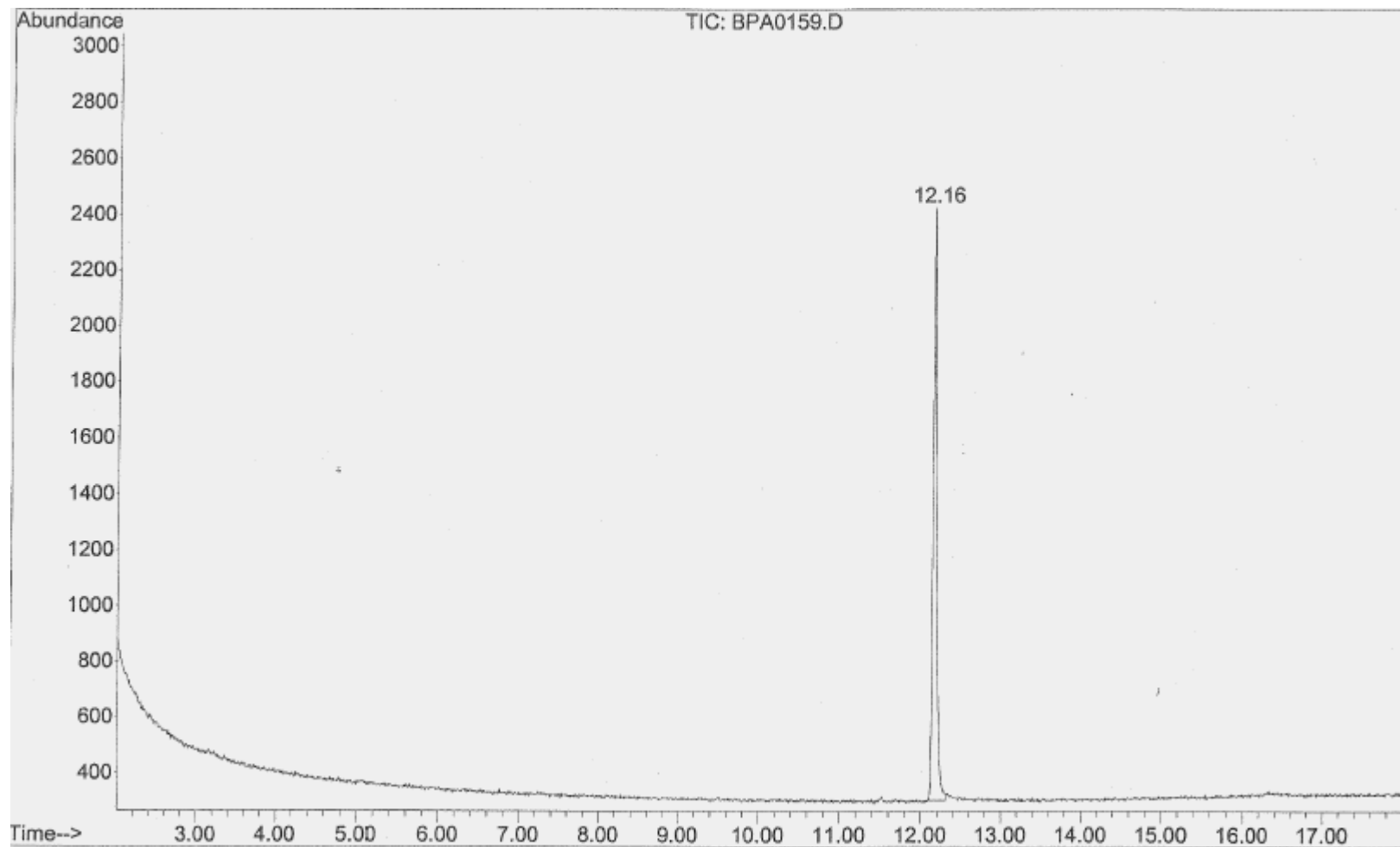
GC Method

- Agilent 6890 GC w/ 5973 MSD
 - HP5-MS column (30 m x 0.25 mm x 0.25 μ m)
 - 2 μ L injection
 - Temperature Program: 150°C to 240°C at 4°C/min
 - Selective Ion Monitoring (SIM)
 - Target Ion: 213 m/z
 - Qualifying Ions: 228, 119 and 135 m/z



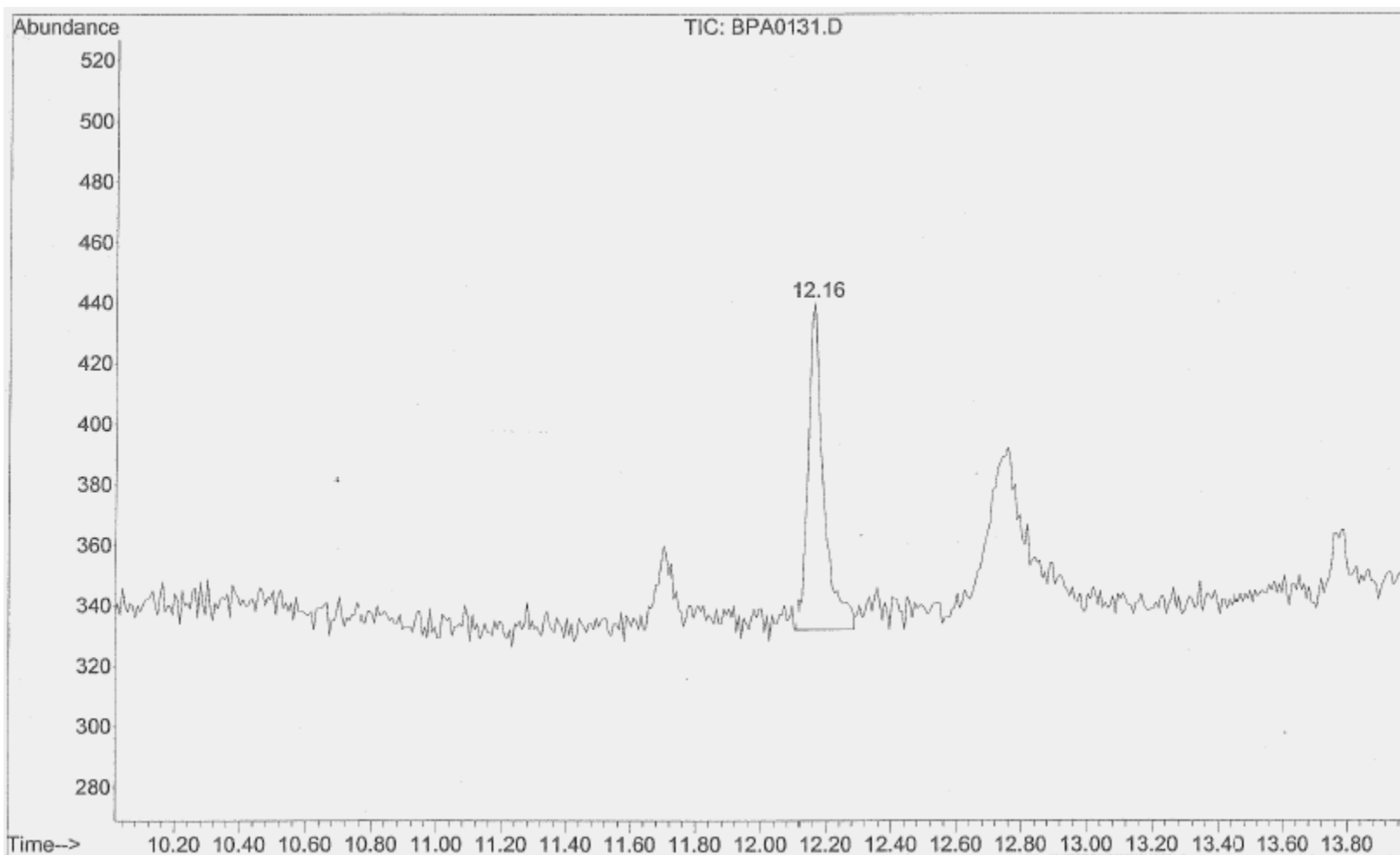
GC Chromatogram Example

- Neat BPA Standard 100 ng/mL



GC Chromatogram Example-LOQ

- Neat BPA Standard 5.0 ng/mL



HPLC Method

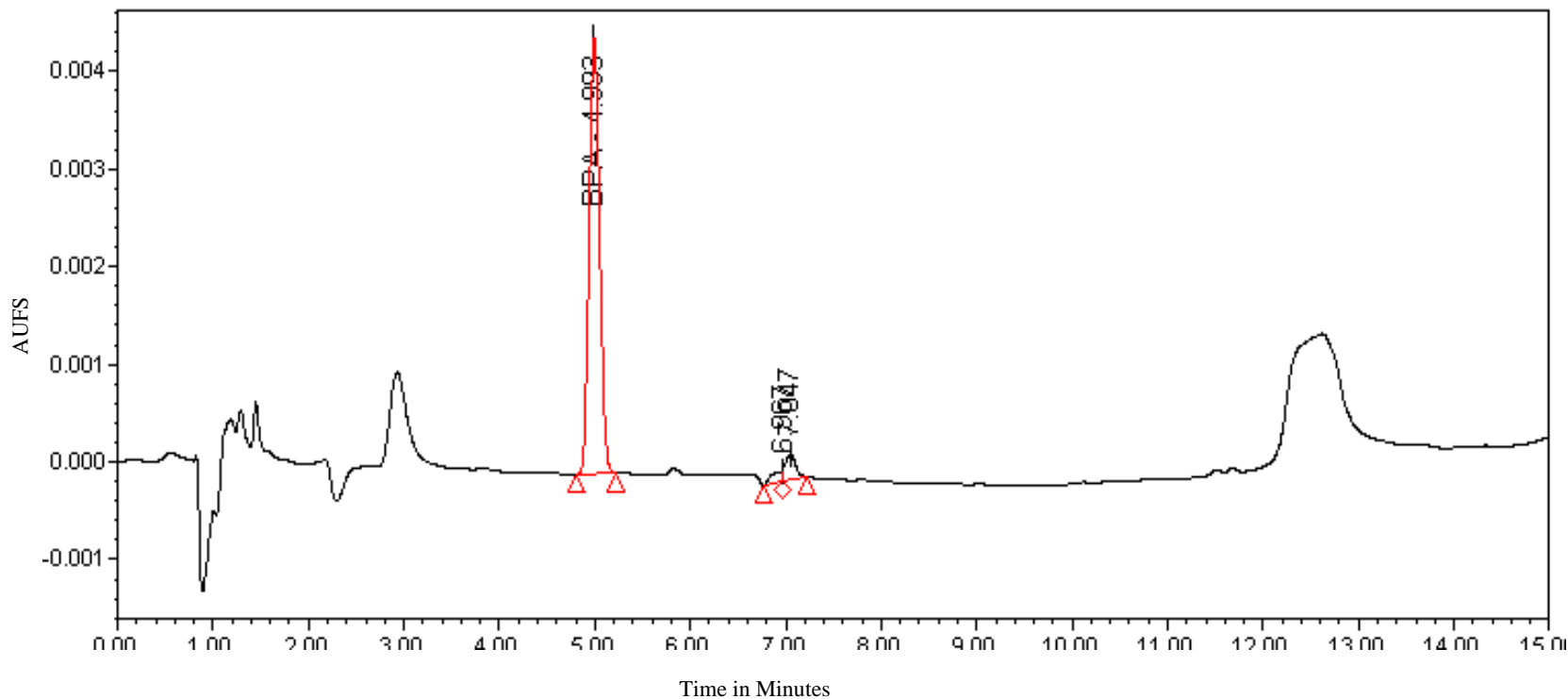
Waters Alliance LC w/ 2487 Dual λ Detector

- Column: Waters Symmetry Shield RP18, 5 μ m, 3.9 x 150 mm
- MPA: 15 mM Phosphate
- MPB: ACN
- Elution gradient: 40 to 100% B in 20 min.
- Flow: 1 ml/min
- 100 μ L injection volume
- Detection λ 225 nm



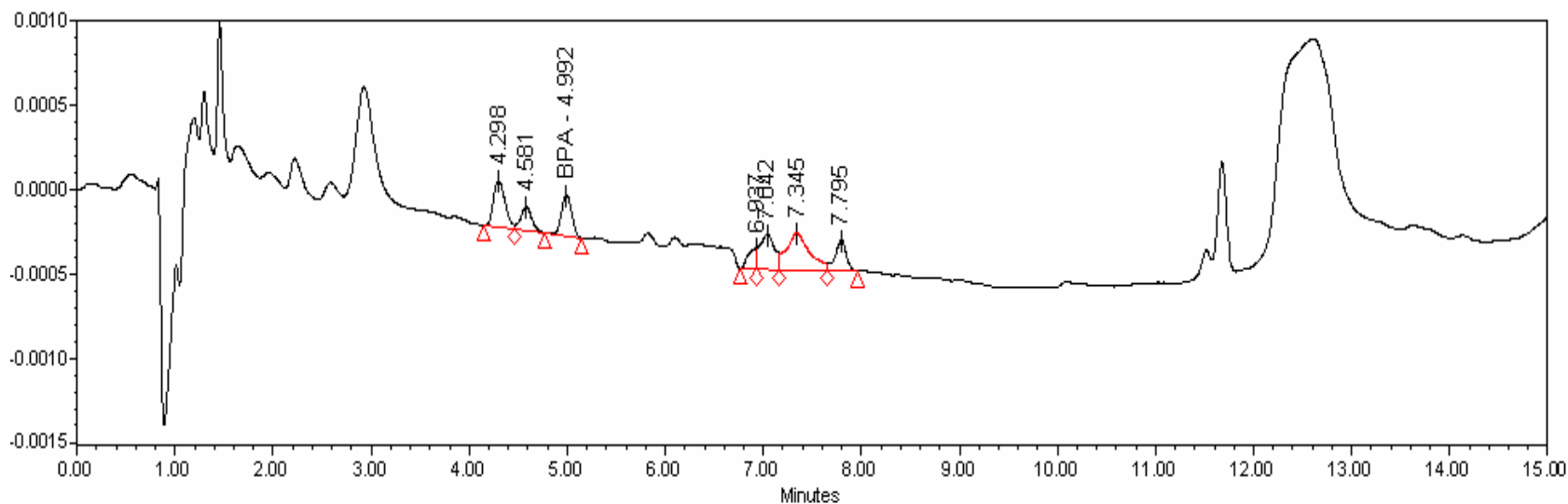
Example HPLC Chromatogram

- Neat Standard: 100 ng/mL



Example HPLC Chromatogram-LOQ

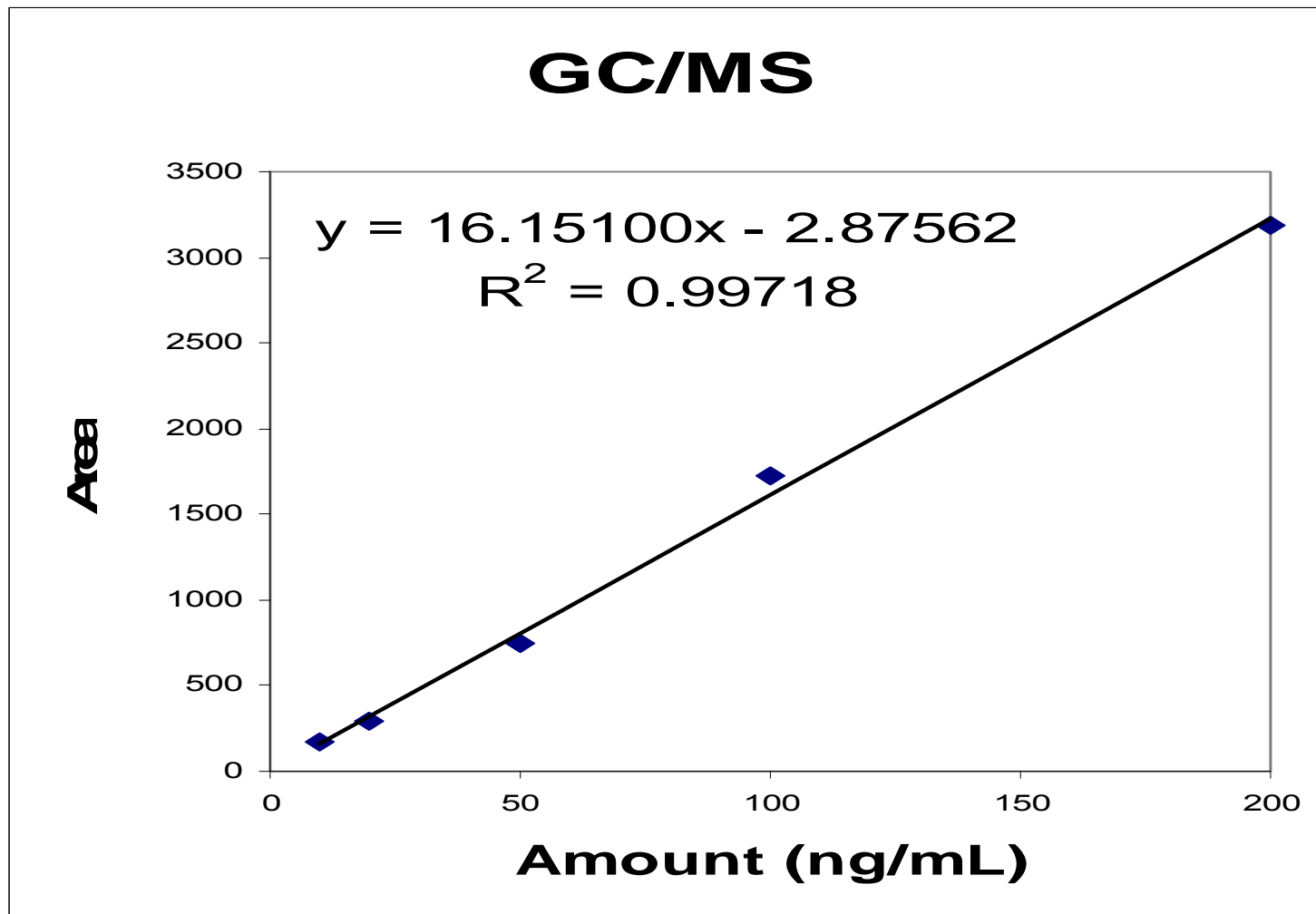
- Neat Standard at 5 ng/mL



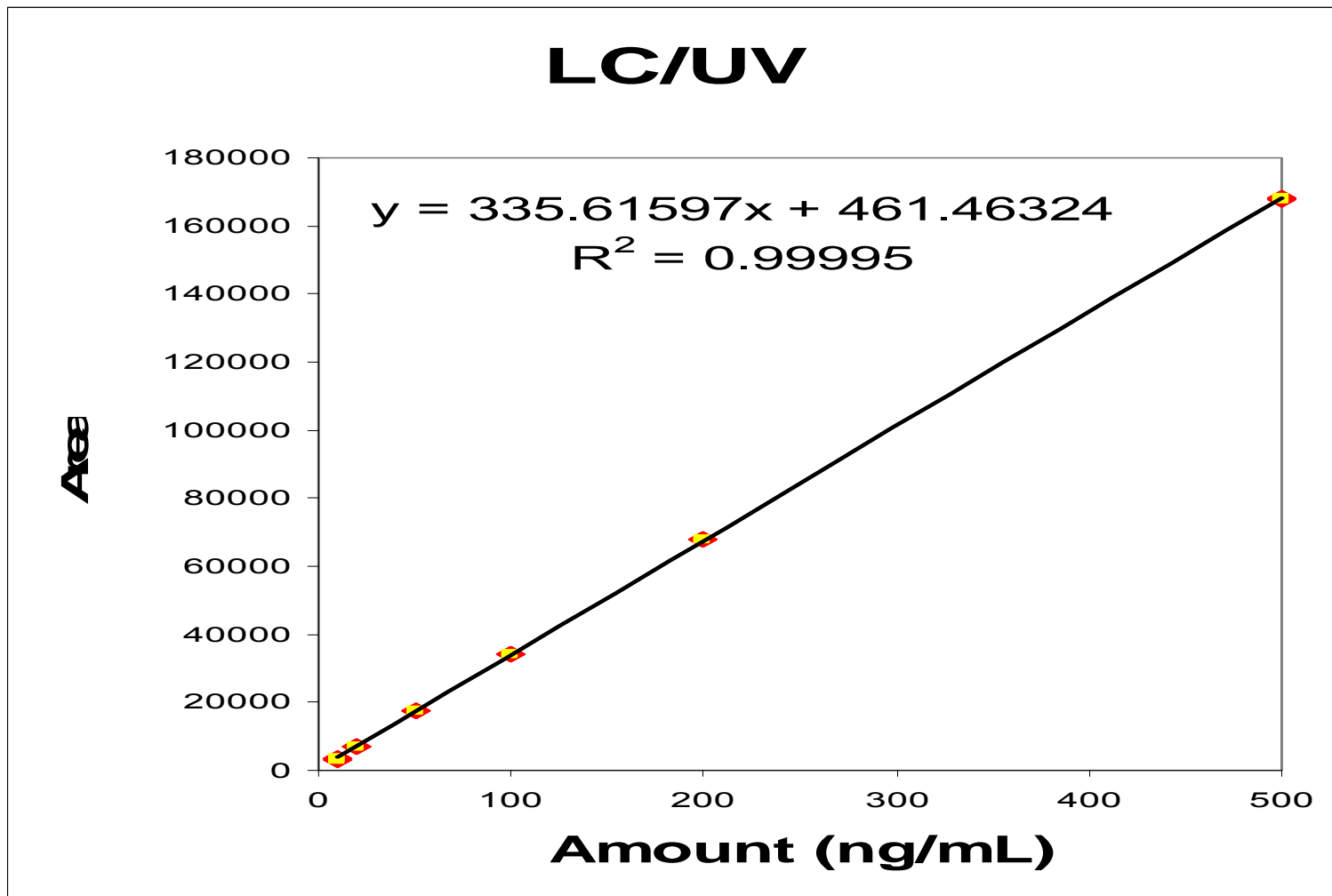
Compare HPLC Method to GC Method

- Linearity
- Accuracy
- Precision
- Recovery
- LOQ
- Ease of Use
- Throughput

GC Linearity



HPLC Linearity



Analytical Method Precision

LC/UV

50 ng/mL:

Mean: 17687
Std. Dev: 59.0
RSD : 0.33 %

5 ng/mL:

Mean: 1692
Std. Dev: 91.84
RSD: 5.4 %

GC/MS

50 ng/mL:

Mean: 1087
Std. Dev: 109
RSD : 10.0 %

5 ng/mL:

Mean: 460
Std. Dev: 110
RSD: 24 %

N = 6, Neat Standards

Extraction Precision, Accuracy/Recovery

Experiment: 200X Extraction of 0.5 ng/mL

LC/UV

% Recovery	Injection RSD	Extraction RSD
88.6	0.19	3.8
88.2		
88.4		
83.9	0.33	
83.8		
84.3		
91.8	0.04	
91.7		
91.7		

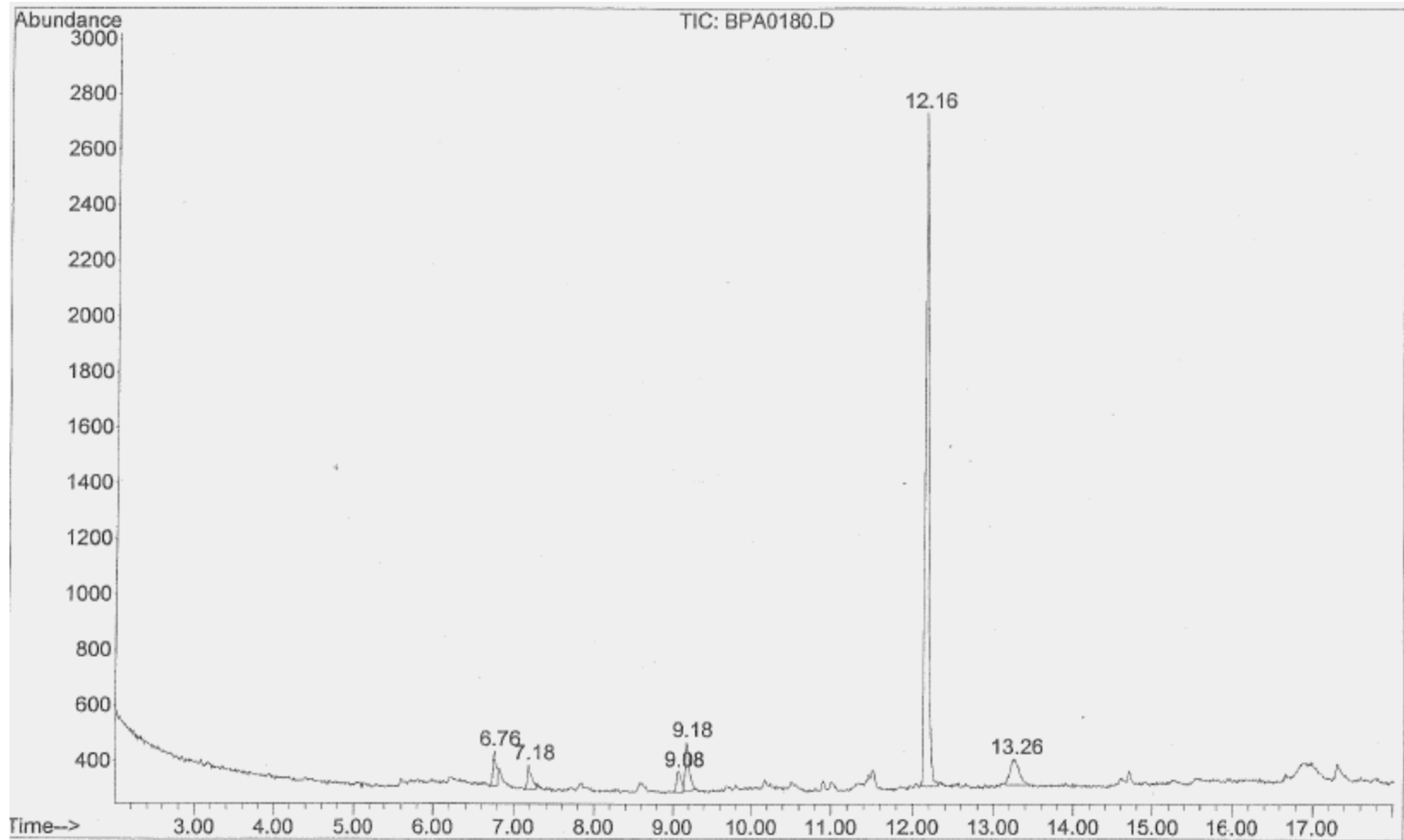
GC/MS

% Recovery	Injection RSD	Extraction RSD
88.3	2.9	5.1
93.6		
91.4		
93.9	6.1	
84.1		
85.2		
97.8	4.8	
95		
89		

Sample represents mid-range of analysis-100 ng/mL

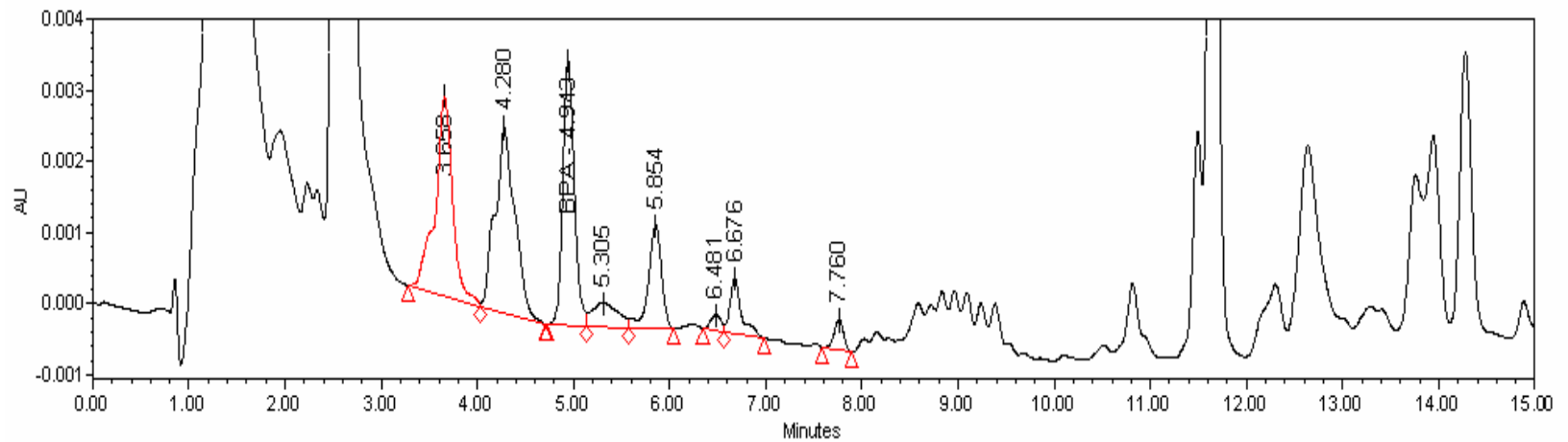
SPE Extraction: Accuracy and Recovery

- 200X extraction of 0.5 ng/mL – GC analysis



SPE Extraction: Accuracy and Recovery

- 200X extraction of 0.5 ng/mL – LC analysis

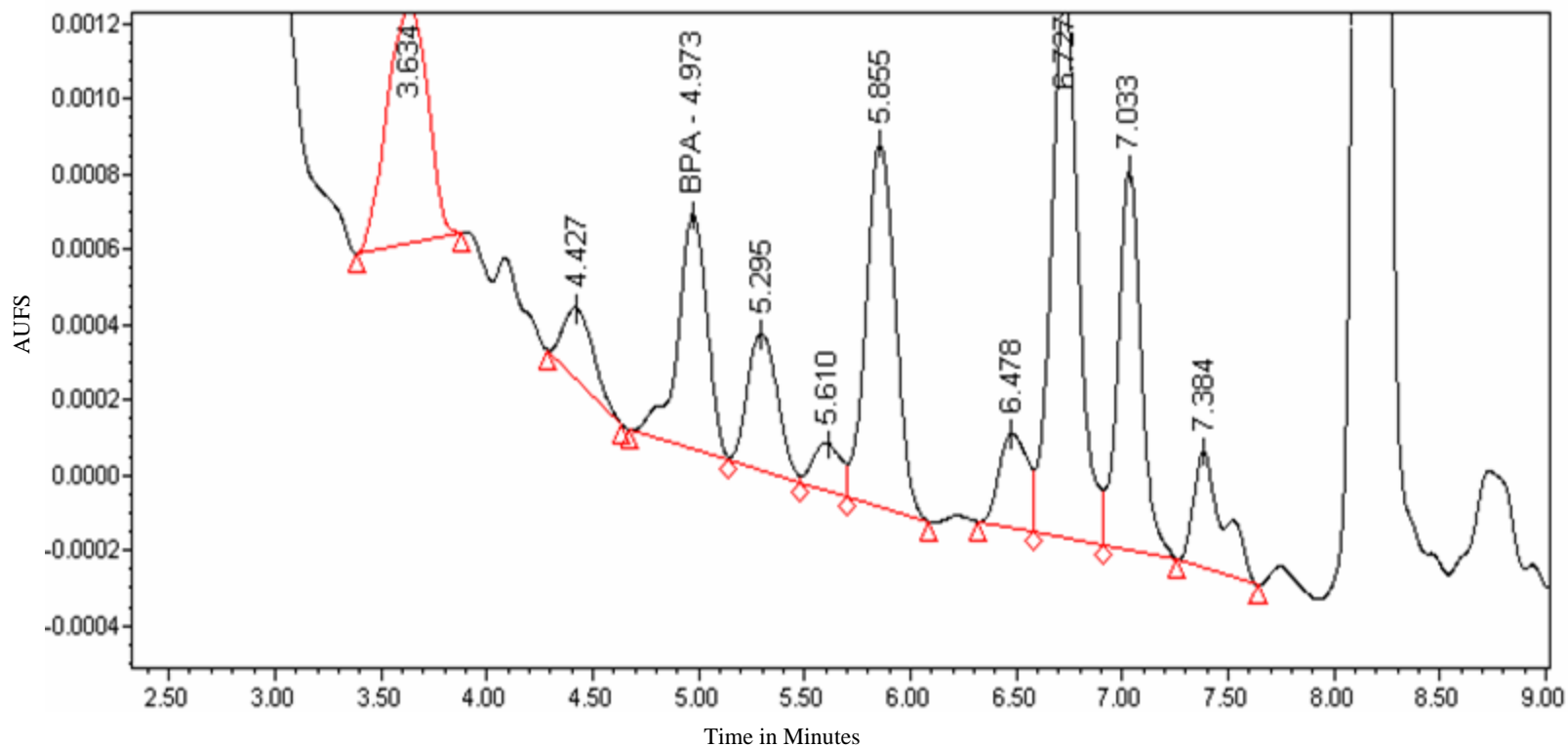


Method Comparison

	HPLC	GC
Linearity	R ² = 0.9999 (5-500 ng)	R ² = 0.997 (5-200 ng)
Accuracy/Recovery	AVG = 88.0, 3.8 %RSD	AVG = 90.9, 5.1 %RSD
Precision-50 ng	0.33 %RSD	10 %RSD
Precision-5 ng	5.4 %RSD	24 %RSD
LOQ	0.5 ng*	0.5 ng*
Retention Time	5.0 min.	12 min.
Cycle Time	30 min.	35 Min.
Selectivity	++	++++

Sample Analysis: Commercial Baby Bottle

HPLC: 50X Concentration Extraction



Future Work

- Standardize on HPLC Method
 - Optimize-improve selectivity
 - Validate
- Evaluate LC/MS/(MS)
 - Buffer change
 - Sensitivity, Accuracy, Precision, etc.
- Look at Additional Samples
- Optimize Sampling Procedure

Summary

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- Method Comparison Results
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Acknowledgements

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And:

