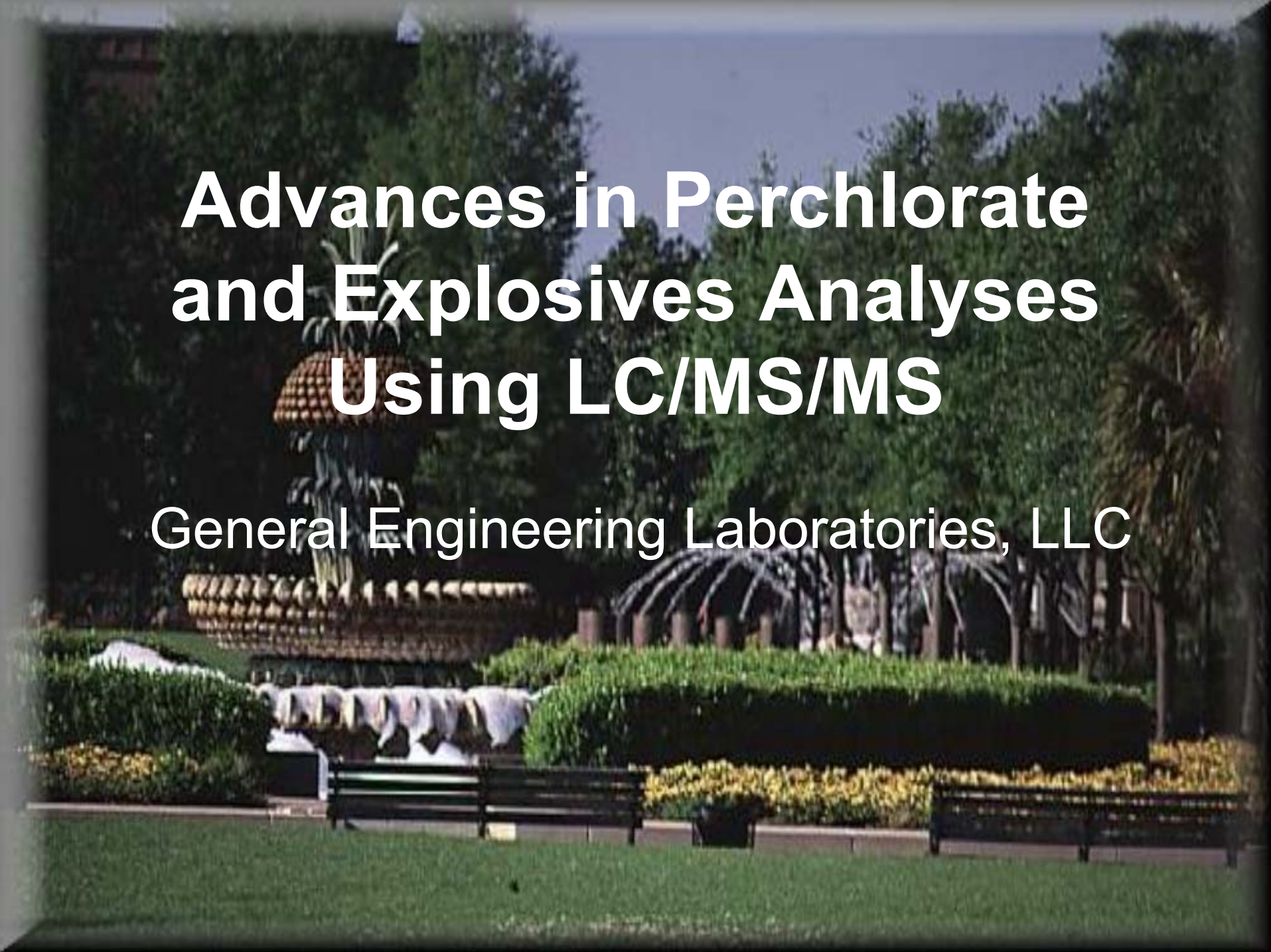
Advances in Perchlorate and Explosives Analyses Using LC/MS/MS

General Engineering Laboratories, LLC



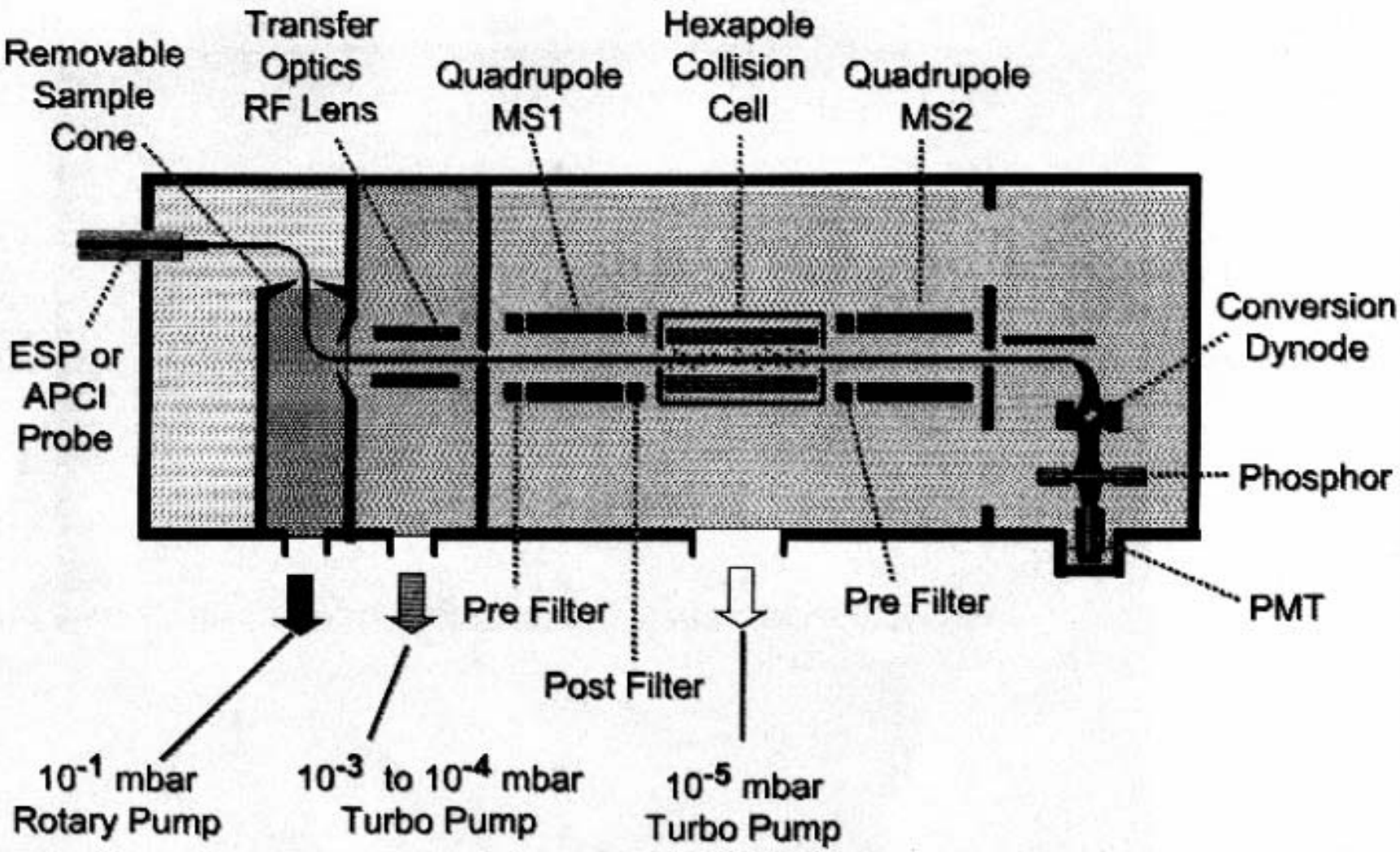
Agenda

- Introduction to LC/MS/MS Instrumentation
- Comparisons of Perchlorate Methods
- Conclusion and Discussion

LC/MS/MS

- System Configuration
  - Waters LC
  - Quattro Micro Dual Mass Spectrometer
  - Electrospray Probe

Schematic Overview of the Quattro micro

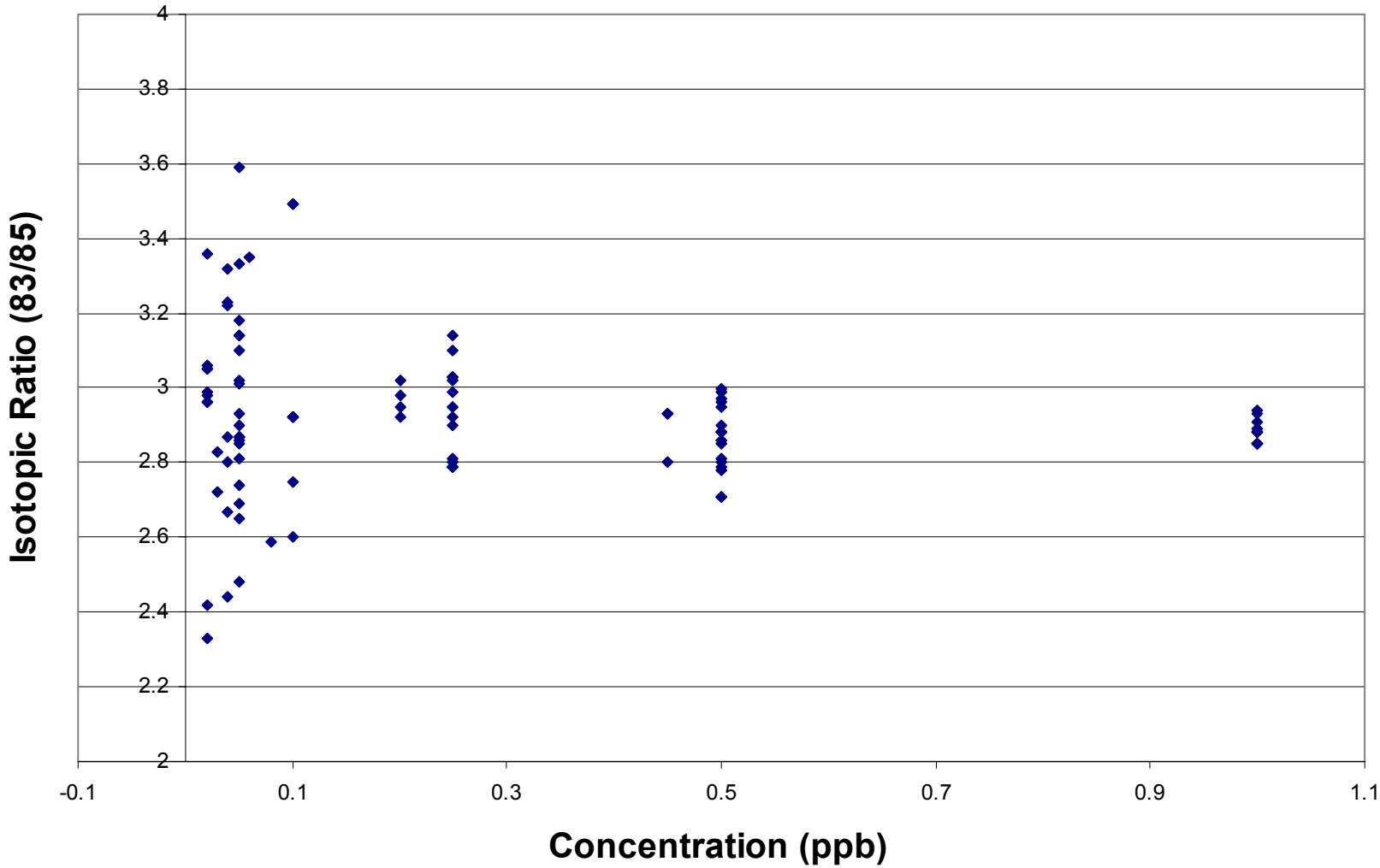


Perchlorate Methods

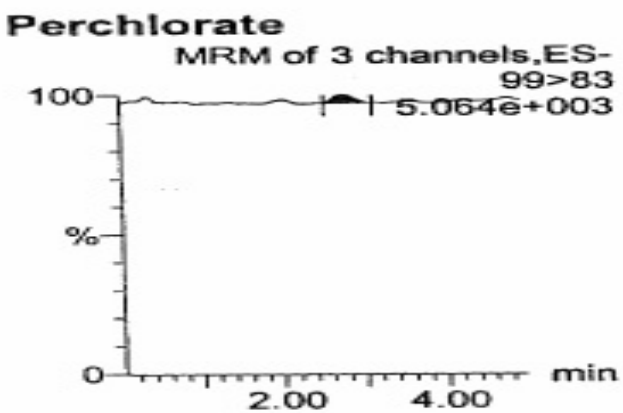
LC/MS/MS vs. IC 314.0

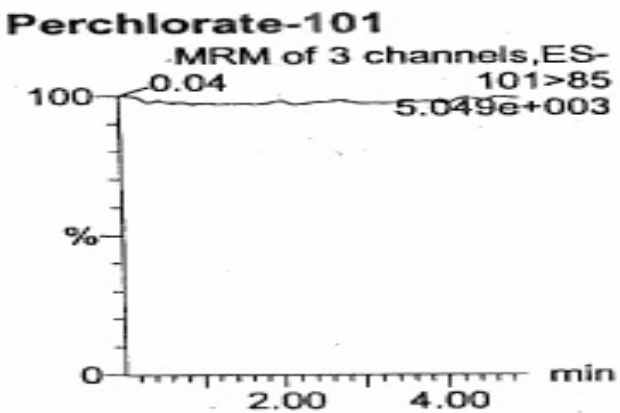
- Selectivity - Isotopic Ratio
- Sensitivity - MDL: 50 ppt, PQL: 200 ppt
- Speed - 6 minute run time
- Reduced Interferences from Difficult Matrices

PERCHLORATE
ISOTOPIC RATIOS OF SAMPLES WITH PERCHLORATE CONCENTRATIONS OF 0.050 ppb AND GREATER



Water Blank
Spiked with High Concentrations of Sulfate, Carbonate, Bicarbonate, And Chloride

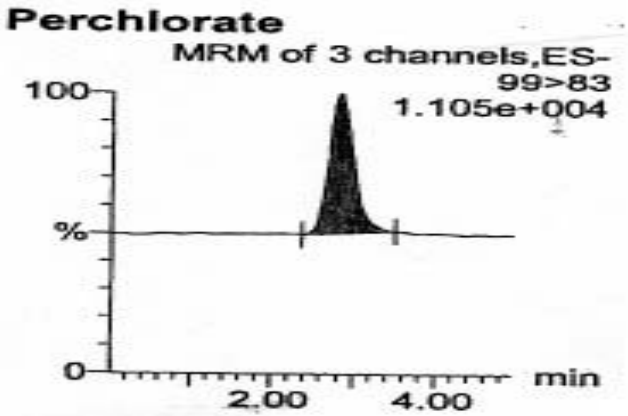


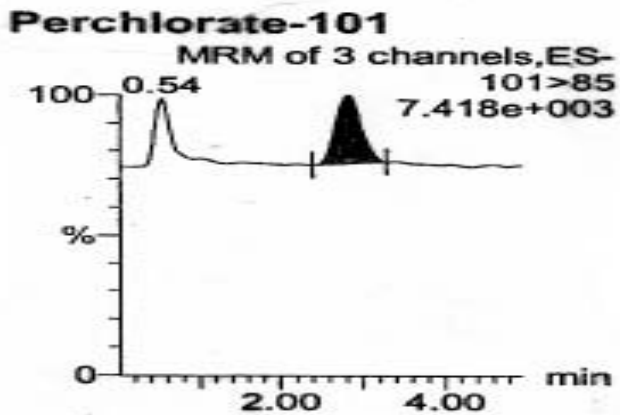


Filtered with one hydrogen and two barium Dionex cartridges

(Perchlorate detected at <0.050ppb)

0.20ppb Perchlorate Standard Spiked with High Concentrations of Sulfate, Carbonate, Bicarbonate, And Chloride

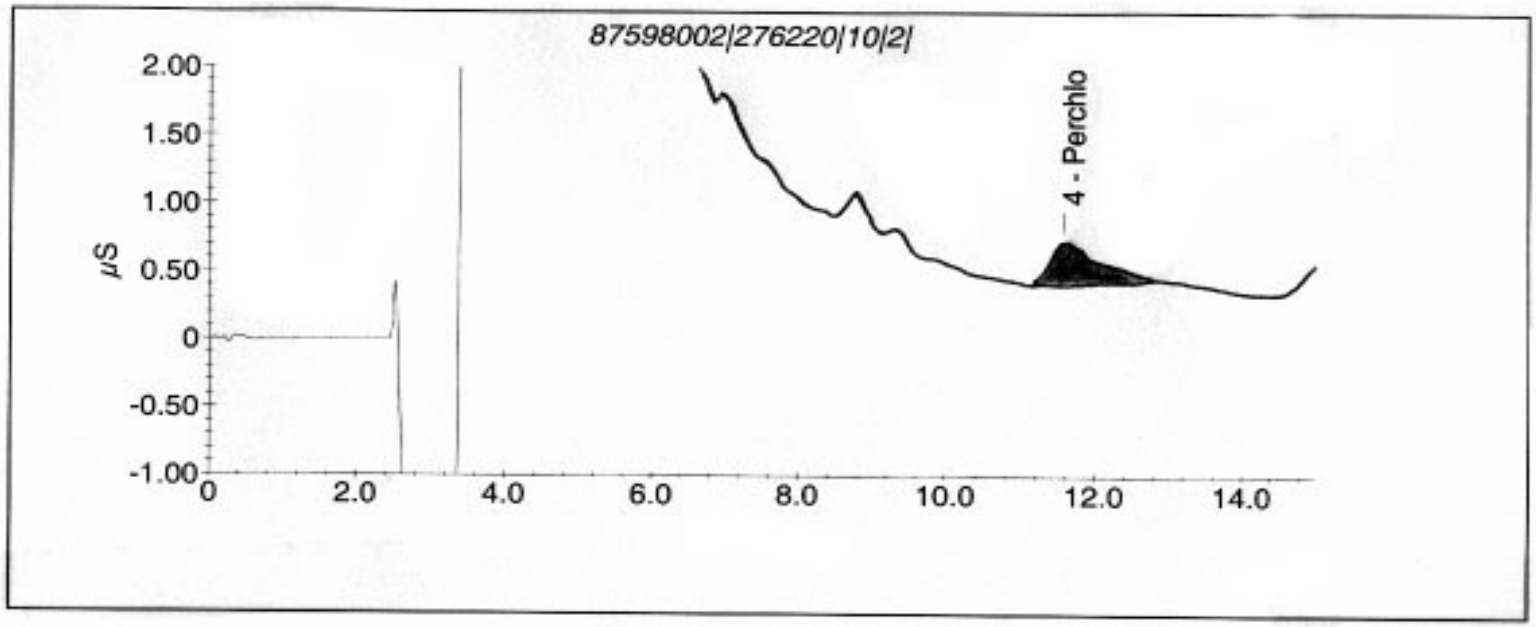




Filtered with one hydrogen and two barium Dionex cartridges

(Perchlorate detected at 0.17ppb)
(Isotopic Ratio of 2.90)

Vegetables by Method 314.0

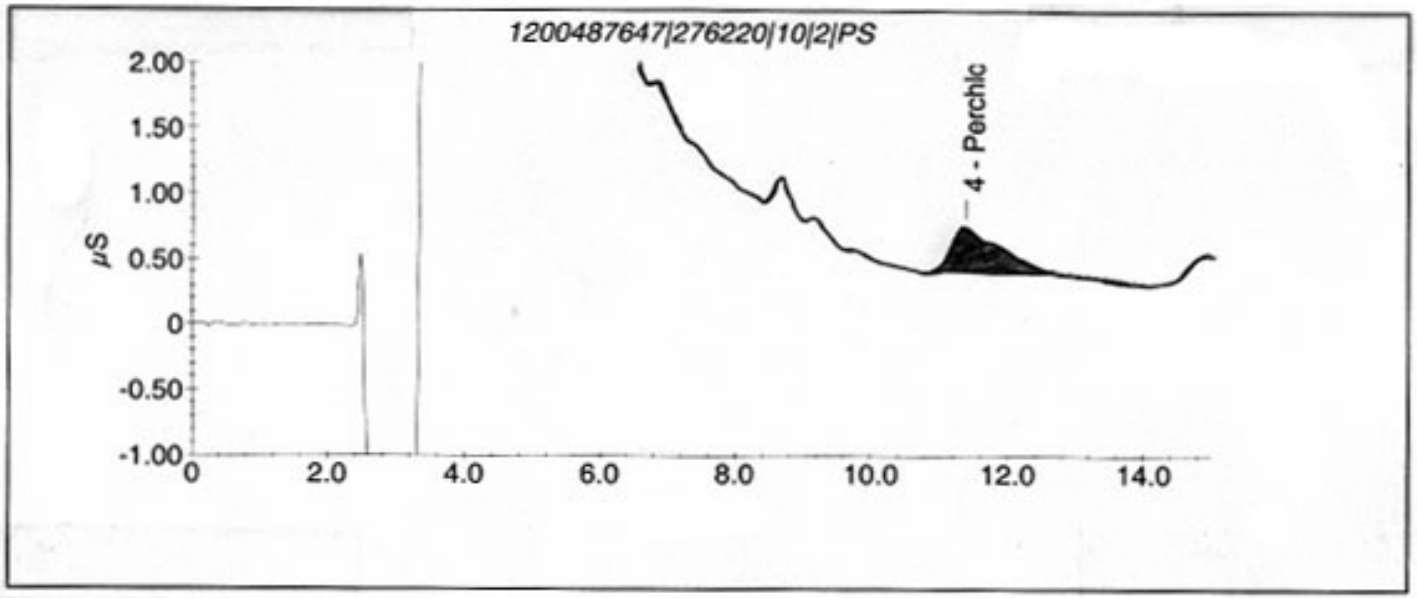


Sample was analyzed at a 1:10 dilution.

Filtered with a hydrogen, barium, and silver Dionex cartridge.

(Perchlorate detected at 1281.6ug/L)

Vegetables by Method 314.0



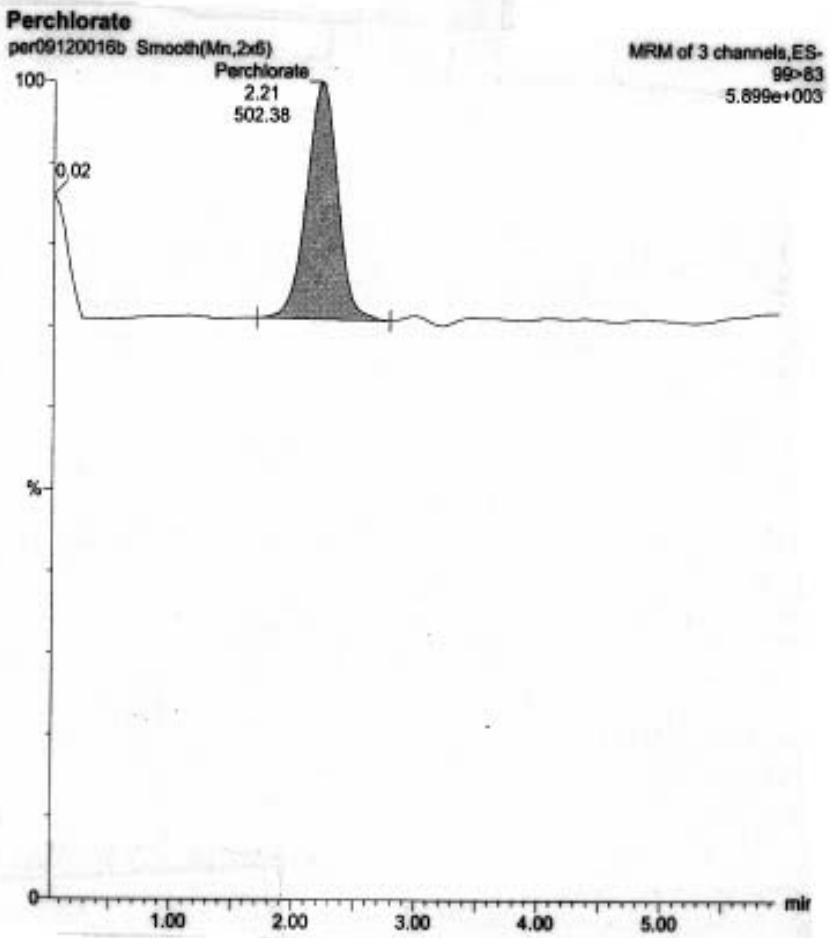
POST SPIKE

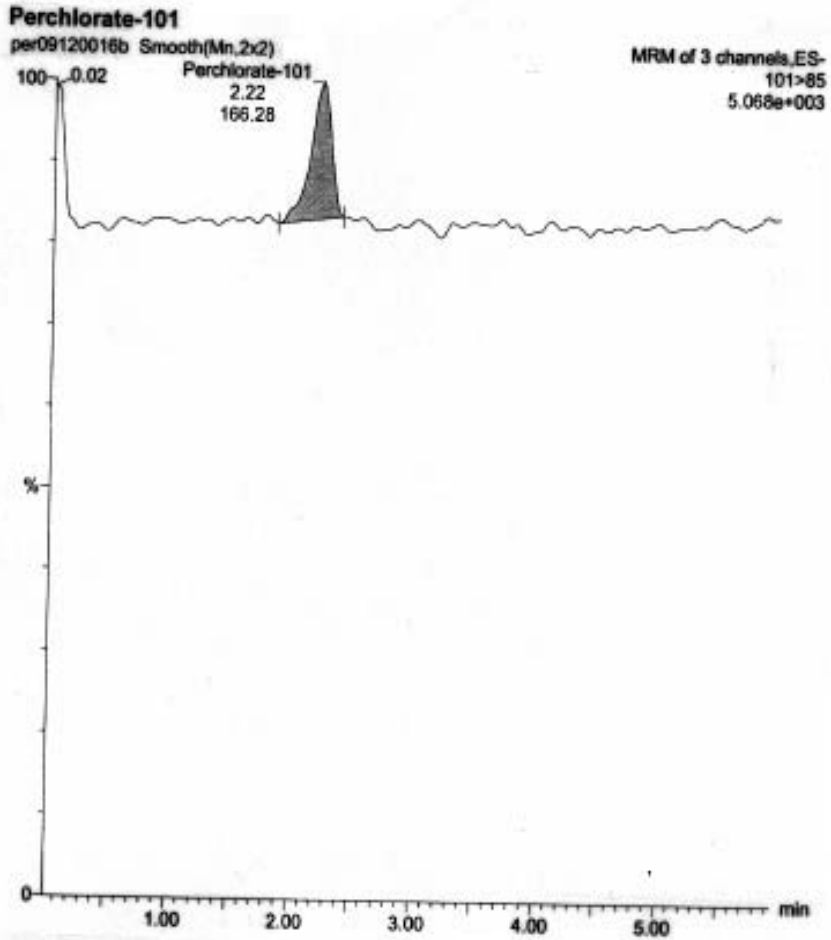
Sample was analyzed at a 1:10 dilution.

Filtered with a hydrogen, barium, and silver Dionex cartridge.

(Perchlorate retention time shifted- Perchlorate is not at 1281.6ug/L in the sample.)

Vegetables by Method 8321A





Sample was analyzed at a 1:10 dilution.

Filtered with a hydrogen and two barium Dionex cartridges.

(Perchlorate was detected at <5.0ppb.)

Perchlorate Method Scorecard

Characteristic	314.0	8321A
Lowest Detection Limit		❍
Shortest analysis time		❍
Lowest detection limits in abnormal matrices		❍
Least possible to be suppressed by chloride, sulfate, or carbonate		❍
Least likely to produce false positive/negative results		❍
Most steps of selectivity		❍
Ability to detect naturally occuring isotope		❍
Price	❍	

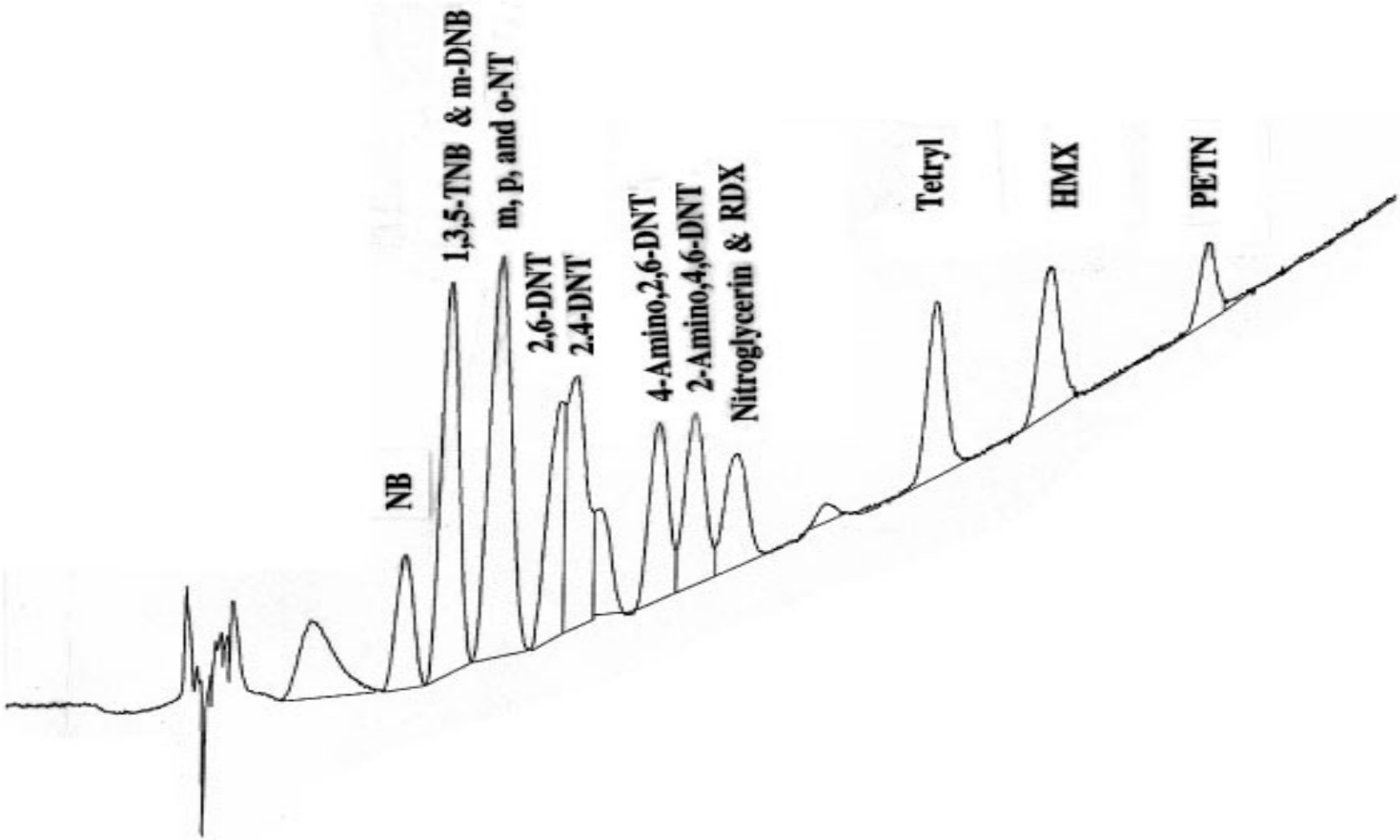
Explosives Methods

LC/MS/MS 8321A vs. HPLC 8330

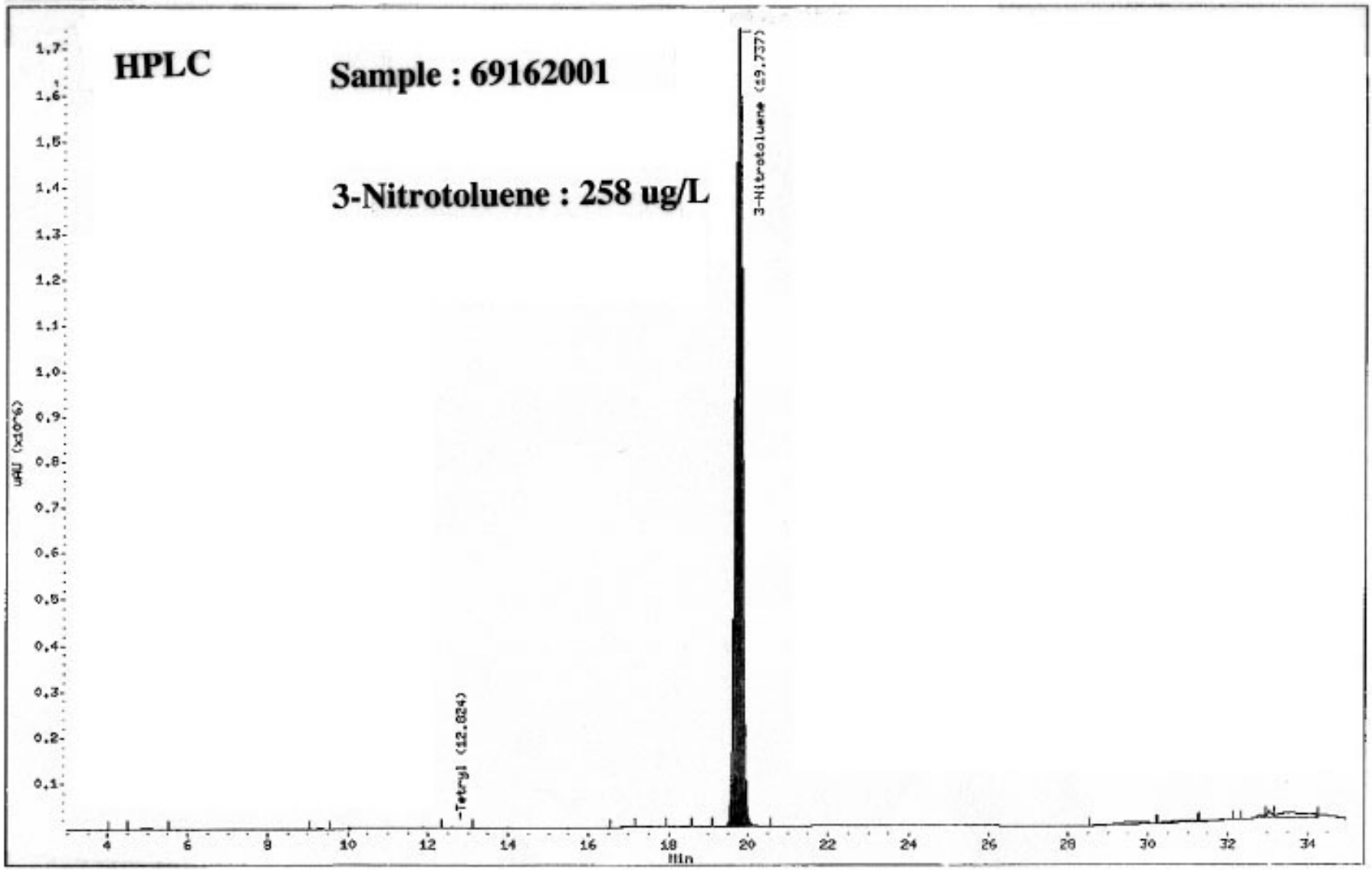
- Selectivity
  - Coelution
  - False Postives
  - False Negatives
- Sensitivity
  - Picric Acid
  - Difficult Matrices
  - Single Analysis

HPLC

CYANO COLUMN COELUTIONS



Well Water

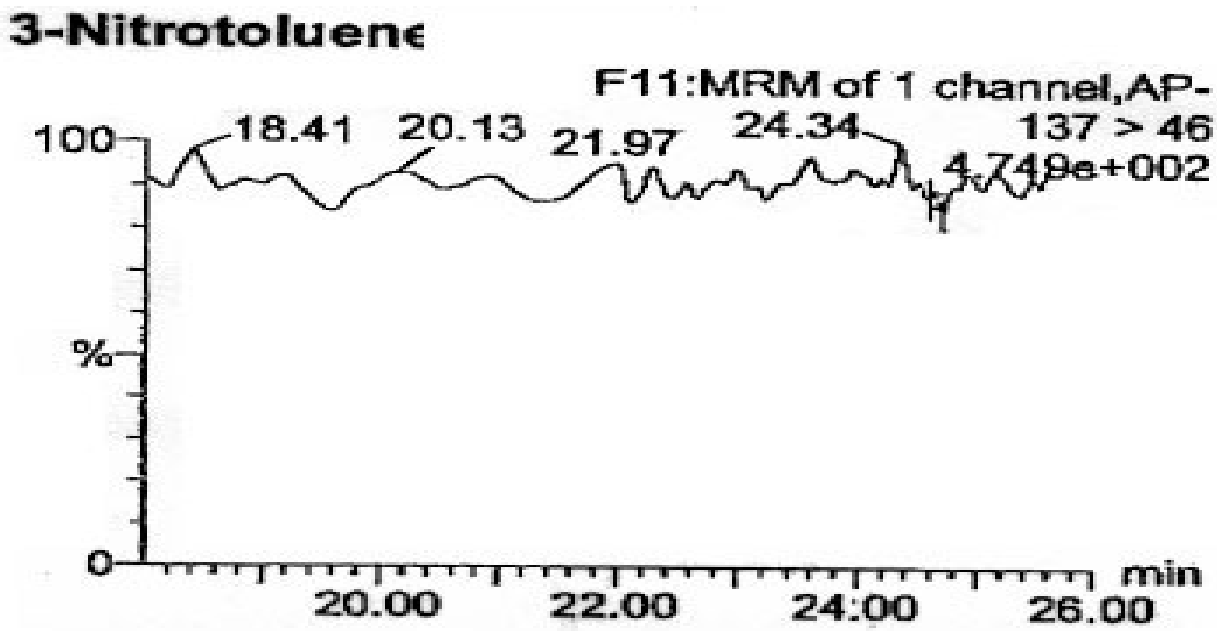

HPLC
Sample : 69162001
3-Nitrotoluene : 258 ug/L
3-Nitrotoluene (19.737)
-Tetryl (12.824)
uAU (x10^6)
Min

Well Water

LCMSMS

Sample : 69162001

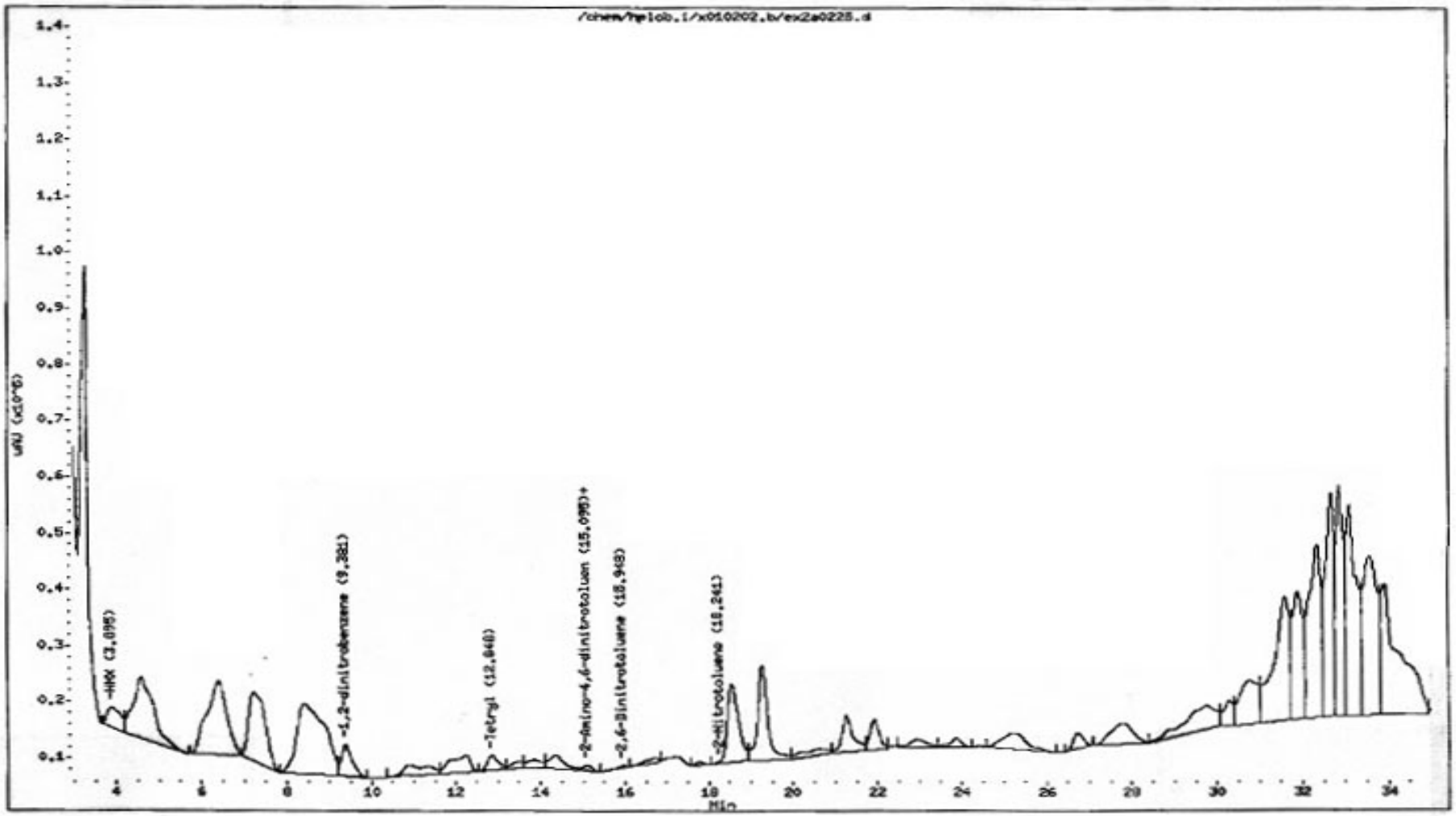
3-Nitrotoluene : < 0.0519 ug/L



HPLC

Mouse

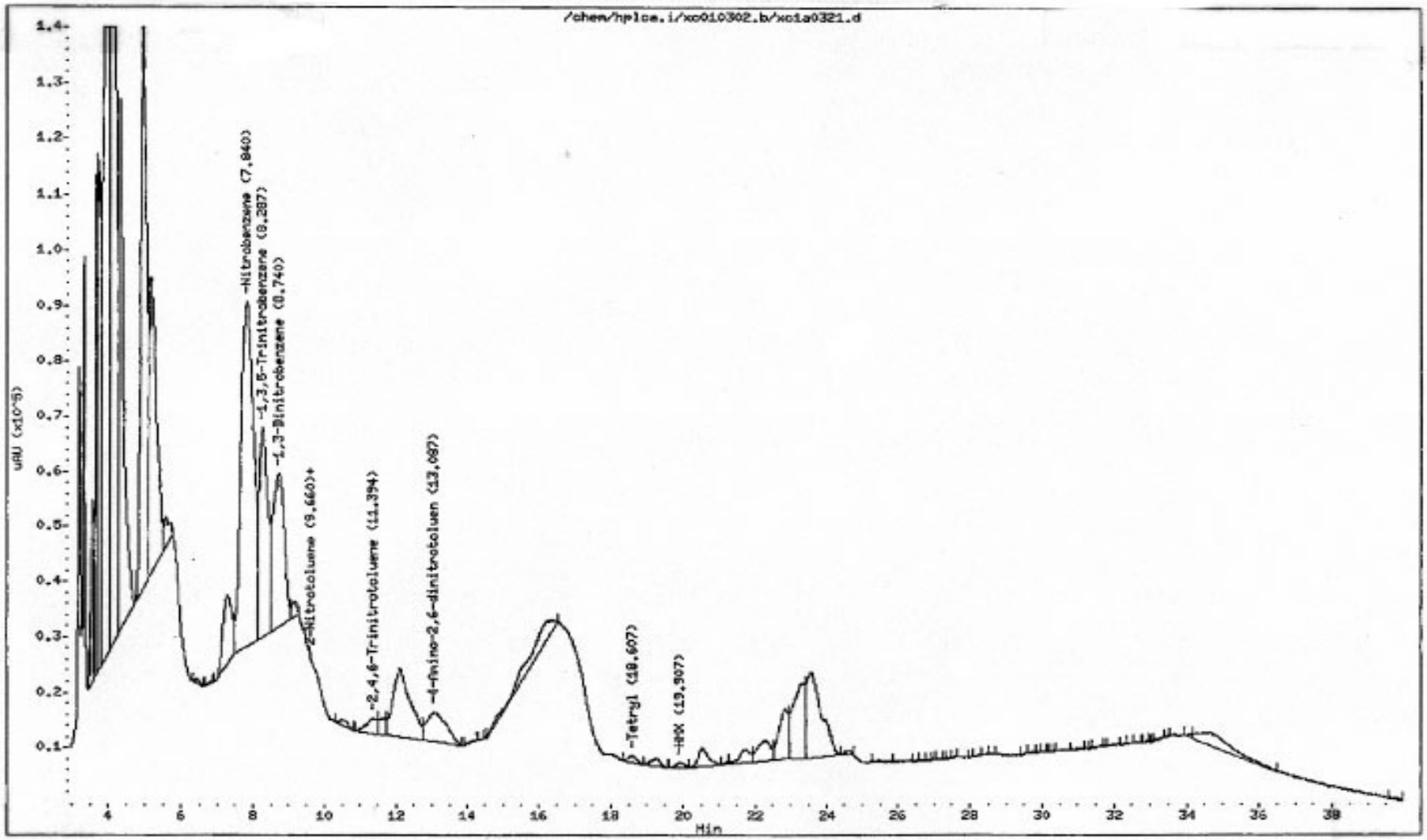
Primary Column



HPLC

Mouse

Confirmation Column



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