Newly Developed EPA Methods for the Determination of Perchlorate in Drinking Water

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EPA Methods for the Analysis of Perchlorate in Drinking Water

- IC/conductivity
- IC/conductivity with preconcentration
- IC/MS or IC/MS-MS
- LC/MS or LC/MS-MS

Analyte Preservation

- Chemical degradation
- Microbiological degradation
- Impact of Degradation
  - False negatives
  - Inaccurate quantification
- Solution
  - Filter samples through sterile 0.2 micron filter
  - Collect filtered sample in sterile bottle
  - Maintain headspace

EPA Method 314.0
IC/Conductivity

- IC method using suppressed conductivity detection
- MRL of approximately 4.0 ug/L (ppb) in moderate to low levels of Cl^-, HCO_3^- and SO_4^{2-}
- Published in 1999

EPA Method 314.0

- Interferences
  - Requires fortifying unfamiliar positive samples
- Matrix
  - Maximum conductivity threshold
  - Dilution
  - Clean-up cartridges
  - Heart cutting
- Sensitivity
  - Use 2 mm column system or inject more sample

EPA Method 314.1
IC/Conductivity with Preconcentration

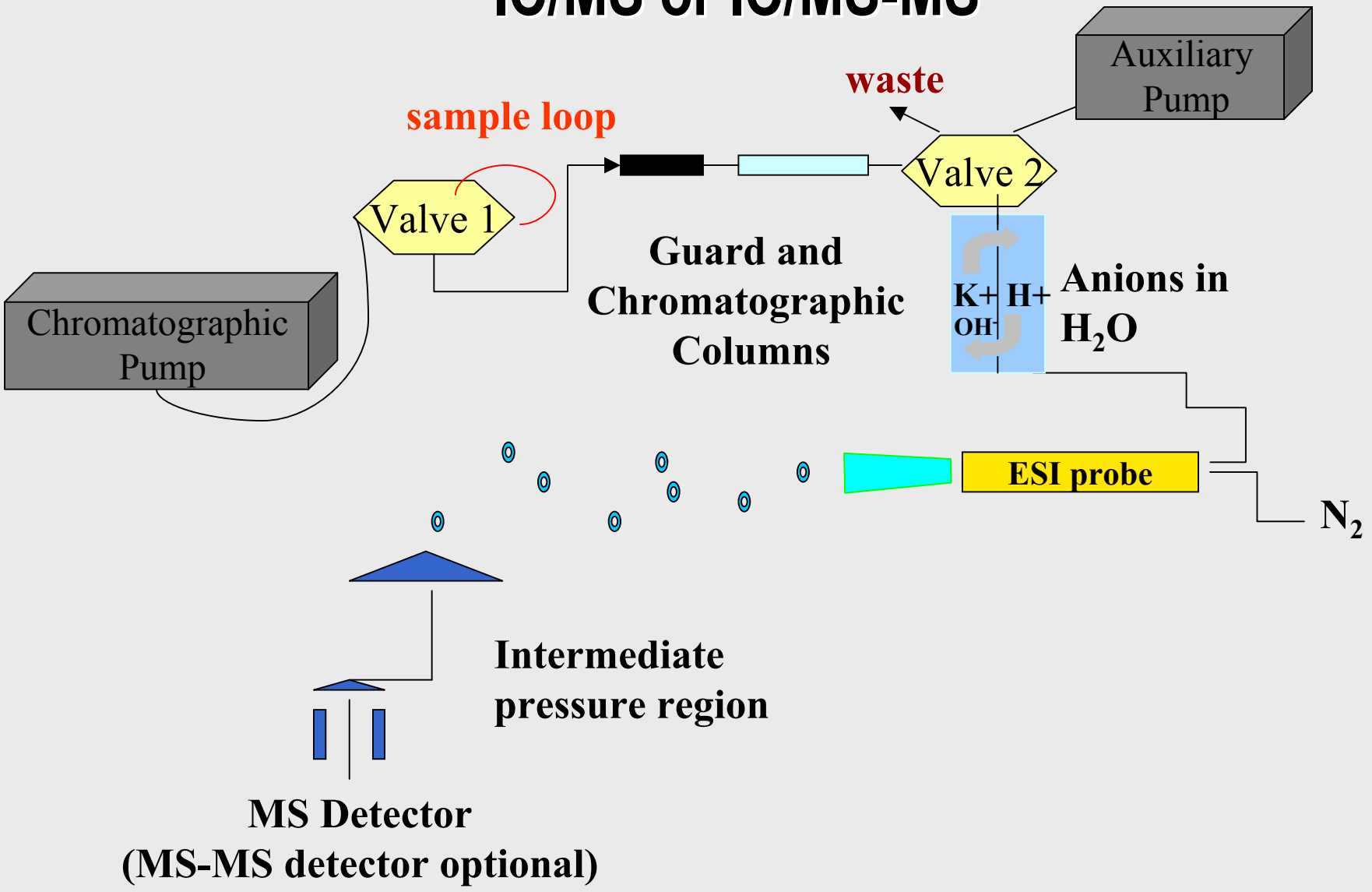
- IC method combining column concentration with matrix elimination and suppressed conductivity detection
- MRL of approximately 0.5 ug/L (ppb) in 1000 mg/L (ppm), each, of Cl^-, HCO_3^- and SO_4^{2-}
- Incorporates new 4 x 35-mm high-capacity Cryptand concentrator column with 2-mm AG16/AS16 analytical column set (Primary), and AG20/AS20 analytical column set (Confirmatory)
- Scheduled for publication in April

EPA Method 314.1

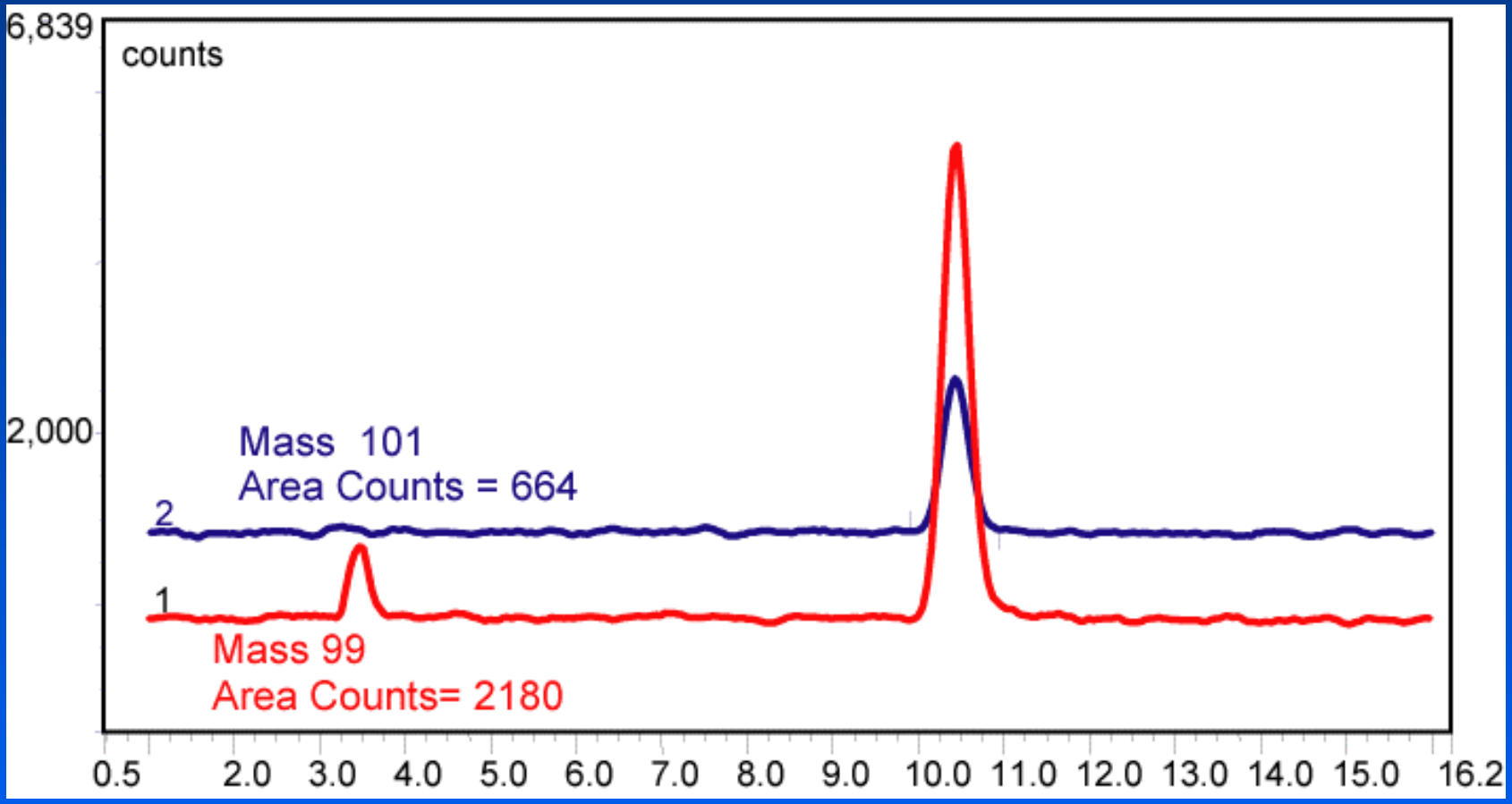
- Interferences
  - Chromatographic Confirmational Column
- Matrix
  - In-line concentration column permits analyses in matricies with higher anion content
  - Dilution
  - Clean-up cartridges

EPA Method 330.0

IC/MS or IC/MS-MS



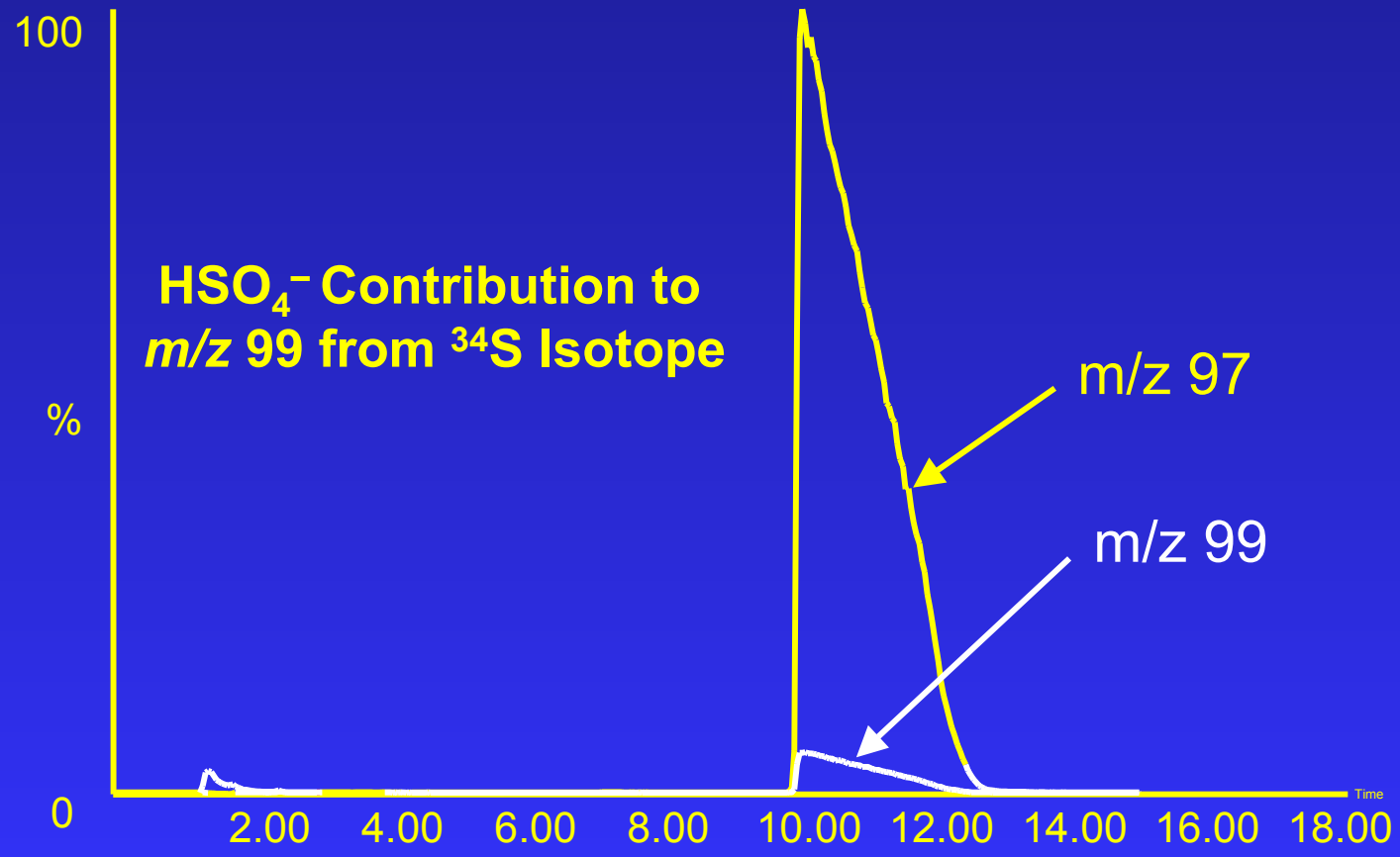
EPA Method 330.0
1.0 µg/L (ppb) Perchlorate in Reagent Water



A Good Thing!

What Can Ruin a Good Thing?

- A contaminant co-elutes with ClO_4^- and has the same *m/z* as ClO_4^-.

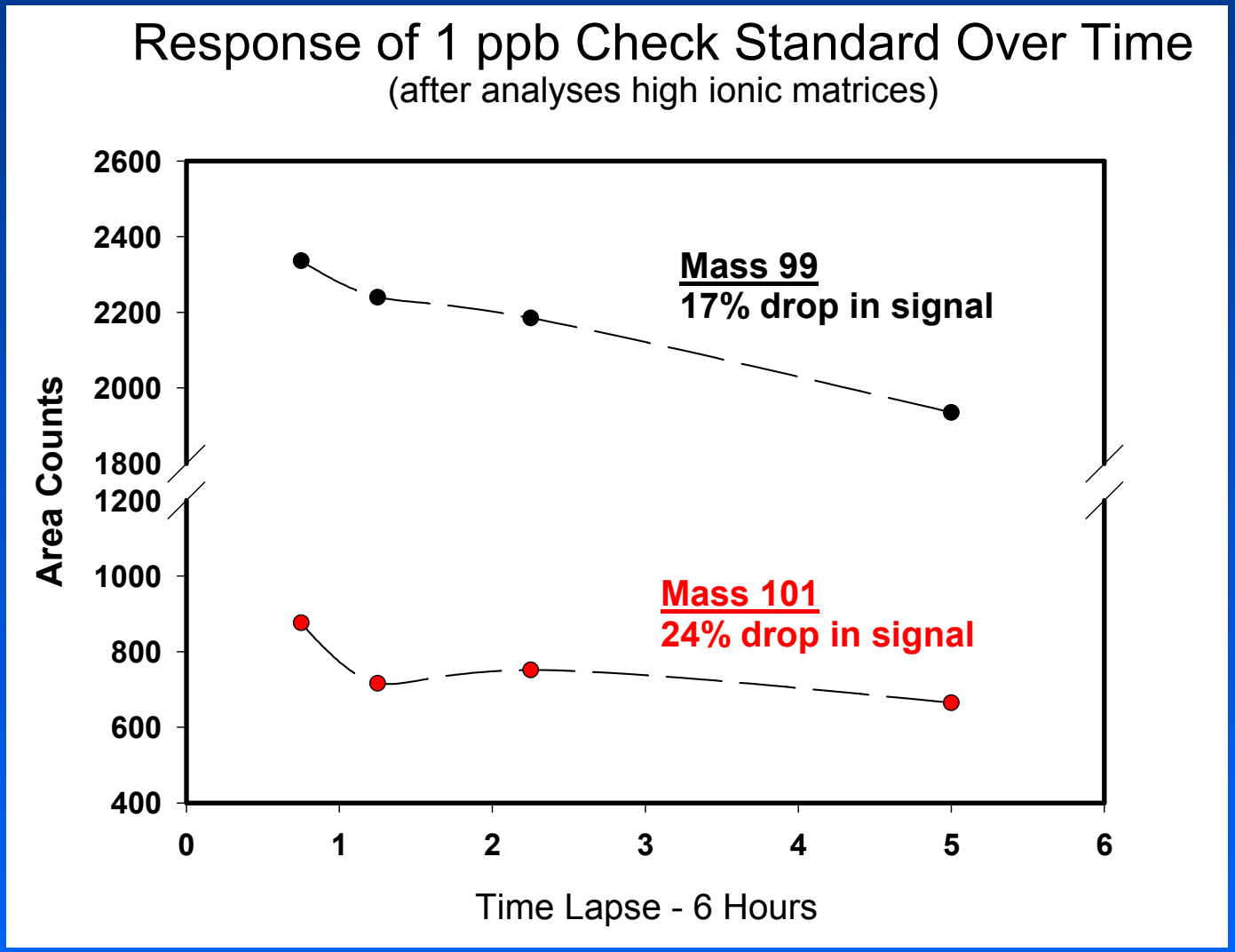


What Else Can Ruin a Good Thing?

- A contaminant co-elutes with ClO_4^- and has the same *m/z* as ClO_4^-.
- **Gradual loss of signal intensity with cone fouling if matrix diversion is not used with high TDS samples.**

EPA Method 330.0

Without Matrix Diversion of High TDS Salts

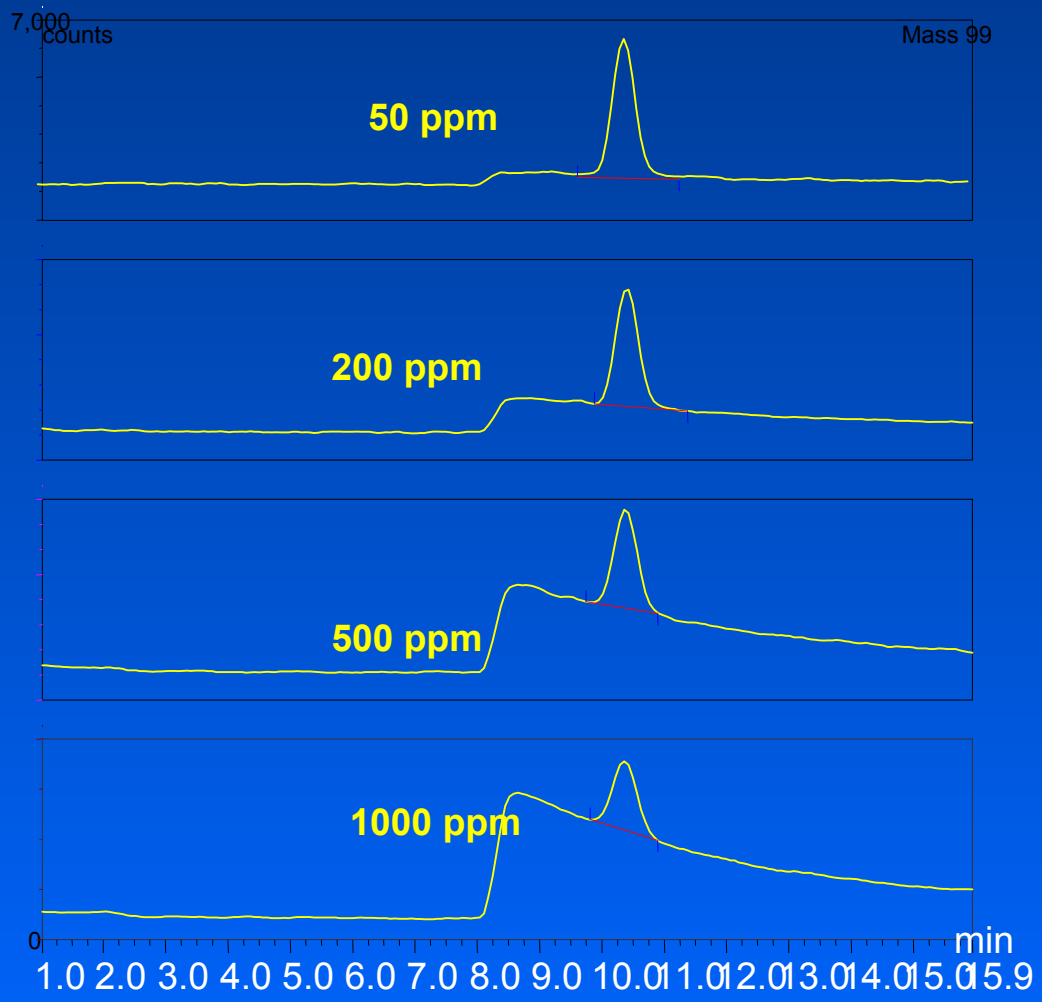


What Else Can Ruin a Good Thing?

- A contaminant co-elutes with ClO_4^-. and has the same *m/z* as ClO_4^-.
- Gradual loss of signal intensity with cone fouling.
- **A contaminant co-elutes with ClO_4^- and causes ion suppression.**

EPA Method 330.0

1.0 ug/L Perchlorate in Varying Concentrations of Cl^-, SO_4^{2-} and CO_3^{2-}



NOTE: Matrix valved to bypass MS until 2 minutes before ClO_4^- elutes

EPA Method 330.0

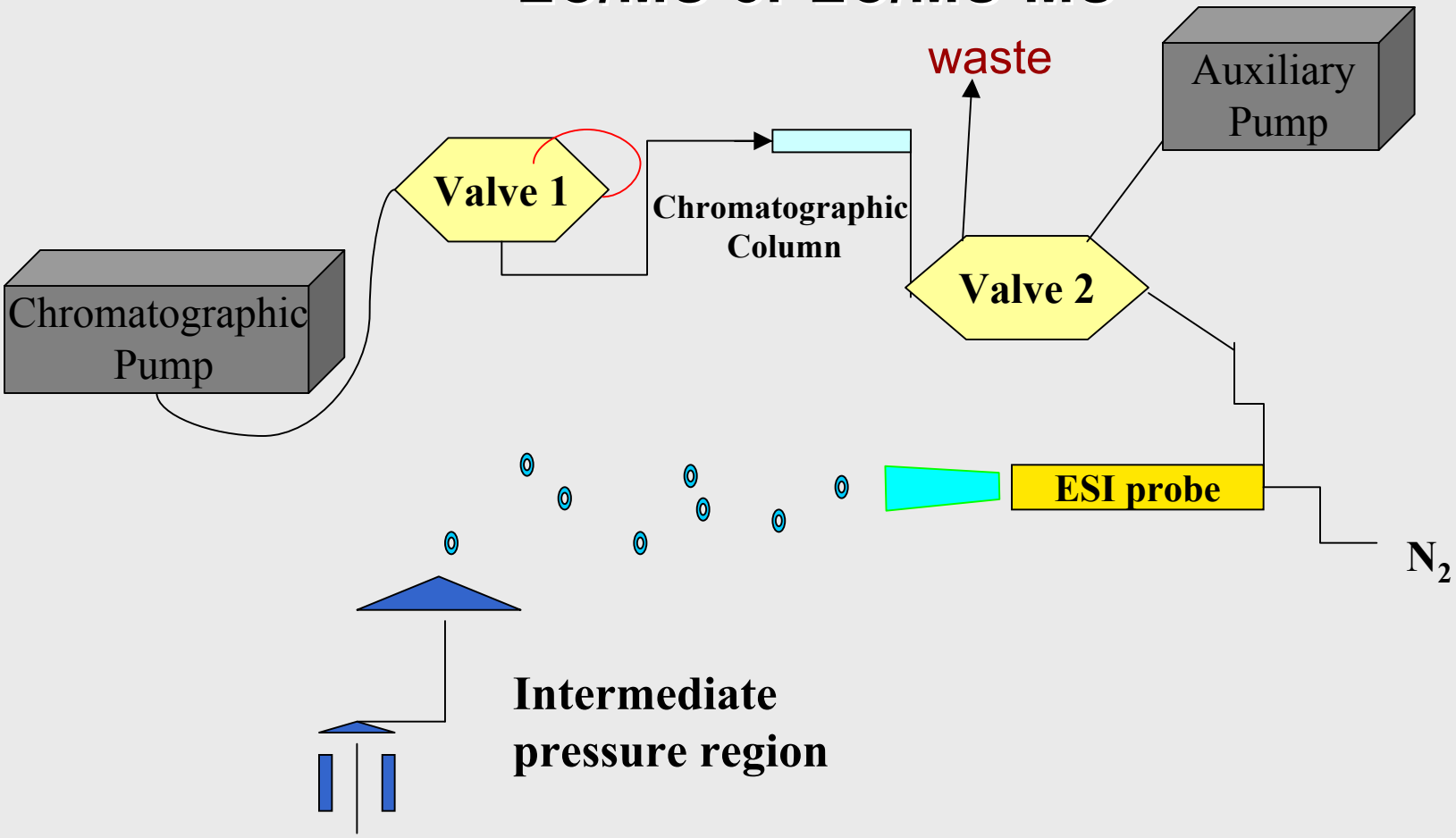
- Interferences
  - Monitor ion ratio
  - Good chromatography
- Matrix
  - Use labeled perchlorate standard to:
    - Correct for suppression (to a point)
    - Instrument drift
  - Good chromatography
  - Matrix diversion

Labeled Perchlorate Standard

- $^{35}Cl^{18}O_4^-$ - *m/z* 107
- We have monitored labeled standard for oxygen exchange in water and in hydroxide over time.

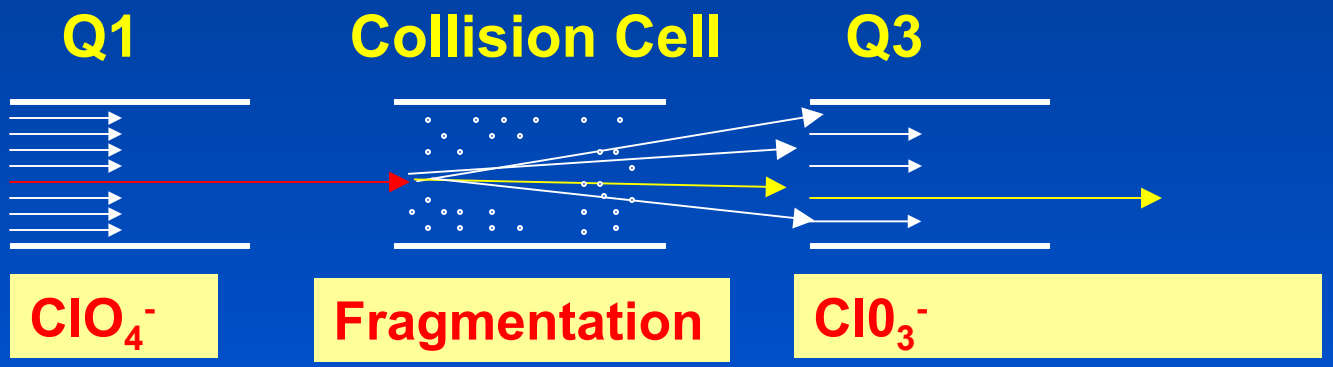
EPA Method 331.0

LC/MS or LC/MS-MS



MS-MS Approach

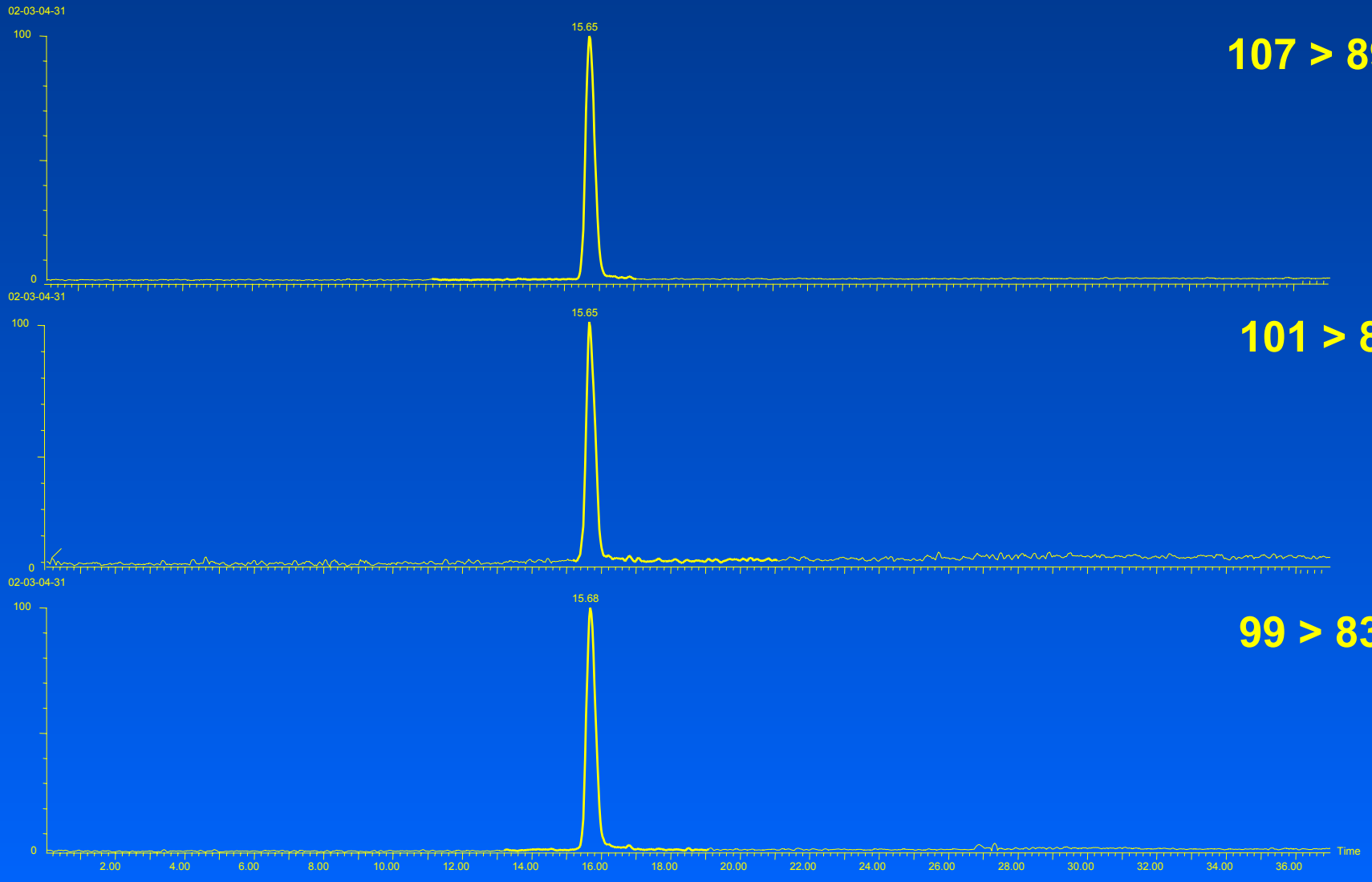
Selected Reaction Monitoring (SRM)
Multiple Reaction Monitoring (MRM)



- Expensive
- Complex
- Selectivity
- Sensitivity in complex matrix

EPA Method 331.0

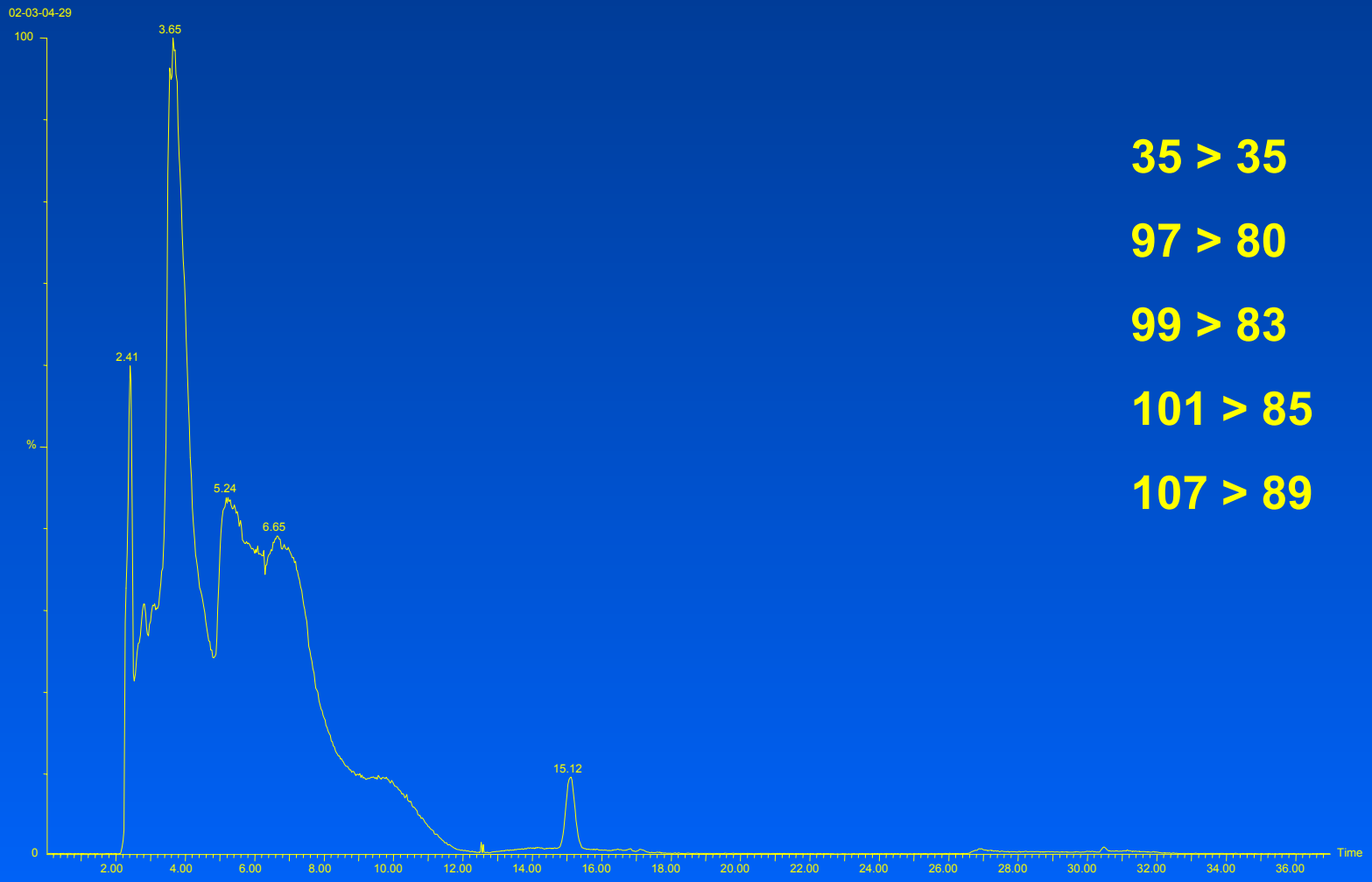
1 ug/L (ppb) Perchlorate + 3000 mg/L Common Anions



EPA Method 331.0

1 µg/L (ppb) Perchlorate + 3000 mg/L Common Anions

MRM of 5 Channels



35 > 35

97 > 80

99 > 83

101 > 85

107 > 89

EPA Method 331.0

- Interferences
  - Monitor ion ratio
  - Good chromatography
- Matrix
  - Use labeled perchlorate standard to:
    - Correct for ion suppression (to a point)
    - Correct for Instrument drift
  - Good chromatography
  - Matrix diversion

Partners

- TSC - Cincinnati, OH
- NERL - Cincinnati, OH
- Dionex - Sunnyvale, CA

- NRMRL - Ada, OK
- Metrohm-Peak - Houston, TX
- Region 2 - Edison, NJ

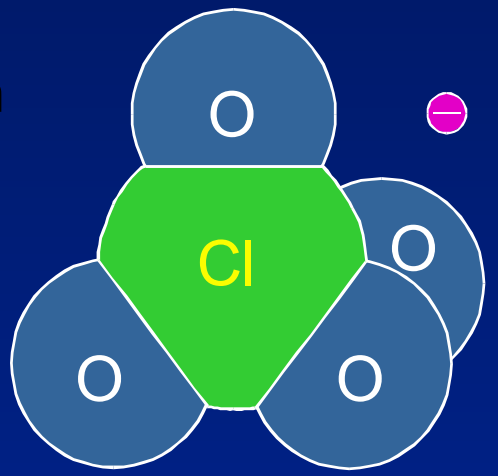
New EPA Perchlorate Methods
Instrumentation and Performance

Robert J. Joyce
Dionex Corporation
Sunnyvale, CA USA

DIONEX

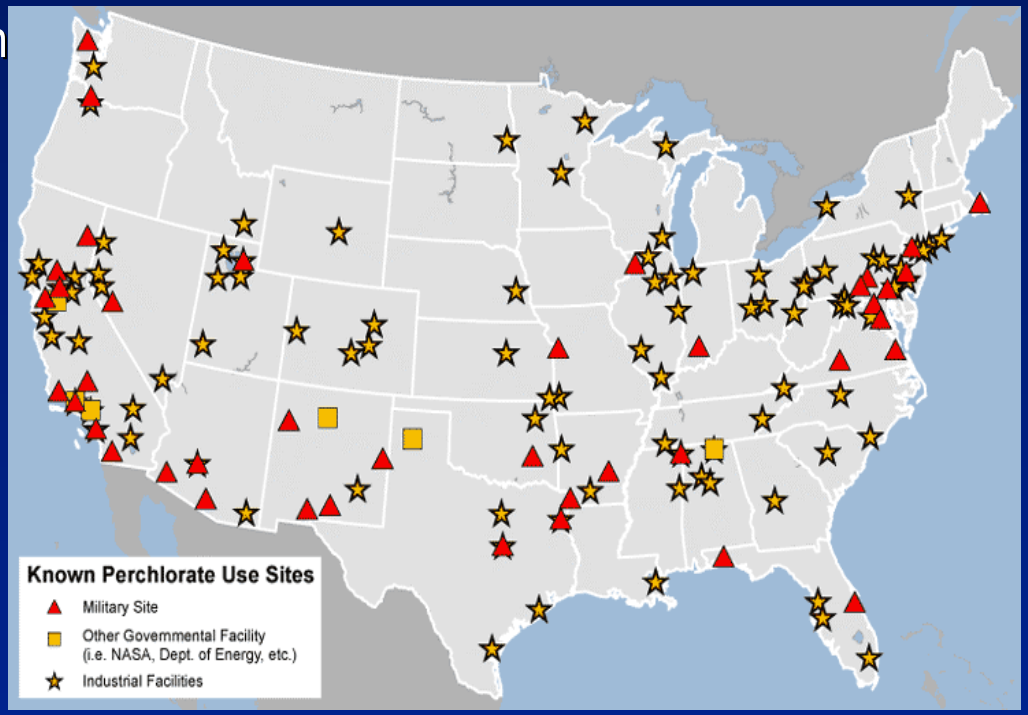
Perchlorate
General Chemistry

- The perchlorate anion (ClO_4^-) is a tetrahedral array of oxygen atoms around a central chlorine atom.
- The oxidation state of the chlorine is +7
- Perchlorate is a strong oxidizing agent (slightly weaker than dichromate or permanganate)
- Perchlorate reduction is extremely nonlabile (slow) and "rarely" observed in chemical systems
- Perchlorate is not reduced in 0.1-4.0 M acid;
- Other than some bacterial systems, perchlorate reduction is not observed
- ***Perchlorate is very stable in the chromatographic conditions encountered IC applications (i.e. sample matrix and eluents)***



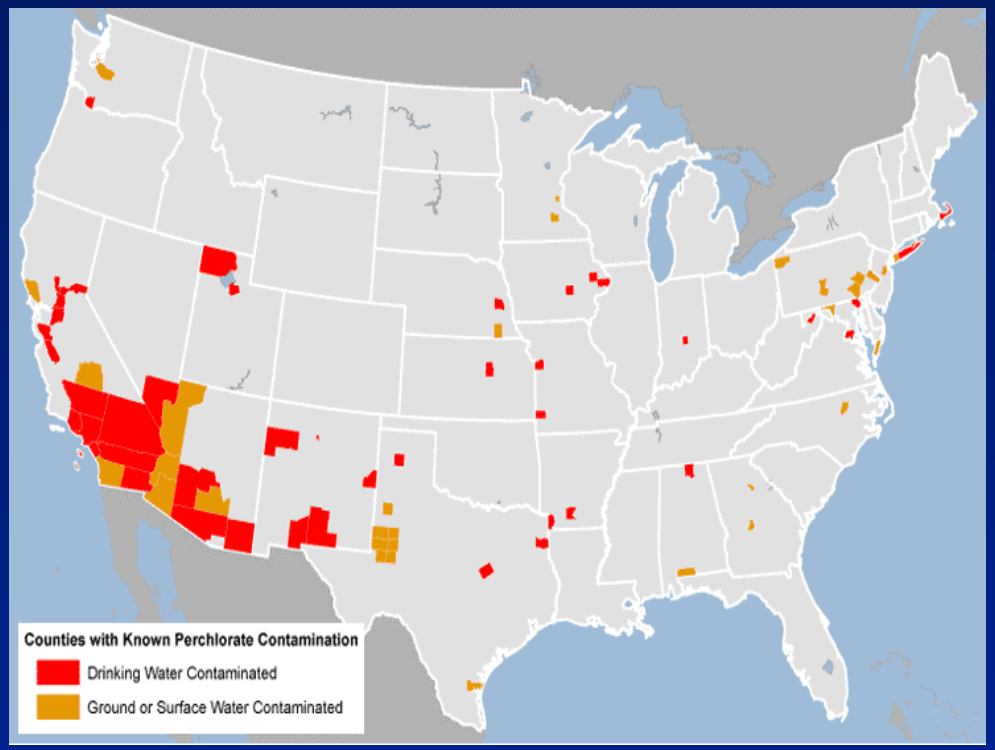
Sources of Perchlorate Contamination

- Rocket fuel production and waste disposal
- Munitions and explosives production and waste disposal
- Fireworks production, use and disposal
- Road flare production and disposal
- Hazardous waste disposal sites
- Phosphoric acid added to food and beverage products
- Indiscriminate chemical disposal



Where Perchlorate Has Been Found in Significant Concentrations (>1 ppb)

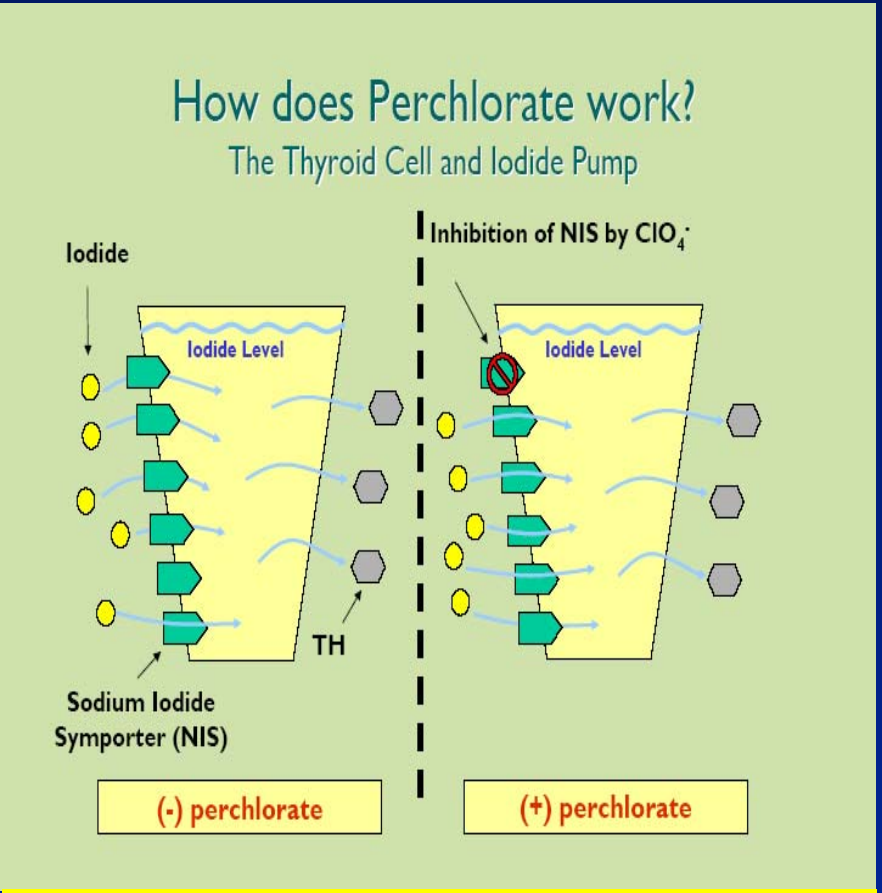
- Drinking water
  - Twenty states in the U.S.
- Aquifers associated with disposal sites
- Lakes and rivers associated with contaminated aquifers
  - Lake Mead (NV)
  - Colorado River (NV, CA, AZ)
- Crops irrigated by contaminated water
- Other foods (e.g., milk)



Perchlorate Health Issues*

"Perchlorate interferes with the iodide uptake into the thyroid gland."

- Interferes with thyroid hormone production
- Interferes with thyroid regulation of metabolism
- Interferes with neurological development of fetus and newborn
  - Behavior changes
  - Delayed development
  - Decreased learning capability
- Changes in thyroid hormone levels may result in thyroid gland tumors



Courtesy of Richard Pleus, Intertox

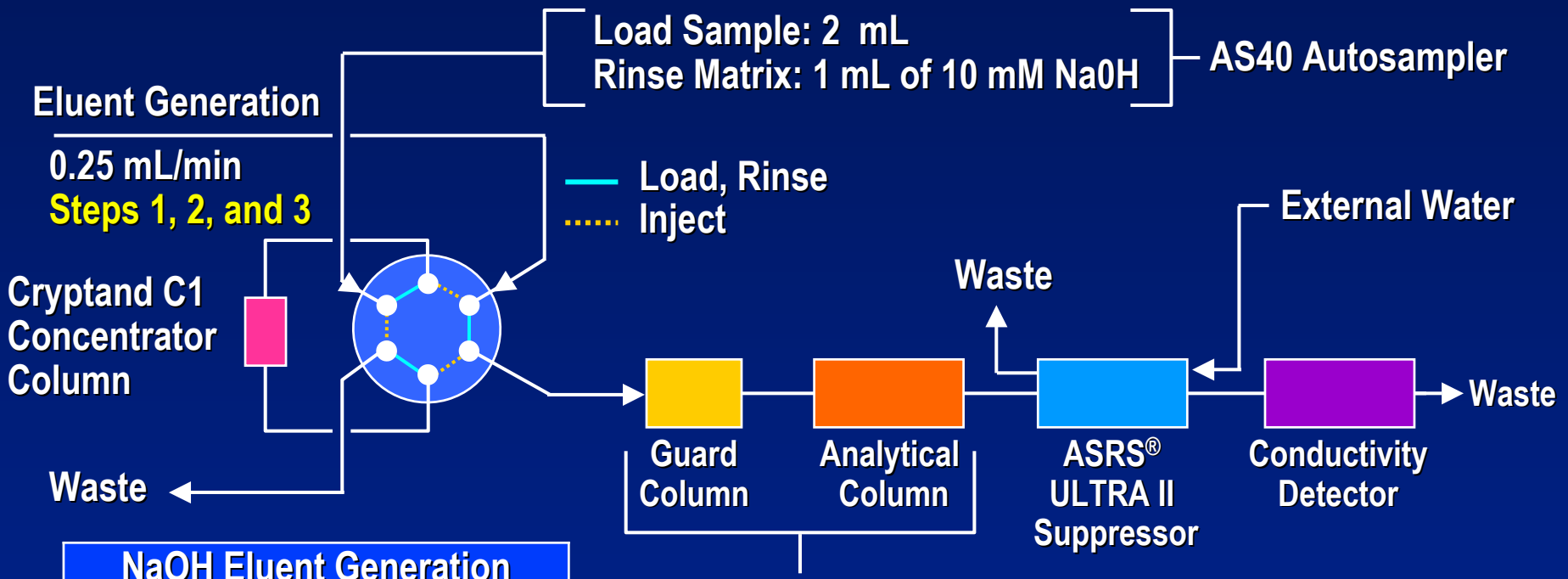
* U.S. EPA website (www.epa.gov)

Perchlorate—Preliminary Draft Toxicity Assessment

- EPA Draft Reference Dose (RfD)*
  - Exposure level, without appreciable risk of adverse affects over a lifetime: 0.00003 mg/kg/day
- Assessment considered perchlorate effect on:
  - Developing nervous system, including sensitive subgroups
  - Thyroid tumors
- Assumptions in conversion of RfD to drinking-water-equivalent level
  - 70 kg body weight
  - 2 L of water consumption per day
- EPA proposed drinking water limit = 1 µg/L (ppb)

*** U.S. EPA website (www.epa.gov)**

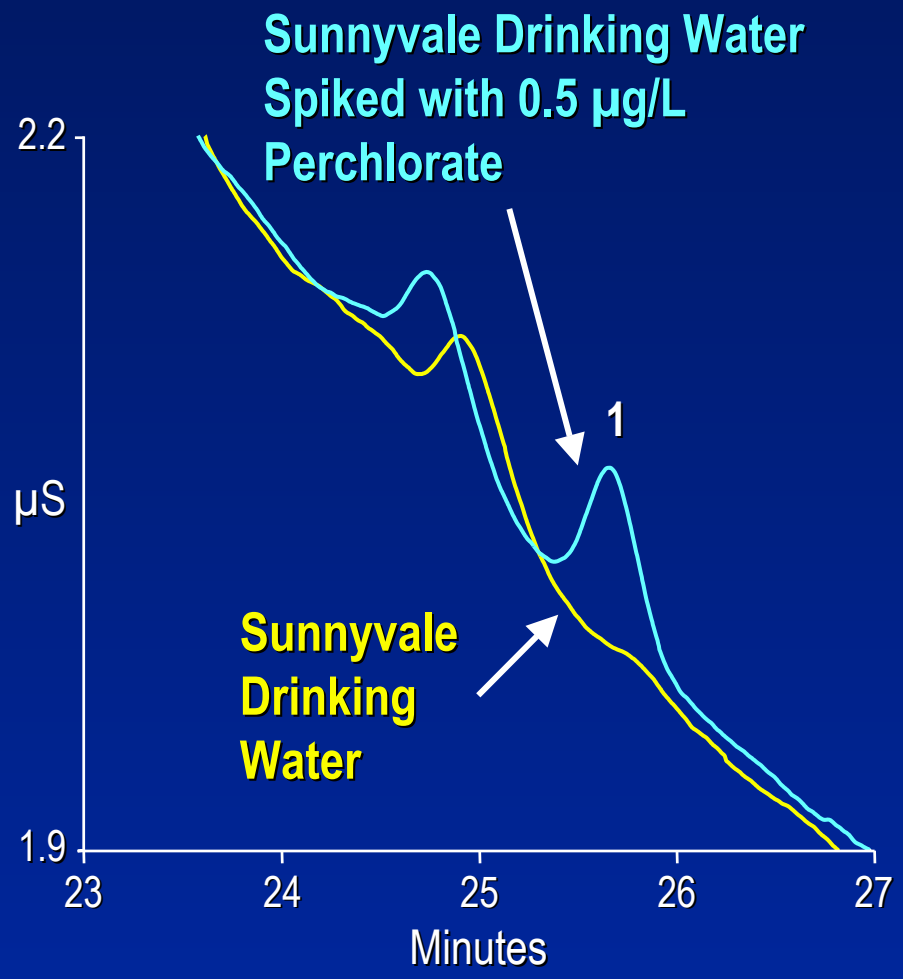
EPA Method 314.1*
Perchlorate Analysis Using RFIC with Preconcentration and Matrix Rinse



NaOH Eluent Generation			
Steps	Function	Conc.	Time
1.	Perchlorate Transfer	0.5 mM	12 Min
2.	Analysis	65 mM	13 Min
3.	Column Cleanup	100 mM	5 Min

* Under joint development by EPA and Dionex

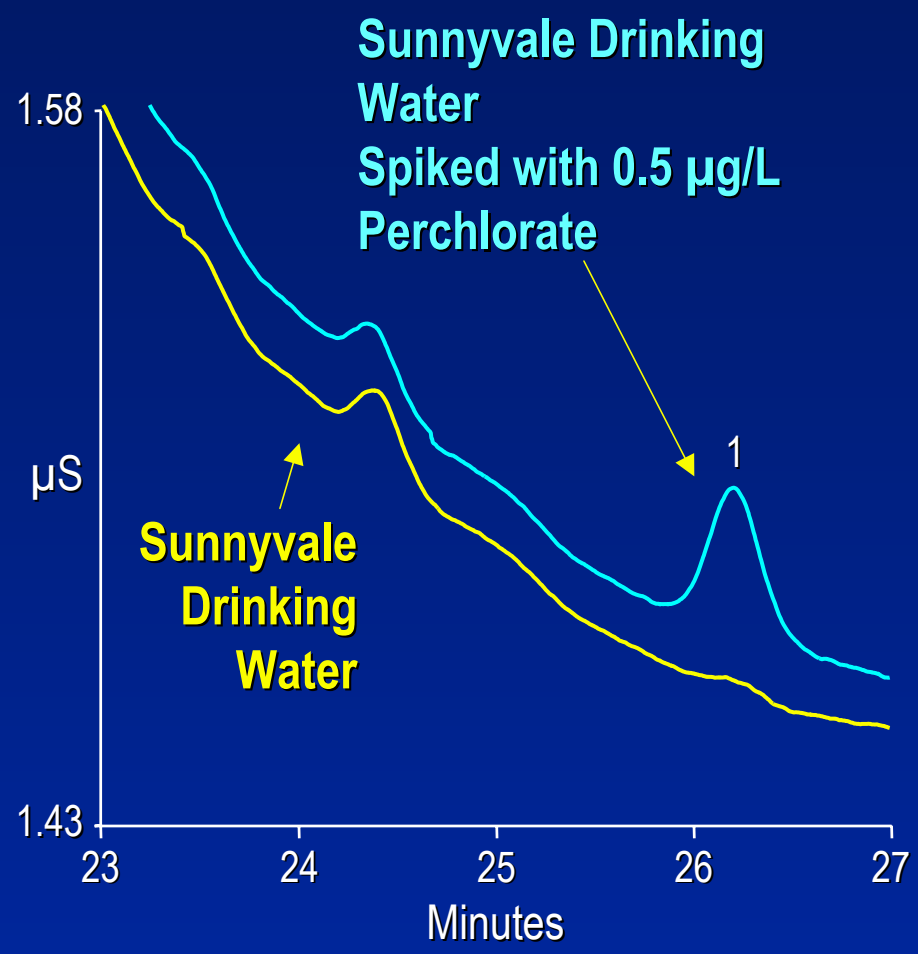
Determination of Trace Perchlorate in Drinking Water Using the AS16 Column and the Cryptand C1 Concentrator Column



Column:	IonPac® AG16, AS16, 2 mm
Concentrator Column:	IonPac Cryptand C1, 4 x 35 mm
Eluent:	Sodium hydroxide: 0.5 mM from 0–12 min, 65 mM from 12.1–28 min, 100 mM from 28.1–30 min.
Eluent Source:	EGC II NaOH cartridge with CR-ATC
Temperature:	35 °C
Flow Rate:	0.25 mL/min
Inj. Volume:	2 mL
Rinse Volume:	1 mL (10 mM NaOH)
Detection:	Suppressed conductivity, ASRS® ULTRA II, 2 mm, AutoSupression® external water mode, 100 mA
Peaks:	1. Perchlorate 0.5 µg/L (ppb) Recovery 92%

21088

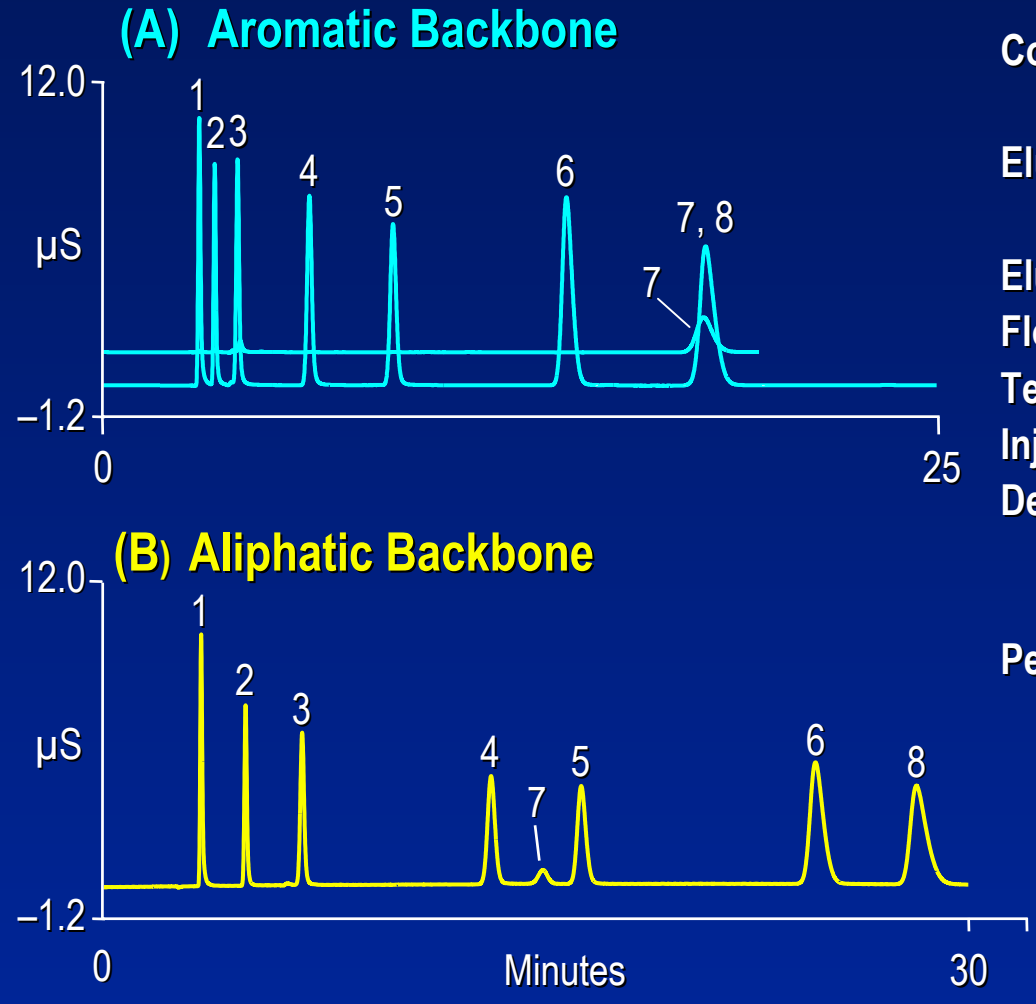
Determination of Trace Perchlorate in Drinking Water Using the AS20 Column and the Cryptand C1 Concentrator Column



Column:	IonPac® AG20, AS20, 2 mm
Conc.Column:	IonPac Cryptand C1, 4 x 35 mm
Eluent:	Sodium hydroxide: 0.5 mM from 0–12 min, 65 mM from 12.1–28 min, 100 mM from 28.1–30 min.
Eluent Source:	EGC II NaOH cartridge with CR-ATC
Temperature:	35 °C
Flow Rate:	0.25 mL/min
Inj. Volume:	2 mL
Rinse Volume:	1 mL (10 mM NaOH)
Detection:	Suppressed conductivity, ASRS® ULTRA II, 2 mm, AutoSupression® external water mode, 100 mA
Peak:	1. Perchlorate 0.5 µg/L (ppb) Recovery 92%

21089

Comparison of Primary and Confirmatory Column Performance When Using EPA Method 314.1* for Perchlorate



Columns:	(A) IonPac® AS16, 2 x 250 mm
	(B) IonPac AS20, 2 x 250 mm
Eluent:	NaOH (A) 35 mM
	(B) 25 mM
Eluent Source:	EGC II NaOH
Flow Rate:	0.25 mL/min
Temperature:	30 °C
Inj. Volume:	25 µL
Detection:	Suppressed conductivity, ASRS® ULTRA, 2 mm, AutoSuppression® recycle mode
Peaks:	1. Fluoride
	2. Chloride
	3. Sulfate
	4. Thiosulfate
	5. Iodide
	6. Thiocynate
	7. 4-Chlorobenzene sulfonate
	8. Perchlorate

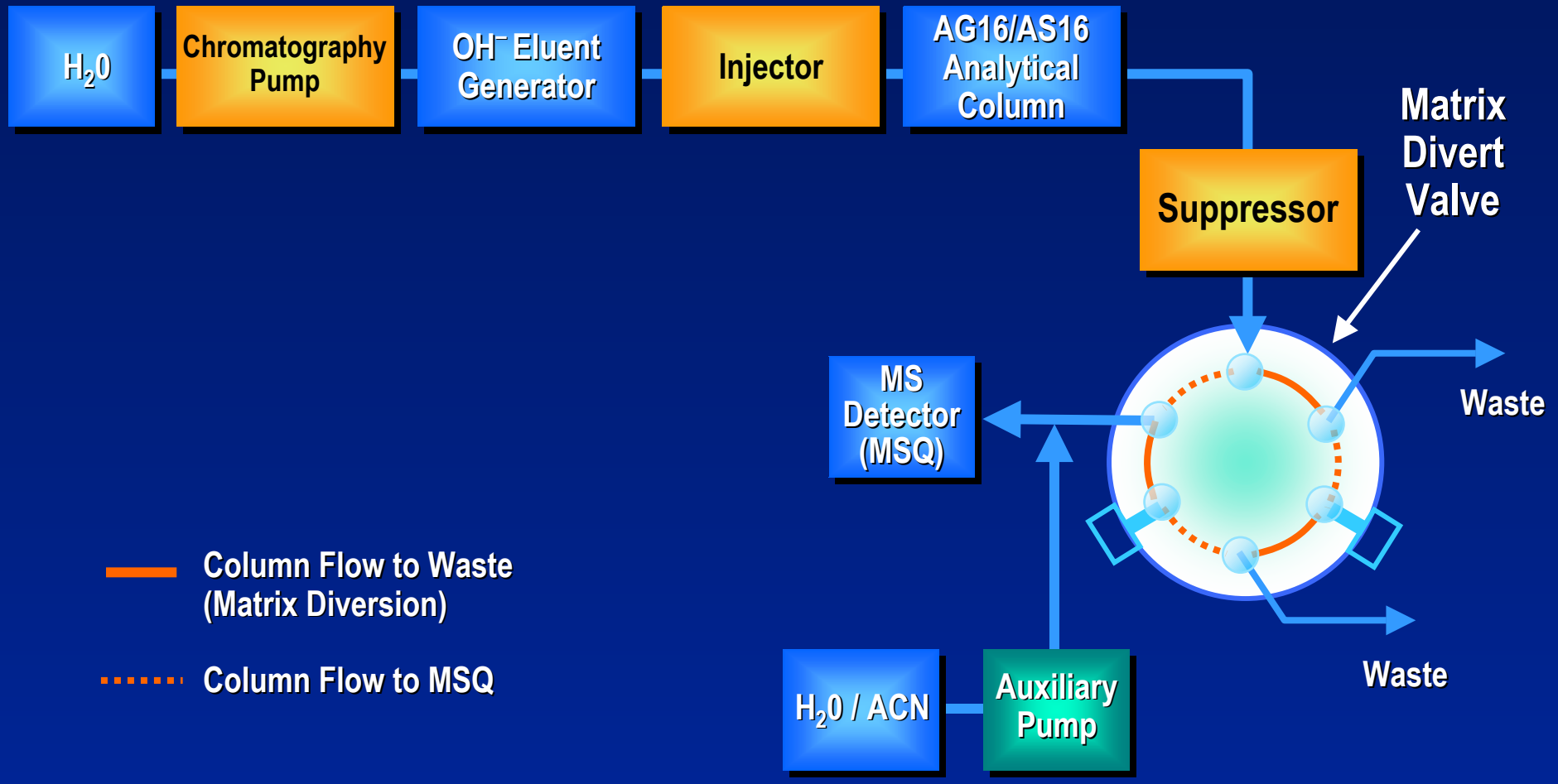
* Under joint development by EPA and Dionex

Benefits of Combining Suppressed IC with Mass Spectrometry Detection

Dionex ICS-2500 with MSQ™ Plus

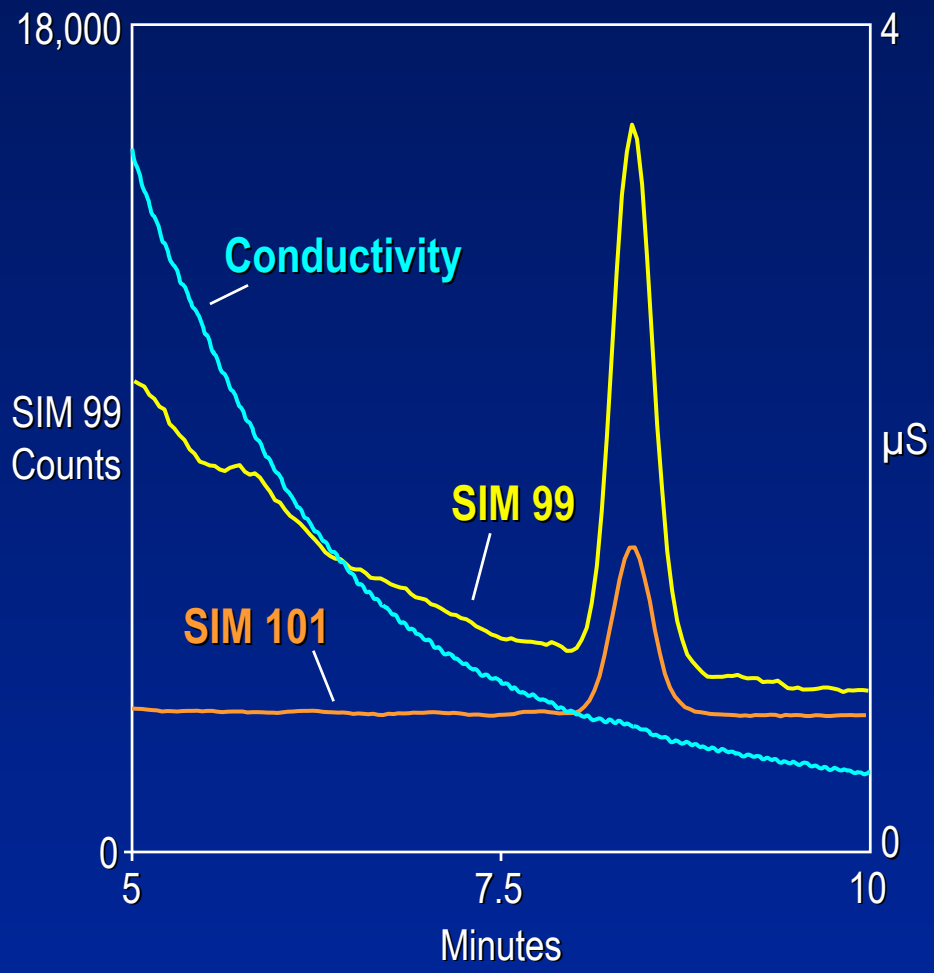
- Separate ionic analytes using standard IC conditions
- Suppressor permits use of high ionic strength eluents to get the benefits of high capacity columns
- Detect and identify analytes with high specificity
  - Avoid coeluting interferences to ensure accurate identification
  - Avoid background interferences to ensure highest analyte sensitivity
  - Identify analytes by mass and isotope ratios for added confirmation
  - Internal standard adds to method robustness
- Identify unknowns

EPA Method 330.0*
IC/MS System with Matrix Diversion



* Under joint development by EPA and Dionex

Perchlorate in California Groundwater
Using EPA Method 330.0 (IC/MS)*

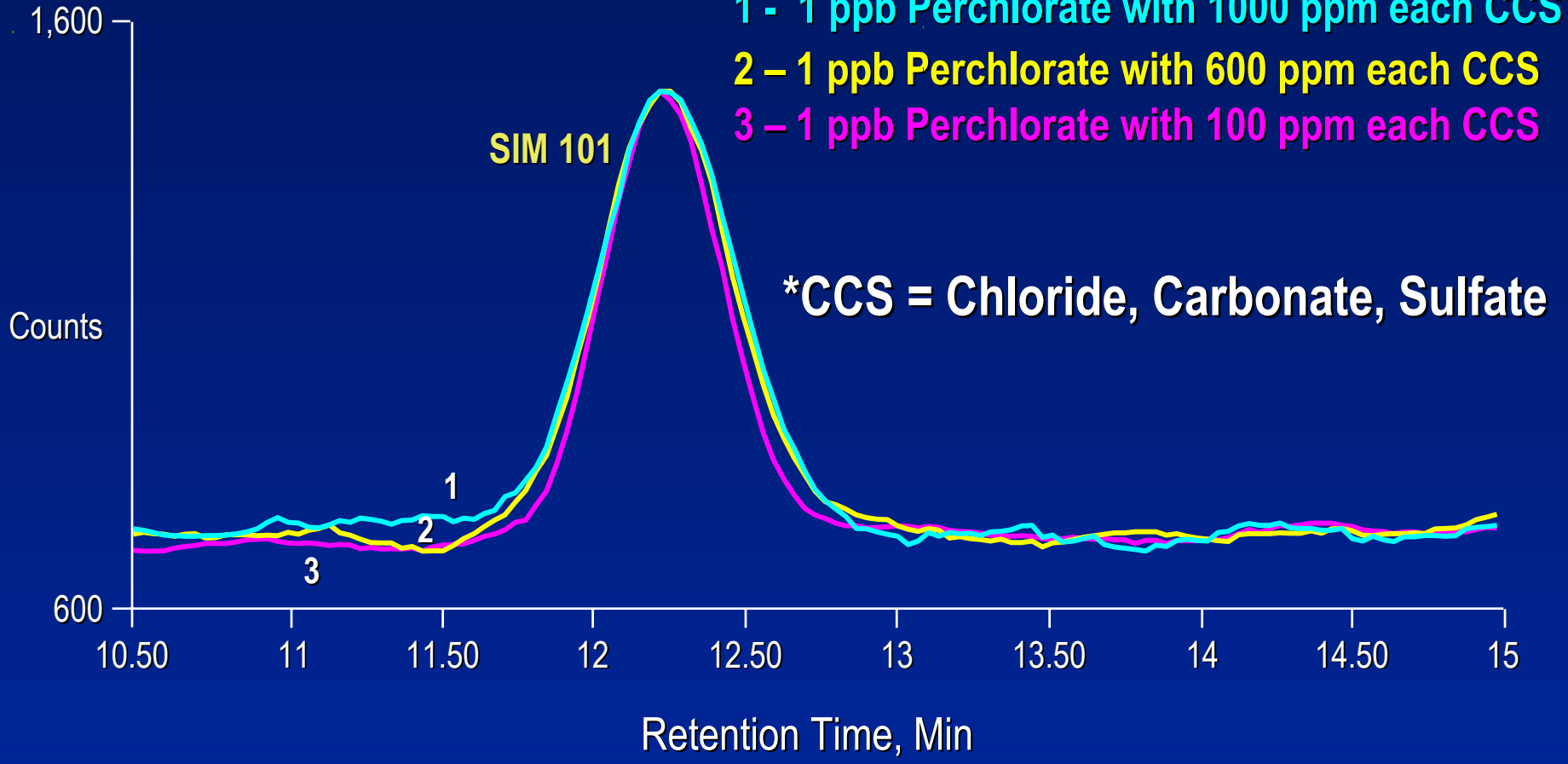


Column:	IonPac® AG16, AS16, 2-mm i.d.
Suppressor:	ASRS® ULTRA, 2 mm
Eluent:	65 mM KOH (EG40)
Flow Rate:	0.30 mL/min
Inj. Volume:	250 µL
Detection:	1. Conductivity
	2. MS, SIM 99, $^{35}ClO_4^-$
	3. MS, SIM 101, $^{37}ClO_4^-$
MS Conditions:	–ESI, 70 V, 350 °C
Sample:	Groundwater diluted 1/10
Peak:	Perchlorate

* Under joint development by EPA and Dionex

Low-Level Perchlorate Analysis Using RFIC-MD-MS with 50% Acetonitrile Solvent Wash

EPA Method 330.0*

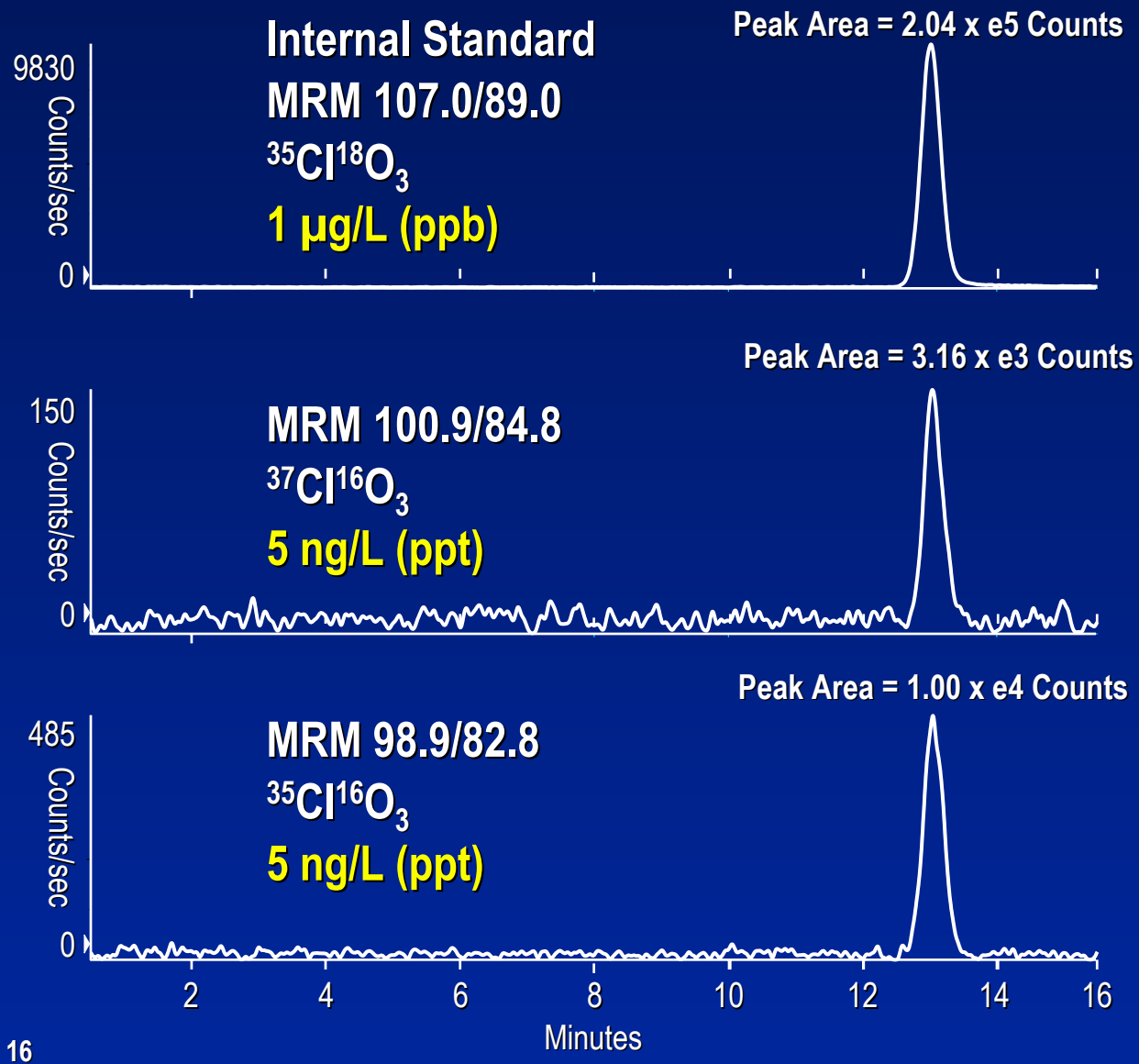


* Under joint development by EPA and Dionex

EPA Method 330.0*
IC/MS-MS System with Matrix Elimination

* Under joint development by EPA and Dionex

EPA Method 330.0
IC/MS-MS



IC System:
Dionex ICS 2500
with Matrix Diversion
and Auxiliary Pump

MS-MS System:
MDS Sciex API 2000

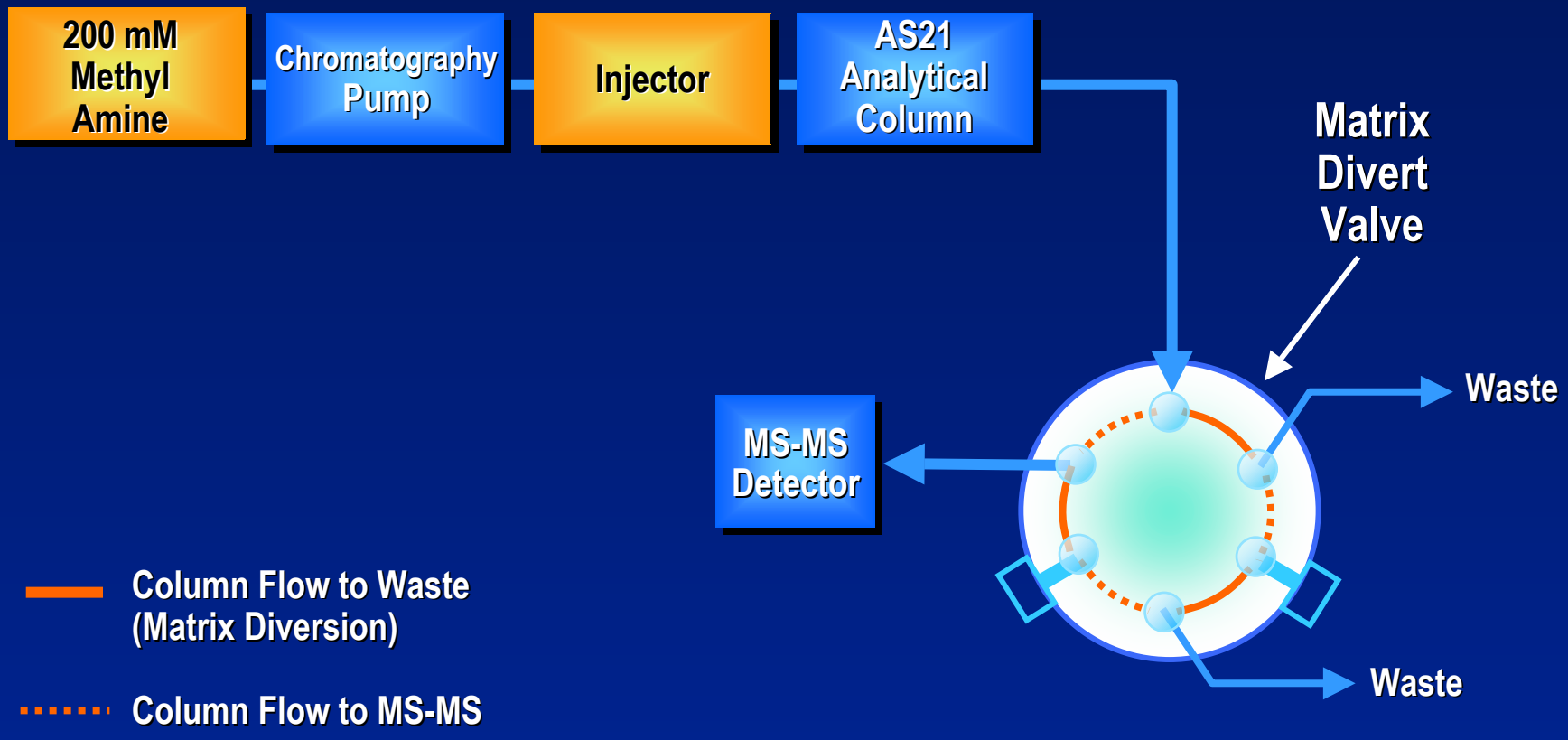
Internal Standard:
0-18 Perchlorate: 1 µg/L*

Inject Volume:
100 µL

Perchlorate Isotope Ratio:
10,000/3160 = 3.16

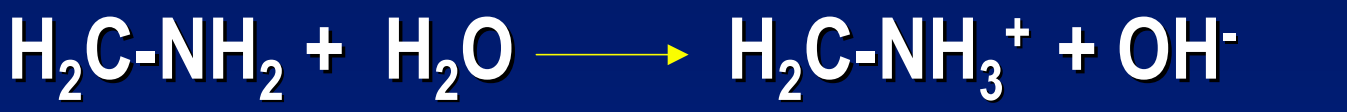
** Prepared from 1 mg/L stock standard (Dionex Corp)*

EPA Method 331.0*
LC/MS-MS System with Matrix Diversion



* Under joint development by EPA and Dionex

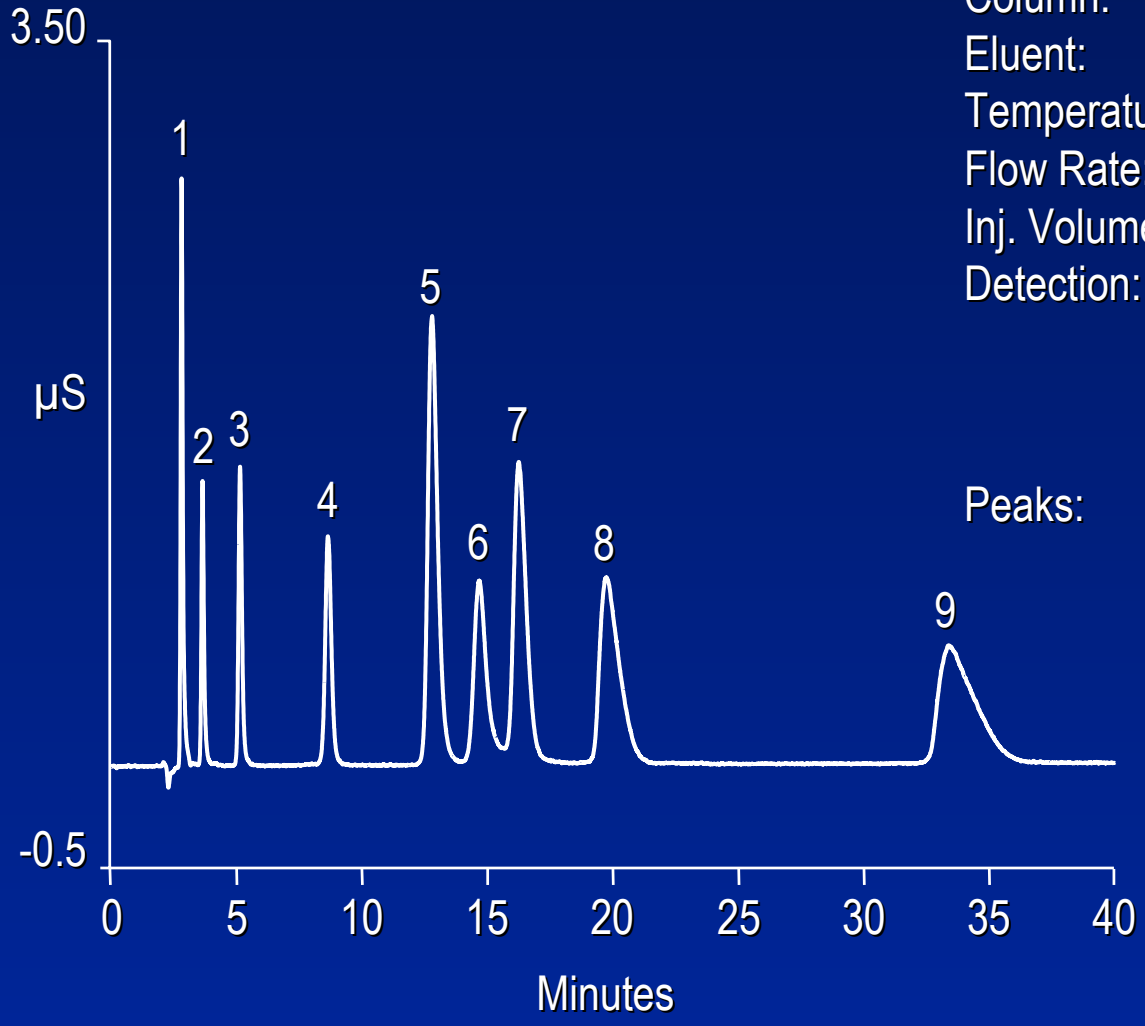
Non-Volatile Eluent for LC/MS-MS
Methylamine



Eluent Concentration: 200 mM (pH = 12)

At pH = 12 Hydroxide Concentration = 10 mM

Analysis of Environmental Anions Using a Methylamine Eluent

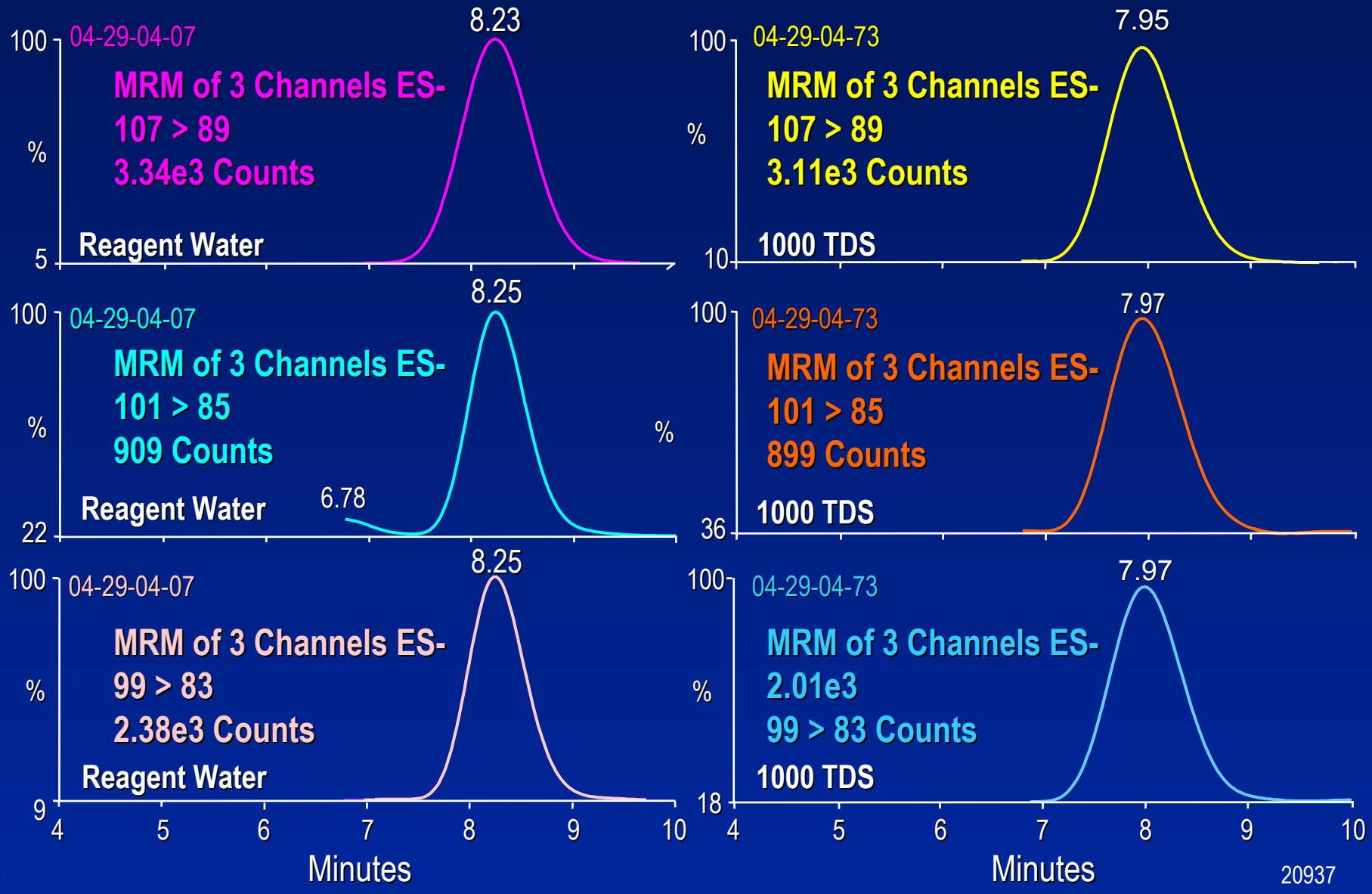


Column:	IonPac® AG21, AS21, 2 mm
Eluent:	200 mM Methylamine
Temperature:	30 °C
Flow Rate:	0.35 mL/min
Inj. Volume:	2.5 µL
Detection:	Suppressed conductivity, ASRS® ULTRA II 2mm, AutoSuppression® external water mode

Peaks:

1. Fluoride	2	mg/L (ppm)
2. Chloride	2	
3. Nitrate	5	
4. Sulfate	5	
5. Perchlorate	30	
6. Tungstate	30	
7. Chromate	30	
8. Phosphate	30	
9. Arsenate	30	

0.5 µg/L (ppb)Perchlorate Analysis Using EPA Method 331.0 (LC / MS-MS)



New EPA Perchlorate Methods
Low-level Performance

Method	Detection Limit (µg/L)	Lowest Concentration Method Reporting Limit (µg/L)
314.1	0.03	0.14
330.0 *m/z* 99 *m/z* 101*	 0.01 0.02	 0.05 0.10
331.0 MRM (*m/z* 83) SIM (*m/z* 101)	 0.005 0.008	 0.022 0.056

****M/Z* 101 used for Quantification**

MRM = Multiple Reaction Monitoring SIM = Single Ion Monitoring

Isotope Ratio Confirmation—$^{35}ClO_4^-/^{37}ClO_4^-$ = 3.1 (+/- 25%)

Summary

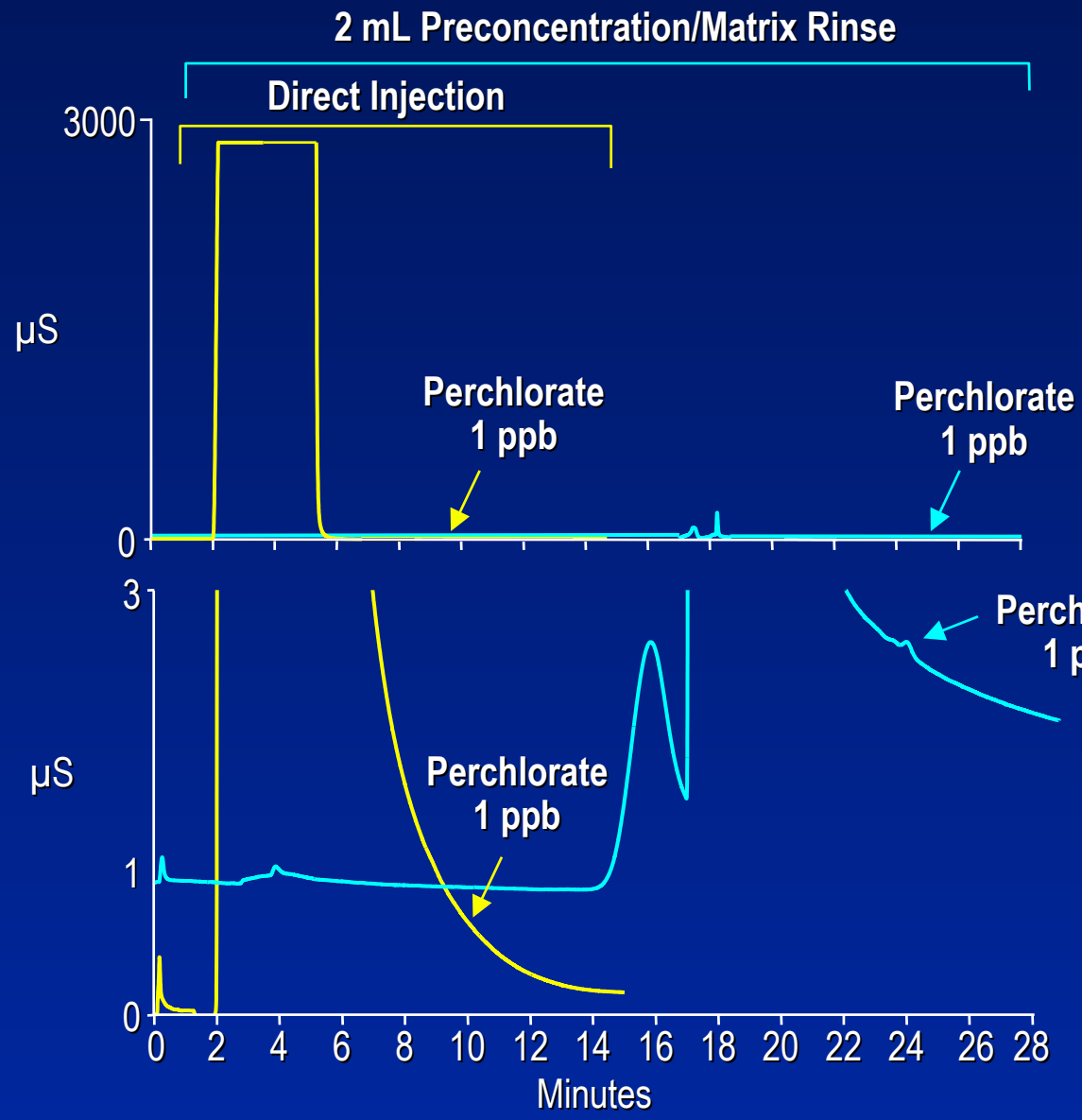
Method 314.1: sub-µg/L(ppb) sensitivity with second column confirmation

Method 330.0: ng/L(ppt) sensitivity with excellent inherent confirmation

Method 331.0: ng/L(ppt) sensitivity with outstanding inherent confirmation

Column performance delivers excellent chromatographic efficiency even when loaded with high TDS salts

Perchlorate by Direct Injection vs Preconcentration/ Matrix Rinse



Matrix	
Chloride	1000 ppm
Bicarbonate	1000
Sulfate	1000

SEVERN TRENT STL

Novel LC/MS-MS and IC/MS-MS Methods for Definitive Identification of Perchlorate in Environmental Samples

February 2005

Richard Burrows

rburrows@stl-inc.com

Leaders in Environmental Testing

SEVERN TRENT STL

MS Methods Used by STL for Perchlorate Analysis

- Conductivity – Method 314.0
- LC/MS, single-stage mass spectrometry
  - First used by STL in late 2001
- LC/MS-MS
  - Used as definitive method for 2.5 yrs, 2 instruments
- IC/MS-MS
  - Initial development began Fall 2003
- MS-MS methods preferred over MS

SEVERN TRENT STL

Characteristics Considered When Validating Methods

Linearity – predictable instrument response

Sensitivity – low concentration reliably detected

Precision – reproducibility of results

Accuracy – proximity of results to true value

Selectivity – ability to differentiate compound of interest from interferences

Ruggedness – ability of method to work properly in a variety of types of samples

SEVERN TRENT STL

MS-MS (Tandem) Detection

SOFT IONIZATION → ISOLATION → FRAGMANTATION

SEVERN TRENT STL

Mass Spectrometry (MS-MS)

- Unlike GC/MS electron impact (EI) source that fragments organic molecules → many ions
- LC/MS or IC/MS electrospray interface (ESI) ion source is lower energy → little or no fragmentation
- LC/MS-MS or IC/MS-MS ESI → fragmentation provides valuable structural information

SEVERN TRENT STL

One Detector System for Both LC/MS-MS & IC/MS-MS

Micromass Quatro Ultima

1st Stage – Molecular Ions Selected

			m/z
ClO_4^-	= $^{35}Cl+4^{16}O$	=	99
	= $^{37}Cl+4^{16}O$	=	101

2nd Stage – Fragmentation with collision gas

3rd Stage – Daughter Ions Analyzed:

ClO_3^-	= $^{35}Cl+3^{16}O$	=	83
	= $^{37}Cl+3^{16}O$	=	85

Expected ion ratio ($^{35}Cl/^{37}Cl$) = 3.08

SEVERN TRENT STL

Improved Selectivity by LC/MS-MS and IC/MS-MS

Identification based on 5 characteristics:

1. Retention time from ion exchange separation
2. Negative ions formed in electrospray interface
3. Molecular ions produced in first stage
4. Transition to characteristic daughter ions, detected in third stage
5. Ion ratios provide confirmation

SEVERN TRENT STL

Limitation of LC/MS-MS (2003 Technique)

- Many separation columns tried while developing LC/MS-MS method
- Separation of Perchlorate from some species, including SO_4^{2-}, incomplete, causing ion suppression
- Reliable identification not an issue, but
- Accuracy must be carefully monitored to avoid low bias

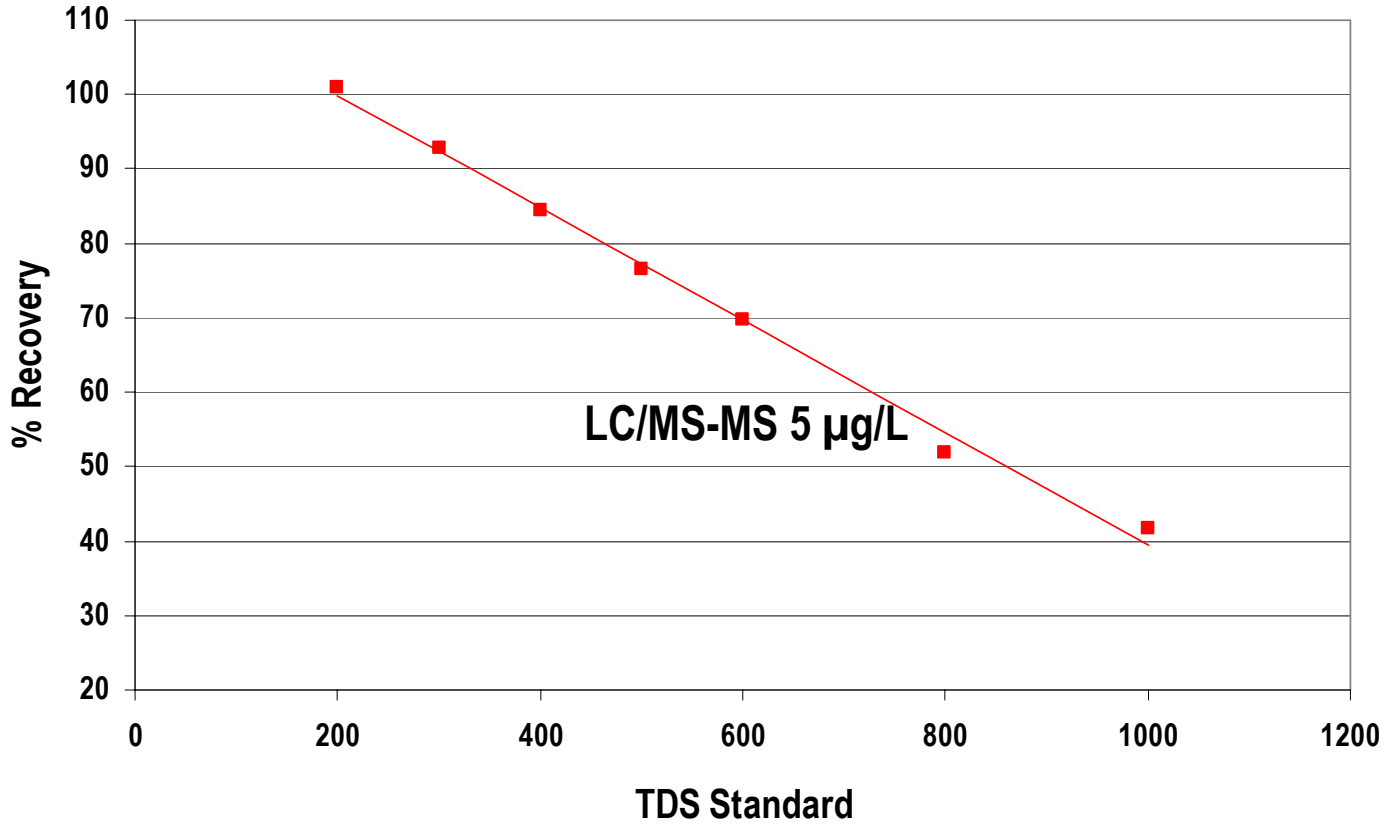
SEVERN TRENT STL

Perchlorate Analysis by IC/MS-MS

- **Extraction**
  - Soils tumbled with DI water
  - Waters are analyzed directly
- **IC Analysis**
  - IonPac® AG16/AS16 column set
  - Eluent generator producing a KOH mobile phase,
  - Eluent suppressor system to convert KOH to H_2O
  - Time actuated valves for sample diversion during the analysis
- **MS—ESI negative ion MS-MS detection, heavy chlorine isotope monitored for confirmation**
- **O–18 labeled perchlorate Internal Standard (IS)**
- **Calibration: 10–500 ng/L (ppt)**

SEVERN TRENT STL

ESI Ionization Suppression Using LC*/MS-MS (Without IS Correction)



TDS = mg/L, each, chloride, bicarbonate, sulfate

* No special effort to separate sulfate from perchlorate

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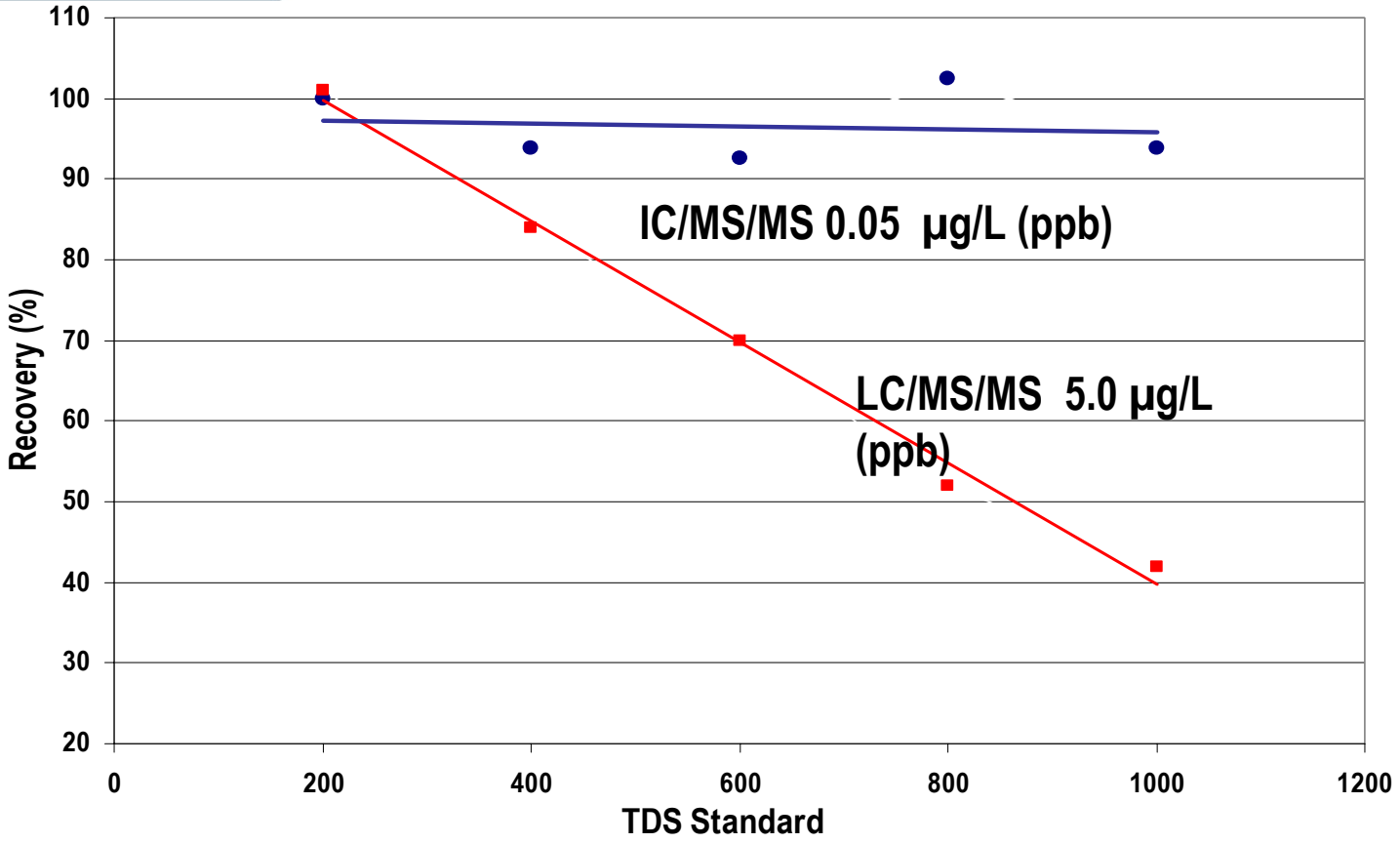
Controlling Ionization Suppression

- O–18 labeled Perchlorate used as an internal standard, improves precision and accuracy
- If internal standard recovery is too low,
  - Can perform pretreatment using cartridges to remove Cl^-, SO_4^{2-}, HCO_3^-, CO_3^{2-}

    (other interfering species may not be affected)
  - Can dilute samples, or better still:
- Use method with improved separation

ESI Ionization Suppression (Without IS correction) LC/MS-MS *vs.* IC/MS-MS



TDS = mg/L each chloride, bicarbonate, sulfate

Leaders in Environmental Testing

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IC/MS-MS Separation Technology

Ion Chromatograph	Dionex Model ICS-2500
Electrolytic Eluent Generation	KOH
Sample Injected	50 µL loop
Electrolytic Eluent Suppression	Yes
Separation Column	AG16/AS16 (2 mm)
Matrix Diversion	1st 10.5 minutes eluent diverted to waste

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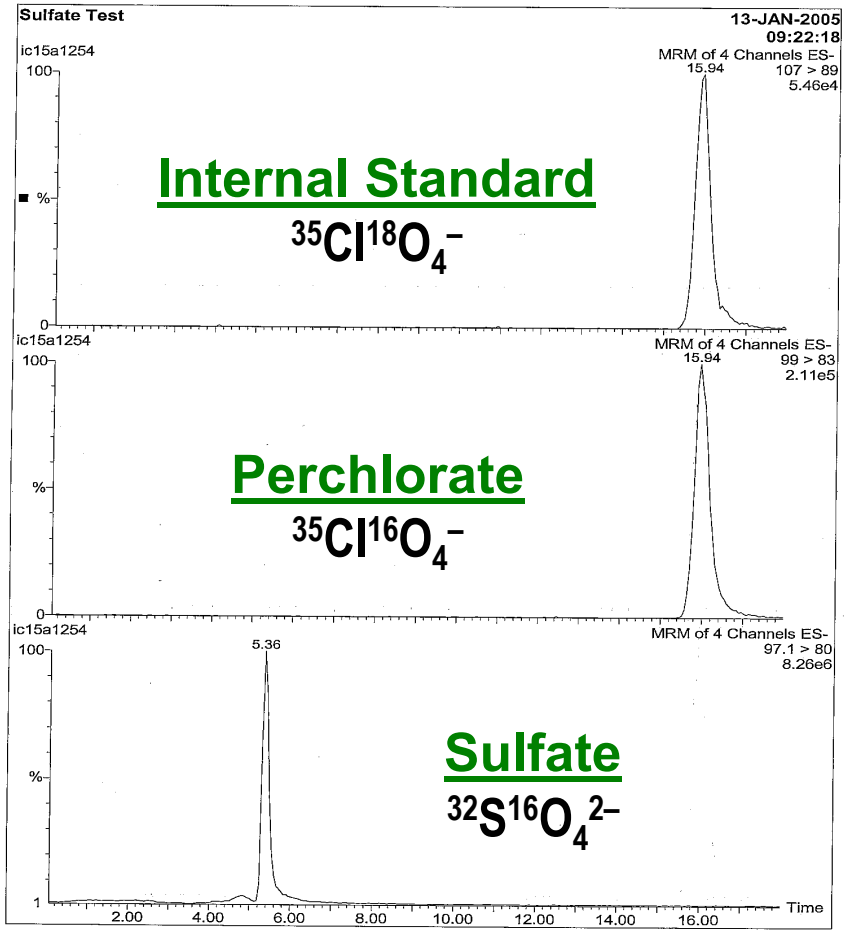
Separation of Perchlorate from Sulfate by IC/MS-MS



Leaders in Environmental Testing

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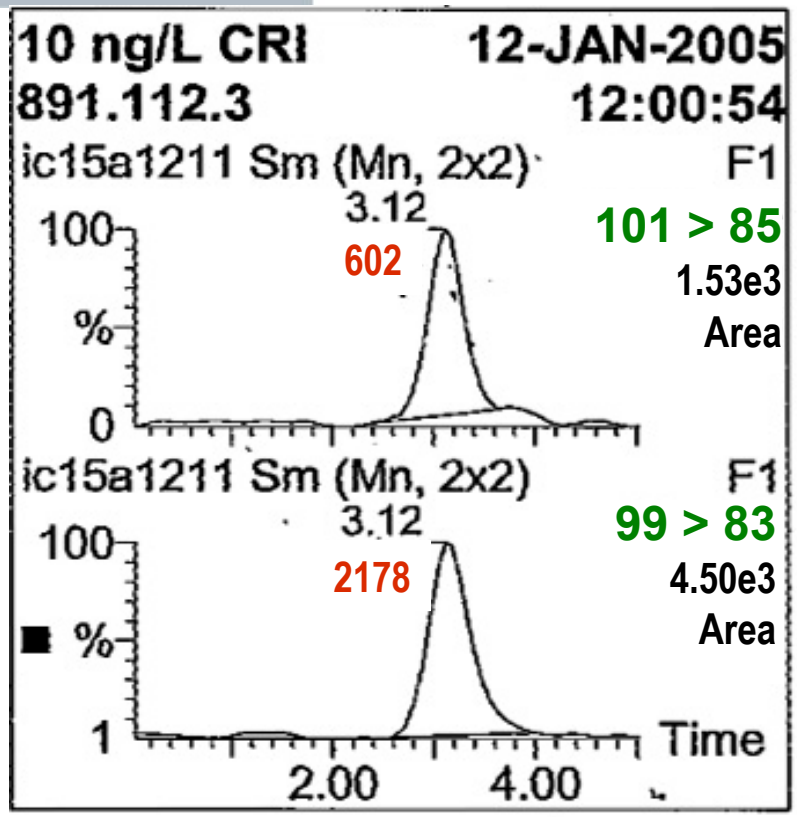
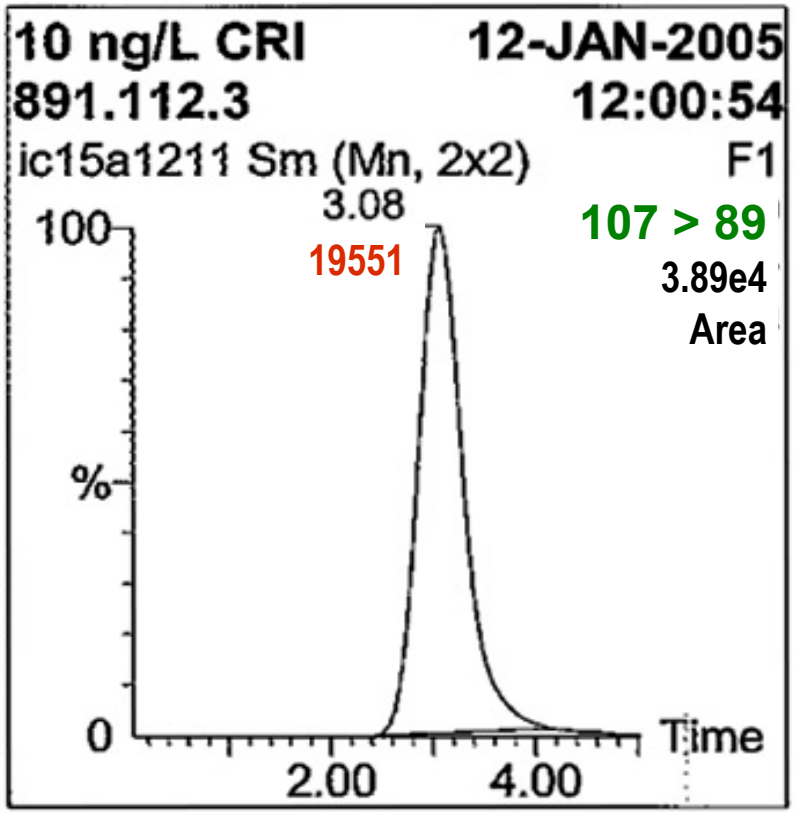
Perchlorate 10 ng/L (ppt) Standard in Reagent Water





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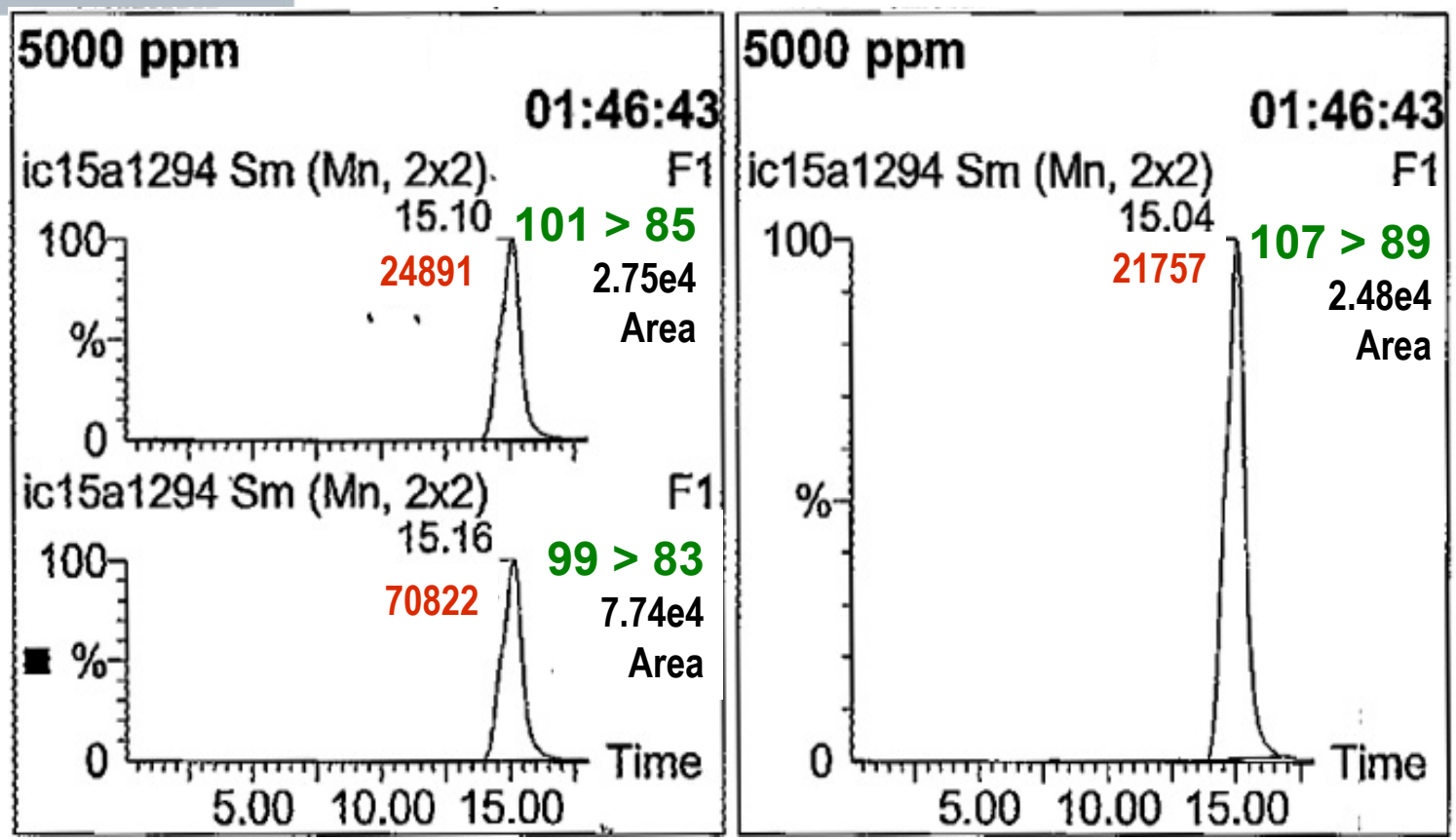
0.5 µg/L (ppb) Perchlorate in 5000 ppm High TDS Solution*



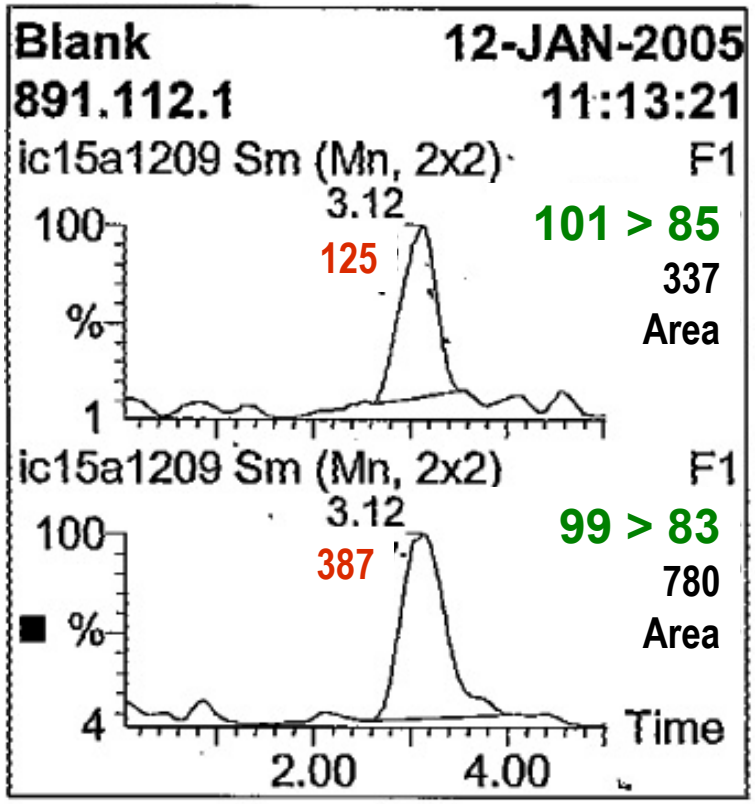
* 5000 ppm, each, chloride, bicarbonate and sulfate

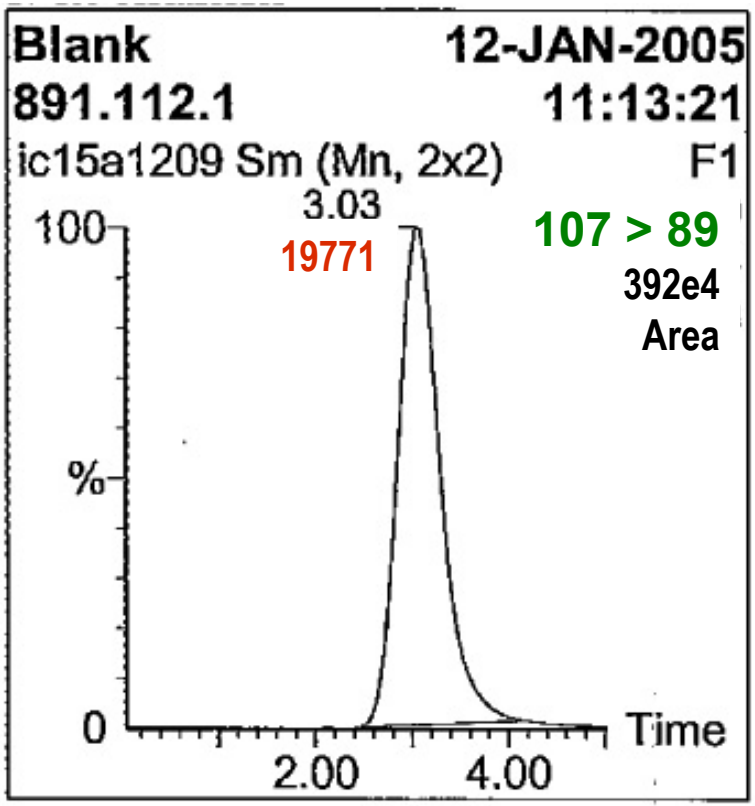
Leaders in Environmental Testing

SEVERN TRENT STL

Perchlorate Blank

Quantitation < 2ppt

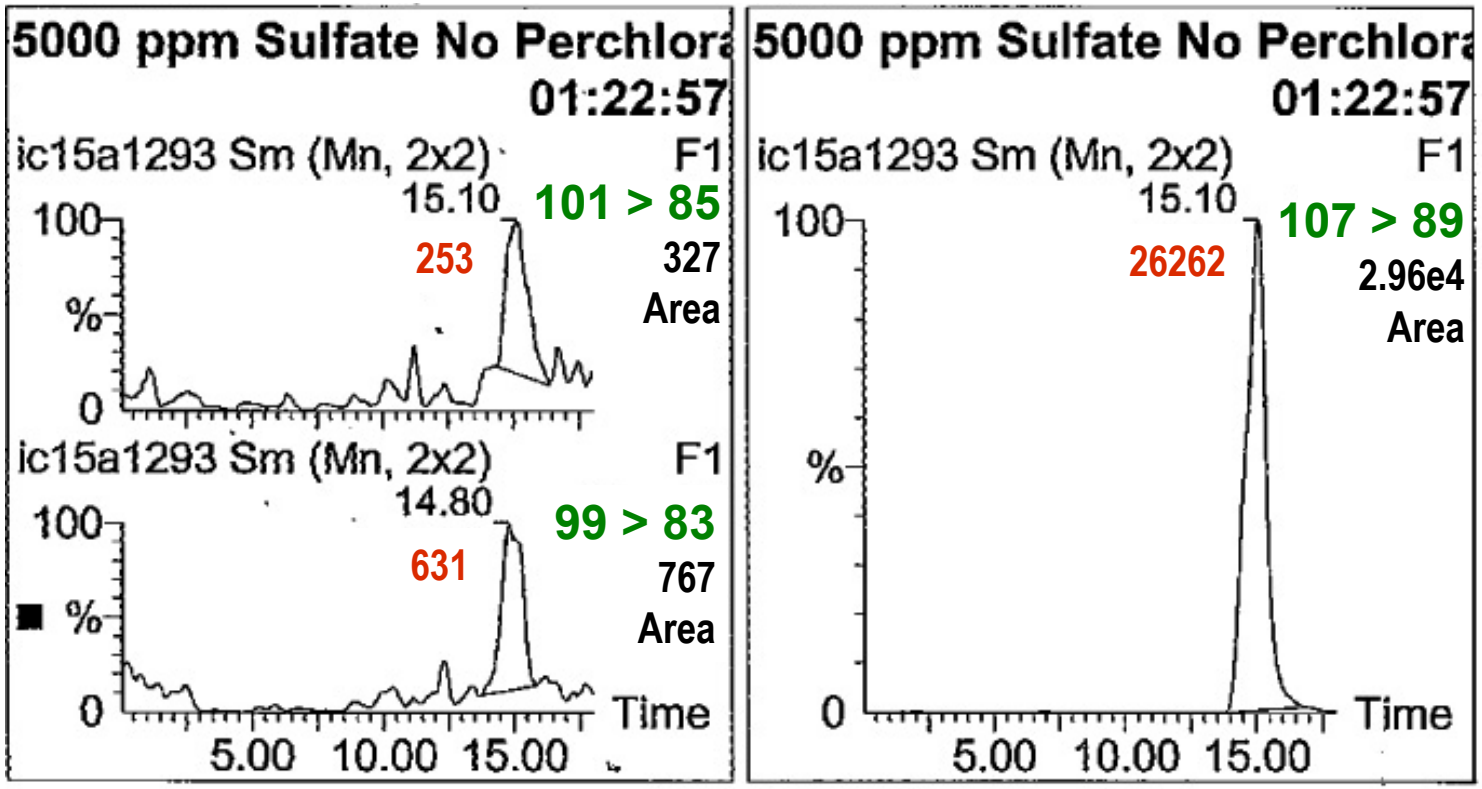




SEVERN TRENT STL

Perchlorate Blank in 5000 ppm High TDS Solution

Quantitation < 2ppt



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IC/MS-MS Precision / Accuracy Data in 5,000 ppm High TDS Water

Day2 & Day3:

Test No.	True Value (µg/L)	Mean Recovery (%)	RSD (%)
1	0.01	116.8	14.9
2	0.25	99.2	2.71
3	0.5	93.6	2.84

- Spikes prepared in water with 22,600 mg/L (2.3%) TDS
- No pretreatment
- 8 replicates tested per concentration, 4 on each of 2 days
- O-18 labeled perchlorate used as internal standard

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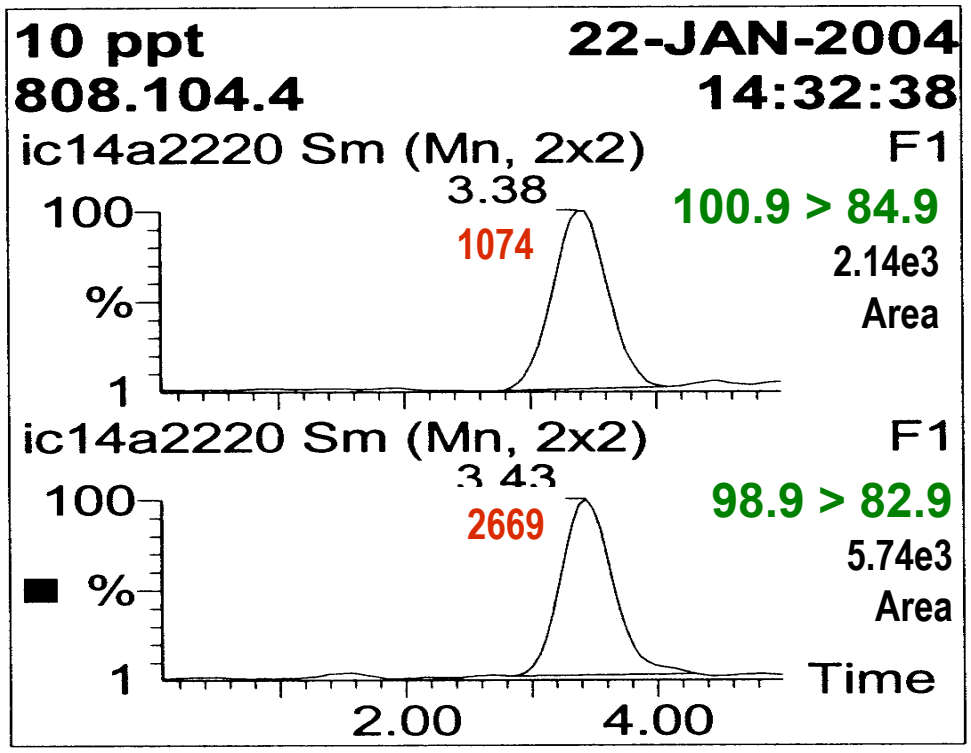
0.01 µg/L (ppb) Perchlorate Calibration Standard



← 85 ion

1,074 area counts

← 83 ion

2,669 area counts

True RT = 10.5+3.4 = 13.9 min.

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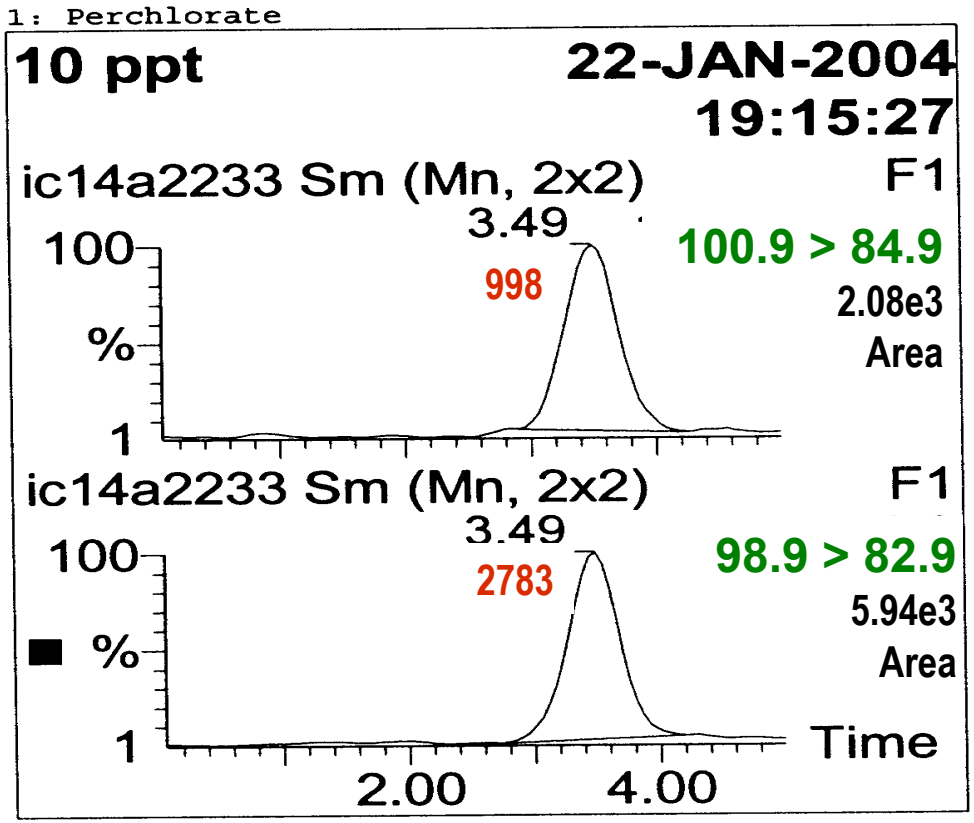
10 ng/L (ppt) Perchlorate In 5,000 ppm High TDS Water

After running high TDS samples for hours:



← 85 ion

998 area counts

← 83 ion

2,783 area counts

True RT = 10.5+3.5 = 14.0 min.

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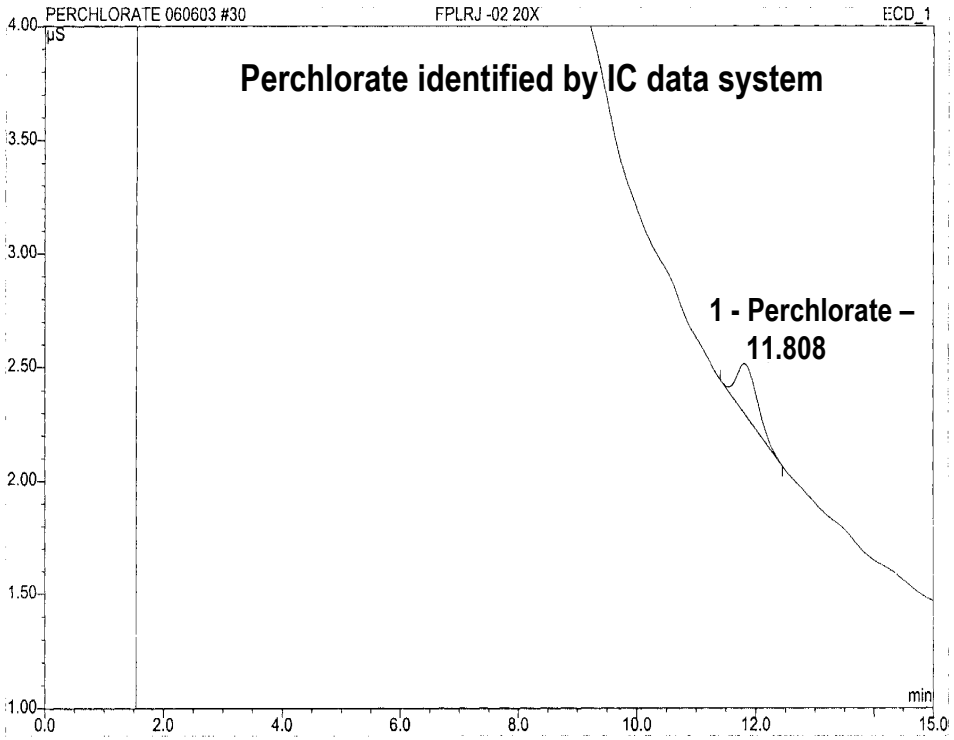
Real World Examples

Leaders in Environmental Testing

Treated Wastewater from Rocket Motor Manufacturer by 314.0

- Sample diluted 20x because of high conductivity
- Method 314.0 result, 670 µg/L perchlorate
- Similar to result reported by another laboratory
- Perchlorate was not detected, < 0.2 µg/L

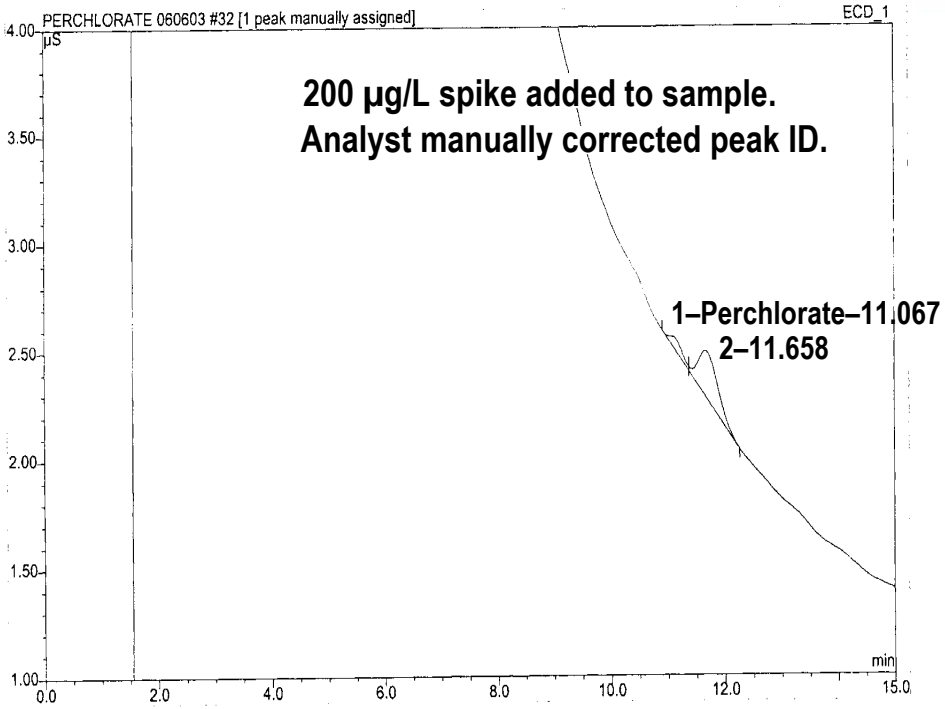
Which is correct?



No.	Ret.Time min	Peak Name	Height µS	Area µS*min	Rel.Area %	Amount ug/L	Type
1	11.81	perchlorate	0.217	0.091	100.00	670.546	BMB
Total:			0.217	0.091	100.00	670.546	

Treated Wastewater from Rocket Motor Manufacturer by 314.0 (Continued)

- 200 µg/L (ppb) perchlorate addition produces 2nd peak
- Original peak cannot be perchlorate

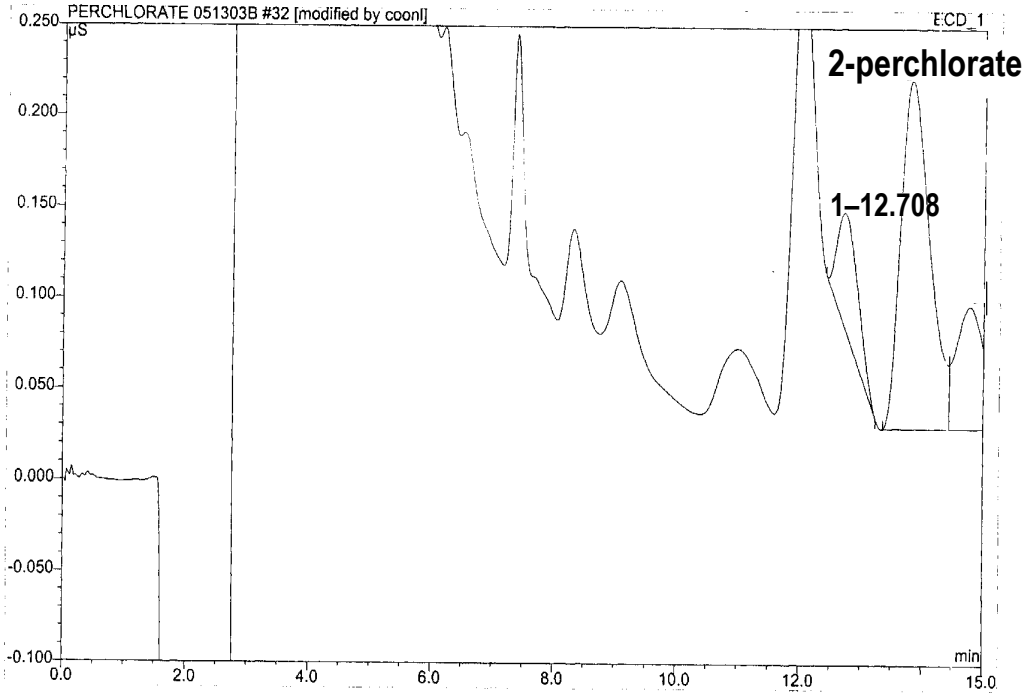


No.	Ret.Time min	Peak Name	Height µS	Area µS*min	Rel.Area %	Amount ug/L	Type
1	11.07	perchlorate	0.035	0.014	14.03	102.412	BM ^
2	11.66	n.a.	0.208	0.086	85.97	n.a.	MB
Total:			0.243	0.100	100.00	102.412	

Cooling Tower Water by Method 314.0

- Sample diluted 5x because of high conductivity
- Calculated result :

  194 µg/L perchlorate

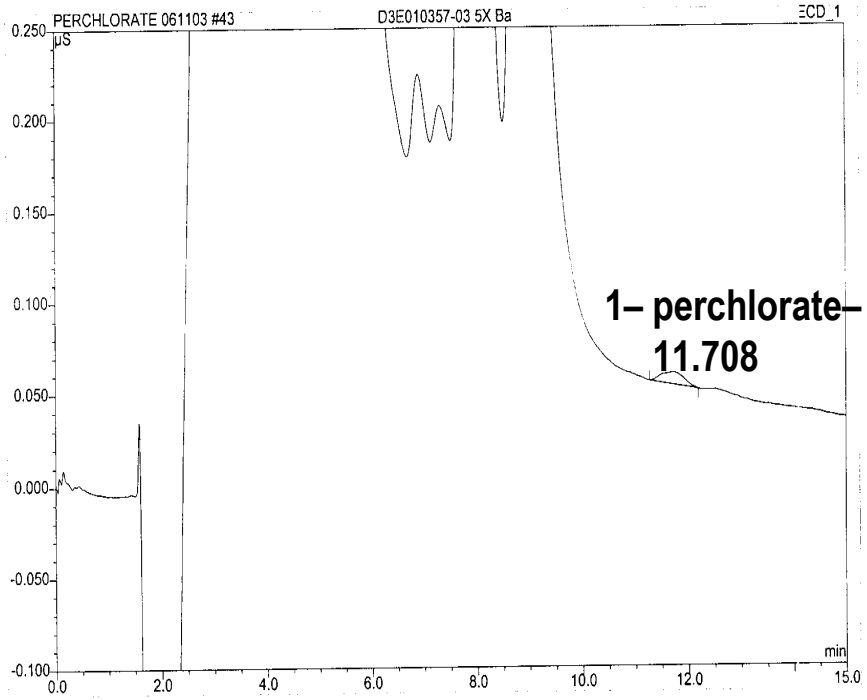


No.	Ret.Time min	Peak Name	Height µS	Area µS*min	Rel.Area %	Amount ug/L	Type
1	12.71	n.a.	0.062	0.026	15.87	n.a.	BMB
2	13.80	perchlorate	0.191	0.106	65.22	193.666	BM *
3	14.78	n.a.	0.067	0.031	18.91	n.a.	M *
Total:			0.321	0.162	100.00	193.666	

Cooling Tower Water by 314.0 with Barium Cartridge* Cleanup

- Cleanup procedure improved analysis considerably
- 6.5 µg/L reported result,
- But complex peak, is this reliable?

* **Barium form cation resin to reduce sulfate**

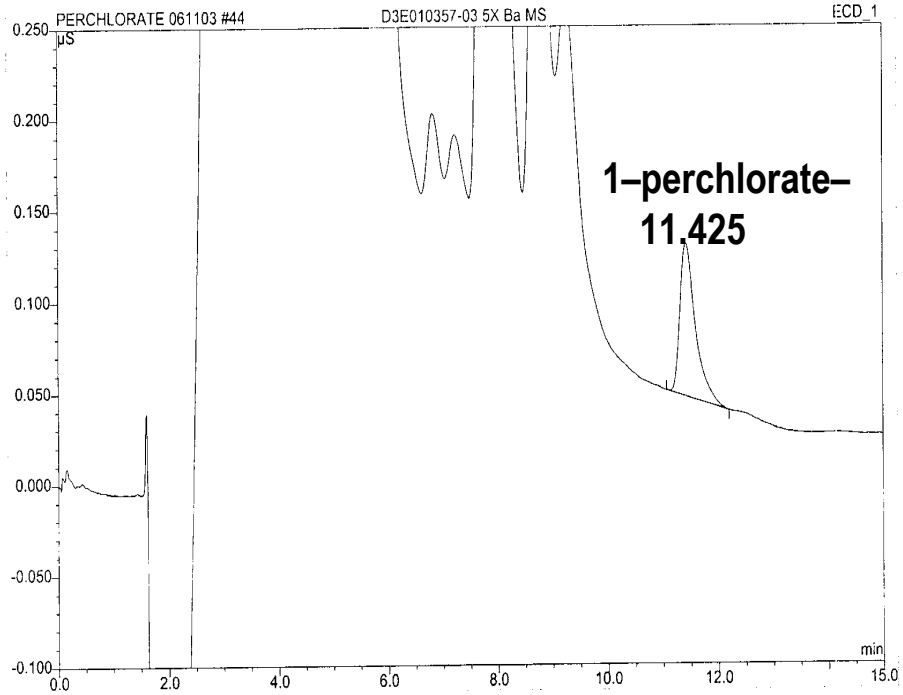


No.	Ret.Time min	Peak Name	Height µS	Area µS*min	Rel.Area %	Amount ug/L	Type
1	11.71	perchlorate	0.007	0.003	100.00	6.530	BMB
Total:			0.007	0.003	100.00	6.530	

Cooling Tower Water by 314.0 with Barium* Cartridge Cleanup

- 50 µg/L (ppb) perchlorate addition
- 98% recovery
- Good recovery, but is this reliable?

* **Barium-form cation resin to reduce sulfate**



No.	Ret.Time min	Peak Name	Height µS	Area µS*min	Rel.Area %	Amount ug/L	Type
1	11.43	perchlorate	0.084	0.027	100.00	55.572	BMB
Total:			0.084	0.027	100.00	55.572	

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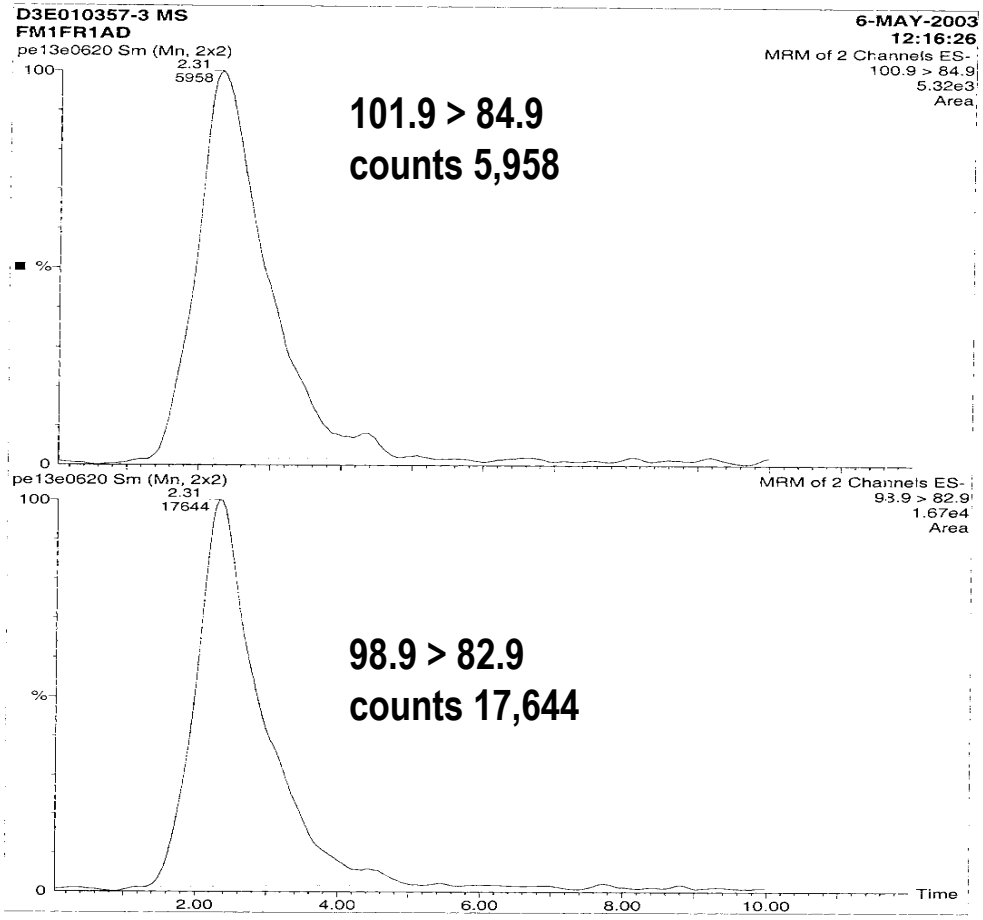
Cooling Tower Water by LC/MS-MS

- 1 µg/L (ppb) perchlorate addition, 80% recovery
- Confirms result 198x lower than original IC result
- Important for this facility, as proposed action level in this state is 1.0 µg/L



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Applications for IC/MS-MS

- IC/MS-MS Used by STL for USGS Research
  - Analysis of ancient brines for naturally occurring perchlorate in continental US
  - Analysis of leaves from deep-rooted desert plants to use as indicator of subterranean perchlorate
- Routine Applications
  - Definitive perchlorate testing
  - Confirmation of 314.0 results

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ERA Reference Material
Single Blind PT Freeze-Dried Spinach — Set 1

- Lab 1 829 µg/kg (IC/MS-MS)
- Lab 2 986 µg/kg (LC/MS-MS)
- Lab 3 780 µg/kg (314.0)
- Lab 4 84 µg/kg (LC/MS-MS)
- Lab 5 78 µg/kg (LC/MS-MS)
- Lab 6 79 µg/kg (LC/MS-MS)

Note: Low results probably due to ion suppression and no internal standard

SEVERN TRENT STL

ERA Reference Material
Single Blind PT Freeze-Dried Spinach—Set 2

- Study mean: 870 µg/kg
- Study SD: 131 µg/kg
- Number of data points: 7

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Summary

- There is no substitute for separating Perchlorate from common anions to prevent negative bias due to ion suppression
- Oxygen-18 Perchlorate internal standard improves quantification but can only go so far in correcting for ion suppression
- IC separation guards against ion suppression
- IC separation combined with MS-MS detection and incorporating Oxygen-18 Perchlorate internal standard delivers a trace Perchlorate method with excellent sensitivity* and outstanding selectivity