

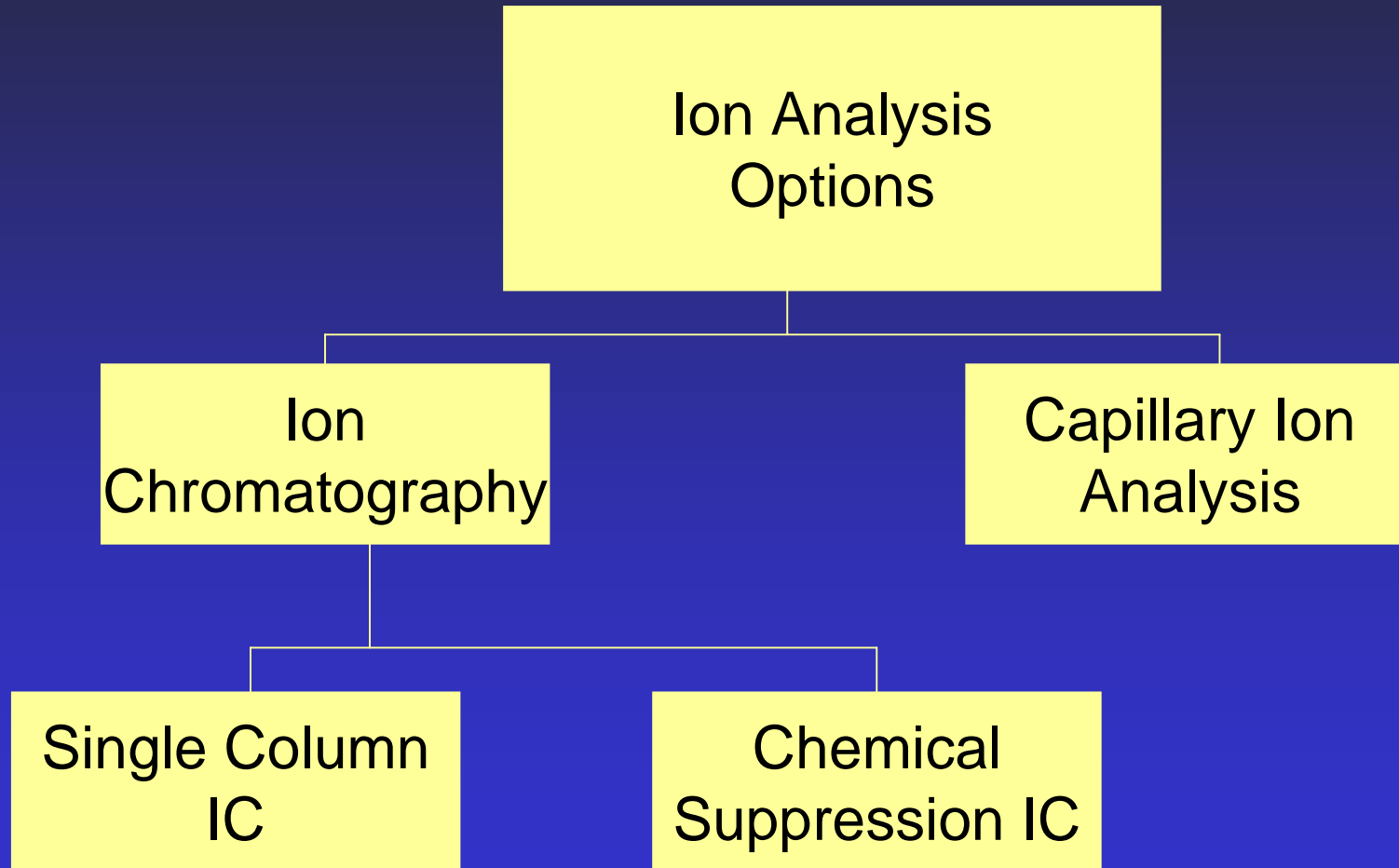
Ion Chromatography Analysis Methods and Issues

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Ion Analysis

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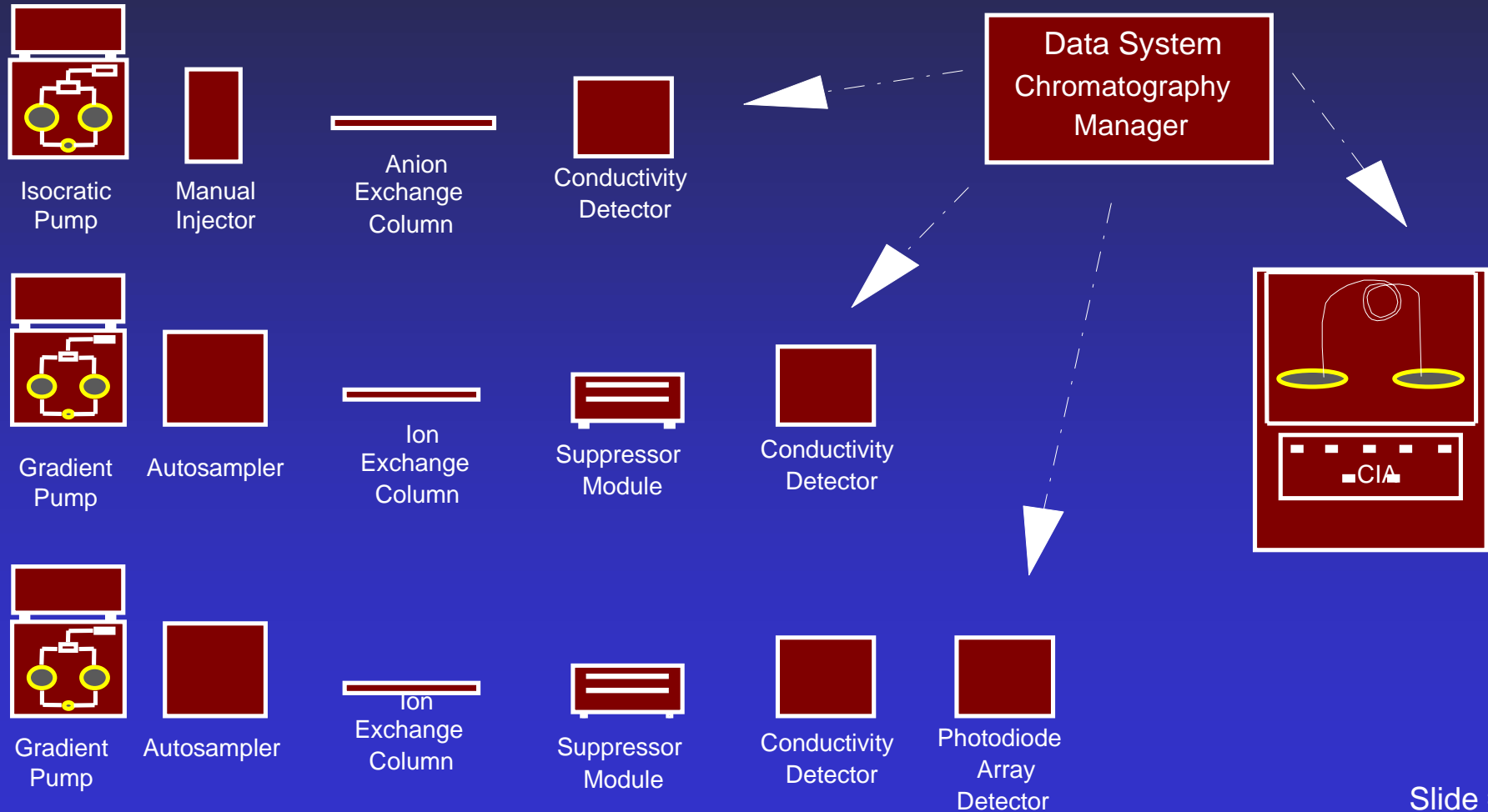
Feb/Mar 2000

You Have a Choice for Ion Analysis



Ion Chromatography

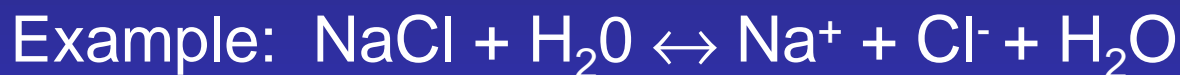
Hardware Configuration Options



What Are Ions?

Any chemical species that carries a electrical charge

- Soluble in water
- Exhibit conductivity in solution



$\text{Na}^+ = \text{Cation}$ $\text{Cl}^- = \text{Anion}$
or

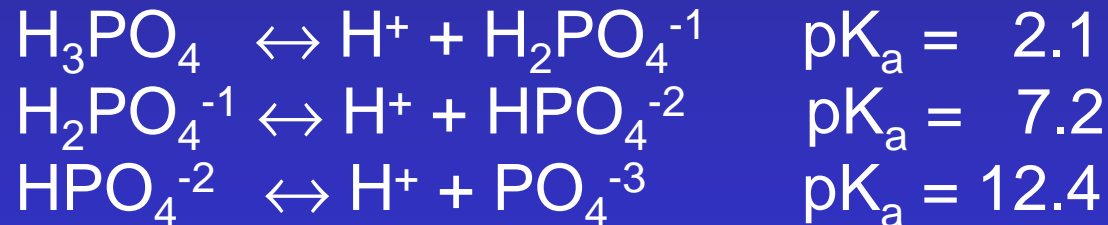


Importance of pH and pK_a

Ions can exist in solution in a number of different forms
Depending on pH of the solution and pK_a of the ion

$$\text{pH} = -\log [\text{H}^+]$$

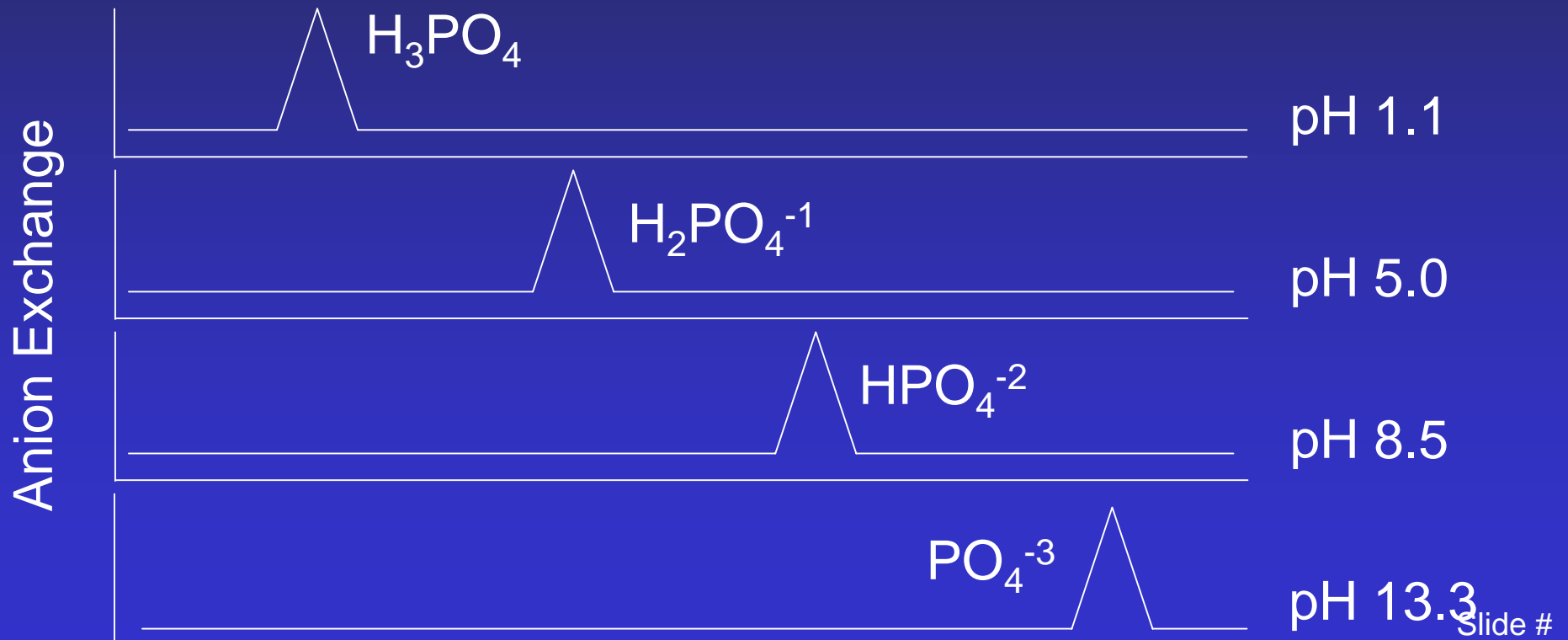
pK_a = the pH where the species is 50% ionized



Importance of pH and pK_a

Why is this important to IC?

All ions in solution will convert to the form favored by the pH of the eluent.



Importance of K_{sp} Solubility Product

Before an ion can be analyzed by IC, it must be in solution.

Depending on the sample matrix, other ions influence solubility.

$$K_{sp} = \frac{[A^-][C^+]}{[AC]}$$

$$pK_{sp} = -\log K_{sp}$$

Example: Analysis of Cl in a sample matrix containing Ag, or
Analysis of SO_4 in a sample with high Ca.

K_{sp} for AgCl is 9.75 implying that Cl is not in solution
 K_{sp} for $CaSO_4$ is 5.04 implying that only a portion of
the SO_4 is in solution.

Important Questions to Ask About the Sample

- 1 What are the analyte ions of interest?
- 2 What is the expected concentration?
- 3 What else is in the sample matrix?

Detection of Ions

Conductivity

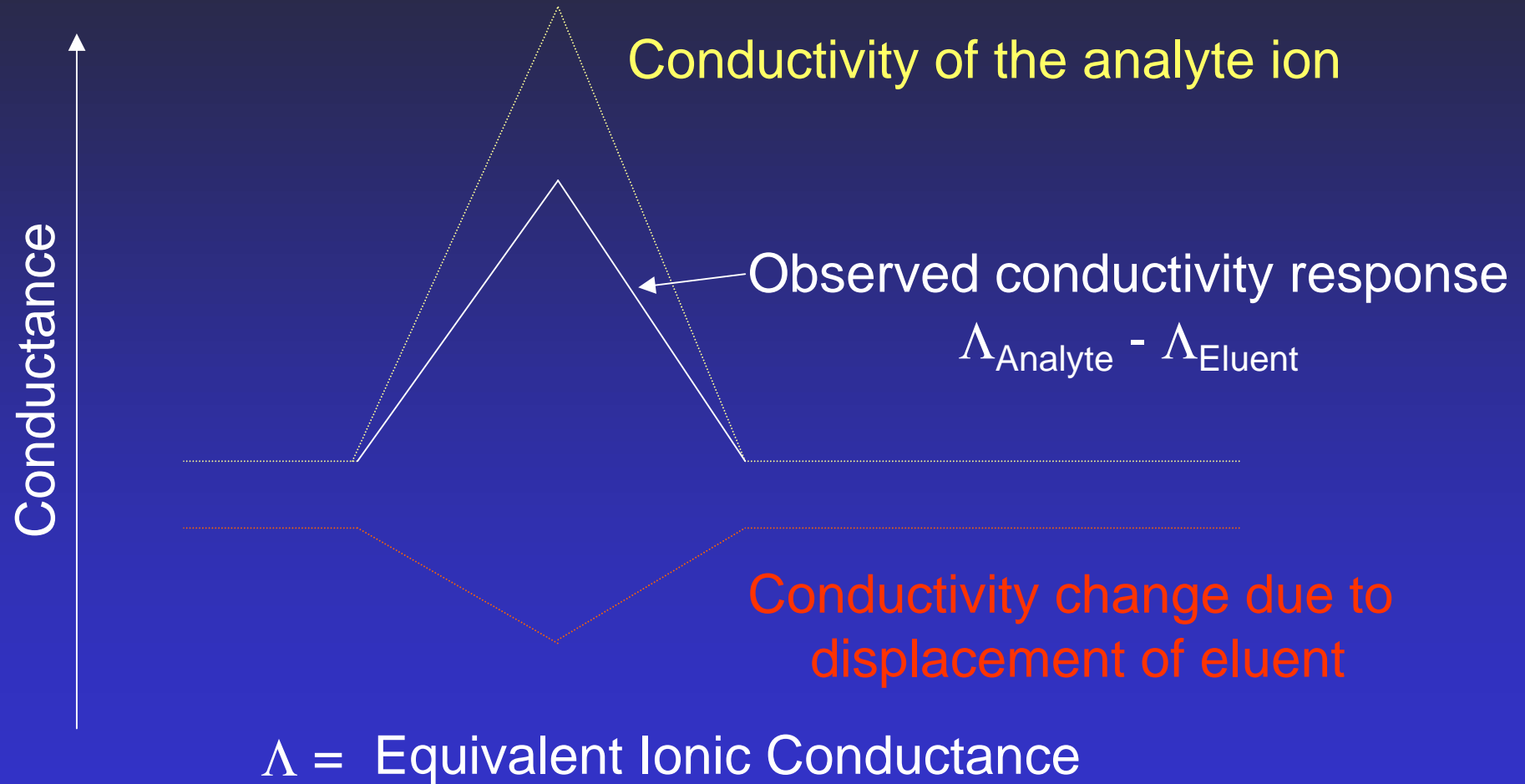
- Direct: Net increase in conductivity;
low background eluent conductivity
- Indirect: Net decrease in conductivity
high background conductivity

UV/VIS

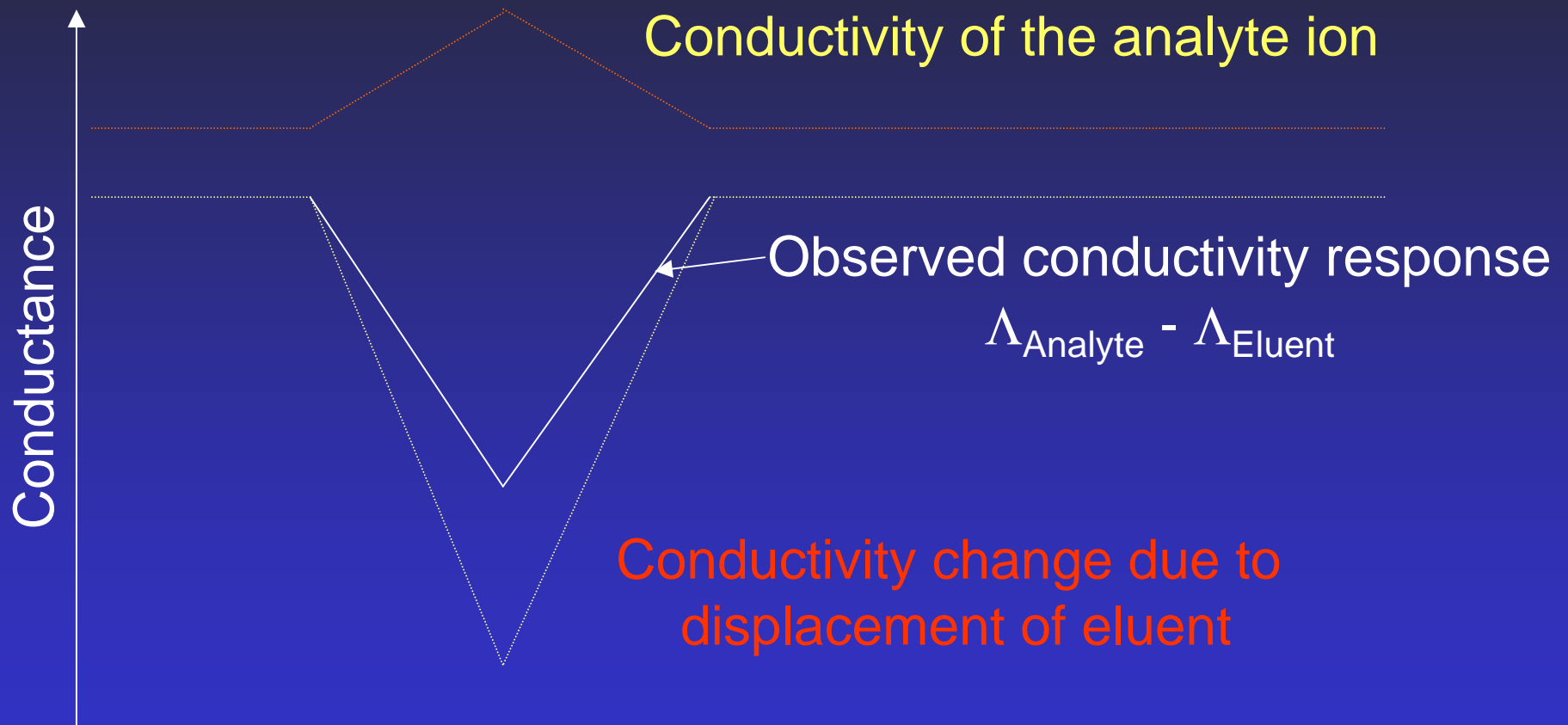
- Direct: Generally between 200 and 220 nm
- Post Column Derivatization

Electrochemical

Direct Conductivity Detection



Indirect Conductivity Detection



Switch detector polarity for positive mV peaks

What is Chemical Suppression Conductivity Detection

A Suppressor is a device placed between the column and the detector, and acts to reduce the background conductivity of the eluent and enhance the conductivity of the analytes.

For anion analysis, the suppressor is a high capacity cation exchange membrane or resin in the acid form.

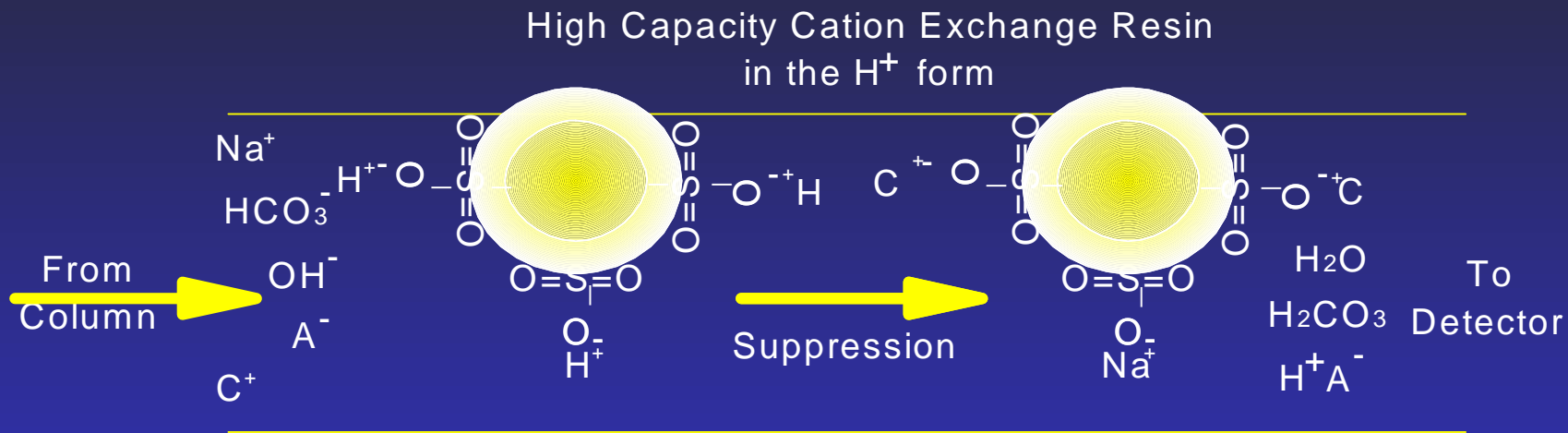
It removes cations from the eluent and replaces them with H^+ .

The H^+ neutralizes the highly conductive HCO_3^-/CO_3^{2-} , or OH^- , to non-conductive H_2CO_3 and H_2O . Puts the anions into their highly conductive acid form for conductivity detection.

Lowers background conductivity and enhances anion conductivity.

Mechanism of Chemical Suppression

Anion Analysis Using Alltech ERIS 1000HP



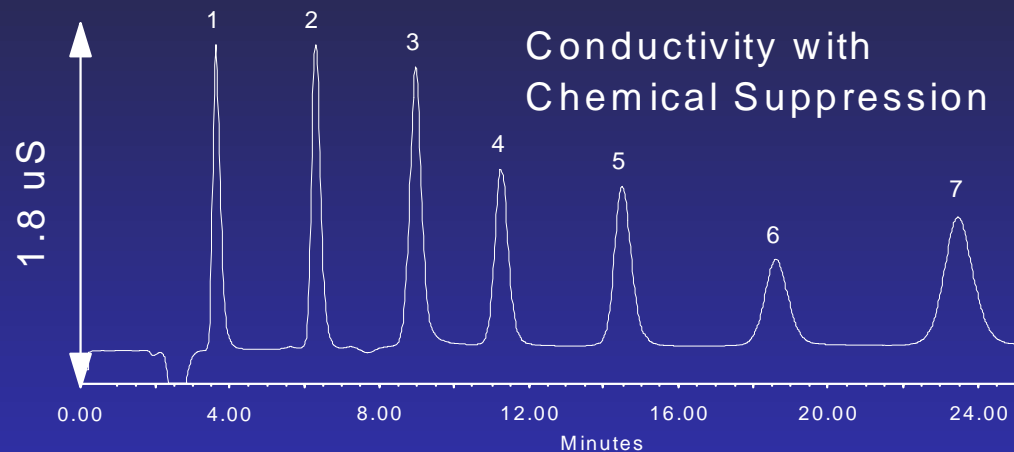
- Eluent ions, Na , OH , and HCO_3/CO_3 exhibit high conductivity
- Resin in H^+ form acts to remove Na and Sample Cations, C^+ , from the eluent, and replaces them with H^+
- H^+ interacts with OH and HCO_3/CO_3 to yield H_2O and H_2CO_3 that have low conductivity and the conductivity of the eluent is significantly reduced
- Simultaneously, the sample anions, \bar{A} , are placed into their highly conductive acid form to enhance their conductivity response
- Overall S/N and sensitivity is enhanced

Direct UV/VIS Detection

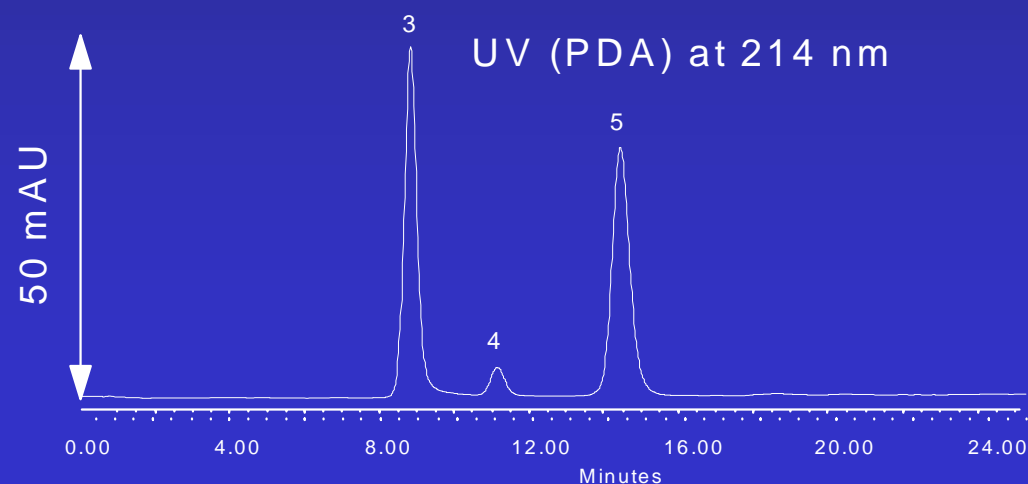
Many anions and all organic acids are UV active
in the general range of 200 to 220 nm

Examples: Nitrite, Nitrate, Sulfite, ThioSulfate, ThioCyanate
Chlorite, Bromide, Bromate, Iodide, Iodate
Arsenite, Arsenate, Selenite, Selenate,
All Organic Acids

Direct UV Detection in Series with Conductivity Detection

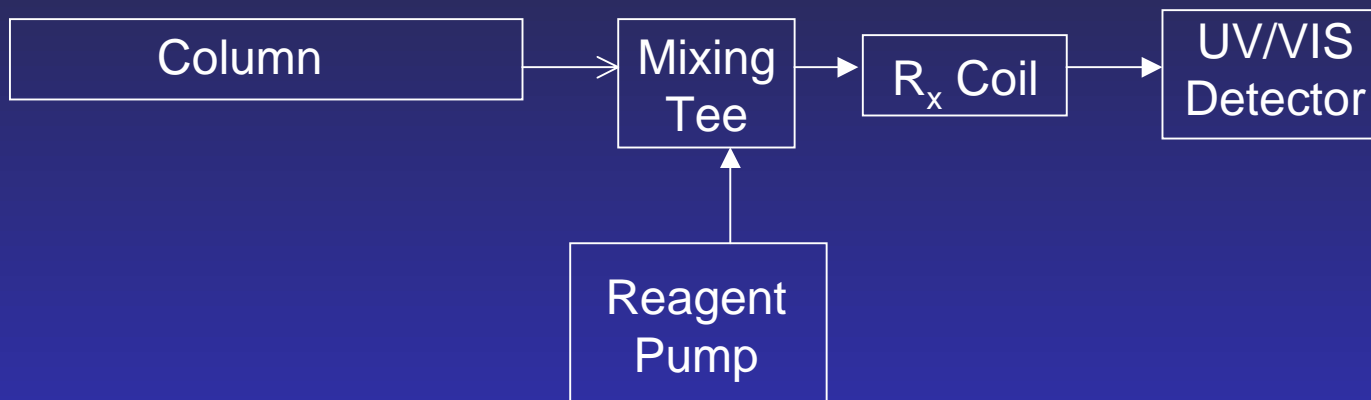


Column: Waters IC-Pak A/ HC
Eluent: 1.2 mM Na_2CO_3 /
1.2 mM NaHCO_3
Flow rate: 2.0 mL/min
Injection Vol: 50 μL



1. Fluoride	1 ppm
2. Chloride	2 ppm
3. Nitrite	4 ppm
4. Bromide	4 ppm
5. Nitrate	4 ppm
6. Phosphate	6 ppm
7. Sulfate	4 ppm

UV/VIS Detection with Post Column Derivatization



Additional hardware is necessary to the basic IC to do this type of application

Used for bromate with EPA 300.2, and for transition metal analysis

Ion Chromatography

Applications and Methods

Common Anion Analysis: EPA 300, Std Mtds 4110, & ASTM D4327;
and Std Mtds 4140 & ASTM Dxxx using Capillary Ion Analysis
Fluoride, Chloride, Bromide, Nitrite, Nitrate, o-Phosphate, and Sulfate

Oxyhalide Analysis: EPA 300.1 & 300.2, ASTM pending
Chlorite, Bromide, and Bromate

Perchlorate Analysis: EPA 314 (?), no ASTM or Std Mtds

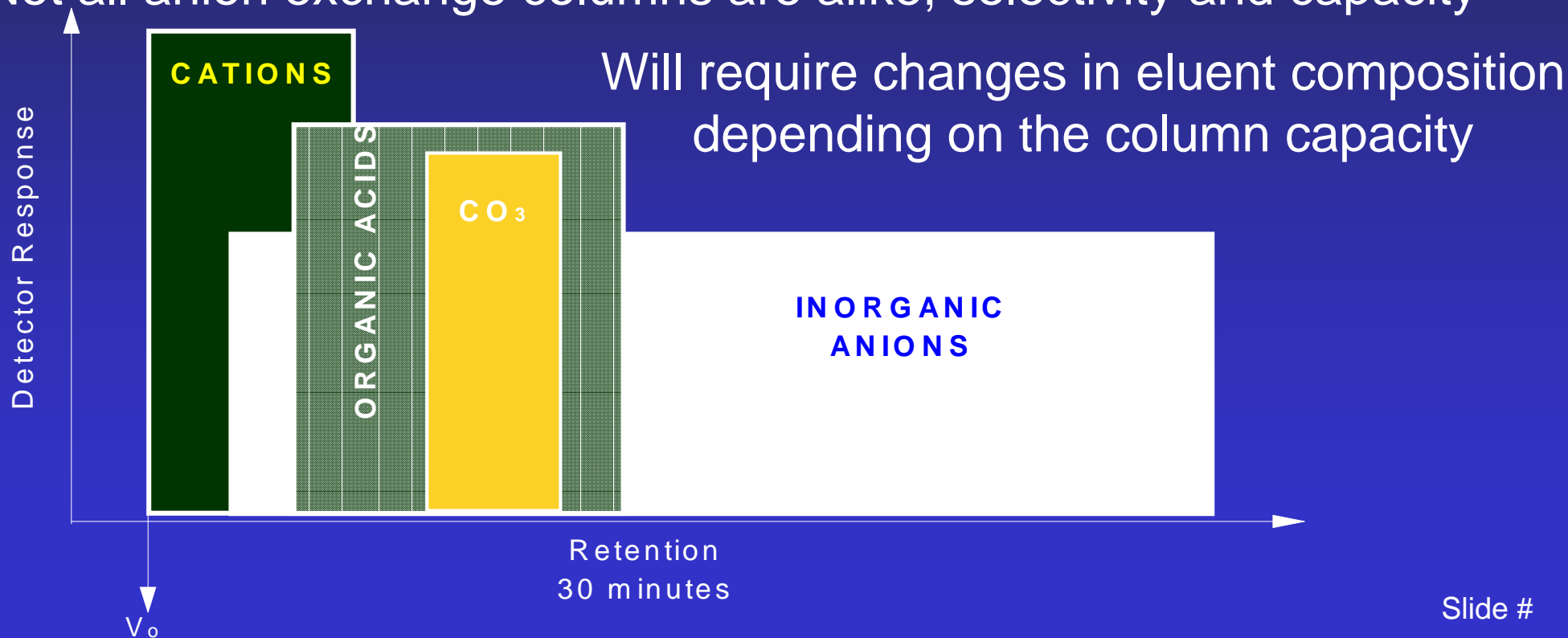
Chromate (Cr^{+6}): EPA 218.6, or Std Mtds 3500, no ASTM

Alkali/Alkaline Earth Cations: No EPA, Std Mtds, or ASTM

IC Anion Exchange Selectivity

All EPA methods use an anion exchange column; Dionex or Equivalent Waters columns and Alltech ERIS 1000HP Auto Suppressor are considered equivalent

Not all anion exchange columns are alike; selectivity and capacity



Ion Chromatography for Anion Analysis

The Validation Issues

Hardware: The Ion Chromatograph

Software: Data Processing

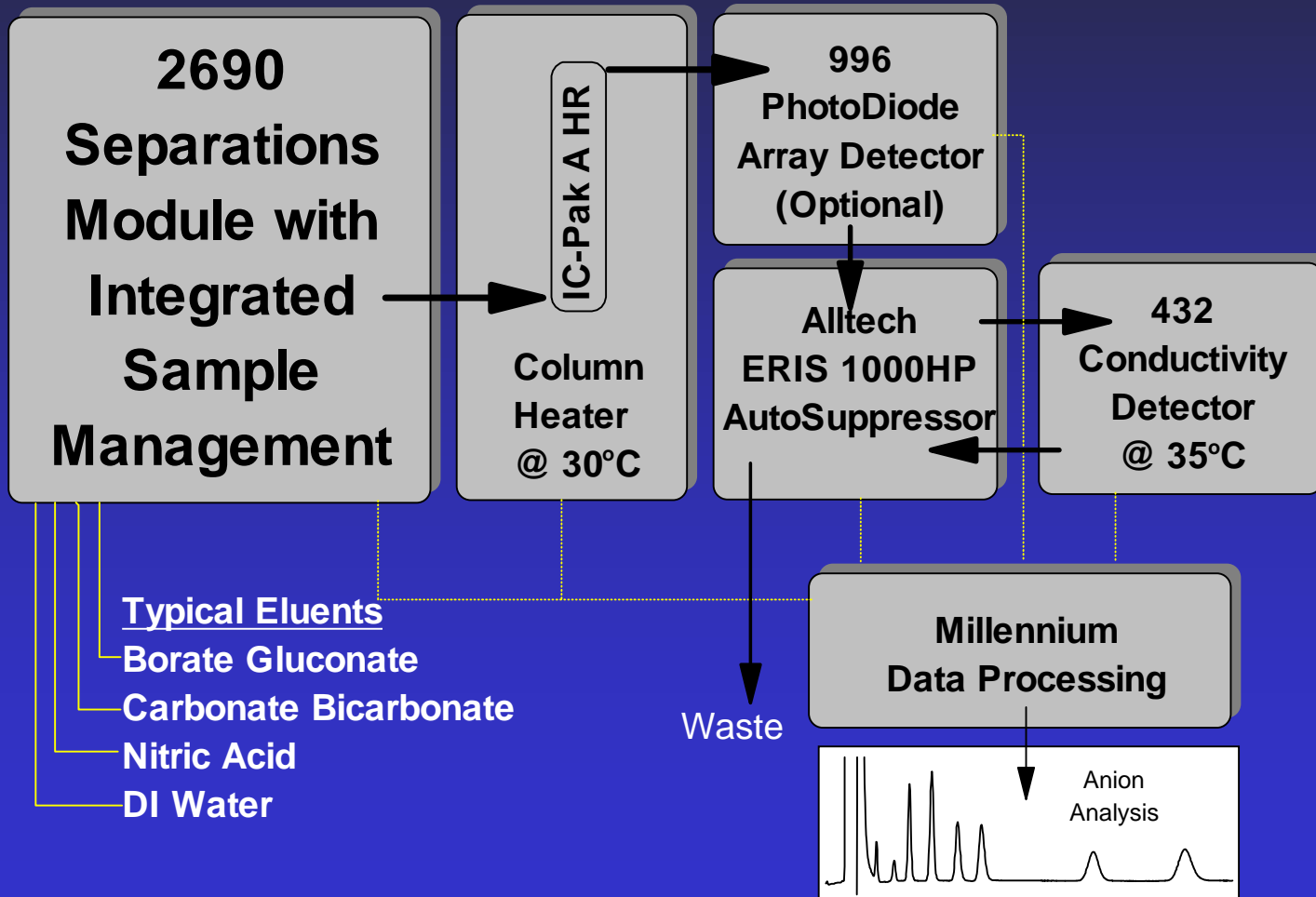
Method: The Chemistry
Sensitivity and Detection Limits
Linearity and Accuracy

System Suitability: QA / QC

Method Validation was Performed Using Waters Alliance™ System for IC

- ★ Waters® 2690 Separations Module
 - 316 Stainless Steel, low dispersion
 - Enhanced Flow Precision, 0.075% RSD
 - Continuous Eluent Vacuum Degassing
 - Integrated Sample Management, 0.5% at 100 µL
- ★ Waters IC-Pak™ Anion HR (300.1 Equivalent Column)
 - 6 µm; 4.6 mm x 75 mm; PolyMethacrylate Based
- ★ Waters 432 Conductivity Detector
 - 5 Electrode Design Providing High Sensitivity at High Background Conductivity
 - Direct Temperature Control
- ★ Alltech ERIS™ 1000HP AutoSuppressor (300.1 Equivalent Suppressor Device)
 - Two High Capacity Cation Exchange Solid Phase Cells in Parallel
 - Electrochemical Self-Regeneration with Eluent
- ★ Waters Millennium® Data Processing

Alliance System for Ion Chromatography



Anion Analysis

Method Validation Design

Individual Youden Pair Standards, in ppm

Analyte Anion		1	2	3	4	5	6	7	8
	Cl	0.7	2.0	3.0	15.0	40.0	20.0	50.0	0.5
	Br	2.0	3.0	15.0	40.0	20.0	50.0	0.7	0.5
	NO ₂	3.0	40.0	20.0	15.0	50.0	0.5	2.0	0.7
	SO ₄	40.0	50.0	0.5	0.7	2.0	3.0	15.0	20.0
	NO ₃	15.0	20.0	40.0	50.0	0.5	0.7	2.0	3.0
	F	2.0	0.7	0.5	3.0	10.0	7.0	20.0	25.0
	PO ₄	50.0	40.0	20.0	0.5	3.0	2.0	0.7	15.0

The collaborative design is intended to demonstrate performance between 0.1 and 50 ppm anion, except for Fluoride between 0.1 and 25 ppm.

Standard 9 is 100 ppb of each anion for detection limit calculations.

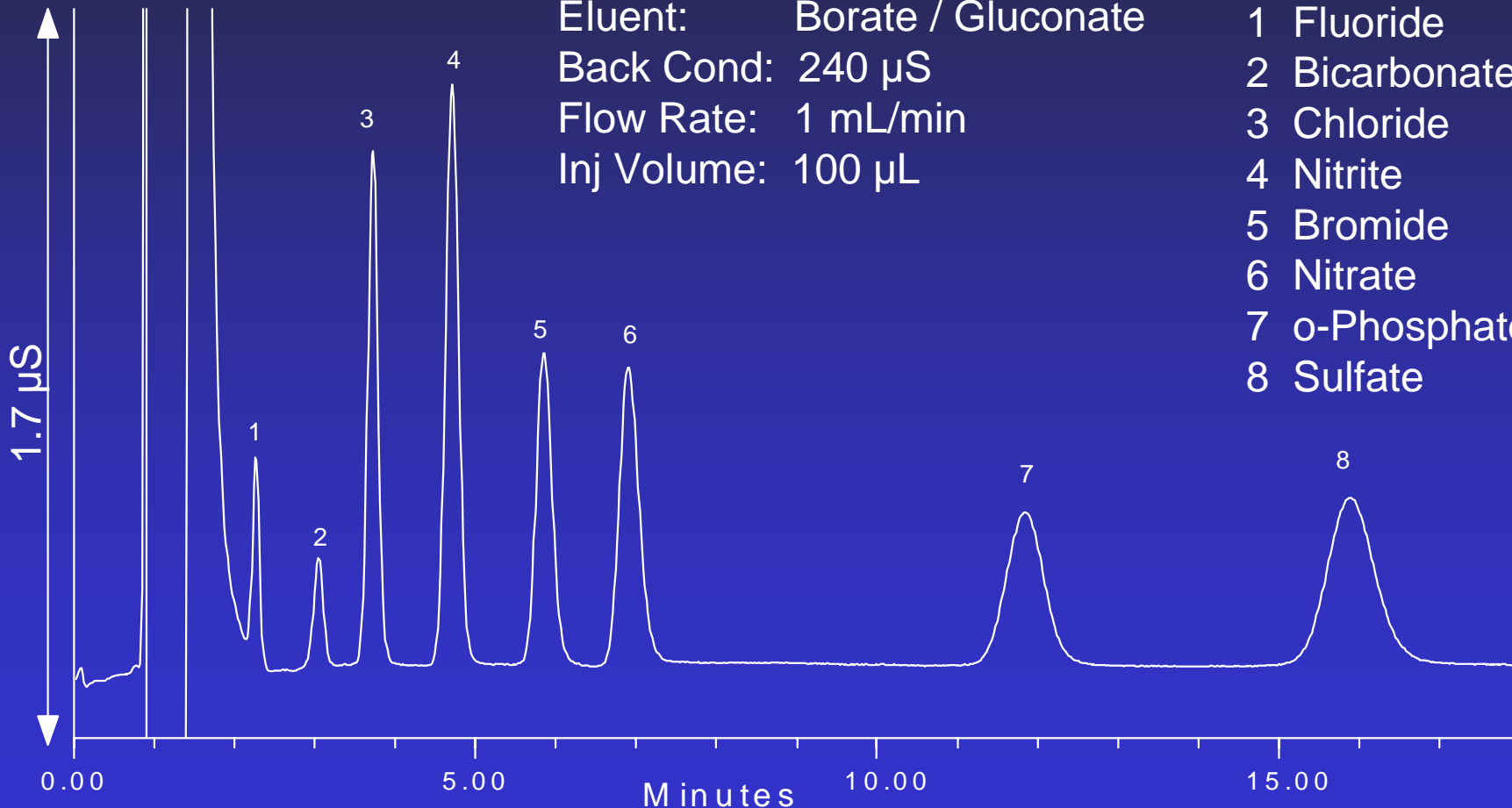
All Standards prepared from Certified 1000 ppm Stock Standards

Single Column Ion Chromatography

Direct Conductivity Detection

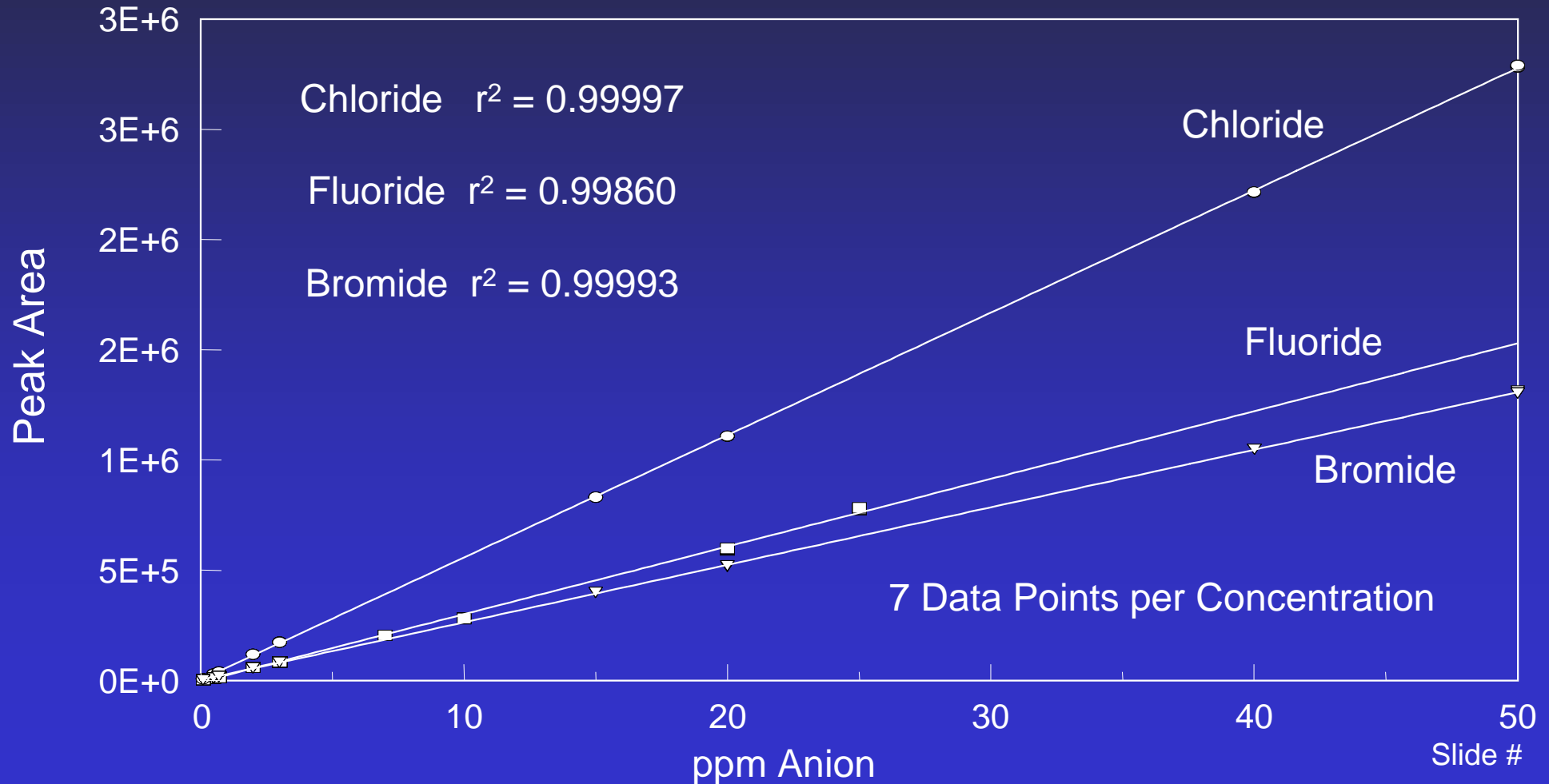
Column: Waters IC Pak A/HR
Eluent: Borate / Gluconate
Back Cond: 240 μ S
Flow Rate: 1 mL/min
Inj Volume: 100 μ L

1	Fluoride	= 1 ppm
2	Bicarbonate	
3	Chloride	= 2
4	Nitrite	= 4
5	Bromide	= 4
6	Nitrate	= 4
7	o-Phosphate	= 6
8	Sulfate	= 4



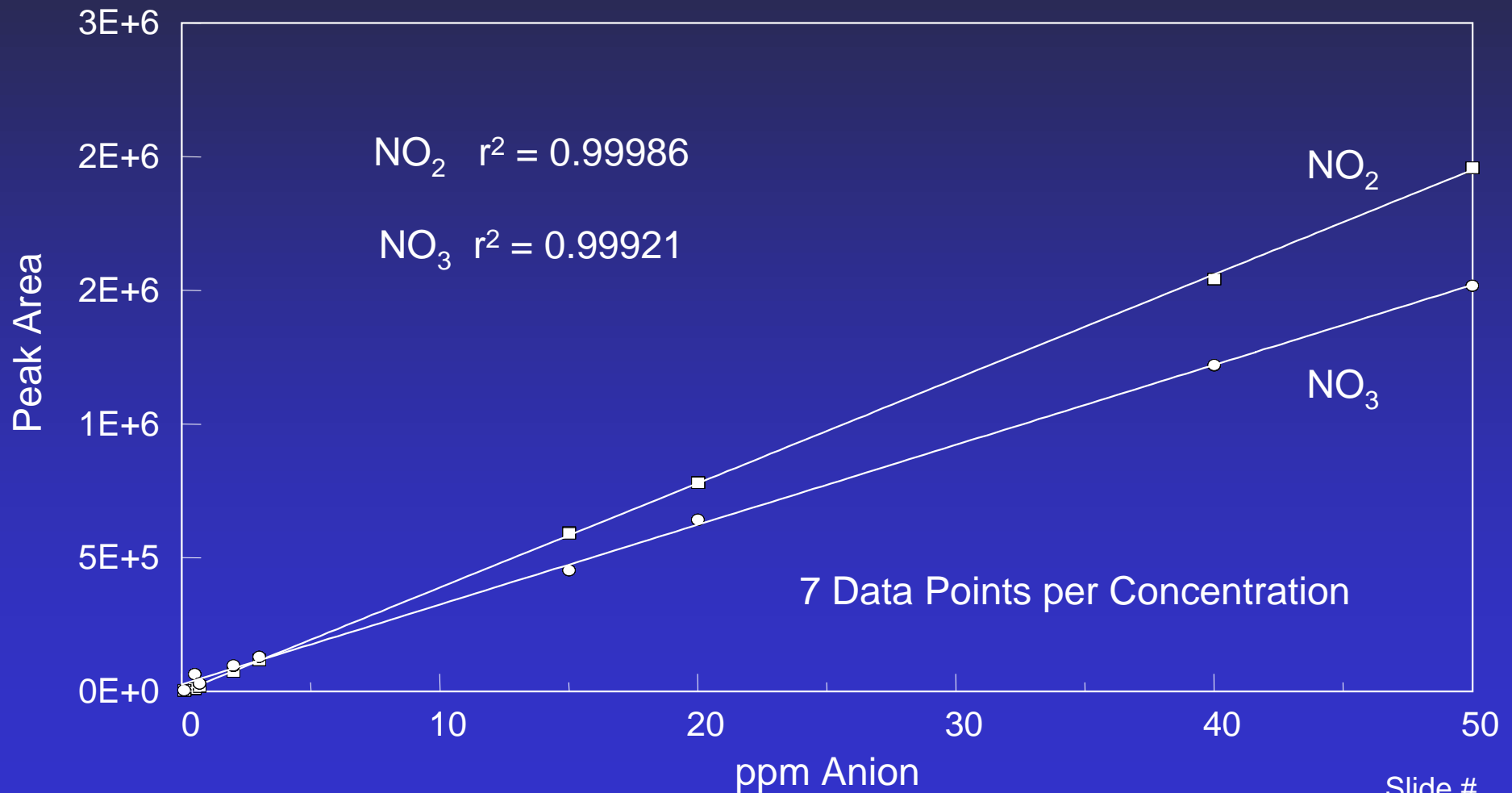
Single Column Ion Chromatography

Peak Area Response Linearity



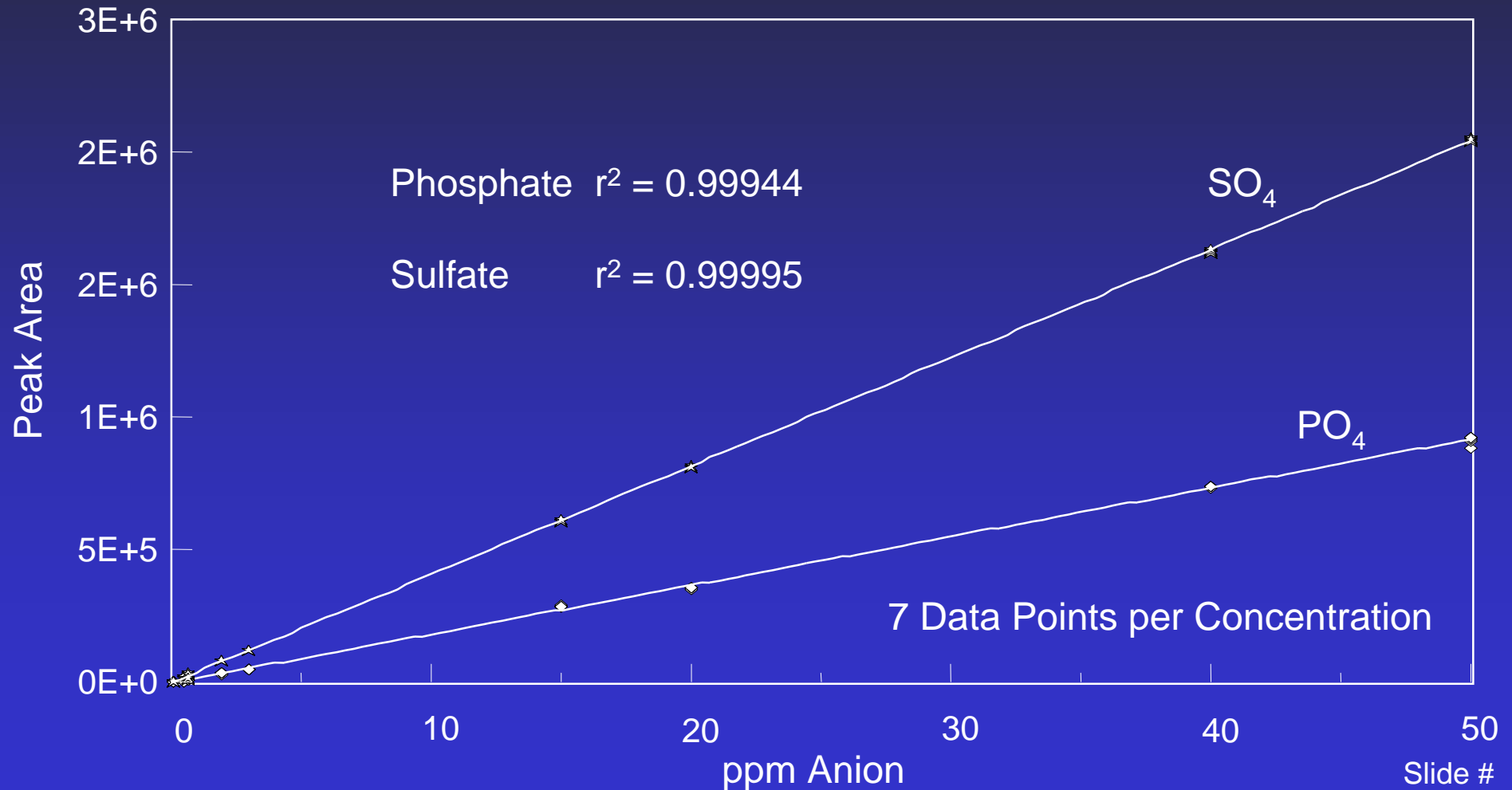
Single Column Ion Chromatography

Peak Area Response Linearity



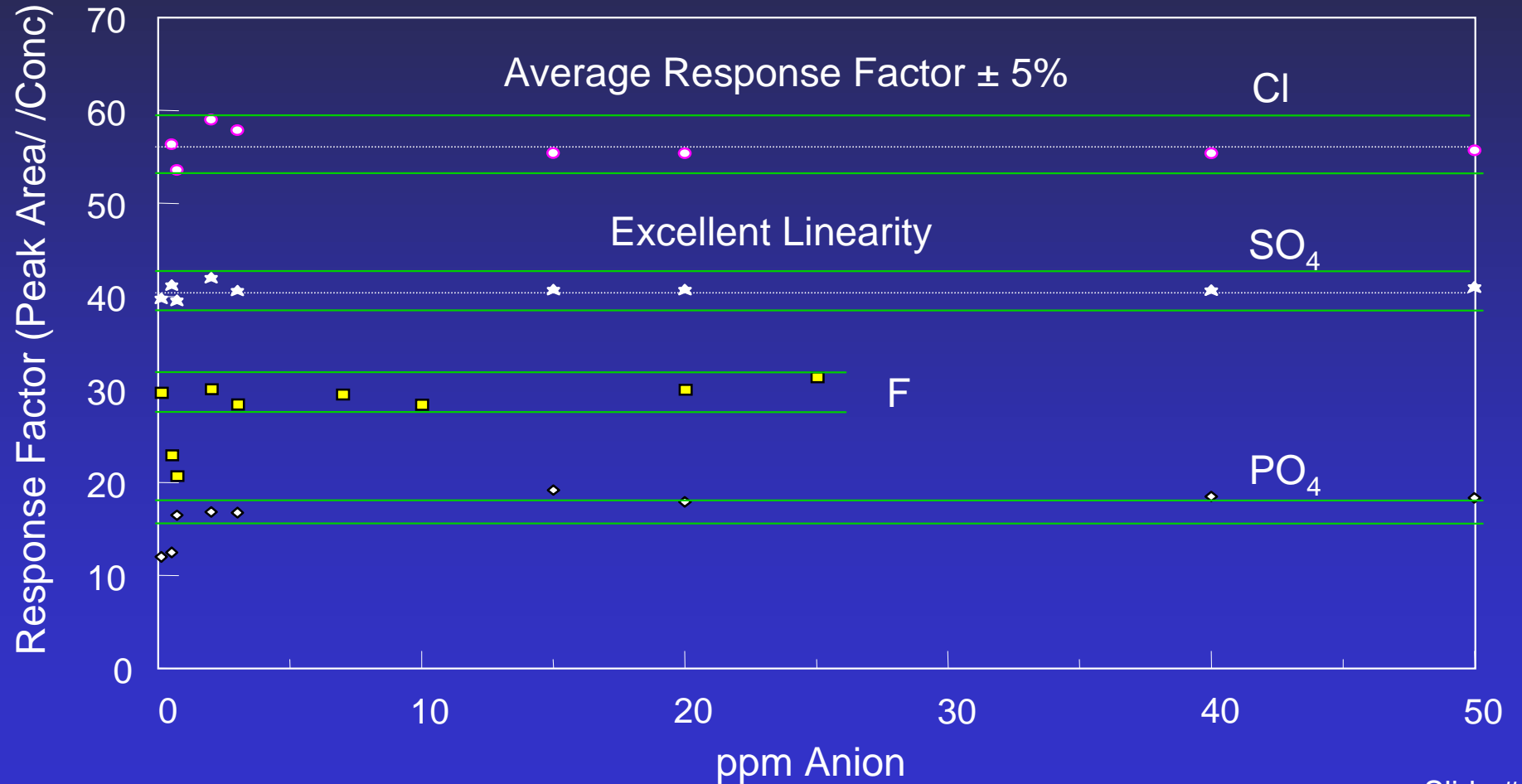
Single Column Ion Chromatography

Peak Area Response Linearity

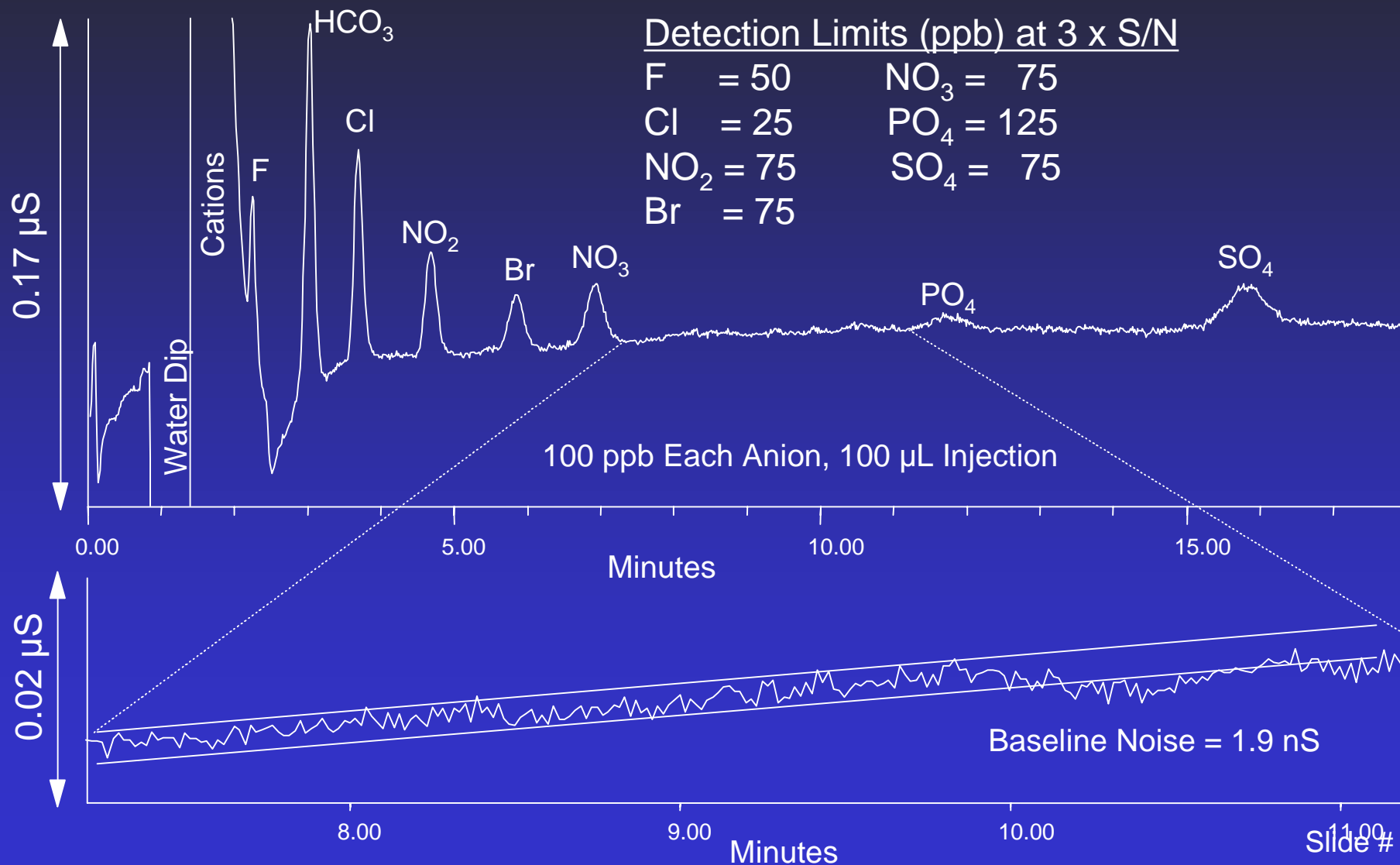


Single Column Ion Chromatography

ASTM Linearity



Single Column Anion Detection Limits Using the Alliance IC System



Single Column IC Peak Area Precision

ppm Concentration	Analyte	F	Cl	NO ₂	Br	NO ₃	PO ₄	SO ₄
	0.5	0.95	1.11	3.44	5.17	0.32	12.98	7.62
	0.7	0.67	1.64	0.78	1.73	0.75	9.29	3.90
	2.0	0.17	0.18	0.56	1.14	0.91	2.91	1.07
	3.0	0.43	0.17	0.20	0.67	0.19	3.49	0.64
	15	0.44	0.05	0.28	0.11	0.16	0.50	0.32
	20	0.45	0.04	0.05	0.30	0.06	0.52	0.25
	40		0.05	0.04	0.03	0.08	0.37	0.26
	50		0.13	0.02	0.26	0.03	1.56	0.16

Data as Peak Area %RSD for 7 Replicate Injections
of Each Standard

Single Column Ion Chromatography

Accuracy Using a Performance Evaluation Standard

	Analyte	F	Cl	NO ₂	NO ₃	PO ₄	SO ₄
Performance Evaluation Standard	True Value in ppm	2.69	43.00	1.77	15.37	6.29	37.20
Official Anion	Measured Mean	2.75	43.30	1.77	15.42	6.38	37.00
Methods Wet Chem & IC	Measured Std Dev	0.26	3.09	0.07	1.15	0.21	2.24
Alliance IC System	Ave IC n=3	2.63 ± 0.05	43.87 ± 0.09	1.93 ± 0.01	15.04 ± 0.06	6.47 ± 0.09	37.03 ± 0.12
IC-Pak A HR	IC/Mean	0.956	1.013	1.090	0.975	1.014	1.001
and B/G Eluent	IC/True Value	0.978	1.020	1.090	0.979	1.029	0.995

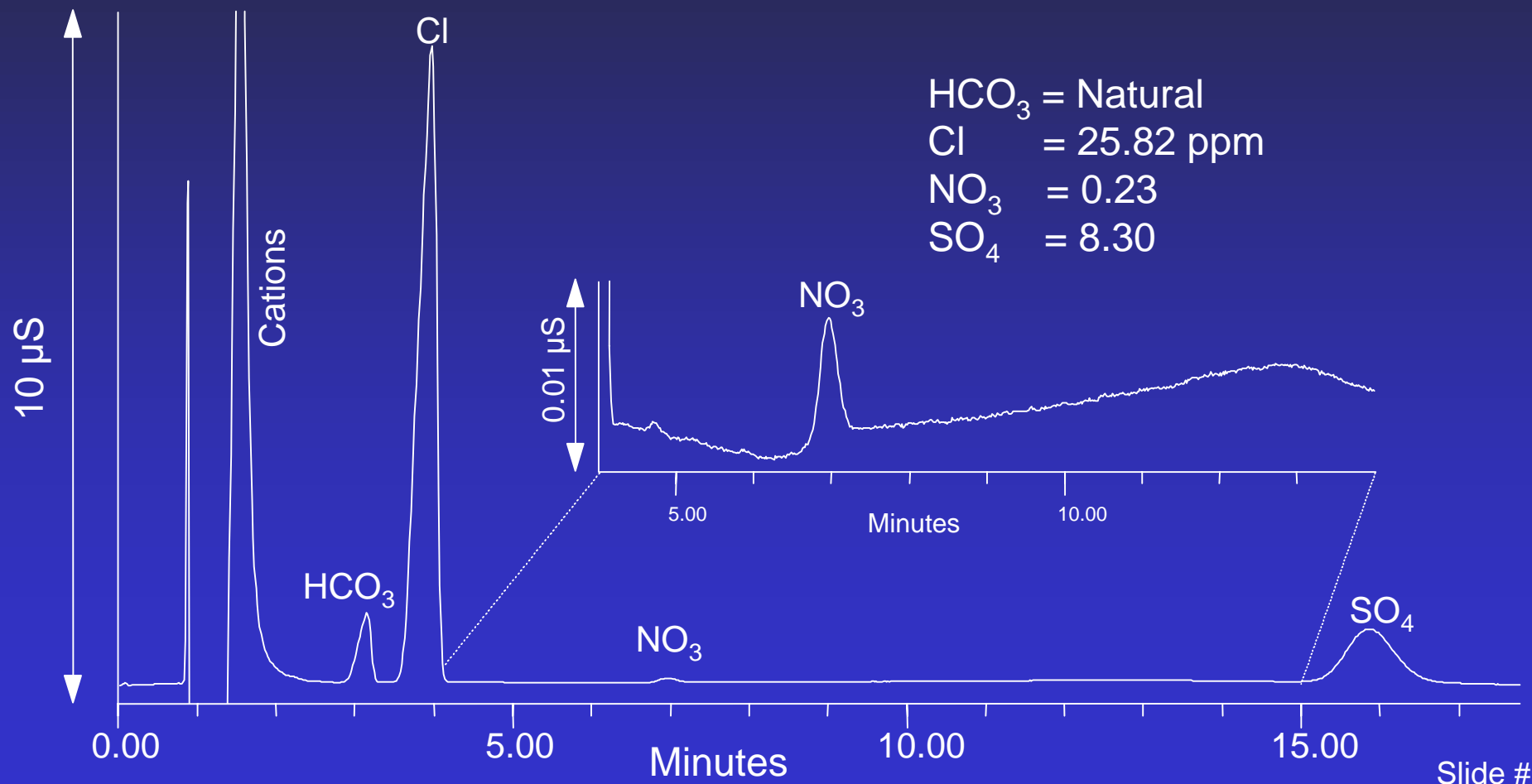
The performance evaluation standards were purchased from APG Laboratories and diluted 1:100 with Type 1 DI Water.

The measured results are the average from numerous laboratories using conventional, EPA approved wet chemistries and IC methods.

An IC/True Value of 1.000 indicates perfect agreement; Note PO₄ = 1.014-1.029.

Single Column Ion Chromatography

Typical Drinking Water



Single Column Ion Chromatography

Recovery of Performance Evaluation Standard from Drinking Water

Analyte	F	Cl	NO ₂	NO ₃	PO ₄	SO ₄
Milford Drinking Water n=3, as ppm	Not Detected	25.82 \pm 0.04	Not Detected	0.23 \pm 0.002	Not Detected	8.30 \pm 0.02
%RSD		0.16		0.92		0.27
Performance Evaluation Std	2.69	43.00	1.77	15.37	6.29	37.20
MDW + PES n=3; as ppm	2.46 \pm 0.04	69.64 \pm 0.08	1.82 \pm 0.004	15.52 \pm 0.02	5.35 \pm 0.05	46.46 \pm 0.17
%RSD	1.51%	0.11%	0.21%	0.10%	0.92%	0.37%
% Recovery	91.4%	102.5%	102.8%	99.5%	85.1%	102.8%

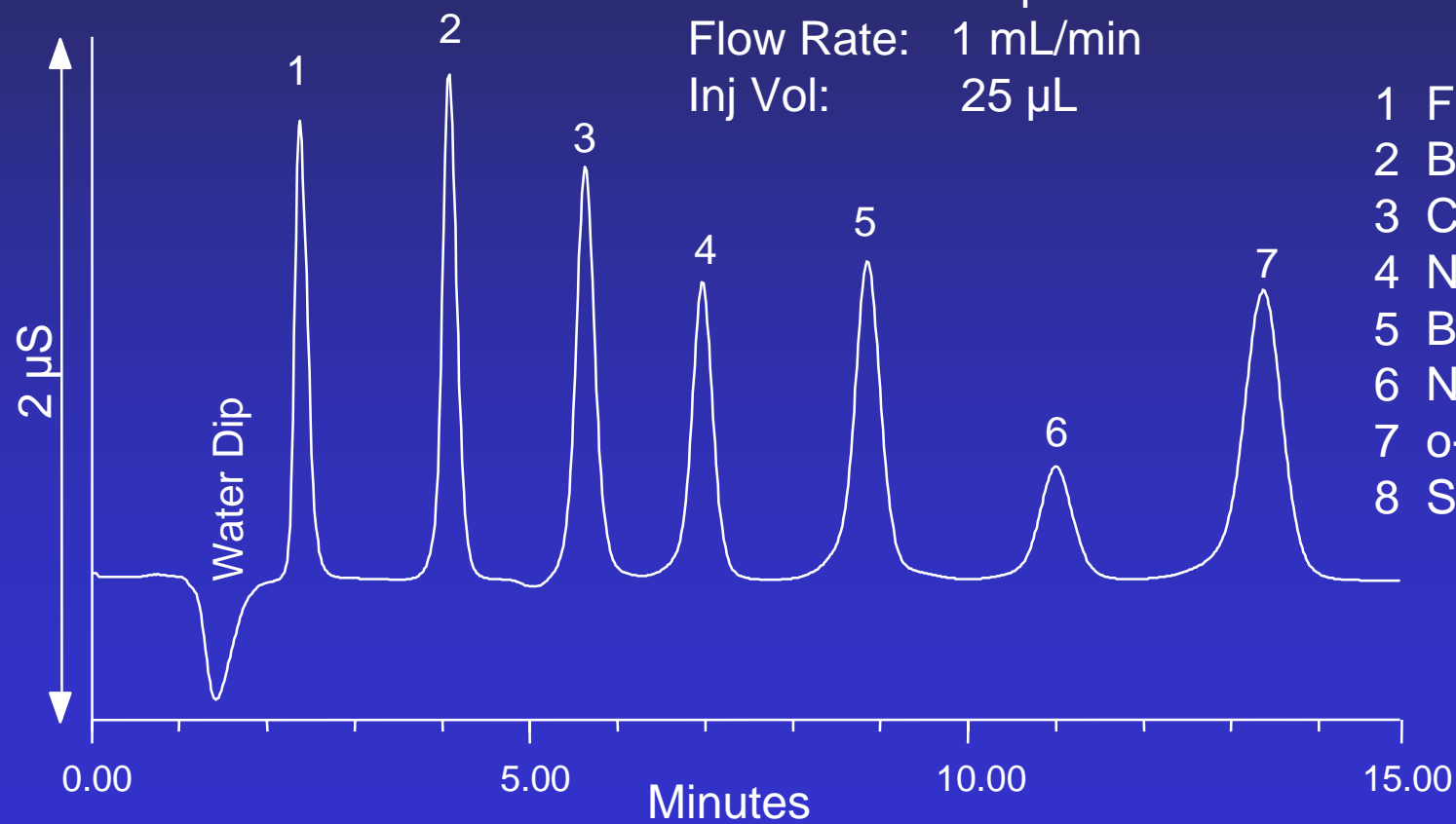
The Performance evaluation standard was diluted 1:100 with typical drinking water.

The low recovery for PO₄ is attributed to low ppb Mg, Ca, Fe, Mn, and Cu in the drinking water. Not from the stainless steel hardware.

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Column: Waters IC Pak A/HR
Eluent: 1.6 mM NaHCO₃ / 1.4 mM Na₂CO₃
Back Cond: 12 µS
Flow Rate: 1 mL/min
Inj Vol: 25 µL

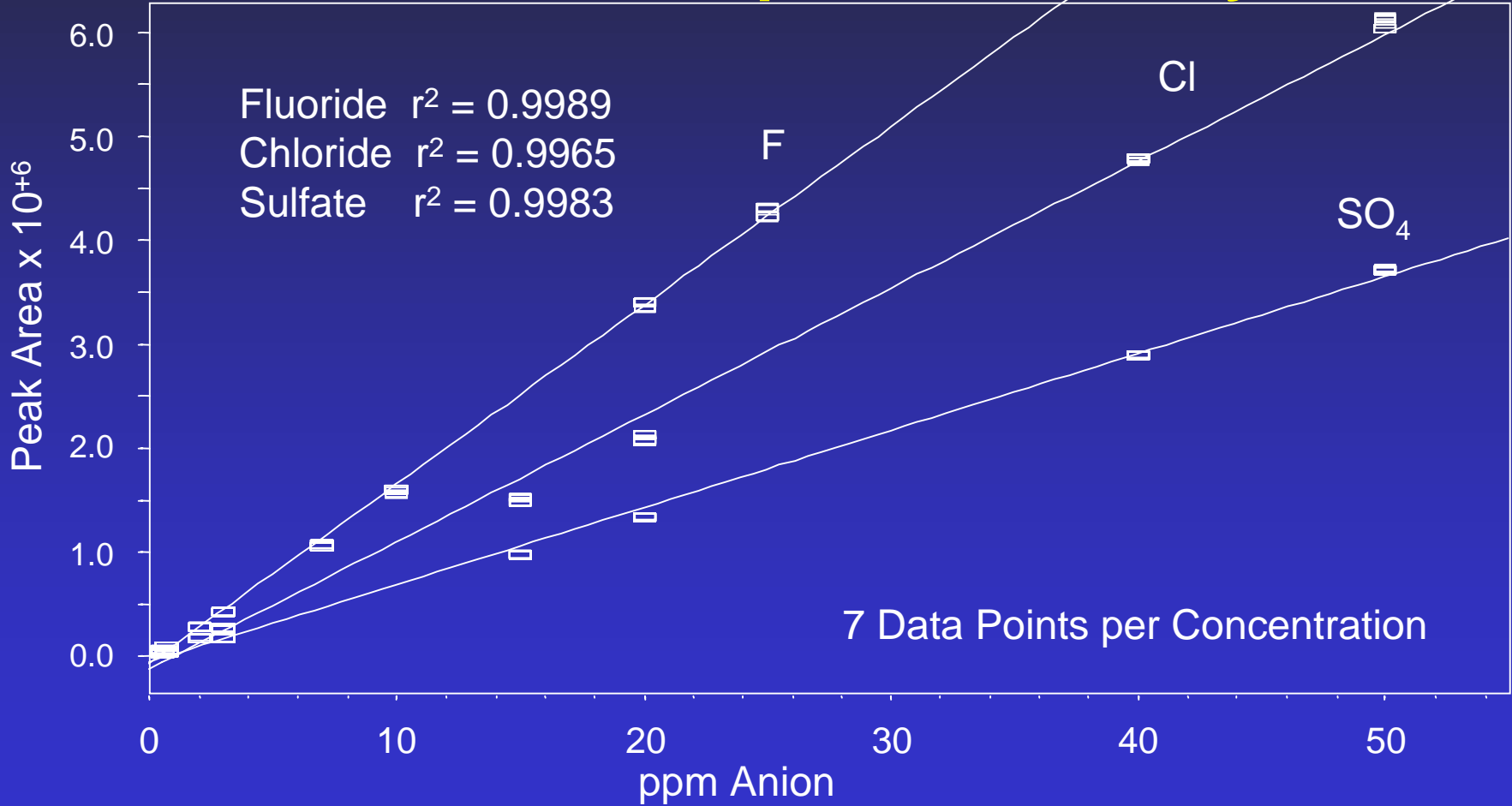


- 1 Fluoride = 1 ppm
- 2 Bicarbonate
- 3 Chloride = 2
- 4 Nitrite = 4
- 5 Bromide = 4
- 6 Nitrate = 4
- 7 o-Phosphate = 6
- 8 Sulfate = 4

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

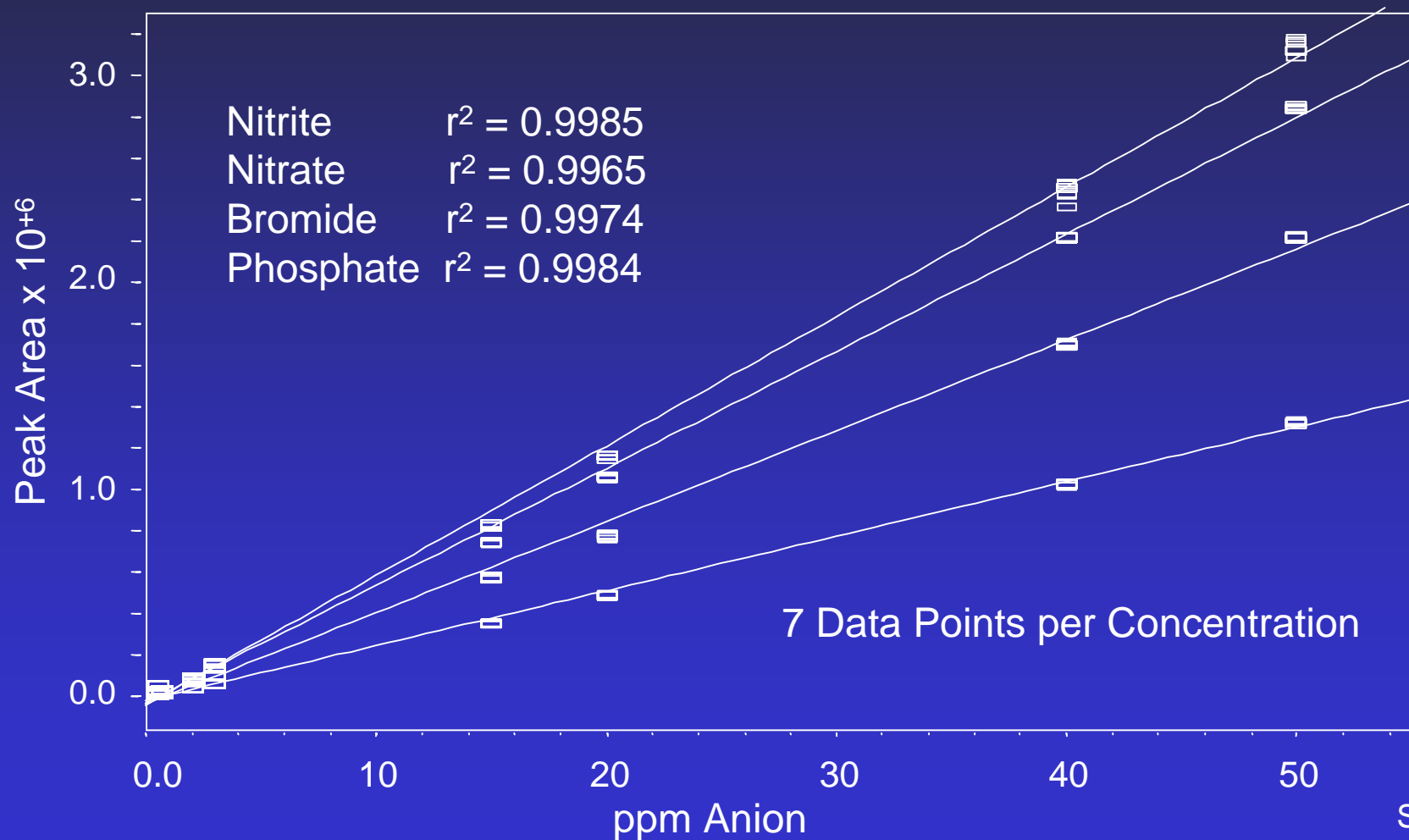
Peak Area Response Linearity



Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

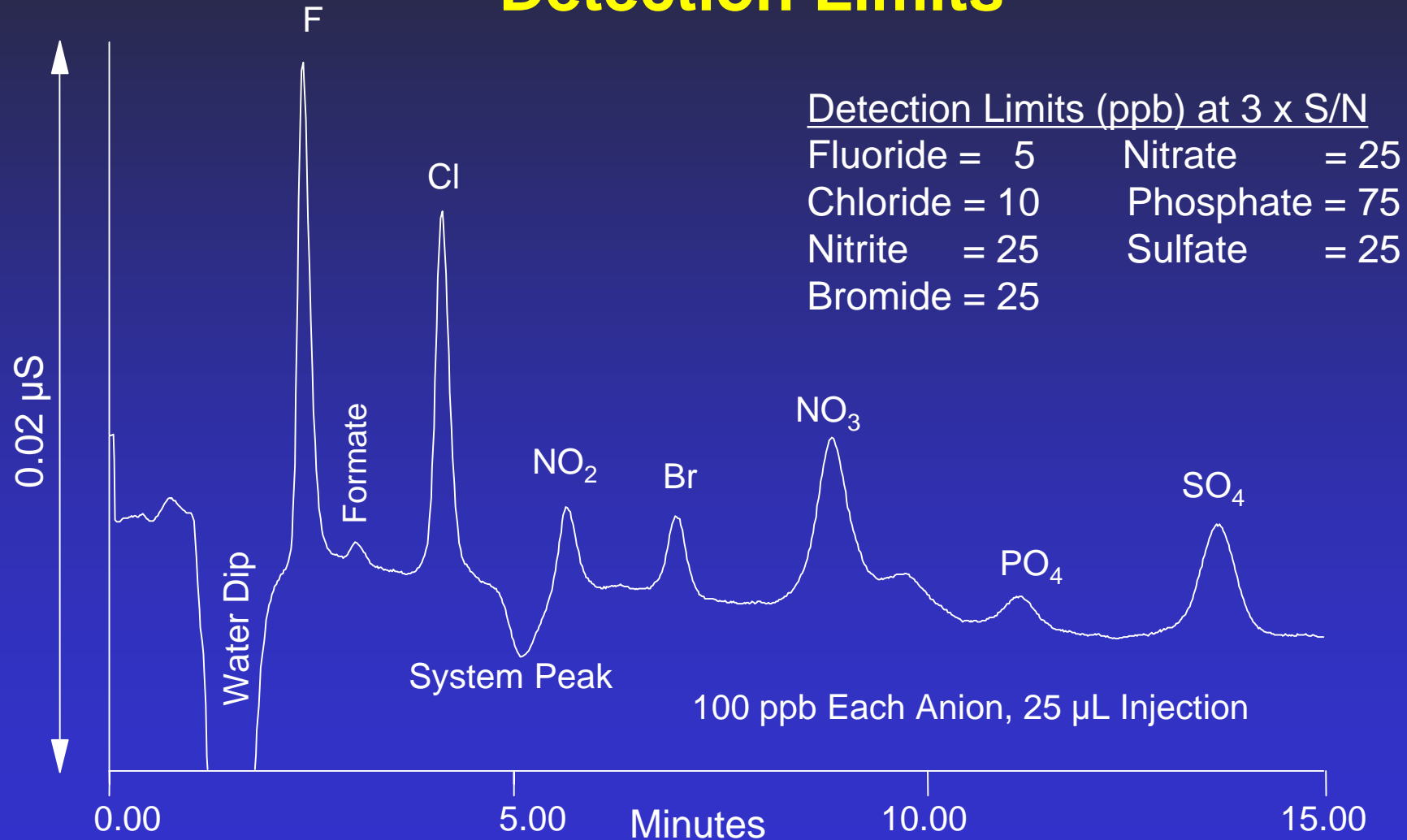
Peak Area Response Linearity



Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

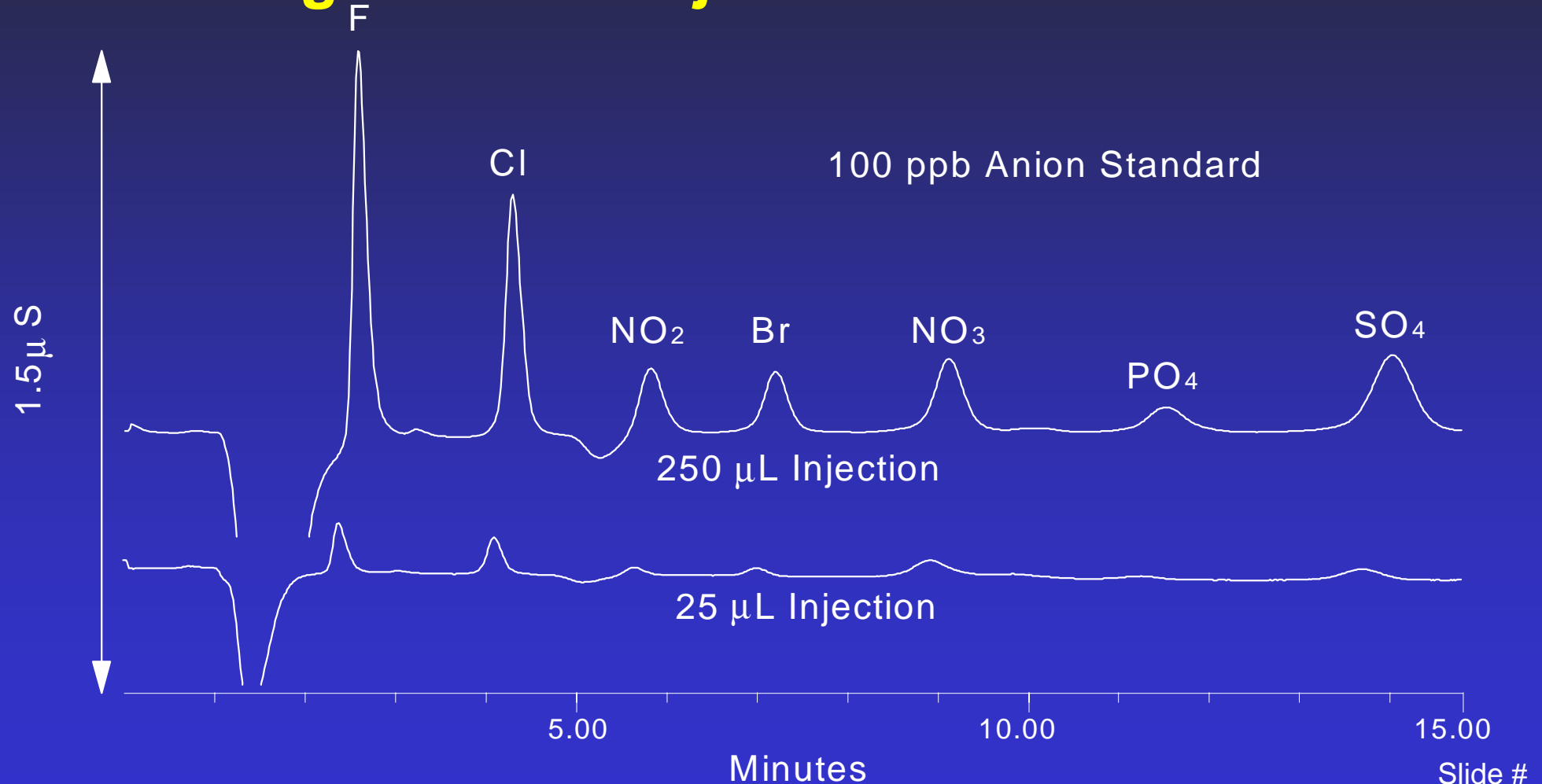
Detection Limits



Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Large Volume Injection Detection Limits



Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Large Volume Injection Detection Limits

Detection Limits at 3 times S/N	25 uL Injection ppb	250 uL Injection ppb
Fluoride	5	<1
Chloride	10	<1
Nitrite	25	5
Bromide	25	5
Nitrate	20	5
Phosphate	75	10
Sulfate	20	5

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Accuracy Using a Performance Evaluation Standard

	Analyte	F	Cl	NO ₂	NO ₃	PO ₄	SO ₄
Performance Evaluation Standard	True Value in ppm	3.43	104.46	3.68	16.64	4.57	56.70
Official Anion Methods Wet Chem	Measured Mean	3.41	107.01	3.61	16.90	4.69	59.51
	Measured Std Dev	0.16	6.48	0.43	1.15	0.25	6.94
Alliance IC System & ERIS 1000HP IC-Pak A HR HCO ₃ /CO ₃ Eluent	Ave IC n = 3	3.21 ± 0.01	116.54 ± 0.40	3.53 ± 0.03	15.43 ± 0.04	4.32 ± 0.04	59.32 ± 0.22
	IC / Mean	0.941	1.088	0.978	0.913	0.921	0.997
	IC / True Value	0.936	1.116	0.959	0.927	0.945	1.046

The performance evaluation standards were purchased from APG Laboratories and diluted 1:100 with Type 1 DI Water.

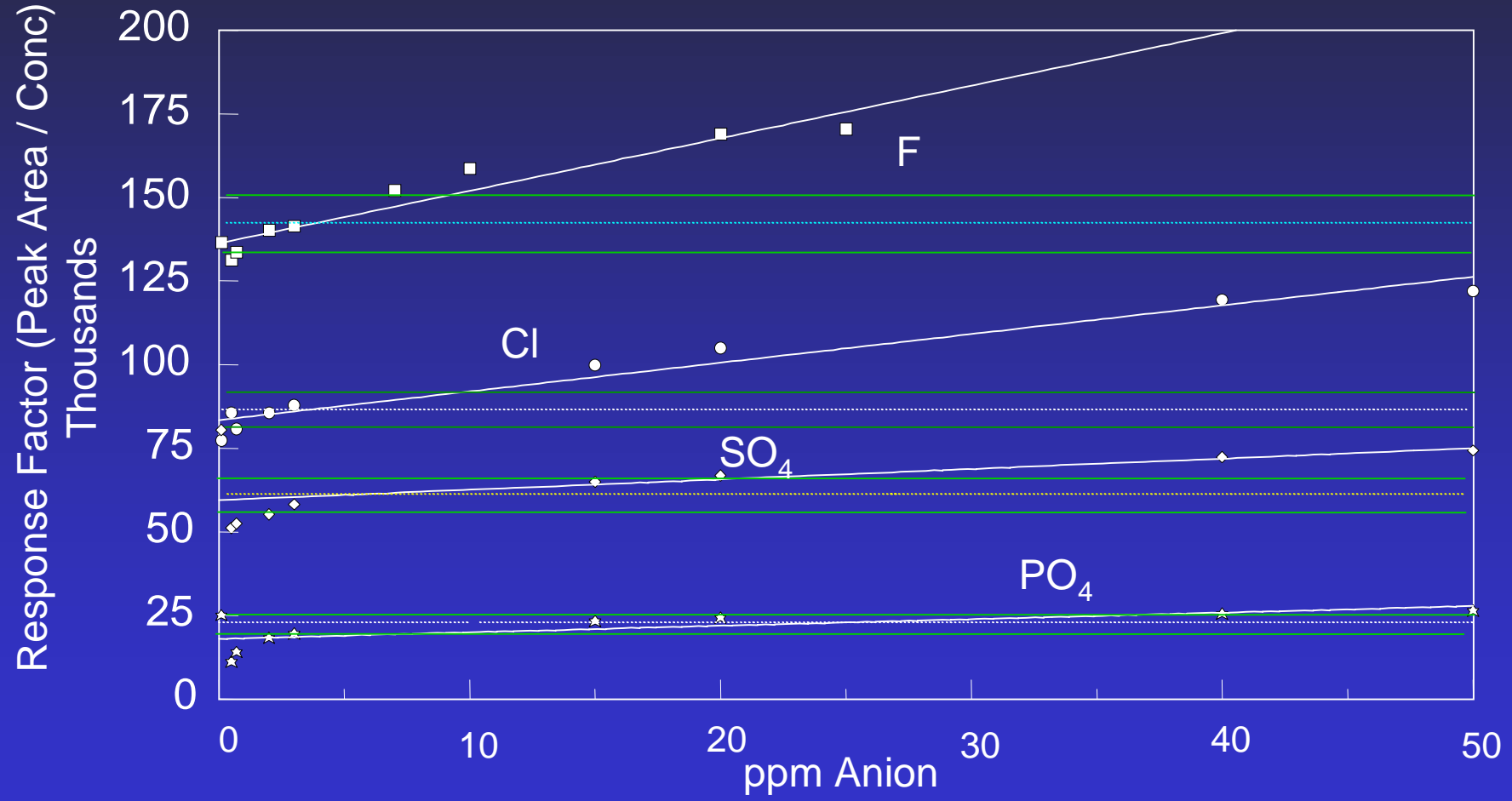
The measured results are the average from numerous laboratories using conventional, EPA approved wet chemistries and IC methods.

An IC/True Value of 1.000 indicates perfect agreement.

Chemical Suppression Ion Chromatography

Using Alltech 1000HP Autosuppressor

Response Factor (ASTM) Linearity

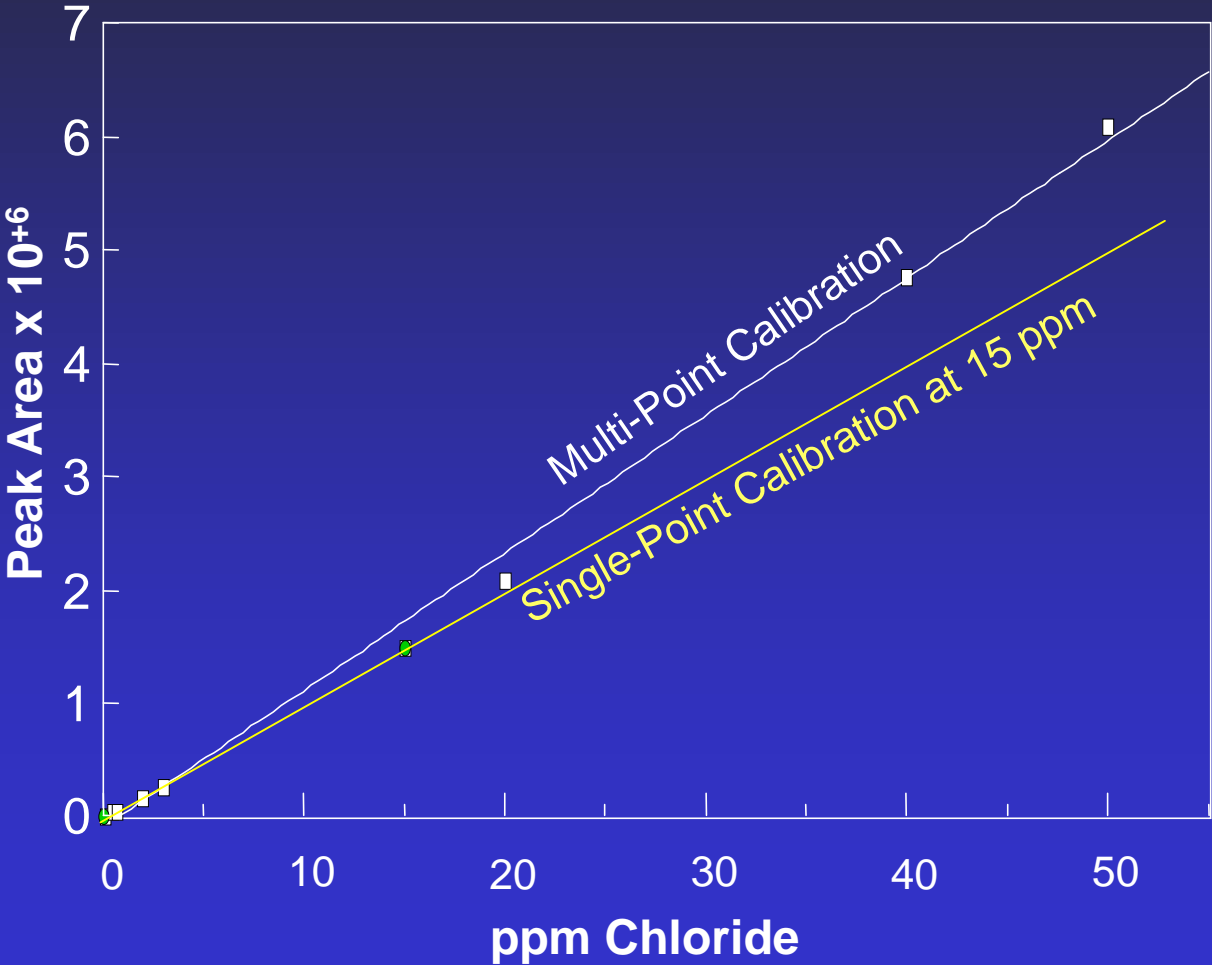


Questionable Linearity?

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Multi-Point vs Single Point Chloride Calibration



Actual ppm Cl	Single Point Calculation	Error Due to Linearity
0.1	0.077	-23.0%
0.5	0.428	-14.4%
0.7	0.566	-19.1%
2	1.713	-14.4%
3	2.643	-11.9%
15	Single Point Calibration	
20	21.028	5.1%
40	47.843	19.6%
50	61.1	22.2%

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Multi-Point vs Single Point Calibration

Multi-Point Calibration between 0.1 and 50 ppm

Used 15 ppm Anion for Single-Point Calibration; 3 ppm for Fluoride

	Certified True Value	Calculated Value	Calculated Value	% Difference From	% Difference From
Anion	Concentration in ppm	Multi-Point Calibration	Single Point Calibration	True Value Using Multi-Point	True Value Using Single Point
F	3.63	3.21	3.47	-6.4%	-1.2%
Cl	104.46	116.54	140.60	+11.6%	+34.6%
NO ₂	3.68	3.53	3.33	-4.1%	-9.5%
NO ₃	16.64	15.43	17.03	-7.3%	+2.3%
PO ₄	4.57	4.32	4.13	-5.5%	-9.6%
SO ₄	56.7	59.32	48.58	+4.6%	-14.3%

Optimum quantitation is achieved using a multi-point calibration
bracketing the expected analyte concentration range, or
A single-point at the mid-point of the expected concentration range

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Peak Area Response Precision

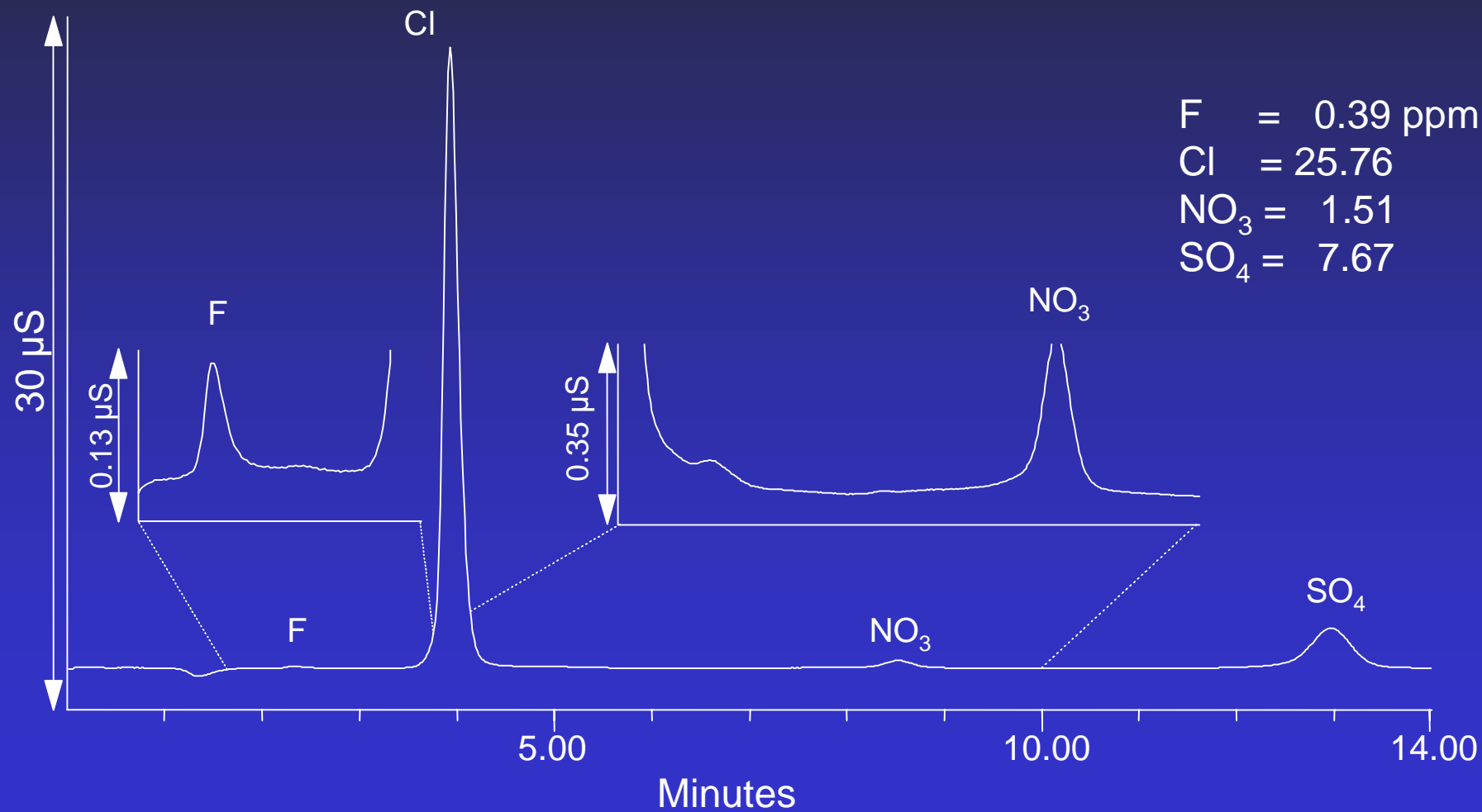
Data as Peak Area %RSD for 7 Replicate Injections
at each Concentration

Analyte		F	Cl	NO ₂	Br	NO ₃	PO ₄	SO ₄
ppm Concentration	0.5	0.62	3.64	6.82	2.57	1.65	8.93	5.58
	0.7	0.71	1.36	4.34	2.39	3.12	3.98	1.28
	2	0.34	3.69	1.95	1.29	1.19	1.58	0.85
	3	0.54	3.91	1.67	1.46	0.74	1.19	0.50
	15	1.36	1.73	0.90	0.84	0.36	0.49	0.34
	20	0.96	1.43	0.80	0.95	0.45	0.64	0.12
	40		0.28	1.64	0.41	0.09	0.48	0.19
	50		0.83	1.05	0.19	0.26	0.46	0.25

Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Typical Drinking Water



Chemical Suppression Ion Chromatography

Using Alltech ERIS 1000HP Autosuppressor

Recovery of Performance Evaluation Standard from Drinking Water

Analyte	F	Cl	NO ₂	NO ₃	PO ₄	SO ₄
Milford	0.39 ±	25.76 ±	Not	1.51 ±	Not	7.67 ±
Drinking Water n=3, as ppm	0.002	0.21	Detected	0.04	Detected	0.01
%RSD	0.40	0.80		2.46		0.16
Performance Evaluation Std	3.43	104.46	3.68	16.64	4.57	56.70
MDW + PES	3.28 ±	138.24 ±	3.37±	17.23±	3.99±	69.40±
n=3, as ppm	0.02	0.91	0.02	0.03	0.05	0.27
%RSD	0.49	0.66	0.43	0.163	1.215	0.39
%Recovery	84.3%	107.7%	91.4%	94.4%	87.3%	108.8%

The performance evaluation standard was diluted 1:100 with typical drinking water. Slide #

Ion Chromatography

Importance of Method Validation

- ★ Single Column IC provides the best linearity and accuracy, but has a detection limit limitation of 100 ppb.
- ★ Chemical Suppression using the Alltech 1000HP Autosuppressor provides the best sensitivity, but has questionable linearity and accuracy limitations.
- ★ The question that needs to be answered,
Do I need sensitivity, or do I need accuracy?
- ★ The choice is based upon the analyte concentration range and the sample matrix.
- ★ Regardless of the answer, the method must be validated in order to have confidence in the results.

What are Oxyhalides?

Oxyhalides are an oxidized form of the common halides

Chloride, Bromide, and Iodide

General formula XO_y , where $y = 2$ to 4

Chlorite	ClO_2	Chlorate	ClO_3	Perchlorate	ClO_4
		Bromate	BrO_3		

Chlorination of drinking water

Ozonization of bottled water

Industrial Bleaching and Oxidizing Agents

Explosive Residues

Oxyhalide IC Methods

- EPA 300.1 Approved, ASTM Pending Std Methods (?)
 - Evolved Dionex Chemistry 2 mm AS9-HC for Common Anions & DBP
 - Uses Suppressed Conductivity Detection with Na_2CO_3
 - Surface , Ground, and Finished Drinking Waters
- EPA 302.0
 - Specific for Bromate and Chlorite using Dionex 4 mm AS9-HC
 - Post Column Derivatization and Visible Detection at 450 nm
- Waters Columns and IC System are Considered Equivalent for both Methods

Common Anions and Oxyhalides

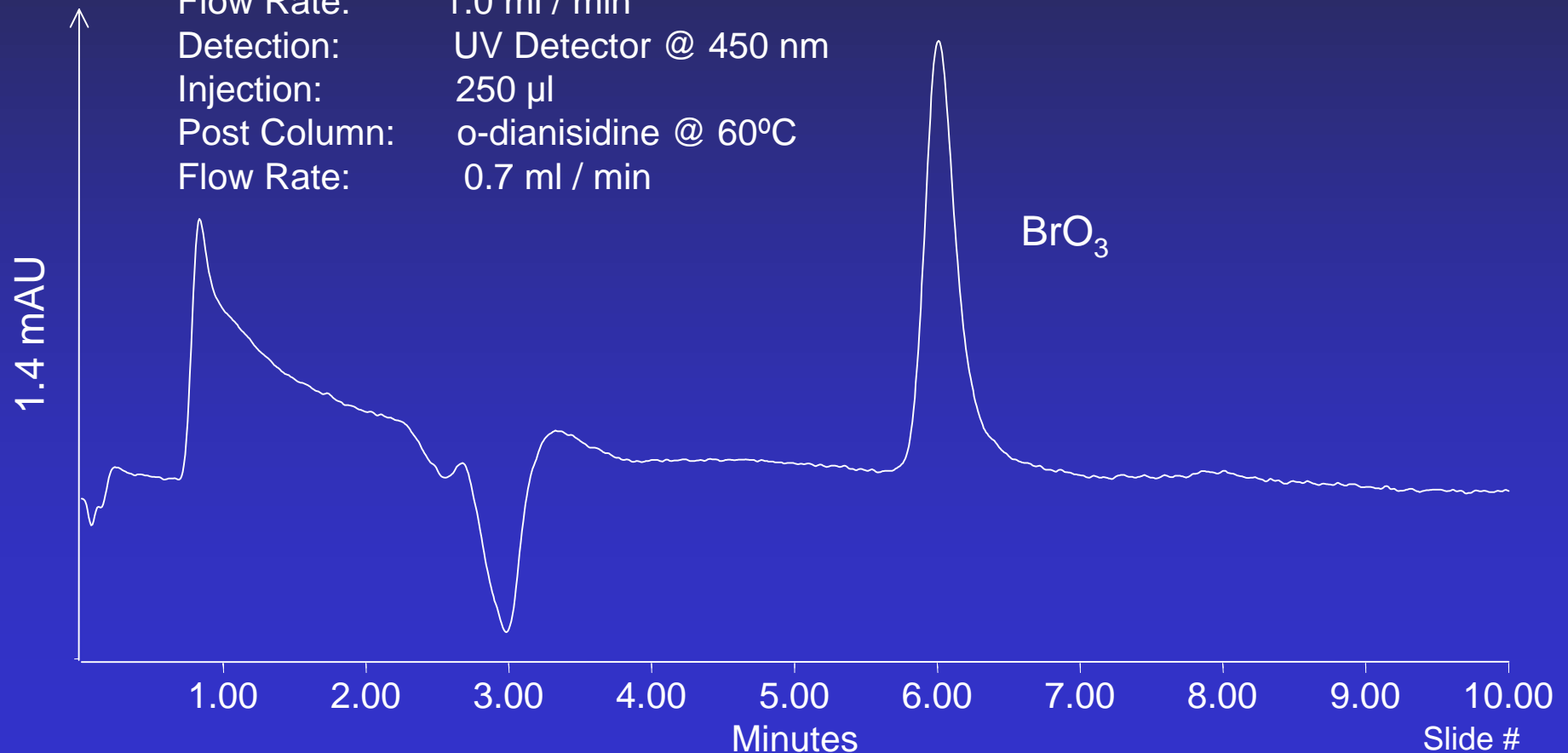
Single Column Conductivity and Direct UV Detection

Column: Waters IC Pak Anion HC
 Eluent: Borate / Gluconate
 Flow Rate: 2 mL/min
 Inj Volume: 100 µL
 Back Cond: 274 µS

<u>Analyte</u>	<u>Detection Limits at 3 x S/N</u>	
	<u>Cond</u>	<u>UV @ 214</u>
F	40	
IO ₃		30
ClO ₂	175	70
BrO ₃	240	100
Cl	40	
NO ₂	63	10
Br	150	80
NO ₃	125	15
PO ₄	450	
SO ₄	185	

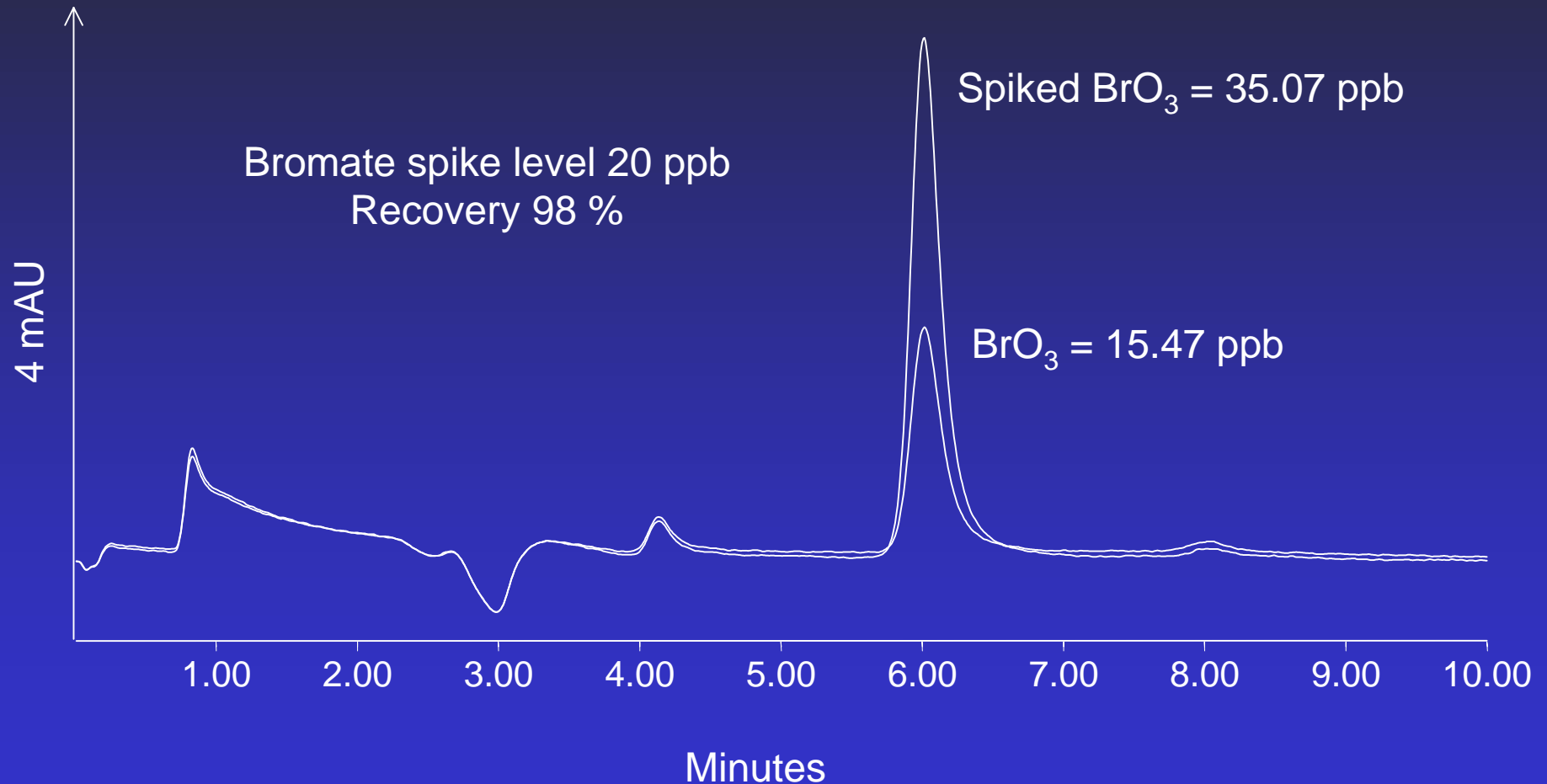
EPA 302.0 Using Waters Alliance IC 10ppb Bromate

Column: IC-PAK™ Anion HC, 4.6X150 mm @ 30°C
Mobile Phase: 5 mM Na₂CO₃, 12% acetonitrile
Flow Rate: 1.0 ml / min
Detection: UV Detector @ 450 nm
Injection: 250 µl
Post Column: o-dianisidine @ 60°C
Flow Rate: 0.7 ml / min

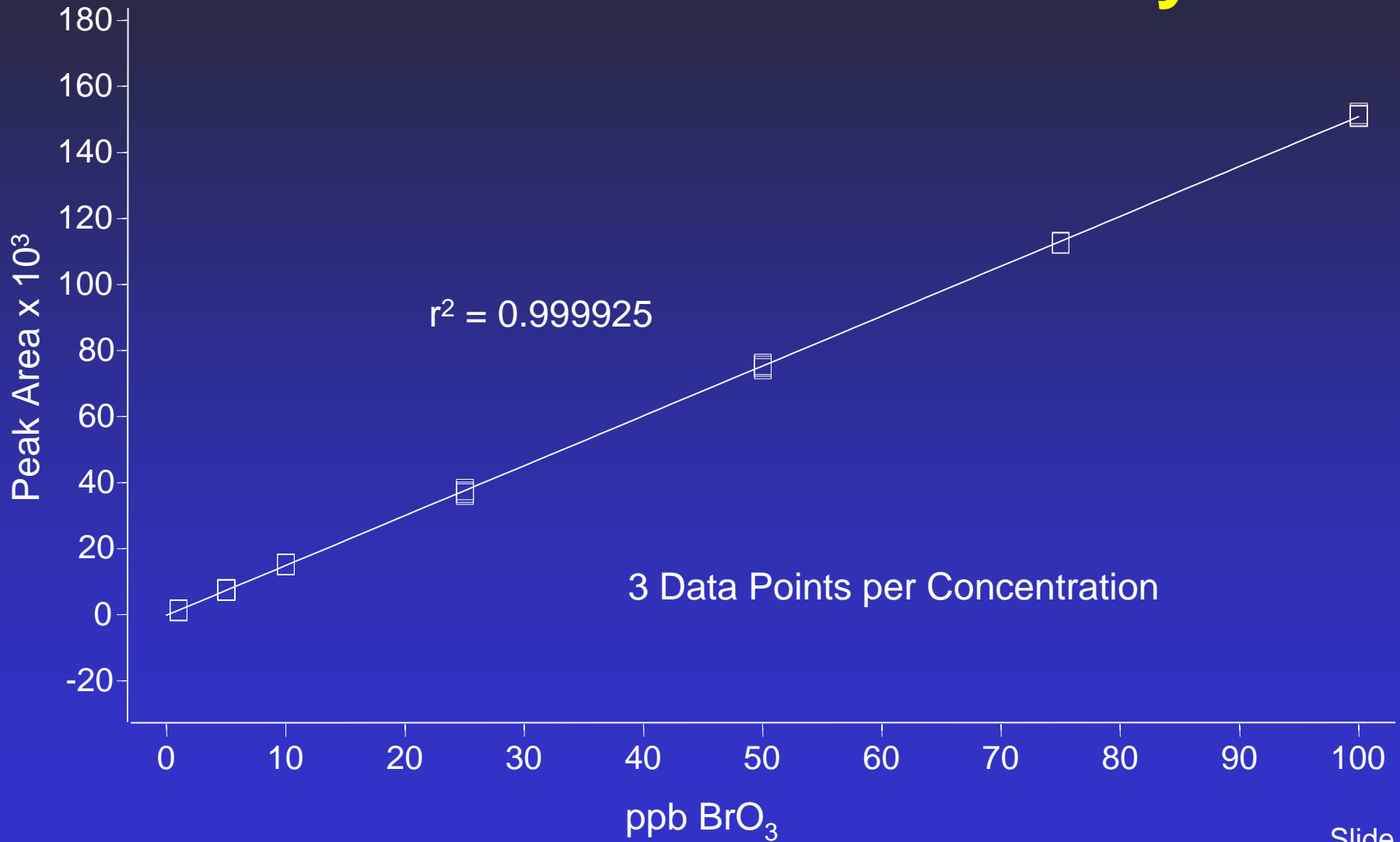


Bromate in Bottled Water

Recovery



EPA 302 Bromate Linearity



Perchlorate Analysis

Suppressed Conductivity Detection

State of California Dept of Health developed (1997) an IC method using Suppressed Conductivity Detection

Uses Dionex AS5 Column
120 mM NaOH / 2 mM p-Cyanophenol
Injection Volume of 740 μL

0.7 ppb Detection Limit in Reagent Water
4 ppb Quantitation Limit at 5 x S/N

Problem with high ionic strength water;
Coelution with void volume response

No EPA, ASTM, or Stds Methods Approval

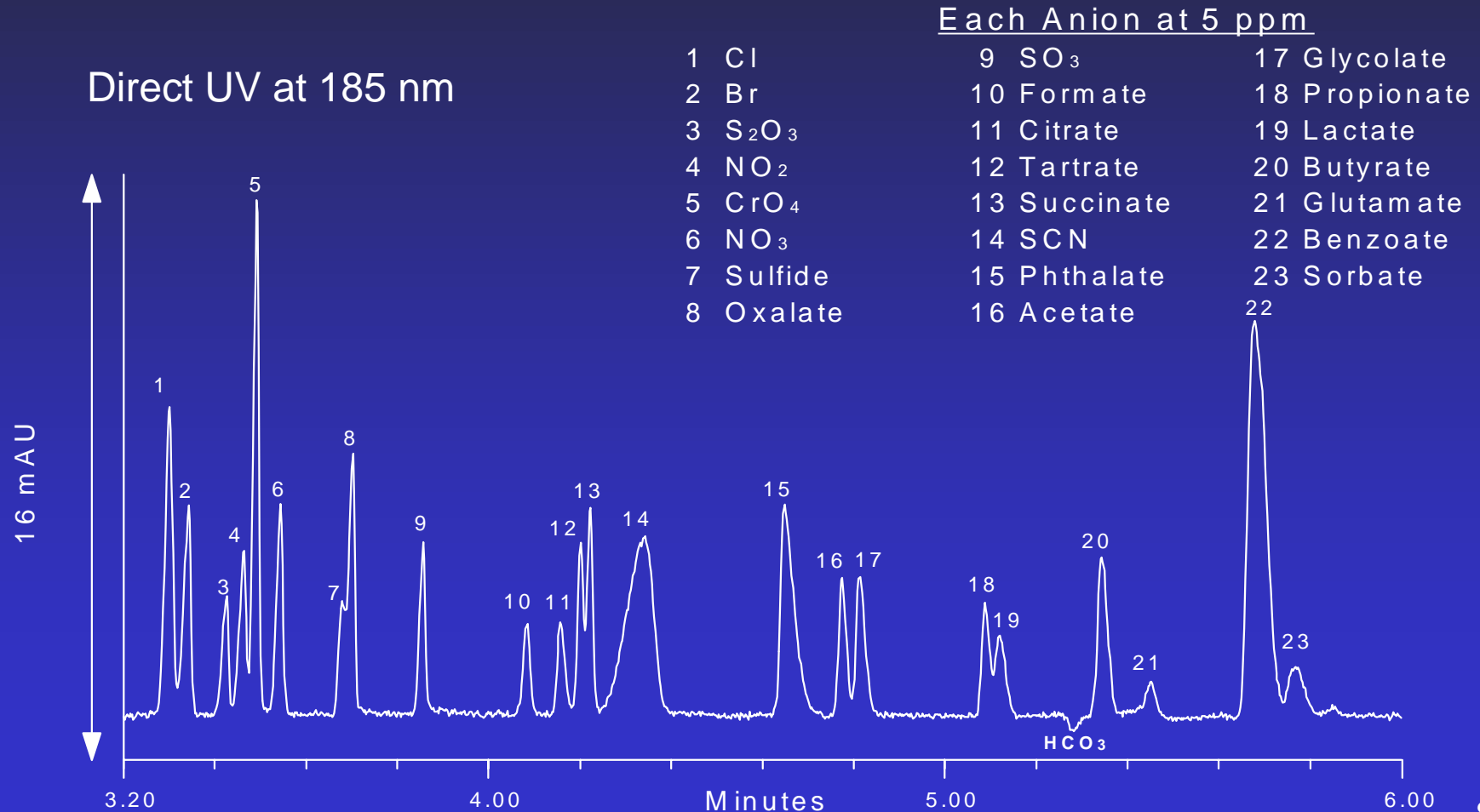
Perchlorate Analysis

Suppressed Conductivity Detection

Column: Waters IC Pak Anion HR
Eluent: 20 mM NaOH / 12% AcCN
Inj. Volume: 500 µL
Detection: Suppressed Conductivity with
Alltech ERIS 1000HP Autosuppressor

Detection Limit: 4 ppb

Capillary Ion Analysis of Inorganic and Organic Acids Phosphate Electrolyte and Direct UV Detection



Chromate Analysis

EPA 218.6, ASTM 5257, and Std Methods 3500

Post Column Derivatization

Uses Dionex AS7 with 250 mM $(\text{NH}_4)_2\text{SO}_4$ / 100 mM NH_4OH
at 1.5 mL/min

Post Column Derivatization and Detection at 530 nm

Ret Time = 3.80 mins Detection Limit = 0.4 ppb with 250 μL

OR EQUIVALENT

Waters IC Pak Anion HC with 25 mM $(\text{NH}_4)_2\text{SO}_4$ / 10 mM NH_4OH
at 1.5 mL/min

Post Column Derivatization and Detection at 530 nm

Ret Time = 5.4 mins

Detection Limit = 1.8 ppb with 100 μL
= 0.7 ppb with 250 μL

Chromate Analysis

EPA 218.6 Modified Direct UV Detection

Waters IC Pak Anion HC with 25 mM $(\text{NH}_4)_2\text{SO}_4$ / 10 mM NH_4OH
at 1.5 mL/min

Direct UV Detection at 365 nm

Ret Time = 5.4 mins

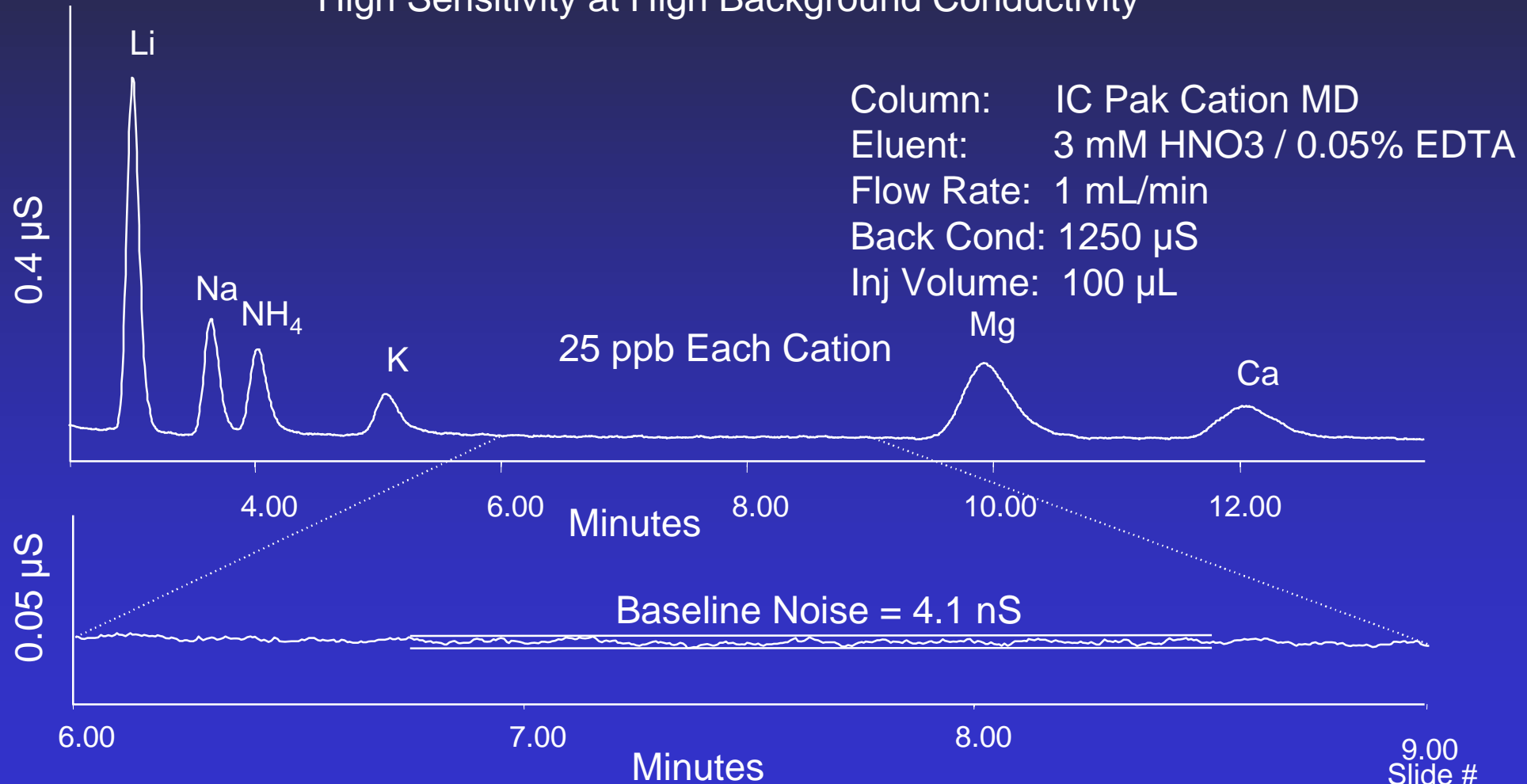
Detection Limit = 4.7 ppb with 100 μL
= 1.9 ppb with 250 μL

Simpler detection alternative with comparable results
Not Approved by EPA, ASTM, or Std Methods

Cation Analysis

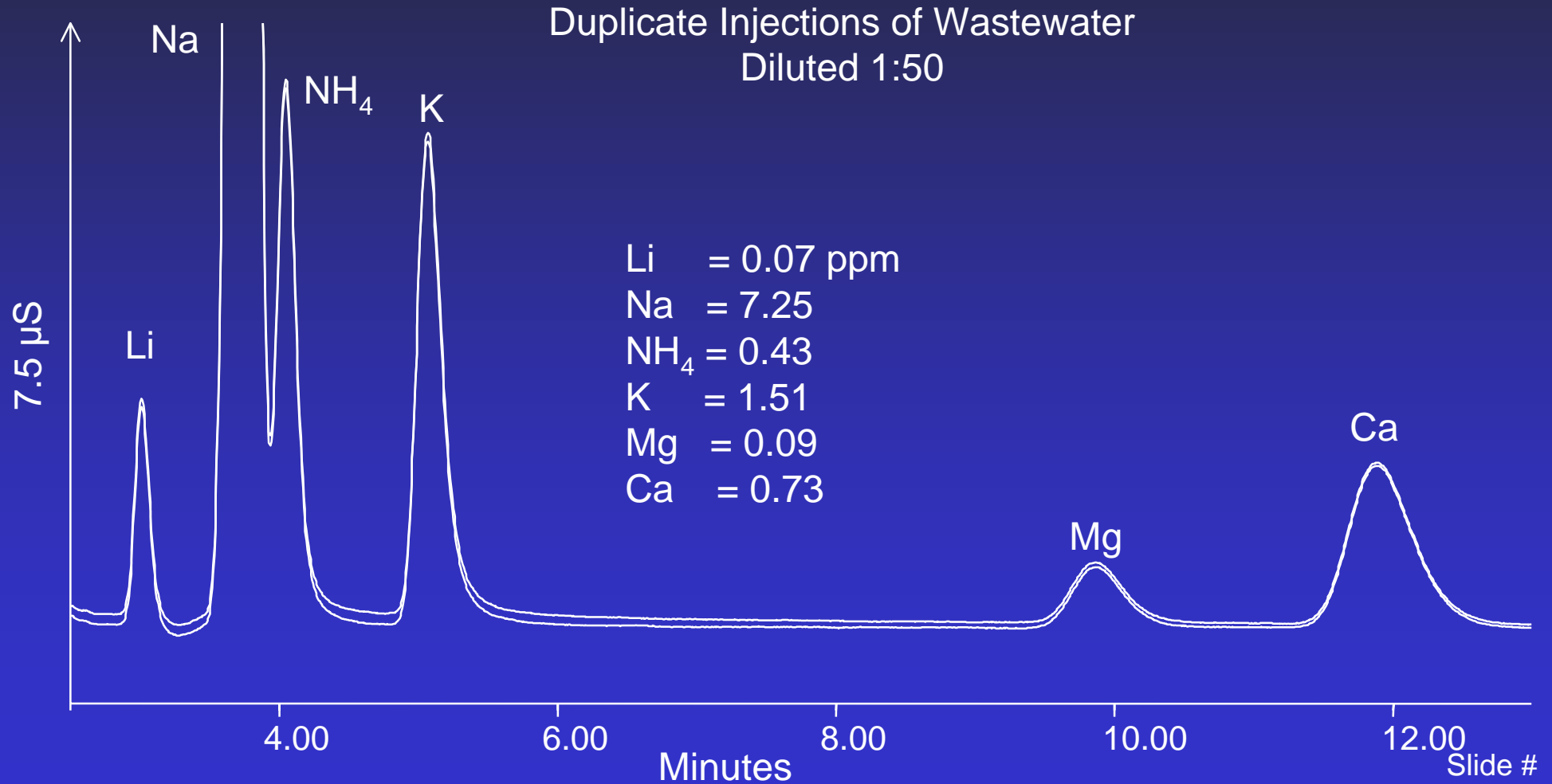
Single Column Conductivity Detection

High Sensitivity at High Background Conductivity



Cation Analysis

Single Column Conductivity Detection



Common Problems Encountered with IC Analysis

- ★ 80% of all IC performance problems can be Attributed to the Quality of DI Water used to Prepare Eluents, Standards, and Sample
- ★ 18 Megohm DI Water contains ppm levels of TOC
- ★ TOC concentrates on the polymeric column surface with all aqueous eluents
- ★ As TOC elutes off it causes
 - ★ Unstable conductivity and UV baselines
 - ★ Loss of Column Efficiency resulting in broad peaks, and
 - ★ Loss of Column Capacity resulting in decreased retention times
 - ★ Acts as potential ion exchange sites

DI Water Quality

- ★ ASTM D1193 Type I DI Water specifies <100 ppb TOC, and Bacteria <10 colonies / L
- ★ TOC is non conductive and is not detected using resistivity
 - ★ TOC in feed water to DI system
 - ★ Plasticizers leached from polymeric tubing
 - ★ Bacterial Contamination
- ★ Indicator is presence of formate and acetate (organic acids) in the eluent and sample
- ★ Always use freshly drawn DI Water!
- ★ Change DI System Cartridges every 6 months

IC Column Fouling

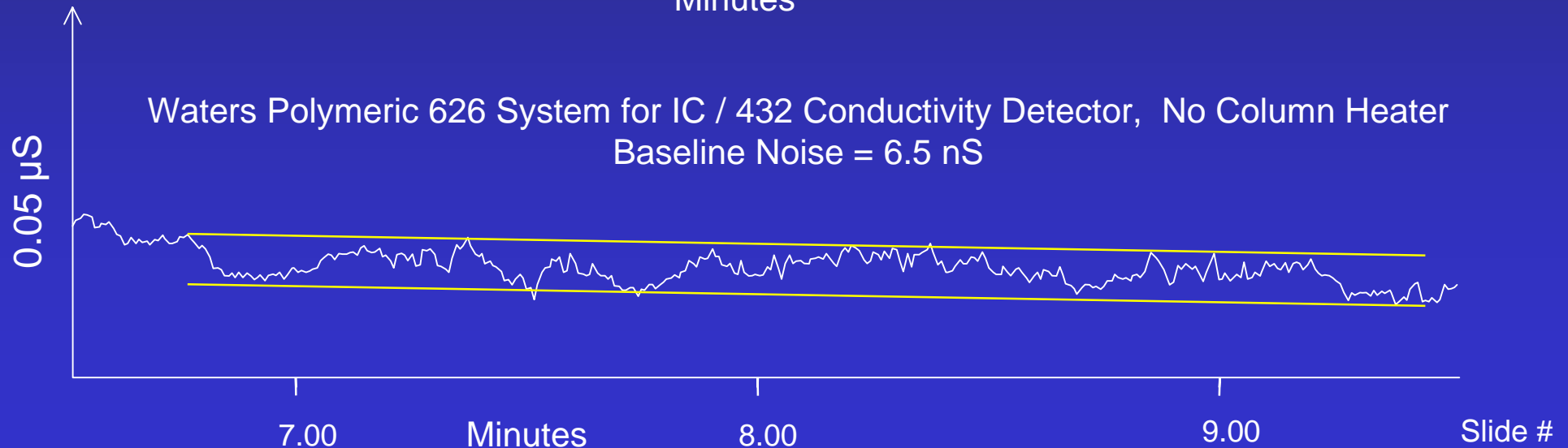
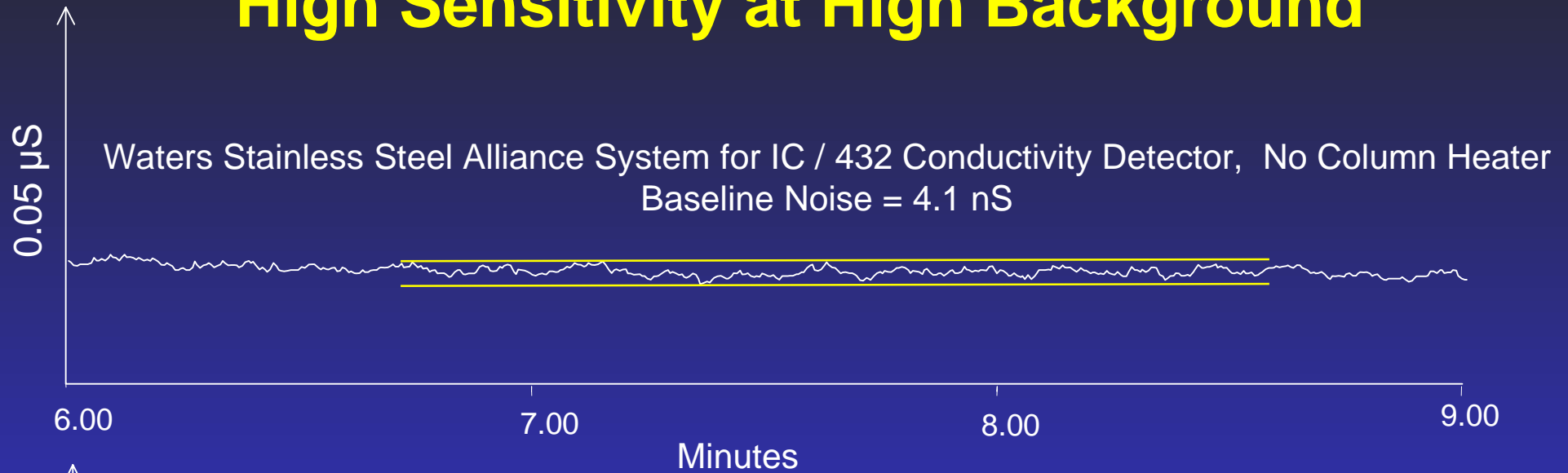
- ★ Absorption of TOC from eluent and samples
- ★ Use Good Quality DI Water
- ★ Remove organics from the sample using Solid Phase Extraction before injection
- ★ Wash column with AcCN periodically to remove TOC from surface
- ★ Wash column with 1% Na₂EDTA & 100 mM HNO₃ to clean ion exchange sites

IC Hardware Considerations

- ★ Polymeric IC or Stainless Steel IC System
- ★ Pump Performance effects baseline noise and RT reproducibility
 - change pump seals routinely
 - always degass eluent; He sparge has marginal effect
- ★ Detector Performance effects baseline noise and drift
 - direct temperature control of the conductivity detector eliminates thermal drift
 - conductivity noise increases as eluent background conductivity increases

Conductivity Baseline Noise

High Sensitivity at High Background



Ion Chromatography

Stainless Steel vs Polymeric Systems

- ★ Stainless steel is “inert” to typical single column and chemical suppression eluents (pH 1 to 13)
 - HNO_3 , H_3PO_4 , H_2SO_4 , MSA
 - Borate / Gluconate, NaOH , NaHCO_3 / Na_2CO_3 , Tetraborate
- ★ Greatest source of polyvalent metal contamination is the sample matrix and eluent impurities; not from SS corrosion
- ★ Polyvalent metals bind to SO_3^- sites on suppressor membranes or columns causing distorted peak shape and loss of specific analyte response, such as PO_4 or divalent organic acids