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Automation of Sample Preparation in Ion Chromatography

Praha, 2nd October 2014

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ACO Sales Manager (IC and SP)

The world leader in serving science

Agenda

- **Thermo Scientific Dionex Sample Preparation Products**
- **Automation of Sample Preparation in Ion Chromatography**
  - pH and Conductivity Measurement
  - Inline Filtration
  - Inline Sample Preparation
  - Matrix Elimination using Dionex InGuard Chemistries
  - Matrix Elimination with Selective Analyte Trapping

Thermo Scientific Dionex Sample Prep Product Line

Thermo Scientific™ Dionex ™ASE™ 150 and 350 Accelerated Solvent Extractor

Thermo Scientific Dionex AutoTrace™ 280 Solid-Phase Extraction (SPE) Instrument

Thermo Scientific Dionex SolEx™ SPE Cartridges

Genevac Rocket™ Evaporator

Novel & Innovative Solutions

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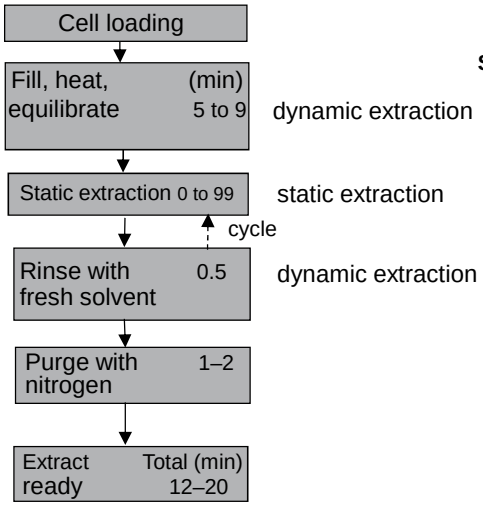
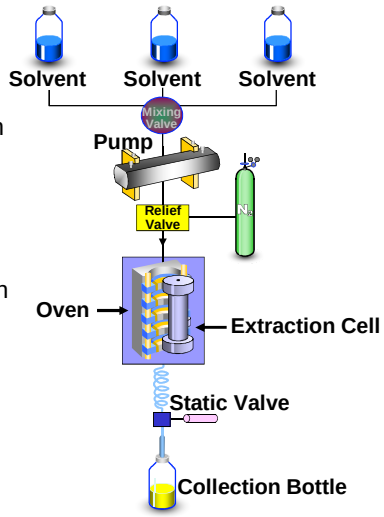
How Does Accelerated Solvent Extraction Work?





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AutoTrace 280 : Automated SPE Extraction of large volume of liquid samples

- Automated SPE instrument for large volume aqueous samples (20 mL to 4 L)
- Extracts 6 samples with unattended operation
- All SPE parameters automatically controlled
- EPA approved for drinking water extraction

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Dionex SolEx Solid-Phase Extraction Cartridges

- **Cartridges designed for fast, and easy preconcentration of contaminants prior to analysis**
- **Offered in a variety of chemistries and sizes to fit your sample extraction needs**
  - **Silica-Based SPE Cartridges**
    SolEx C8
    SolEx C18
    SolEx C8-Clean (Phthalate-Free)
    SolEx C18-Clean (Phthalate-Free)
    SolEx C18-525
    SolEx Unbonded Silica
  - **Carbon Based SPE Cartridges**
    SolEx Activated Carbon
    SolEx Graphitized Carbon
  - **Polymeric New SolEx Phases**
    SolEx HRPHS
    SolEx SAX
    SolEx SCX
    SolEx WAX
    SolEx WCX

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Rocket Evaporator

Rocket Evaporator

ASE Pucks

Flip Flop Vials

- Fully Automated Workflow: Fills gap not addressed by ASE or AutoTrace
- Reduces Laboratory Error: Pucks allow direct sample transfer from the ASE
- Expedites Sample Processing: Flip Flops evaporator directly into autosampler vials

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Total Workflow Solution: Our Key Advantage

Key Applications:
- Fracking: ASE and LC/MS
- Cr(VI) in soil: ASE and IC
- Pesticides in beverages: AutoTrace and GC/MS

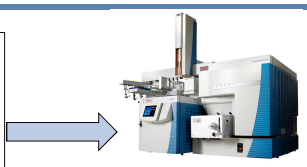
Key Regulations:
- China GB5749-2006 and GB3878-2002: SVOC in drinking water and wastewater
- EPA Method 3545A: POPs in solid matrices
- EU Water Framework Directives

Productivity:
- Thermo Scientific as the single sources of complete analytical workflow
- Reduced solvent use by as much as 90%
- Reduced extraction time to 12 min/sample

ASE 150 and ASE 350

AutoTrace 280

Rocket Evaporator



GC and GC/MS

LC/MS

IC

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Automation of Sample Preparation in Ion Chromatography

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pH-Conductivity Accessory

- A small unit mounts inside an AS-AP autosampler
- Conductivity detector and pH flow cells in series
  - Measurements done prior (!) to injection
  - Conditionals in CM
- USB connection for power and control/communication
- Fluidics connections
- Conductivity mode only- (no pH) just leave plug in place

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pH-Conductivity Accessory

- Easy to install
  - USB Connection for Power and Control
- Leave plug for conductivity only
  - Install pH electrode, if pH measurement is desired

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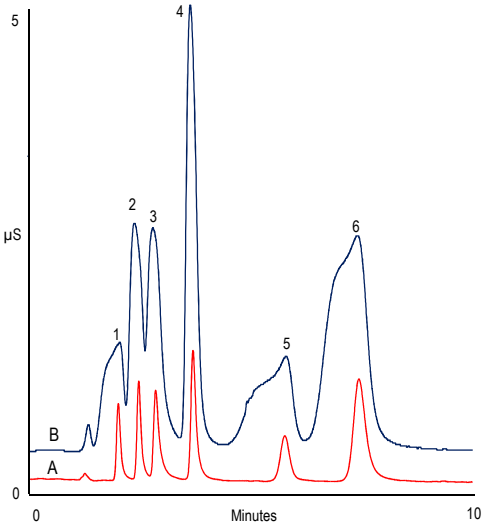
Conductivity based Method Selection



Column:	Thermo Scientific™ Dionex ™ IonPac™ CS12A, 3 × 150 mm
Eluent Source:	EGC-MSA
Eluent:	20 mM MSA
Column Temp.:	Ambient
Inj. Volume:	A: 2.5 µL
	B: 25 µL
Flow Rate:	0.5 mL/min
Detection:	Suppressed conductivity

Sample:	6 cation standard in 100 mM HCl
1. Lithium	0.5 mg/mL
2. Sodium	2
3. Ammonium	2.5
4. Potassium	5
5. Magnesium	2.5
6. Calcium	5

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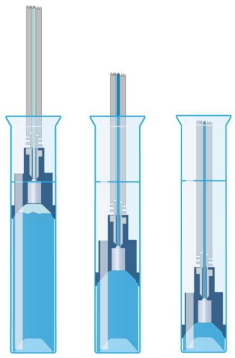
Inline Filtration with Thermo Scientific Dionex AS-DV

- Thermo Scientific™ Dionex™ AS-DV positive displacement autosampler
  - Each sample uses a separate filter, NO carryover
  - Filter caps (20 µm pore size)
  - Very low dead volume
  - Particulates tend to settle to the bottom, filter moves from top down
  - Loop and concentrator loading
  - No sample waste
  - Low cost autosampler
  - No sample pump needed



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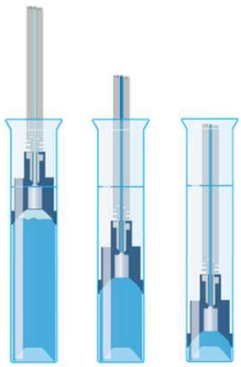
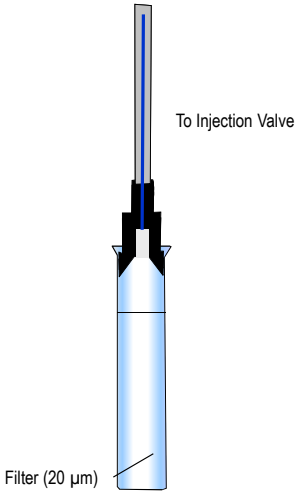
Dionex AS-DV: How Sample Injection Works





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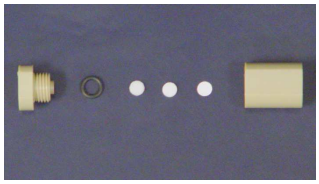
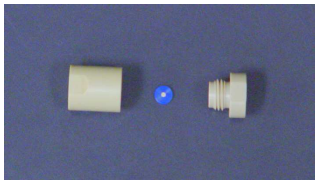
Inline Filtration with Cartridges and Valves

High pressure In-Line filters

- **Multiple configurations available**
  - Filter in sample line before injection valve
  - Inline on high pressure side
  - Dual filters and 10-port valve with backflush pump
  - High capacity, high volume filter stack of 35 µm, 7 µm and 0.45 µm filters
  - Low capacity, low volume filter frit, 0.5 µm
- **Advantages**
  - Entire sample is filtered- no sample loss
  - Small sample volume loaded onto filter maximizes capacity
  - Filter backflush minimizes carryover and extends filter lifetime





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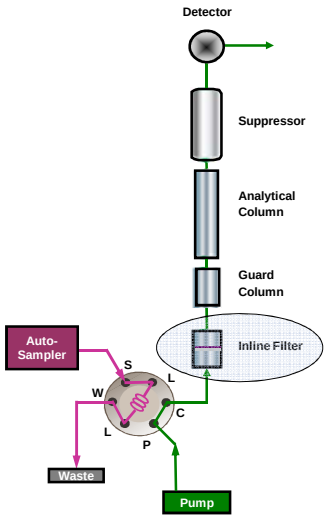
Inline Filtration: Easy to implement!



System Backpressure Indicates When to Exchange Filter

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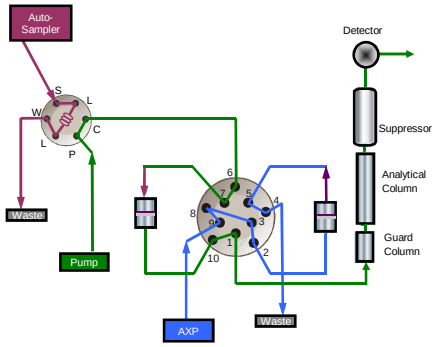
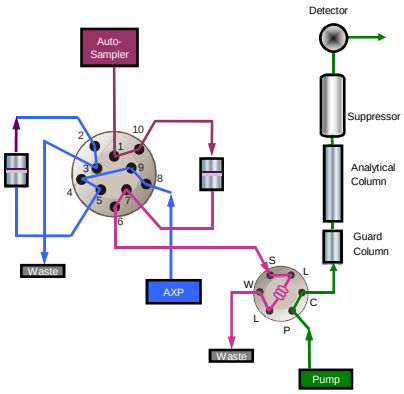
Inline Filtration: Low Pressure or High Pressure



Filter on the high pressure side



Filter on the low pressure side

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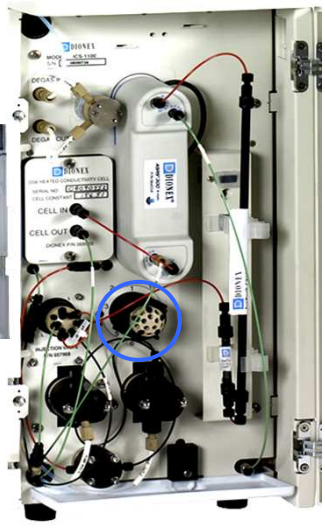
Auxiliary Valves, ICS-5000 and ICS-2100



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Thermo Scientific Dionex InGuard Cartridges

- Cartridges are high capacity

Cartridge Type	Cartridge Capacity
Ag	5 to 5.5 mEq
H	5 to 5.5 mEq
Na	5 to 5.5 mEq
HRP	2 g resin
Na/HRP	50% Na / 50% HRP

- Inline approach- sample volume per cycle equals injection volume
  - Small amount loaded onto cartridge increases effective capacity to many samples per cartridge
  - Reduced sample processing time, labor and cost

High Capacity and Small Sample Volume: Many Samples / Cartridge

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Description of Matrix-Elimination using Thermo Scientific Dionex InGuard Products

- An autosampler or pump is used to pass the sample through the cartridge to remove matrix components.
- The cartridges have very low backpressure but the chemistries usually function optimally at flow rates between 0.5 and 2 mL/min.
- This Sample Preparation is suitable for both standard bore and microbore IC systems
- The number of samples that can be treated with an Dionex InGuardproduct depends on the matrix composition, concentration of compounds to be removed and the sample injection volume.

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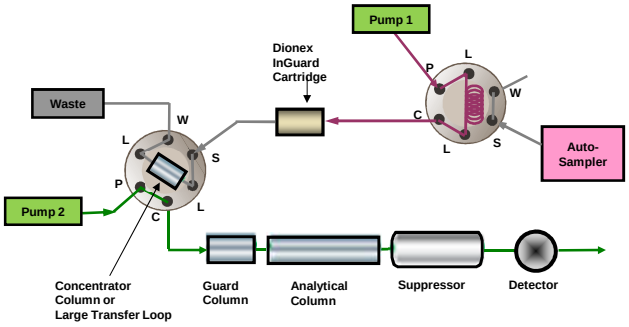
What is the General Setup?



Autosampler: Thermo Scientific™ Dionex™ AS-DV, AS-AP

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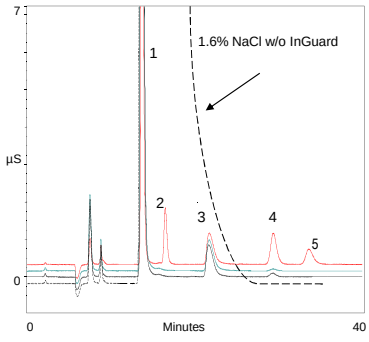
Thermo Scientific Dionex InGuard Cartridges

Application example: Anion analysis in 1.6% NaCl, treat with Dionex InGuard Ag followed by Dionex InGuard Na



Column:	Thermo Scientific™ Dionex™ IonPac™ AG15 and AS15, 4 mm
Eluent:	23 mM KOH, EGC
Flow rate:	1 mL/min
Sample Prep:	Dionex InGuard Ag and Na cartridges
Injection vol:	100 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS 300

- 1.6% NaCl blank without InGuard
- 1.6% NaCl with Dionex InGuardAg and Na
- Water blank with Dionex InGuard
- Std of 2ppm nitrite, sulfate, nitrate in 1.6% NaCl using Dionex™ InGuard™

Peaks:	mg/L (in standard)
1 Chloride	
2 Nitrite	2
3 Carbonate	
4 Nitrate	2
5 Sulfate	2

Dionex InGuardeasily removes interfering chloride, automatically

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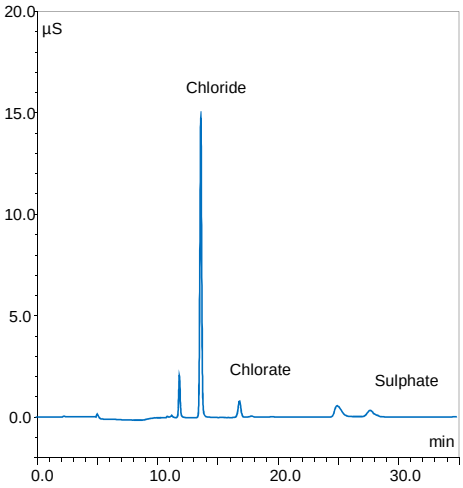
Anions in 50% NaOH



Column:	Thermo Scientific™ Dionex™ IonPac™ AS19 w/ guard
Eluent:	15 mmol/L KOH
Transfer Eluent:	Water
Flow:	0.25 mL/min
Transfer Flow:	0.5 mL/min
Sample	
Pretreatment:	InGuard-H
Detection:	Suppressed conductivity
Background Signal:	< 1.5 µS/cm
Suppressor:	Thermo Scientific™ Dionex™ Anion ASRS 300, recycle mode
Suppressor Current:	10 mA
Temperature:	30°C
Injection Volume:	50 µL
Sample Transfer Loop:	1 mL
Sample Preparation:	The samples "50% NaOH" were diluted 1 ad 50 with ultrapure water and immediately analysed.

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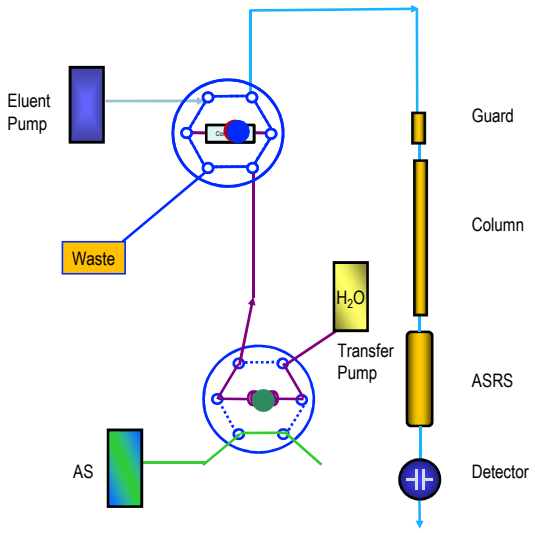
Matrix-Elimination Using Analyte Trapping



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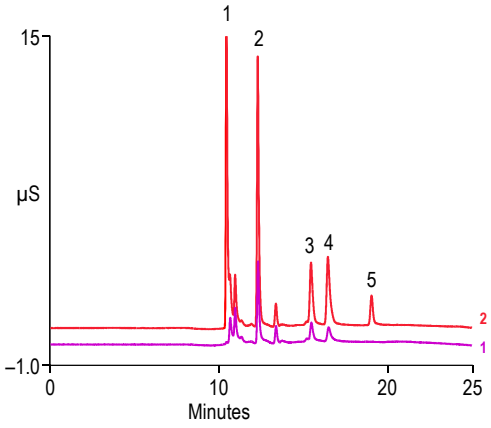
Determination of Anions in 2-Propanol



Column:	Thermo Scientific™ Dionex™ IonPac™ AS18, 2 x 250 mm
Eluent:	18 to 48 mM KOH (EG) in 10 min
Flow Rate:	0.25 mL/min
Inj. Volume:	5 mL concentrated on UTAC-ULP1
Detection:	Suppressed conductivity
Temperature:	30 °C
Suppressor:	Thermo Scientific™ Dionex™ ASRS™, recycle mode with CRD
Sample 1:	**SEMI-grade IPA**
Sample 2:	**4 µg/L spiked SEMI-grade IPA**
Peaks:	1. Fluoride 2. Chloride 3. Sulfate 4. Nitrate 5. Phosphate

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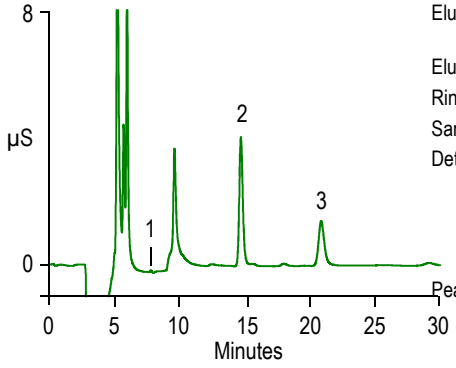
Trace Anion Analysis of Electronic Grade Acetone



Columns:	Thermo Scientific™ Dionex™ IonPac™ AG9-HC, AS9-HC, 2 mm
Concentrator Column:	Dionex™ IonPac™ AG9-HC, 4 mm
Eluent:	8.0 mM Sodium carbonate 1.5 mM Sodium hydroxide
Eluent Flow Rate:	0.25 mL/min
Rinsing Flow Rate:	2.0 mL/min
Sample Volume:	5.0 mL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ II, AutoSuppression™ external water mode
Peaks:	1. Chloride 0.010 µg/L (ppb) 2. Nitrate 20 3. Sulfate 5.4

13508

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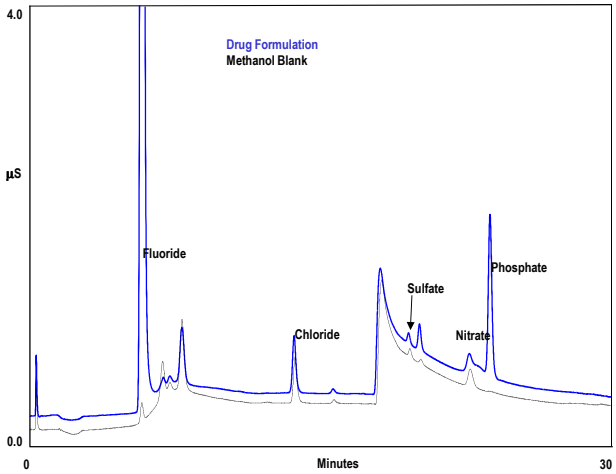
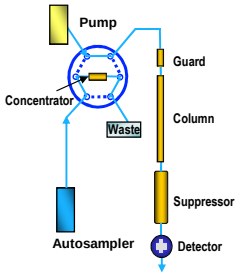
Anions from Water Insoluble Drug



Instrument: Thermo Scientific ™Dionex™ ICS-3000 and AS
Concentrator: UTAC-ULP1 (5 x 23 mm)
Column: Thermo Scientific ™Dionex ™ IonPac™ AG15 (mm)/ AS15 (2 mm)
RFIC-EG: KOH Gradient
Temperature: 30°C
Flow Rate: 0.4 mL/min
Inj. Vol.: 100 µL
Detection: Suppressed conductivity
Sample: Drug formulation in Methanol
Procedure: 1. Sample passed through UTAC
2. Anions collected; drug passed to waste
3. UTAC flushed with water from AS to remove MeOH
4. Anions injected onto column



Anions Trapped and Drug Substance Removed

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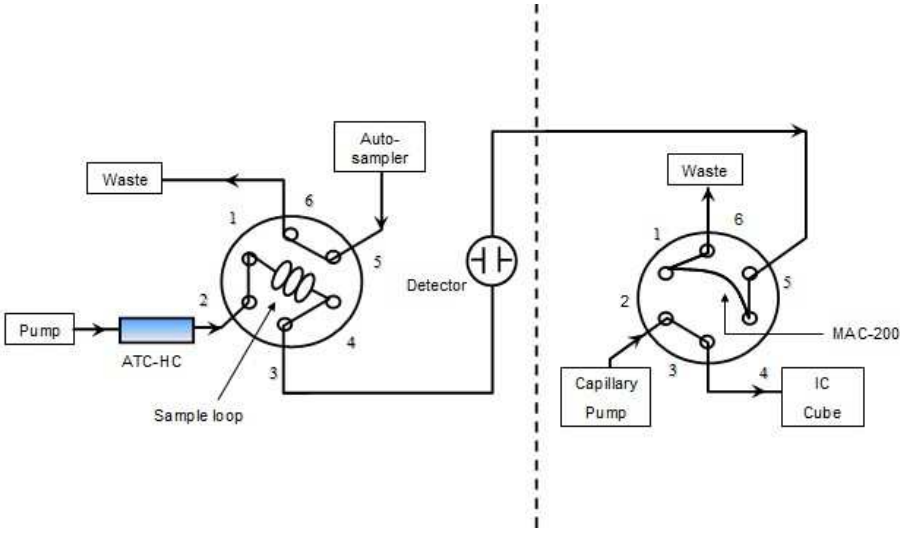
Determination of Trace Anions in Organic Solvent



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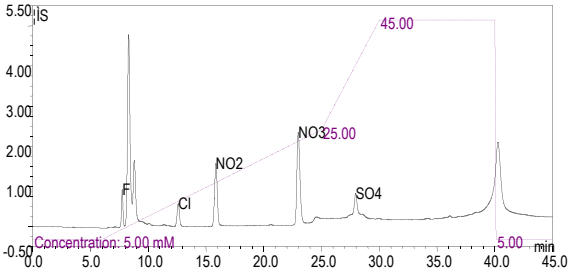
Determination of Trace Anions in Organic Solvent



- Collector: IonSwift™ MAC-200 (0.75×80 mm)
- Column: IonPac™ AG18-Fast capillary Guard(0.4×35mm) +IonPac™ AG18-Fast capillary(0.4×150mm)
- Switching-time: 0-1 min
- Eluent (KOH): 0-6min 5mM; 6-25min 5-25mM; 25-30min 25-45mM; 30-40min 45mM
- Flow-rate ： 10μL/min
- Sample volume: 50 μL

	F	Cl	NO2	NO3	SO4	PO4
Methanol	4.7748	3.6944	13.4431	17.972	4.7748	N.D.
Acetonitrile	3.5126	2.5040	14.3851	17.7439	3.5126	N.D.
Isopropanol	3.3276	2.7701	13.9012	17.0331	3.3276	1.4484
Acetone	3.1213	2.3065	14.0539	17.1718	3.1213	N.D.

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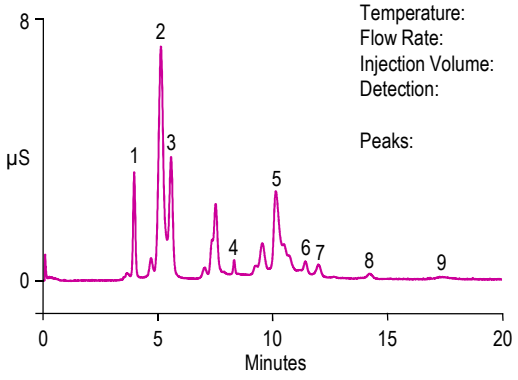
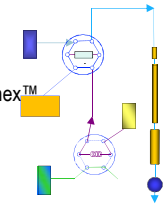
Anions in Hydrogen Peroxide



Concentrator Column:	TAC-LP1
Trap Column:	ATC-1
Column:	Thermo Scientific™ Dionex™ IonPac™ AG15, AS15
Eluent:	KOH (EG40)
Temperature:	35° C
Flow Rate:	2 mL/min
Injection Volume:	400 µL
Detection:	Suppressed conductivity



Peaks:

1.	Fluoride	9.28 µL (ppb)
2.	Acetate	—
3.	Formate	—
4.	Chloride	1.66
5.	Carbonate	—
6.	Sulfate	3.63
7.	Oxalate	—
8.	Nitrate	3.22
9.	Phosphate	3.64

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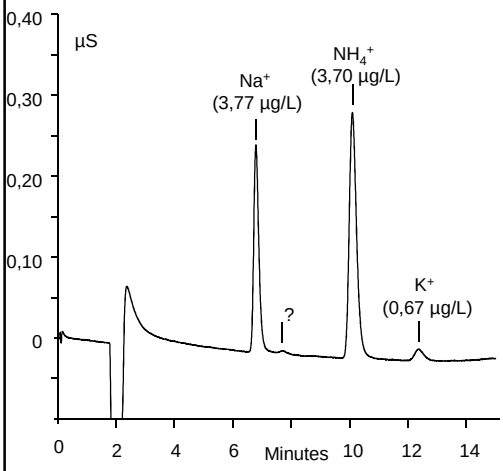
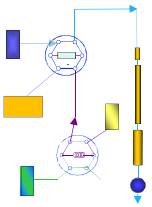
Cations in “Electronic Grade” Hydrogen Peroxide





Column:	Thermo Scientific™ Dionex™ IonPac™ CS10 with Guard (4mm)
Concentrator:	Thermo™ Scientific™ Dionex™ IonPac™ CG12A (4mm)
Eluent:	40 mmol/L MSA
Flow Rate:	1 mL/min
Injection volume:	1000 µL
Detection:	Suppressed conductivity
Suppressor:	Thermo Scientific™ Dionex™CSRS-Ultra, ext. Water

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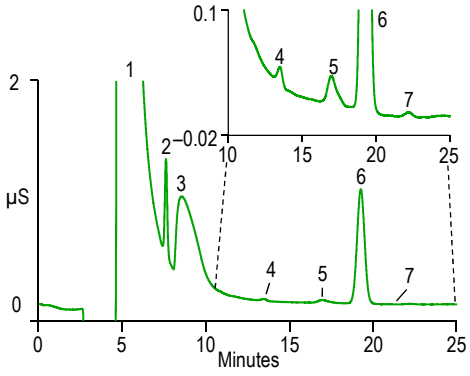
Trace Anions in High Purity 24.5% HF

Pretreatment Column:	Thermo Scientific™ Dionex™ IonPac™ ICE-AS6
ICE Eluent:	Deionized water
ICE Flow Rate:	0.55 mL/min
Sample Volume:	750 µL



Column:	Thermo Scientific™ Dionex™ IonPac™ AG9-HC, AS9-HC, 2 mm
Concentrator:	Dionex™ IonPac™ AG9-HC, 4 mm
IC Eluent:	8.0 mM Sodium carbonate 1.5 mM Sodium hydroxide
IC Flow Rate:	0.25 mL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ ULTRA, AutoSuppression™, external water mode

Peaks:		µg/L (ppb)
1.	Fluoride	—
2.	Chloride	7.9
3.	Carbonate	—
4.	Nitrate	0.89
5.	Unidentified	—
6.	Sulfate	10.1
7.	Phosphate	2.4

14242

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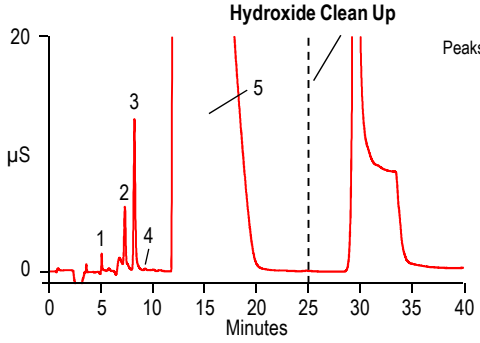
Trace Anions in High Purity 85% Phosphoric Acid

Ion Exclusion (ICE)	
Pretreatment Column:	Thermo Scientific™ Dionex™ IonPac™ ICE-AS6
ICE Eluent:	Deionized water
ICE Flow Rate:	0.50 mL/min
Sample Volume:	200 µL

200 mM Sodium Hydroxide Clean Up



Column:	Thermo Scientific™ Dionex™ IonPac™ AG11-HC, AS11-HC, 2 mm
Concentrator:	Dionex™ IonPac™ AG11-HC, 4 mm
IC Eluent:	20 mM Sodium hydroxide
IC Flow Rate:	0.38 mL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™, AutoSuppression™, external water mode

Peaks:			
1.	Chloride	36	µg/L (ppb)
2.	Unidentified	—	
3.	Sulfate	750	
4.	Nitrate	15	
5.	Phosphate	—	

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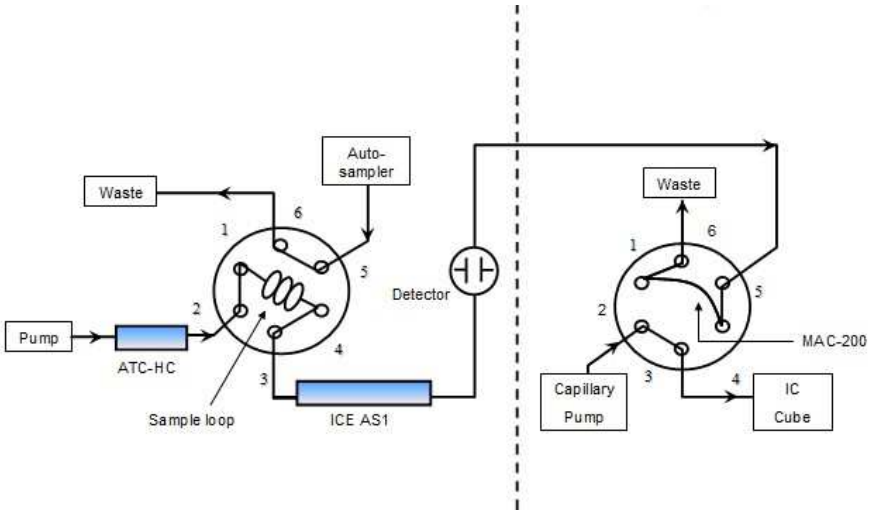
Determination of Trace Anions in Weak Acids



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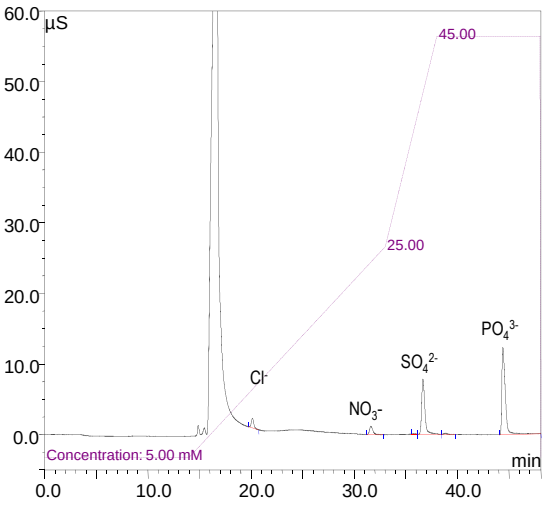
Trace Anions in Formic acid – Capillary IC



1st Dimension
Column: Thermo Scientific™ Dionex™ IonPac™ ICE-AS1(250×9 mm) ion-exclusion column
Sample volume: 50 µL

2nd Dimension
Concentr. Col.: Thermo Scientific™ Dionex™ IonSwift™ MAC-200 (0.75×80 mm)
Column: Thermo Scientific™ Dionex™ IonPac™ AG18-Fast w/ guard (0.4×150mm)
Eluent: KOH-Gradient (RFIC)
Flow rate: 10 µL/min
Detection: Suppressed Conductivity
Suppressor: Thermo Scientific™ Dionex™ ACES (Recycle Mode)

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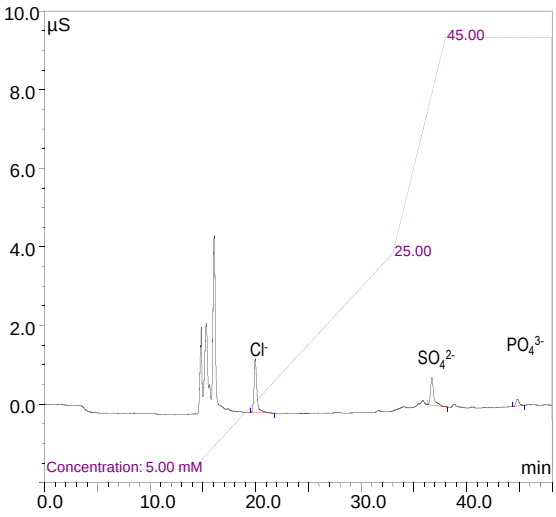
Trace Anions in Acetic Acid – Capillary IC



1st Dimension
Column: Thermo Scientific™ Dionex™ IonPac™ ICE-AS1(250×9 mm) ion-exclusion column
Sample volume: 50 µL

2nd Dimension
Concentr. Col.: Thermo Scientific™ Dionex™ IonSwift™ MAC-200 (0.75×80 mm)
Column: Thermo Scientific™ Dionex™ IonPac™ AG18-Fast w/ guard (0.4×150mm)
Eluent: KOH-Gradient (RFIC)
Flow rate: 10 µL/min
Detection: Suppressed Conductivity
Suppressor: Thermo Scientific™ Dionex™ ACES (Recycle Mode)

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Thank you!

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