Sample Preparation for Ion Chromatography

Comprehensive Solutions to Sample Preparation Challenges

Cathode (-)

OH⁻

OH⁻

OH⁻

Anion Exchange Membrane

C^+
A^- $C^+A^- \longrightarrow C^+OH^-$ C^+
A^-

Anion Exchange Membrane

H^+A^- H^+A^-

H^+

Anode (+)
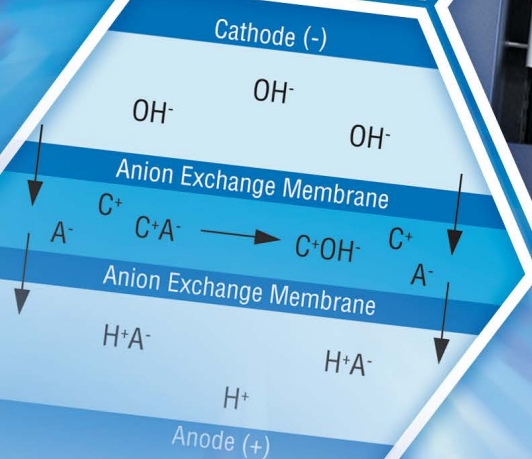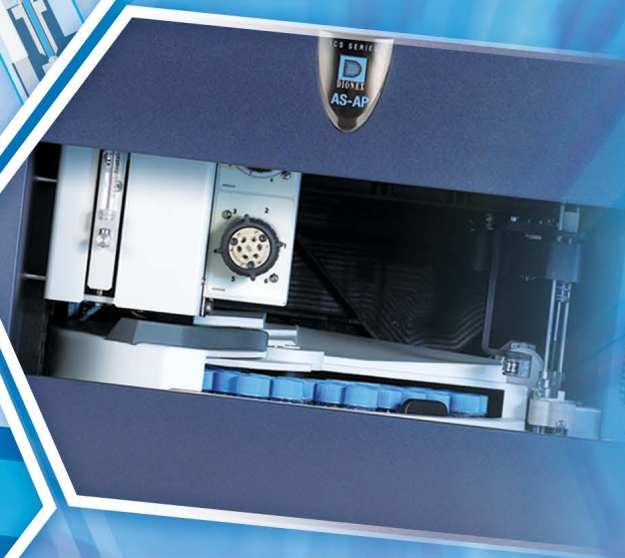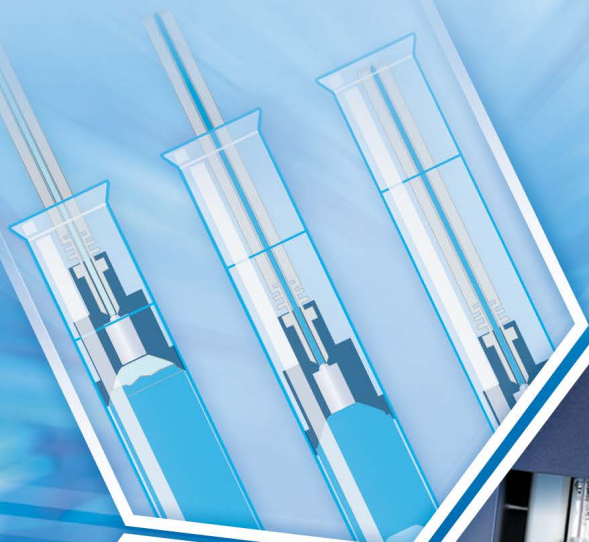
DIONEX

Speed · Simplicity · Solutions

# Choose from a Broad Range of Solutions

No matter what your needs are, turn to Dionex—the ion chromatography (IC) technology leader—for a comprehensive range of proven sample preparation solutions.

- Easy to understand and implement

- Simple to operate

- Highly reliable and reproducible

- Saves time and lowers operating costs

The simplicity and ruggedness of IC often requires no need to prepare a sample before injection. In many cases, a simple dilution of the sample with deionized water is all that's needed. Sometimes more is required to protect the column by removing contaminants or particles or to enhance the sample to make it detectable. Any sample preparation technique must maintain sample integrity, be reproducible, and require minimal labor to accomplish.

Dionex solutions span:

- Off-line solutions that are easy and practical to implement

- In-line and automated technologies using proprietary technologies

- Specialized and high-throughput solutions
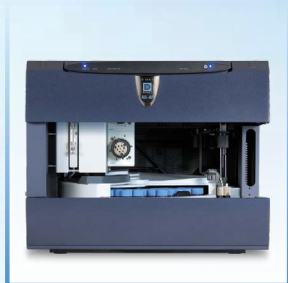
Autosampler Solutions
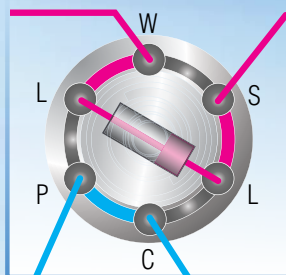
In-line

RFIC-ESP

Chromeleon Software

Cartridge Technologies

Off-line

Electrolytic Trap Devices

IC Integration

Dionex Sample Preparation Solutions

AutoDilution
Conditional determination of high analyte concentration with automatic dilution and reinjection

- Autosampler with Chromeleon® Chromatography Data System (CDS) Software

Matrix Elimination
Remove interfering matrix ions and organic contaminants

- Off-line OnGuard® II Cartridges
- In-line InGuard® Cartridges
- In-line IonPac® NG-1 Columns

Concentrator Columns
Capture and concentrate ions from large, dilute sample volumes

- 15 Anion IC and RFIC Concentrator Columns
- 3 Cation IC and RFIC Columns
- 3 Transition Metal IC and RFIC Trap Columns

Trap Columns
Purify eluent or polish samples

- Anion IC and RFIC Trap Columns
- Cation IC and RFIC Concentrator Columns

RFIC-ESP
Reagent-Free™ Ion Chromatography (RFIC™)-Electrolytic Sample Preparation (ESP)

- Continuously Regenerated Trap Columns (CR-TC)
- AutoNeutralization™ Cartridges
- Electrolytic Water Purifiers (EWPs) for Trace Analysis
- AutoPrep with ESP for Trace Analysis of Anions or Cations

Sample Filtration
Remove particulates that can damage your system; remove microbes to prevent analyte degradation

- Off-line Syringe Filters
- In-line AS-DV Autosampler Filters
- In-line Filter Frit Cartridges

Specialty Systems
Combustion IC and Air Sampling systems

- Combustion IC for Halogen and Sulfur Analysis
- Air Sampling System for Ionic Analysis

Chromeleon Chromatography Data System Software
Plays an integral role in most Dionex automated solutions

- Monitor system pressure to detect filter clogs
- Optimum injection parameters for injection loops and concentrator columns
- Visualization tools for matrix elimination and analyte concentration
- Conditionally controlled autodilution and electrolytic sample preparation techniques

AutoDilution and Analysis
Automatic Detection, Dilution, and Re-injection of Highly Concentrated Samples

Challenge

Sample too concentrated for analysis

Sample needs dilution

Delay in analysis time

Extra work

### Solution—Dionex AutoDilution Capabilities

Automated detection of concentrated samples

Automatic dilution, reinjection, and analysis

Faster results

Saves effort, time, and money

IC systems equipped with an autosampler, Chromeleon CDS software, and the AutoDilution license can monitor chromatographic results. If peak results fall outside a user-specified range, the system will automatically dilute the sample and reinject it. Decisions to dilute and reinject can be based on a single parameter such as peak area, height, amount, resolution or other chromatographic value, or on a combination of values.

AutoDilution: Large Loop/Small Loop Conditional Injection
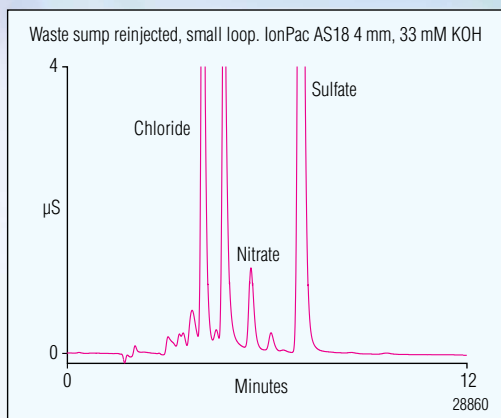
Sample is too concentrated, and analytes are out of the calibration range.
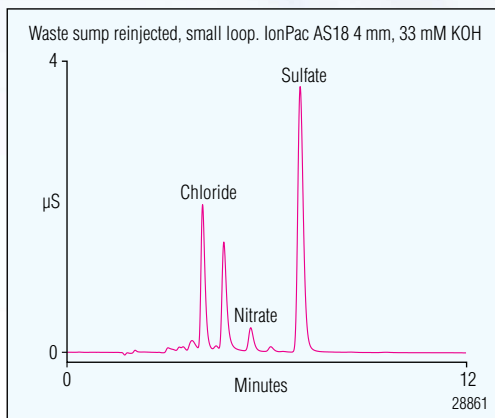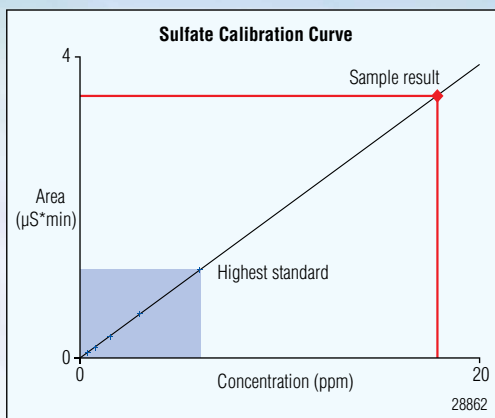
Data system detects concentrated sample, and then autosampler dilutes and reinjects it. Analytes are now in the calibration range.

Matrix Elimination

Selectively Remove Interfering Ions from Matrices Using Solid-Phase Extractants
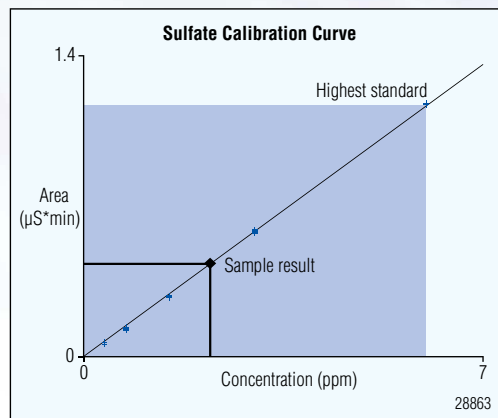
Challenge

Interfering ions

Poor IC performance

Short consumables lifetime

High cost of ownership

Solution—Dionex Matrix Elimination Cartridges

Eliminate interfering ions

Automated and manual methods

Superior IC performance

Extended lifetime of consumables

**Lower cost of ownership**

Off-line Matrix Ion Elimination
OnGuard II Cartridges

OnGuard II Cartridges have Luer inlets for manual processing of a limited number of samples. A wide range of cartridges loaded with resins in 1 and 2.5 cc capacity formats remove matrix interferences encountered in many ion chromatography applications, (see table at right).

These cartridges have wide pH stability and allow low-level ion analysis. They remove both column-fouling substances and interfering ions where several mL of sample is available. An OnGuard Sample Prep Station is available for simultaneous matrix elimination of multiple samples.

OnGuard II Cartridges*	
Resin	**Removes from Sample Matrices**
A	Anionic contaminants; Neutralization of highly acidic samples
Ag	Chloride, bromide, and iodide
Ba	High concentrations of sulfate
H	High levels of alkaline earth and transition metals; Neutralizes highly alkaline samples (e.g., sodium hydroxide or sodium carbonate)
Na	High levels of alkaline earth and transition metals without acidifying the sample
M	Transition metals; also for matrix elimination of alkali and alkaline earth metals
P	Phenolic fraction of humic acids, tannic acids, lignins, anthocyanins, and azodyes prior to analysis by ion exchange
RP	Hydrophobic substances such as aromatic dyes, some aromatic carboxylic acids, hydrocarbons, and surfactants
Ag/H	Chloride, bromide, and iodide from concentrated matrices such as brines
Ba/Ag/H	Respective contaminants using a layered resin cartridge containing the Ba, Ag, and H styrene-based sulfonic acid resins

Cartridges can be used singly or in series.

Iodide Area = 1 mg/L

RSD (peak area) = 1.4%

RSD (retention time) = 0.4%

15651
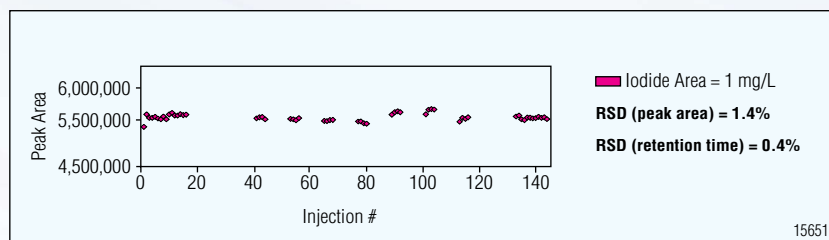
OnGuard II RP for the removal of fat from milk. Failure to remove fat prior to IC analysis leads to high column backpressures, loss in column capacity, and decreased retention times.
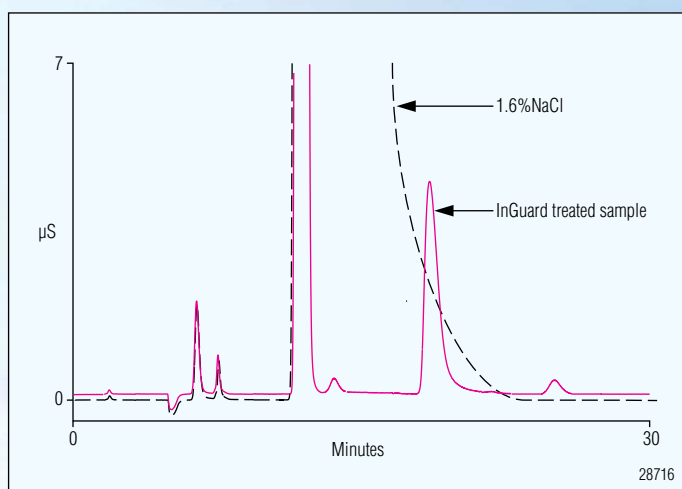
Matrix Elimination

Continued

In-line Matrix Ion Elimination
InGuard Cartridges

InGuard Cartridges provide high-throughput automated removal of matrix interferences encountered in many IC applications. Depending on the cartridge's location in the IC system, the autosampler (low pressure) or pump (high pressure) pushes the sample through the InGuard Cartridge. The number of samples that can be treated with an InGuard Cartridge depends on the matrix composition, concentration of compounds to be removed, and the sample injection volume. Chemistries operate optimally at flow rates between 0.5 and 2 mL/min. They are suitable for both standard-bore and microbore IC systems.

Treatment of a brine sample with InGuard Ag Cartridge removes interfering chloride ions to permit analysis of small amounts of other anions.

InGuard Cartridges for High-Throughput Automated Matrix Elimination*	
Resin	**Removes from Sample Matrices**
Ag	Halides
H	Cations and pH adjustment
Na	Cations
HRP	Hydrophobic species, azo- and cyano-containing species
Na/HRP	Metals (Na) plus organics (HRP)

*InGuard Kits are available that contain everything you need for high-throughput matrix elimination of organics, halides, sample pH reduction, and more.

IonPac NG-1 Columns

These columns are packed with highly hydrophobic cross-linked polystyrene resin beads for the removal of organic contaminants from samples, such as humic acids. The option of a two-column setup with a 10-port valve enables automatic resin cleaning of one trap column while the other is being used for sample preparation. NG-1 kits are available for organic removal for anion and cation analysis.
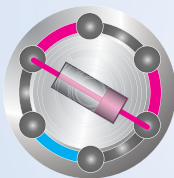
Concentrator Columns
## Capture and Concentrate Sample Ions from Large Sample Volumes

Challenge

Sample ions too dilute

Manual methods prone to errors and not reproducible

Time consuming

Solution—Dionex Sample Pre-Concentration

Preconcentrate ions, increase sensitivity

Fully automated, highly reproducible

Reduced errors and contamination

Saves time and money

IonPac Concentrator Columns can be used to preconcentrate ions as required for high-purity water analysis in, for example, the power and semiconductor industries. The columns retain ions from a measured volume of aqueous sample matrix, thereby concentrating the analyte species and lowering detection limits. Concentrator Columns enable the analysis of ions at µg/L (ppb) to ng/L (ppt) levels.
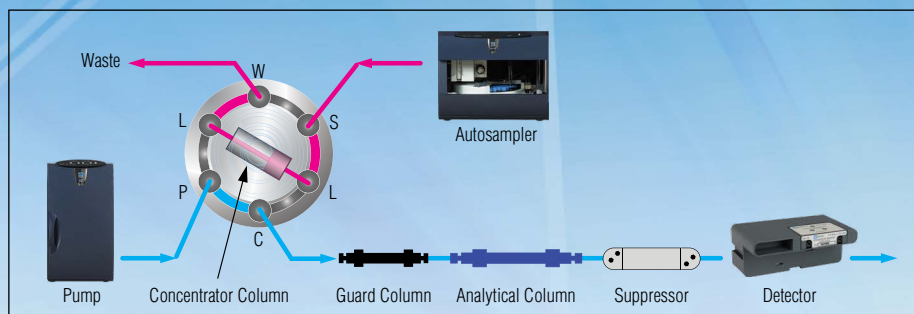
The concentrator column is typically placed on the injection valve in place of the sample loop, just before the analytical column. Sample is first collected on the concentrator column, which strips and retains the ions as the sample flows to waste. With a switch of the valve, eluent then flows through the column eluting the analytes to the analytical column.
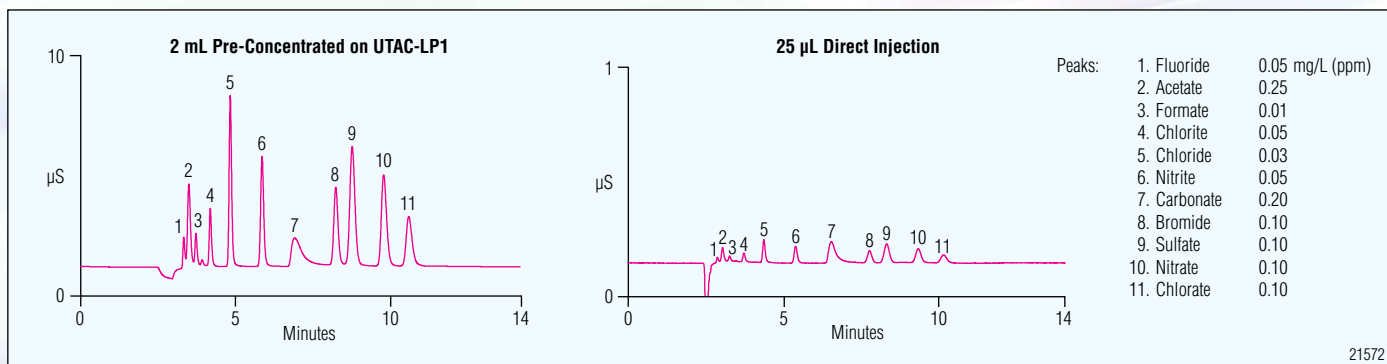
Anion IC and RFIC Concentrator Columns

Dionex offers 15 Anion Concentrator Columns with differing sizes and capacities for applications using hydroxide, carbonate/bicarbonate, or borate eluents.

Cation IC and RFIC Concentrator Columns

Cation Concentrator Columns are fully functionalized with carboxylic acid for methane sulfonic acid (MSA) and sulfuric acid eluents. One column has a sulfonic acid functional group for HCl/DAP•HCl eluents.

Transition Metal IC and RFIC Concentrator Columns

The Trace Metal Concentrator Column is a high-capacity, cation concentrator column used for coupling the MetPac CC-1 column to the IonPac CS5 or CS5A analytical columns when performing chelation IC.

Peaks:		
1. Fluoride	0.05 mg/L (ppm)
2. Acetate	0.25
3. Formate	0.01
4. Chlorite	0.05
5. Chloride	0.03
6. Nitrite	0.05
7. Carbonate	0.20
8. Bromide	0.10
9. Sulfate	0.10
10. Nitrate	0.10
11. Chlorate	0.10

Comparison of direct injection and preconcentration using the UTAC-LP1 for the determination of trace anions.

Trap Columns
Purify Eluent or Polish Samples

Challenge

Unwanted analytes interfering with separation

Contaminant buildup on guard and/or analytical column

Poor results

Solution—Dionex Trap Columns

Remove eluent and sample contamination and improve results

Easy to use

Prolong lifetime of consumables

Lower cost of ownership

IonPac Trap Columns prevent unwanted analytes from interfering with the separation of your analytes of interest. They contain high-capacity, low-efficiency, ion-exchange resins that strip and retain trace contaminants from the eluent and prevent their concentration on the guard and analytical columns.
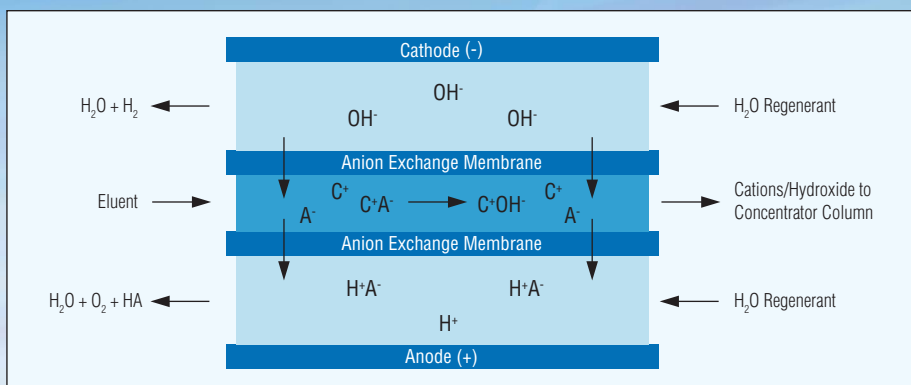
CR-CTC II neutralizes acidic samples, removes anion contaminants.

Trap Columns and RFIC Devices

These columns contain high-capacity resins designed to efficiently trap contaminants in eluents or samples. Back pressure is strictly controlled to provide the most useful devices available. Included are ATC-3 and ATC-HC anion trap devices, as well as the borate form ATC column. The CTC Cation Trap column provides high efficiency trapping of cations from samples, and in the hydrogen form it acts to neutralize basic samples.

Continuously Regenerated Trap Columns (CR-TC)

CR-TC technology provides continuously regenerated sample purification devices for maintenance-free operation. Both anion removal (CR-ATC) and cation removal (CR-CTC II) devices are available. CR-CTC II removes lithium and neutralizes hydroxide of borated water samples from nuclear power plants.

MFC-1 Metal-Free Trap Columns

The IonPac MFC-1 Metal-Free Column contains a special chelating resin. This column is placed in the eluent line prior to the injection valve to remove transition metals from high-pH eluents.

Cation Polisher Columns for Anion Analysis

These columns remove metallic contaminants and other cations such as calcium and magnesium from the sample stream while performing anion analysis, and include the:

- Cation Polisher Na$^+$ Column for anion analysis, which is a cation-exchange column in the sodium form and packed in a low-pressure format for autosampler operation.

- Cation Polisher H$^+$ Column for anion analysis, which is a higher-capacity version in the hydronium form.

RFIC-ESP

Dionex Reagent-Free Ion Chromatography Electrolytic Sample Preparation

Challenge

Ionic contamination in eluent stream

Matrix contamination

Cross-contamination for trace analysis

Method difficult to transfer between systems

Solution—RFIC-ESP Options

RFIC ESP

Automated ion removal from eluent stream

Easy to understand

Simple transfer to another system

Highly reproducible and precise

Reduced handling and human error

Electrolytic Devices for Automated Sample Pretreatment

Several Dionex IC systems integrate hardware and software to enable automated electrolytic removal of ions from an eluent stream using protocols that are easily understandable, simple to transfer to another system, and highly reproducible and precise.

- Highly reproducible results, instrument-to-instrument and lab-to-lab

- Reduced human error and inconsistencies

- Reduced manual operation with reduction in labor costs and sample processing time

- Reduced system complexity by eliminating sample loading pump

- Ultralow backgrounds from on-line electrolytic water purification with routine trace (ng/L) analysis

- Automated purification, concentration, and calibrations providing reduced labor costs and improved quality of results

AutoNeutralization Cartridges

The determination of anions in concentrated bases and the determination of cations in concentrated acids are important applications in industrial, health and safety, and environmental fields. Direct injection of concentrated acids or bases will overload the column resulting in poor chromatography and making ion quantification difficult or impossible. AutoNeutralization is a high-capacity electrolytic ion-exchange device that neutralizes basic or acidic samples prior to injection. A sample can be trapped in the AutoNeutralization device for as long as necessary to fully neutralize the sample matrix. The sample is then transferred to a concentrator column, and then to the analytical separation column.

Electrolytic Water Purifiers (EWPs)

EWP units are devices packed with various ion-exchange resins, and produce low flow rate streams for water of exceptional purity. These water streams can be used to displace water for loading samples onto concentrator columns, either directly or via another sample preparation device such as the CR-TC.

RFIC-ESP and AutoPrep

AutoPrep on the ICS-2100 Ion Chromatography System utilizes RFIC-ESP techniques that simplify the analysis of trace levels of anions or cations. The use of EWPs to transfer large sample volumes to the concentrator column virtually eliminates background contamination, allowing for low ng/L concentrations of ions to be conveniently analyzed. Proportional volume loops enable injection of standards of concentrations 1000 times higher than samples. Automated multilevel calibration is easily accomplished using Chromeleon software. Anion and cation determinations as low as 1 ng/L (ppt) are easily achievable.

RFIC-ESP Advantageous for:

- Trace analysis of ions in power plant and semiconductor ultrapure water samples

- Determination of ions in concentrated acids and bases after neutralization

- Cation elimination from power plant borated waters with continuous regeneration of the trap device

- In-line neutralization of dilute samples

- Continuous desalting of an ion-exchange eluent for coupling to mass spectrometers

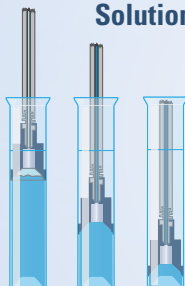- Continuous desalting of ion-exchange eluents before fraction collection

Sample Filtration

Remove Particulates to Protect Valuable Chromatography System Components

Challenge

Particulate blockage in system

High backpressure

Instrument downtime

Microbial contamination

Sample degradation

Solution—Dionex Filtration Devices

Eliminates column contamination and high system back pressures

Manual and automated methods

Reduces cost of ownership

Improves reliability and reproducibility of results

Off-line
Particle Filters

- Practical and easy to use

- Manually process samples with a 0.45 µm nylon membrane

The off-line particle filter has a sterile syringe Luer fitting for injecting a sample through a 0.45 µm nylon filter. This device is perfect for filtering a limited number of samples, such as collecting samples in the field to comply with EPA requirements to filter microbes.

In-line
AS-DV Autosampler Vial Filters

- Automated, robust, inexpensive, and easy to use

- Efficient filtration for highly contaminated samples

- No sample carryover

Used with the AS-DV high-performance autosampler, these 20 µm filtering devices sit atop the sample in either the 5 mL or 0.5 mL sample vial for automatic, high-capacity removal of particulates prior to injection. Because particulates settle to the bottom of the vial, the least contaminated sample is filtered first, thus preventing clogging and enhancing the filter's capacity. Individual filters are used for each sample, eliminating carryover concerns.

In-line Filters

- Housing made of PEEK™ prevents ion contamination

- Minimal dead volume (~1.3 µL) for use in various in-line locations

- High pressure rating of 5000 psi

- Several in-line filtering devices to match your application

Two types of in-line sample filters are available:

High-Capacity In-line Filter Cartridges

Used in the sample and/or eluent line for high-capacity filtration, this in-line filter cartridge has 35, 5, and 0.45 µm filters.

Low-Dead Volume In-line Filter Cartridges

These in-line filter cartridges have 0.5 µm PEEK filter frits and provide low dead-volume sample filtration and added system protection when low particulate matter is expected.

Kits Add Convenience for In-line Filtering

- A variety of in-line filter kits are available with everything needed to configure your IC system.

Specialized Systems

Combustion Ion Chromatography
Automated analysis of halogens and sulfur in petrochemicals, industrial materials, and consumer products

Combustion IC enables anion analysis in a variety of difficult-to-process samples such as ores, printed circuit boards, solders, plastics, and petroleum products, among others. The sample combustion process automates conversion of a solid, liquid, or gaseous sample to an aqueous solution that can be injected directly into an IC system for fully automated sample processing. Samples are efficiently oxidized by heating to 1000 °C in an air or oxygen stream for just a few minutes. Volatile combustion products are carried by the combustion gas stream into a sparger where they are trapped in water. The sample is then loaded onto the IC injection valve and analyzed.

The Combustion Ion Chromatography System comprises (from right to left) an autosampler, combustion unit, absorption unit, and ion chromatography system.

Air Sampling System
Automated analysis of volatile ionic constituents in air from emissions contributing to acid rain

Volatile ionic constituents in air result from emissions ultimately become acid rain. Particulate-borne ions can be dissolved in precipitation. The URG air sampling system easily conducts air monitoring on a continuous basis. The system separates and collects particulate material and volatiles, and captures the volatile constituents using an aqueous sparging system. Particulates, dissolved in water automatically, and samples are then automatically injected and analyzed by ion chromatography. The system can be operated for days without user intervention.

The URG Air Sampling System with integrated ion chromatographic analysis provide near real-time results.

Enjoy Industry-Leading Support

Customer Support Centers for Dionex products are located in the Americas, Europe, and Asia. State-of-the-art laboratories are equipped with the full line of Dionex consumables, instruments, and software capabilities. Support Centers provide accessible locations for advanced training and enhanced application development capabilities. Users can attend these laboratories to learn new skills in addressing challenging applications, receive training and support, and discover new, innovative IC and sample preparation solutions.

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