WATERS® ALLIANCE SYSTEMS FOR ION CHROMATOGRAPHY (IC) ANALYSIS

EXPERIENCE A HIGHER LEVEL OF PERFORMANCE FROM YOUR IC SYSTEM

For years, Waters® has played a pivotal role in advancing ion chromatography technology by providing traditional individual components such as pumps, injectors, detectors, columns, and software. With the innovative Alliance® System, Waters has taken ion chromatography to a higher level of performance and productivity. The heart of the Alliance System is the Waters 2695 Separations Module, a fully functionally-integrated solvent and sample management platform. Add to it the Waters 432 Conductivity Detector, Millennium®32 4.0 Chromatography Manager Software, and IC-Pak™ Columns, and you have a total system approach that addresses many of the concerns and sources of variability in ion chromatography.

Waters gives you a choice of single-column IC or chemical suppression IC by incorporating the Alltech ERIS™ 1000HP Autosuppressor, further enhancing the Alliance total system solution. The addition of a Waters 2487 Dual λ Absorbance Detector or 2996 Photodiode Array Detector provides you with the most capable, versatile chromatographic system for any analysis, for IC or traditional liquid chromatography.

The Alliance System offers an unprecedented level of flexibility, providing high sensitivity conductivity detection, ion retention selectivity using different eluent chemistries, high throughput multi-sample analysis, and choice of IC detection strategies without compromising ease-of-use or reliability.

alliance®

Waters

What are the main concerns that can interfere with the performance of your IC analysis? They are the complications that cause you to work harder and longer just to keep up with the sample load. They decrease your productivity, increase the cost of ownership and erode the confidence you need in your results.

Quantitation Reliability

Linearity. Accuracy. Precision. Reproducibility. These qualities are essential to you and your customers' analyses. Nothing less than the most reliable system will do to meet your most demanding analytical requirements.

Choice of Ion Analysis Technologies

When it comes to ion analysis, there are several options to consider: Ion Chromatography (Single-Column Ion Chromatography or Chemical Suppression Technology). The choice is yours.

Conductivity Baseline Noise

Baseline noise has always been a major concern, particularly when high sensitivity is required. There are several techniques used to decrease baseline noise, but none of them address the true reason for conductivity baseline noise: eluent flow stability.

System Downtime

In today's time-pressed lab environment, there is simply no room for an unreliable instrument. Precious time and money are lost and productivity is compromised.

Sample Backlog

The workload is high. The last thing you need is a sample backlog, but sometimes it's inevitable, or so it seems. You can only get so many samples run in a day. It would be great if you could run samples overnight, but something might go wrong with the equipment. With nobody there to monitor it, your work could be compromised.

Data Retrieval: Strategy or Struggle?

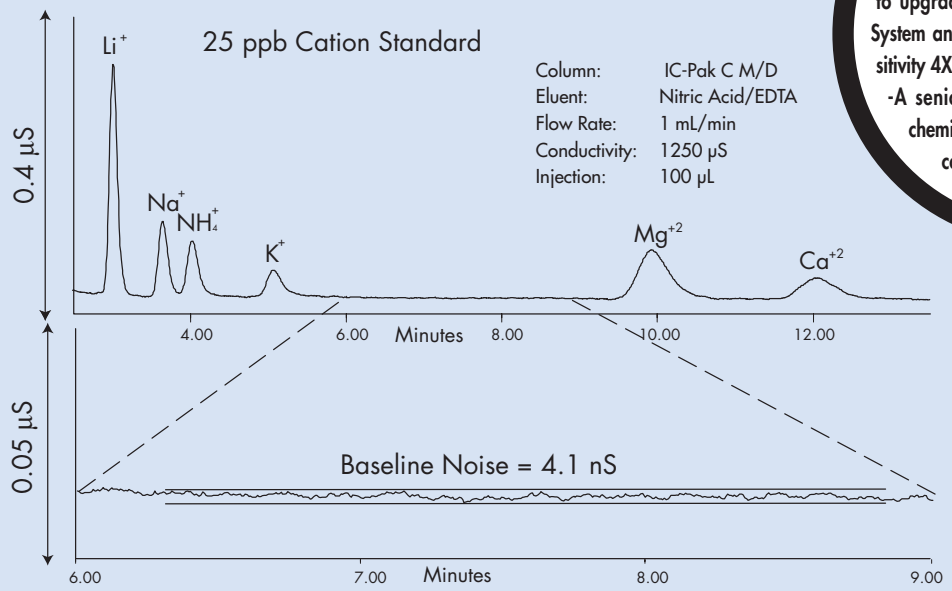
Be honest. No rhyme or reason behind your data retrieval strategy? Do you worry that a regulatory agency will audit your lab? Are you concerned about how to manage your DQOs (Data Quality Objectives) and MQOs (Method Quality Objectives) or compliance-ready data for EPA Electronic Data Reporting and Electronic Records & Electronic Signatures Rule (21 CFR Part 11)?

MAKES ROUTINE WORK OF THE MOST DEMANDING IC APPLICATIONS

The analysis of alkali and alkaline earth cations and amines is a demanding application for any IC system. Using the Alliance system along with the Waters IC-Pak C M/D Column makes this analysis routine[1]. Figure 1 demonstrates high sensitivity in the presence of high background conductivity. The sensitivity calculated at 3x the signal-to-noise (S/N) gives method detection limits below 10 ppb[2].

"I was using an older Waters system to do single-column IC analyses. When it came time to upgrade, I chose the Waters Alliance System and immediately increased my sensitivity 4X running the same analyses."
-A senior chemist with an advanced chemical specialties and adhesives company

FIGURE 1: ALLIANCE SYSTEM FOR ION CHROMATOGRAPHY CATION ANALYSIS



Low part per billion detection of cations can be attained without the need for chemical suppression.

HIGH SENSITIVITY AT HIGH BACKGROUND CONDUCTIVITY

The Waters 432 Conductivity Detector was engineered with single-column IC in mind. In this mode, the background conductivity is the natural conductivity of the eluent of choice. The 432 detector uses a patented 5-electrode design that is significantly different than the traditional 2-electrode design of most IC conductivity detectors. This design monitors the eluent conductivity, regardless of its magnitude, and assigns it a value of zero. The change in conductivity due to the analyte, relative to zero, is then the signal response of the analyte. Thus, a small differential in conductivity can be accurately determined, regardless of the background conductivity. This 5-electrode design eliminates the capacitance and Faradaic effects inherent with the traditional 2-electrode design[3]. The result is a quiet baseline even at high eluent background conductivity.

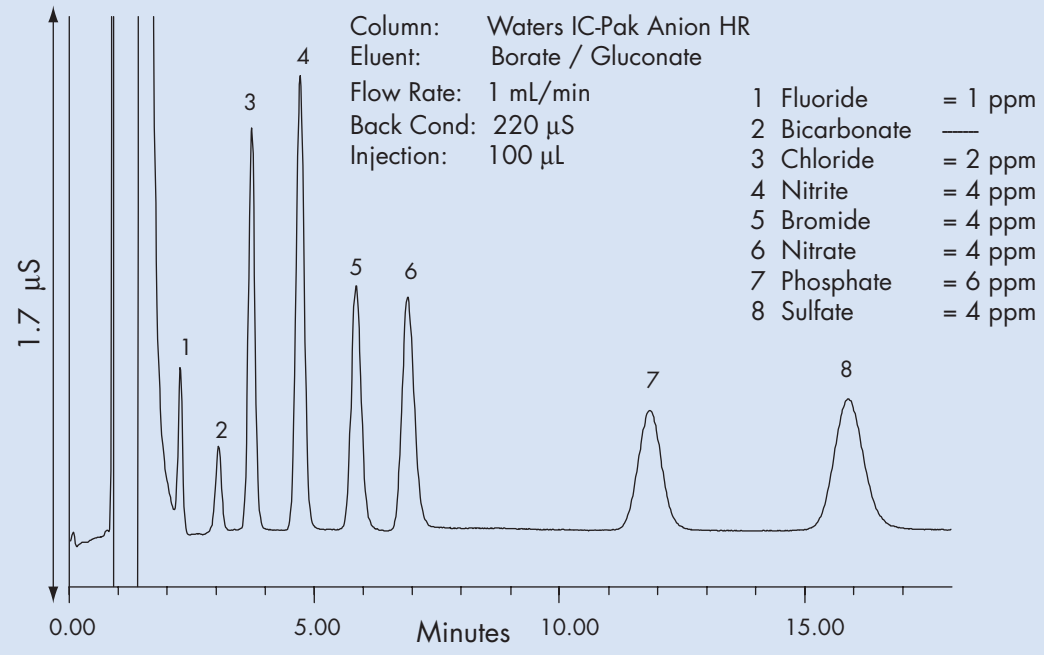
All conductance measurements are affected by changes in solution temperature. Generally there is approximately a 2% change in conductivity per °C. The 432 detector incorporates a direct temperature control heater unit in the cell compartment, ensuring a stable conductivity baseline over the course of the day. This control heater can be set between 30° C and 65° C to assure constant temperature conductivity measurements in even the most demanding environments.

METHOD PERFORMANCE FOR IC APPLICATIONS

The performance of an analytical method is a function of IC system performance, data processing, as well as the method itself. This is the ultimate measure of ion analysis method performance. The method for the most common IC application: the analysis of common inorganic anions (Fluoride, Chloride, Bromide, Nitrite, Nitrate, o-Phosphate, and Sulfate in aqueous matrices) as shown in Figure 2, was validated with the Waters Alliance System for Ion Chromatography. This Alliance System consists of Waters 2695 Seperations Module, 432 Conductivity Detector, with the IC-Pak Anion HR Column and Borate/Gluconate Eluent. A series of nine certified standards in the concentration range of 0.1 - 50 ppm were prepared, and method performance evaluated using ASTM D-2777-98[4] procedures.

This analytical protocol was fully automated via Millennium[32] Software, which provides comprehensive documentation of the analysis. With Millennium[32] Software, it's never a struggle to determine your quality assurance and quality control data trends over time and establish your laboratory's DQOs and MQOs.

FIGURE 2: ALLIANCE SYSTEM FOR ION CHROMATOGRAPHY ANION ANALYSIS



Excellent resolution and sensitivity can be achieved for the most common IC application.

SUPERIOR CONDUCTIVITY RESPONSE LINEARITY FOR SINGLE-POINT CALIBRATION

The series of eight certified standards used for linearity, shown in Figures 3A-3C and 4, were formulated to represent "real" sample solutions with varying concentrations of each anion between 0.5 ppm and 50 ppm, rather than having the anions all at low or all at high concentrations. A more rigorous treatment to determine linearity is the ASTM Response Factor Linearity, which is shown in Figure 4. The response factor, defined as response per unit of concentration, remains within the limits of ASTM linearity for all anions tested*. Other experiments extend the linearity range to over 200 ppm.

*With the exception of 0.5 ppm of PO_4 (phosphate)

FIGURES 3A-3C: LINEARITY USING A BORATE/GLUCONATE ELUENT

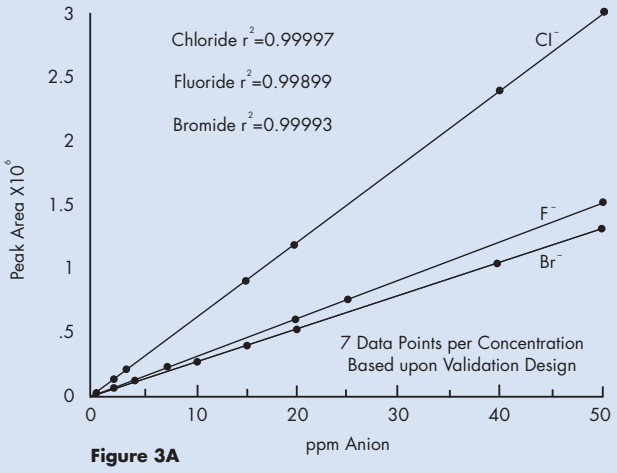


Figure 3A

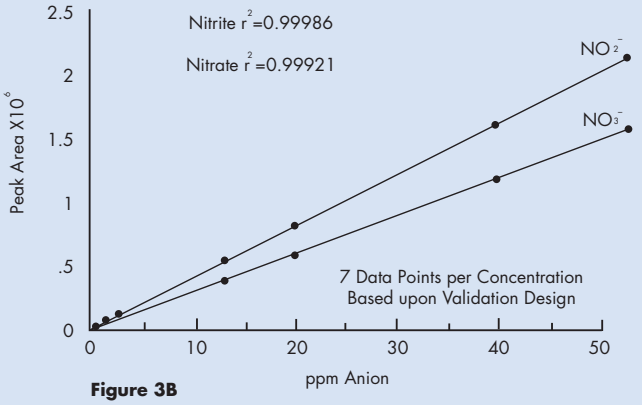


Figure 3B

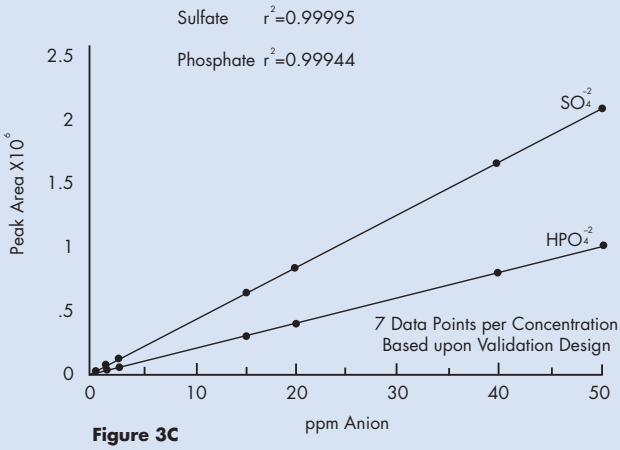
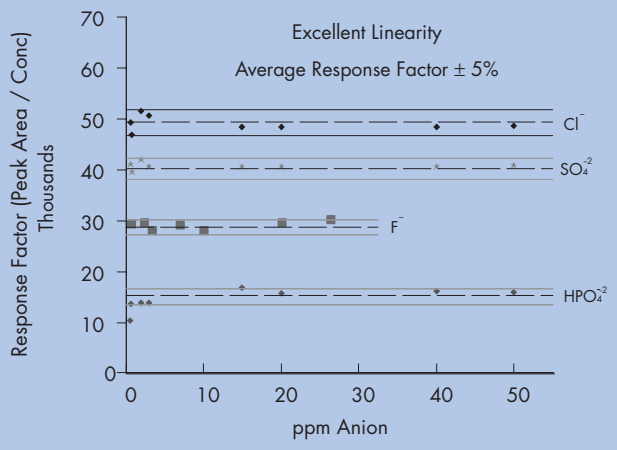


Figure 3C

Figures 3A-3C demonstrate the linearity of direct conductivity response greater than 0.999.

FIGURE 4: ASTM LINEARITY



Since conductivity response is linear with the 432 Conductivity Detector, you can use single-point calibration, saving you time and increasing your productivity.

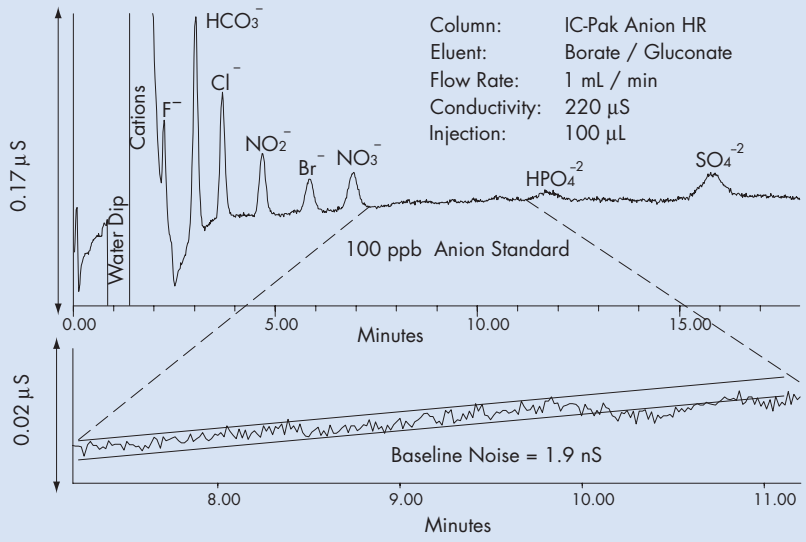
The detection limits for this method were determined using EPA protocols [EPA 40 CFR Ch.1 (July 1, 1992 Edition), Pt. 136, App. B, p. 565-567] with 7 replicates of a 100 µL injection of the ninth 100 ppb mixed anion standard shown in Figure 5. The conductivity baseline noise of 1.9 nS over a three-minute time interval provides for sub 100-ppb detection limits at high detector attenuation of less than 0.1 µS full scale[2]. This degree of performance is routine for the Waters Alliance System.

ACCURATE, REPRODUCIBLE AND RELIABLE RESULTS

Today's analytical procedures often include method quality control to verify the accuracy of results for unknown samples. Accuracy is documented by analyzing performance evaluation standards or QC standards from an outside source as an unknown sample.

A certified, mixed anion performance evaluation standard was purchased from an analytical standards manufacturer, analyzed and quantitated using the single-column anion method linearity shown in Figures 3A through 3C. The results, shown in Table 1, reveal excellent agreement with the true value concentrations. The 95% Confidence Limits were derived from reported data from numerous laboratories using conventional EPA-approved wet chemistry and IC methods. These data unequivocally demonstrate that the Waters Alliance System for IC anion analysis yields accurate, reproducible, and reliable results you can report with complete confidence.

FIGURE 5: ANION METHOD DETECTION LIMITS



The exceptional flow stability of the 2695 Separations Module reduces system noise, providing quieter baselines and resulting in higher sensitivity detection.

TABLE 1. SINGLE-COLUMN IC METHOD ACCURACY

	Analyte	F^-	Cl^-	NO_2^-	NO_3^-	HPO_4^{-2}	SO_4^{-2}
Performance Evaluation Standard	True Value in ppm	2.69	43.00	1.77	15.37	6.29	37.20
Results Using Several Offical EPA Methods	95% Confidence Limits	2.24 to 3.25	37.15 to 49.25	1.50 to 2.02	13.21 to 17.64	5.95 to 6.81	32.61 to 41.39
Results Using Alliance System for IC with IC-Pak Anion HR & B/G Eluent	Average IC Result 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 / True Value	0.978	1.020	1.090	0.979	1.029	0.995

An IC / True Value equal to 1.000 indicates exact agreement.

The Alliance System for Ion Analysis demonstrates excellent agreement with the True Value.

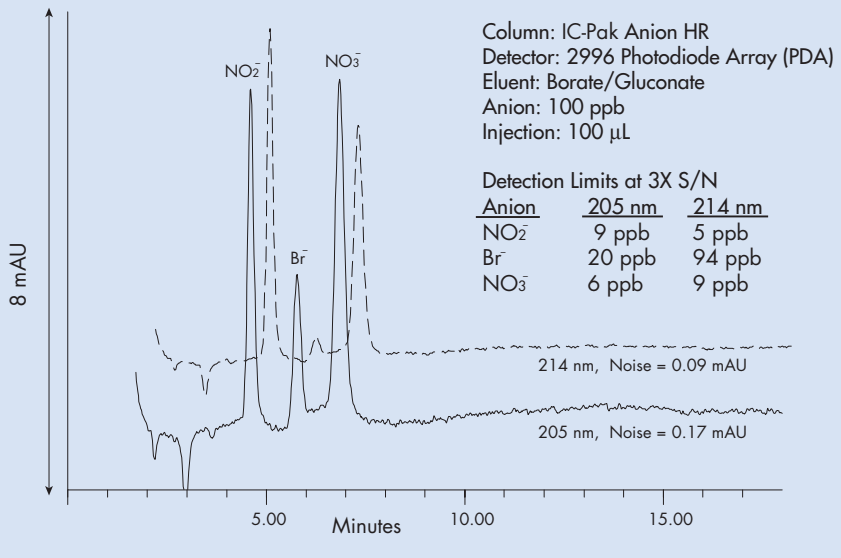
"As a leading provider of calibration standards and reference materials to the environmental industry, accuracy and precision are essential to our daily analyses. We found that the Waters Alliance System for Ion Chromatography is the only system capable of meeting our customers' exacting needs."

-Tom Coyner, President, Analytical Product Group, Inc., an analytical standards manufacturer

DETECTION SELECTIVITY AND SENSITIVITY

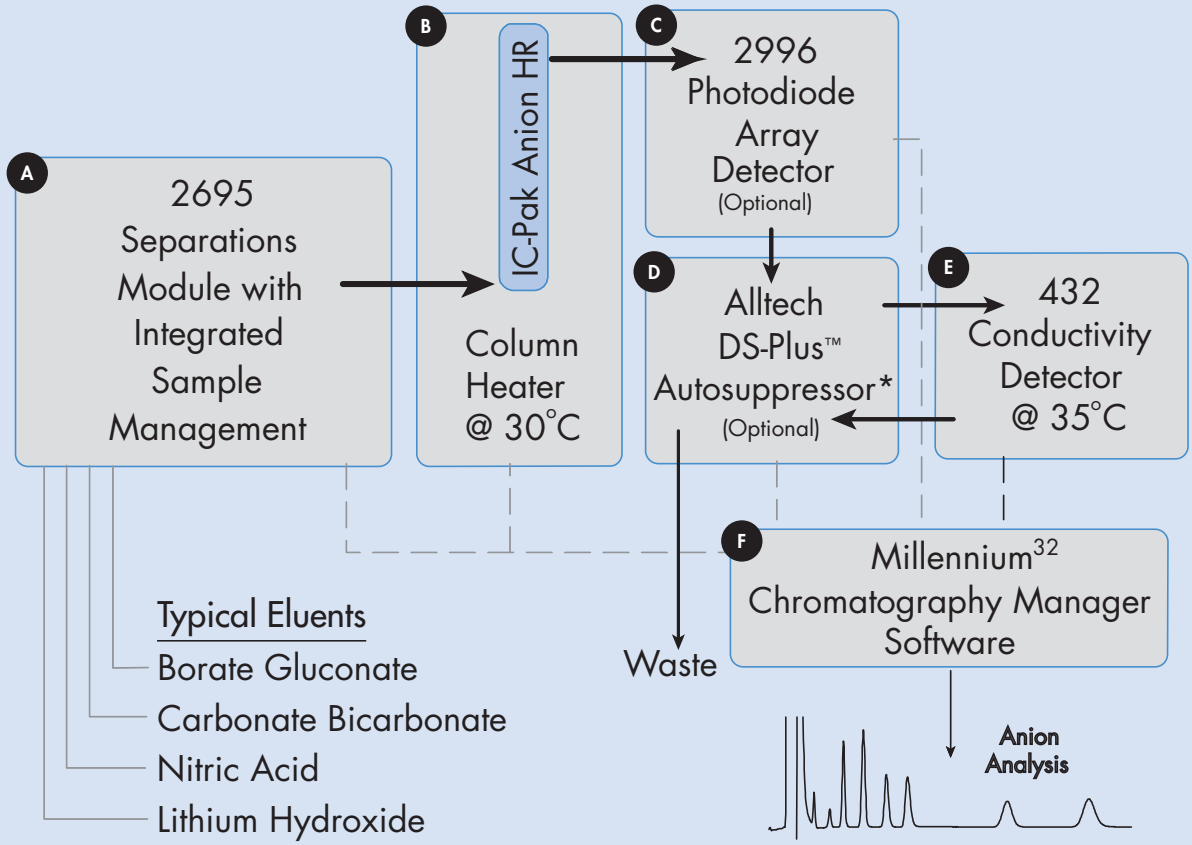
Conductivity detection is not the only means of detecting inorganic and organic ions. Many anions, such as Bromide, Iodide, Nitrite, Nitrate, Sulfite, Thiosulfate, Thiocyanate and organic acids, are UV active at wavelengths below 215 nm. The UV response of these anions may be greater than conductivity. This detection strategy is the basis of the EPA Method B-1011, the determination of nitrite/nitrate in water using single-column IC[5]. The chromatogram in Figure 6 was obtained simultaneously with the chromatogram in Figure 5, using a single injection, by connecting the Waters 2996 Photodiode Array Detector in series with the Waters 432 Conductivity Detector. This combination of detectors provides a wealth of analytical information about an analyte and is invaluable for identifying unknown analytes.

FIGURE 6: DIRECT UV ANION DETECTION SENSITIVITY



Comparison of anions standard at two different wavelengths.

FIGURE 7: SYSTEM CONFIGURATION FOR THE ALLIANCE SYSTEM FOR IC ANALYSIS



*Not required for single-column IC

A 2695 Separations Module
- Innovative Design
- 120-Sample Capacity
- Low Dispersion
- Temperature Control
- Transferability of Methods
- Real-Time Running Log
- Compact, Small Footprint
- No-Tools Maintenance

B IC-Pak Anion HR Column
- For the high resolution analysis of inorganic anions
- 7µm, polymethacrylate-based anion exchange resin
- Easy changeover to another eluent type
- Enables multiple applications on the same column

C 2996 Photodiode Array Detector
- Enhanced absorbance detection
- Advanced TaperBeam™ optics
- Unmatched linearity and sensitivity
- IC applications versatility

D Alltech DS-Plus Autosuppressor
- Chemical suppression+Alliance System = EPA Method 300.1
- Removes cations from eluents and sample
- Increased analyte anion conductivity response
- Meets most challenging sensitivity needs

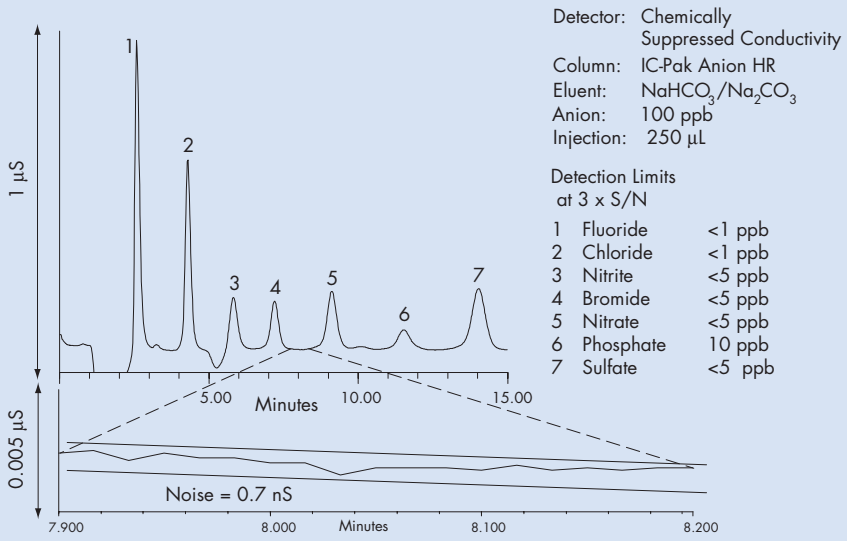
E 432 Conductivity Detector
- For single-column IC or chemical suppression
- Patented five-electrode flow cell
- Precise temperature control
- Reduced volume design

F Millennium^{32} Software
- An easy to learn and use interface
- On-line help
- Fully scalable architecture
- Unique, integrated database
- Upgradable to PDA and MS

ALLIANCE SYSTEM + CHEMICAL SUPPRESSION TECHNOLOGY = EPA METHOD 300.1

Chemical suppression technology can be added to the Alliance System to meet the requirements of EPA Method 300.1, the determination of inorganic anions by ion chromatography. The Alltech Autosuppressor removes cations from the $NaHCO_3/Na_2CO_3$ or NaOH eluents and from the sample, and increases the conductivity response of the analyte anions. The EPA has recognized this approach as equivalent methodology to EPA 300.1, assuring compliance for regulatory mandates.

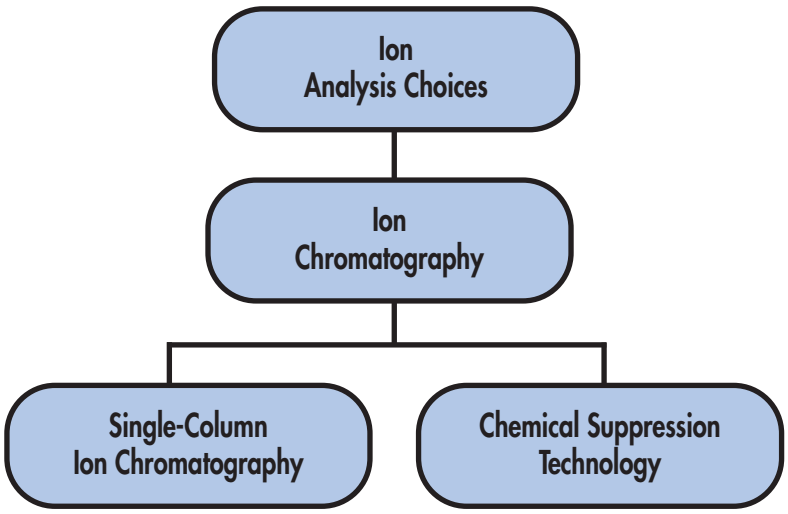
FIGURE 8: ALLIANCE SYSTEM FOR IC ANALYSIS WITH CHEMICAL SUPPRESSION TECHNOLOGY



Single ppb anion detection limits can be obtained to meet the most challenging sensitivity needs.

WATERS OFFERS REAL CHOICES IN ION ANALYSIS TECHNOLOGY

There are several options to consider when choosing technology for ion analysis. For ion chromatography, the Waters Alliance System is superior to other traditional systems in detection strategy, signal-to-noise sensitivity, reproducibility, and system reliability, making it the most capable and versatile system for ion chromatography analysis in the industry.



ADVANCED FLUIDICS DESIGN ENSURES FLOW PRECISION AND ACCURACY PLUS HIGH SENSITIVITY CONDUCTIVITY DETECTION

High sensitivity conductivity detection is an analyte signal-to-baseline noise relationship. The performance of the solvent management system and the conductivity detector are inexorably linked. In the past, baseline noise was decreased by minimizing the pressure drop between piston strokes in a traditional single-piston or dual reciprocating pump via mechanical pulse dampers or software flow feedback control. While baseline noise was decreased with these techniques, they did not address the true reason for conductivity baseline noise: eluent flow stability. The innovative design of the Waters 2695 Separations Module does just that.

The solvent management system of the Waters 2695 Separations Module is the first to incorporate a digitally controlled, independently driven dual piston mechanism and serial flow path to ensure pulse-free eluent delivery that compensates for changes in eluent viscosity. Eluent degassing is performed in-line in order to purge the eluent of any dissolved gases, minimizing baseline noise. The vacuum degasser enhances the flow precision and accuracy for both isocratic and gradient applications. Pistons are continually washed to eliminate buffer buildup, extending the lifetime of the seals and minimizing seal leaks that can detract from flow precision. Waters Alliance Systems have set a new standard of performance – a flow precision specification of ≤ 0.075% RSD or ≤ 0.02 min. standard deviation.

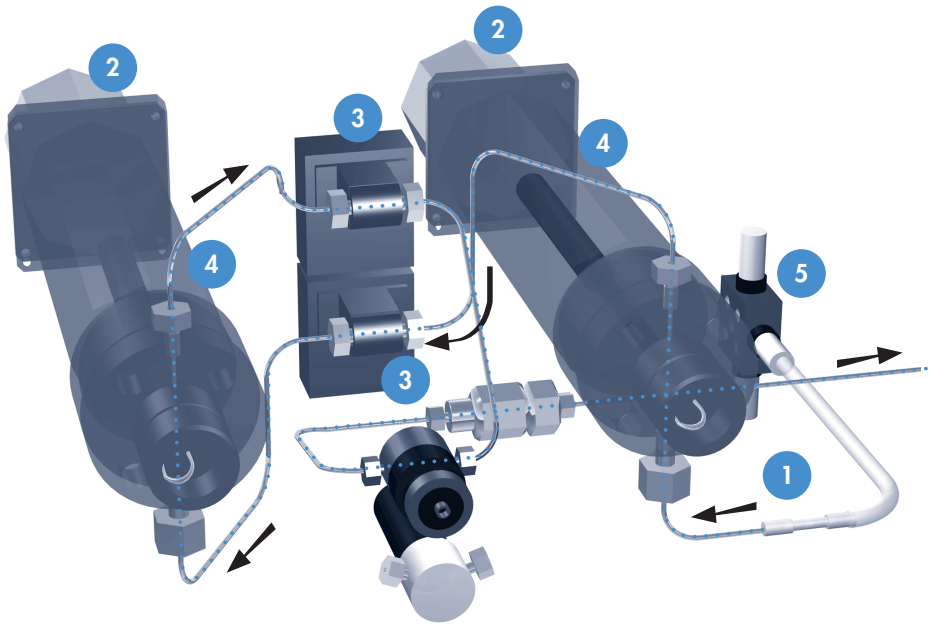


FIGURE 9: The design of the Waters 2695 Separations Module incorporates independently controlled pistons, serial flow, and dual pressure transducer feedback to provide smooth, precise isocratic and gradient operation.

A CLOSER LOOK

Figure 9

1. SERIAL FLOW
- Reduced number of check valves (2)
- First-in, first-out solvent delivery
- Solvent blending in the piston heads

2. INDEPENDENT PISTON DRIVE
- Dual drive motors
- Innovative linear piston motion control
- Precisely monitored and coordinated pistons

3. DUAL PRESSURE TRANSDUCERS
- Matched and dynamically calibrated pressure transducers
- Two-point performance monitoring
- Point of delivery flow optimization

4. VARIABLE PISTON VOLUME
- Piston volume balanced with flow rate
- Coordinated mixing with flow rate
- Automatically controlled for best performance

5. GRADIENT PROPORTIONING VALVE (GPV)
- Low volume design
- Solvent proportioning matched to piston stroke volume
- Synchronized through feedback control software

SINGLE SYSTEM CONTROL

Waters Alliance System for Ion Chromatography is designed to interface with Waters Millennium[32] Software, which enables system control of operating parameters and analytical results to be managed by individual PCs or full, enterprise-wide client/server networks. The integrated Oracle® database links results with conditions and makes audits virtually painless. This software delivers both powerful and flexible functionality in a single package.

COLUMNS AND CHEMISTRIES

Waters offers several multi-application columns for anion, cation and weak organic acids analysis. The Waters IC-Pak Anion HR Column is a 7 µm, polymethacrylate-based anion exchange resin in the borate/gluconate form. Changing to another eluent type, such as hydroxide or carbonate/bicarbonate, is as easy as re-equilibrating the column. This allows for multi-applications on the same column. Also available are the IC-PaK Anion and IC-PaK Anion High Capacity Columns.

The Waters IC-Pak C M/D Column is a 5 µm, silica-base (coated with a polybutadiene/maleic acid copolymer) cation exchange column designed for the analysis of alkali and alkaline earth cations. Its pH range and reverse phase characteristics aid in the separation of low molecular amines.

Weak organic acids, with pKa greater than 3, such as formate and acetate, are easily resolved using the Waters Ion Exclusion Column, a high capacity, sulfonated resin.

For the most common ion analysis problems, Waters has the appropriate column and methods.

WATERS CONNECTIONS®-YOUR LINK TO WORLD-RENOWNED SERVICE AND SUPPORT

Waters Connections provides the solutions you need to maintain maximum uptime with your Waters systems.

- Analytical Instrumentation and Software Services include Total Assurance Plans that extend and enhance the original warranty you receive when you buy a Waters product. These plans minimize the level of insurance investment and deliver the value you need to avoid costly and time-consuming system downtime.
- Connections Compliance Services provide you with timely and cost-efficient solutions to your regulatory compliance problems. You can use our Compliance Services to effectively train and certify personnel in equipment and regulatory requirements for cGMP/GLP compliance, significantly reducing operating costs.
- Connections University is the center of our Educational Services, providing extensive HPLC and LC/MS training and education at your site, at our corporate headquarters or at our local offices around the world.
- Representatives of our global Customer Assurance Organization—trained and certified in all our products and current in HPLC and LC/MC applications—are available in person, on the phone, via FAX or at www.waters.com to answer questions and provide you with around-the-clock support and information services.

References

[1]Krol, Jim, et al., "Ion Chromatography of Alkylamines and Alkanolamines Using Conductivity Detection," J. Chrom. (626): pp. 165-170 (1992).

[2]Krol, Benvenuti, and Romano, Waters Ion Analysis Methods Book, April 1998. (Waters Literature Code WT139)

[3]Haddad and Jackson, "Ion Chromatography: Principles and Applications", J. of Chromatography Library (46): pp. 255-260 (1990).

[4]Annual Book of ASTM Standards, Vol. 11.01(1999)

[5]Heckenburg, Alden, Wildman, Krol, Romano, Jackson, Jandik and Jones, Waters Innovative Methods for IC Analysis, 1989

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