

FEBRUARY 2016

ADVANCING ION CHROMATOGRAPHY WITH HIGH PRESSURE



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INTRODUCTION

High pressures and columns with small particle sizes have been used in high performance liquid chromatography (HPLC) for a long time, and HPLC practitioners know that working with small particle sizes and high pressures can provide significant advantages. In ion chromatography (IC), however, the move to higher pressures has taken much longer, but is finally ready to take off, as the articles in this e-book make clear.

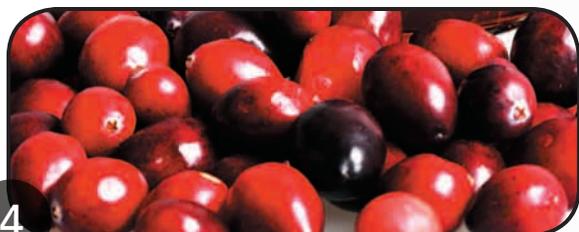
First, Mike Doyle explains the advantages of small column particle size and the benefits of high-pressure IC, and illustrates these benefits with examples from the analysis of food and beverages, such as cranberry juice, orange juice, and beer.

In the next piece, Peter Bodsky outlines the hardware considerations involved in high-pressure IC. He then provides example of the advantages of this technique—and particularly the speed it can provide—in environmental applications, such as the analysis of drinking water and treated wastewater.

We wrap up the e-book with an interview with Chris Pohl. He puts the trend to high-pressure IC in context, and discusses the accompanying move to high-capacity IC columns. He also explains the features that Thermo Fisher Scientific has included in its new high-pressure IC instrument that make it easier to use.

We hope you find this new e-book informative and helpful in your work.





4

Food & Beverage Applications

High-Pressure Ion Chromatography Applications in Food and Beverage Analysis

Mike Doyle



13

Environmental Applications

Fast Ion Chromatography for High Throughput with Environmental Samples

Peter Bodsky



20

Trends

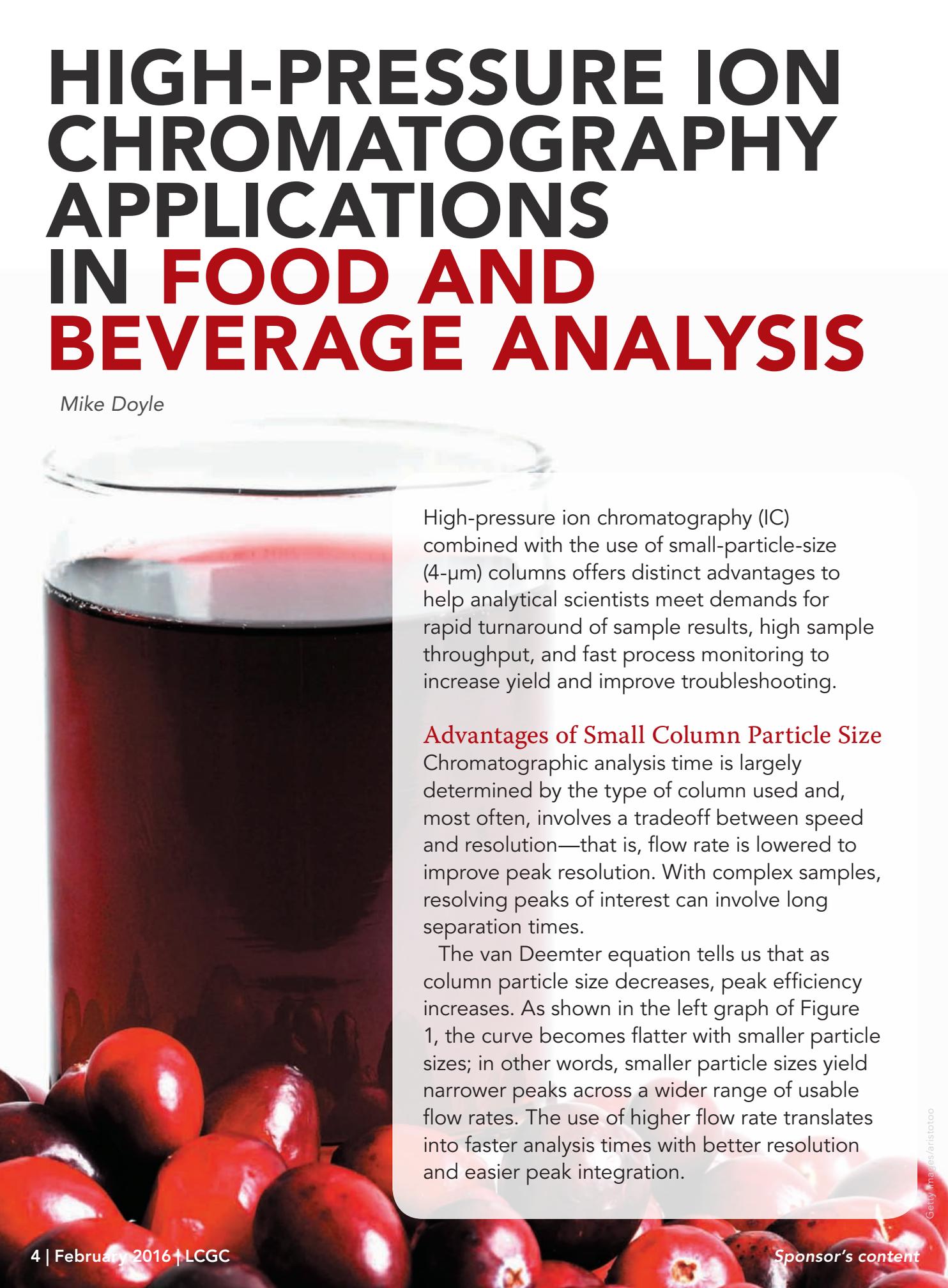
Trends in Ion Chromatography: User-Friendly Instruments, Higher-Capacity Columns, and the Move to High Pressure

An interview with Chris Pohl



HIGH-PRESSURE ION CHROMATOGRAPHY APPLICATIONS IN FOOD AND BEVERAGE ANALYSIS

Mike Doyle



High-pressure ion chromatography (IC) combined with the use of small-particle-size (4- μm) columns offers distinct advantages to help analytical scientists meet demands for rapid turnaround of sample results, high sample throughput, and fast process monitoring to increase yield and improve troubleshooting.

Advantages of Small Column Particle Size

Chromatographic analysis time is largely determined by the type of column used and, most often, involves a tradeoff between speed and resolution—that is, flow rate is lowered to improve peak resolution. With complex samples, resolving peaks of interest can involve long separation times.

The van Deemter equation tells us that as column particle size decreases, peak efficiency increases. As shown in the left graph of Figure 1, the curve becomes flatter with smaller particle sizes; in other words, smaller particle sizes yield narrower peaks across a wider range of usable flow rates. The use of higher flow rate translates into faster analysis times with better resolution and easier peak integration.

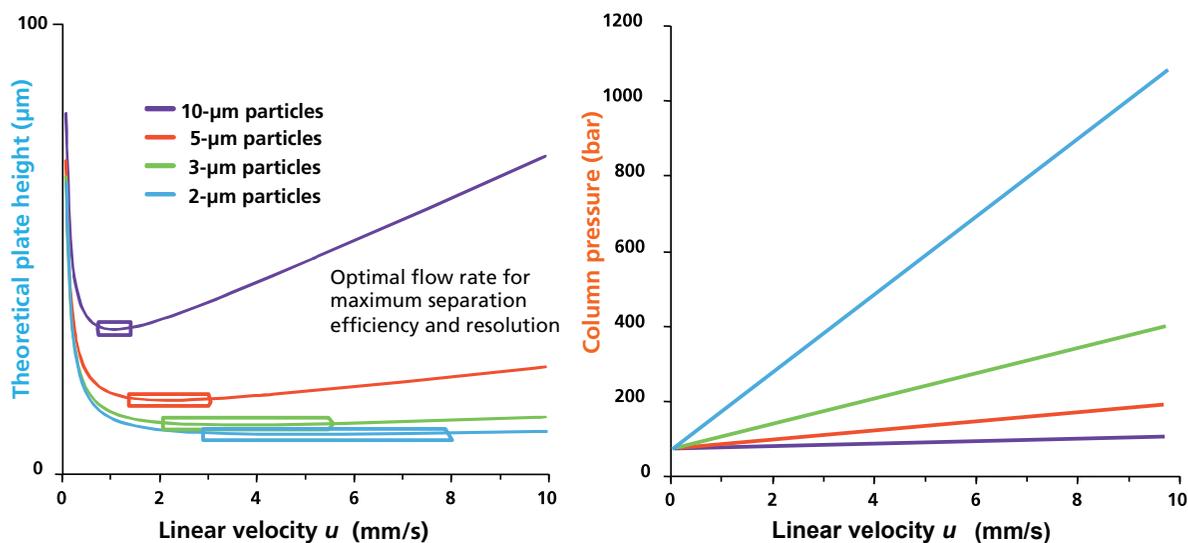


Figure 1: Influence of the particle diameter on efficiency and pressure..

Reducing the particle size, however, dramatically increases column back pressure (Figure 1, right), which presents a challenge in the design of chromatography systems that can operate reliably at higher pressures.

Benefits of High-Pressure Ion Chromatography

High-pressure ion chromatography uses smaller-particle-size resin columns on systems that support high operating pressures and thus facilitates faster runs without sacrificing resolution. Working with small-particle-size resins in various column lengths helps optimize speed and resolution relative to column back pressure.

A 4- μm , 150-mm column, for example,

allows for higher flow rates and faster analysis times at a lower back pressure than a 4- μm , 250-mm column. Using the shorter column will improve resolution for less complex samples, while providing a fast analysis. The longer column will help maximize resolution for more complex samples, but the maximum usable flow rate, and therefore analysis speed, is limited by high back pressure.

Thermo Fisher Scientific has developed a range of both standard and 4- μm high-pressure IC columns in a variety of dimensions to allow analysts to tailor the column to the application by finding the right balance between speed and resolution. In this article, we examine a series of high-pressure IC applications to food and beverage analyses, where high

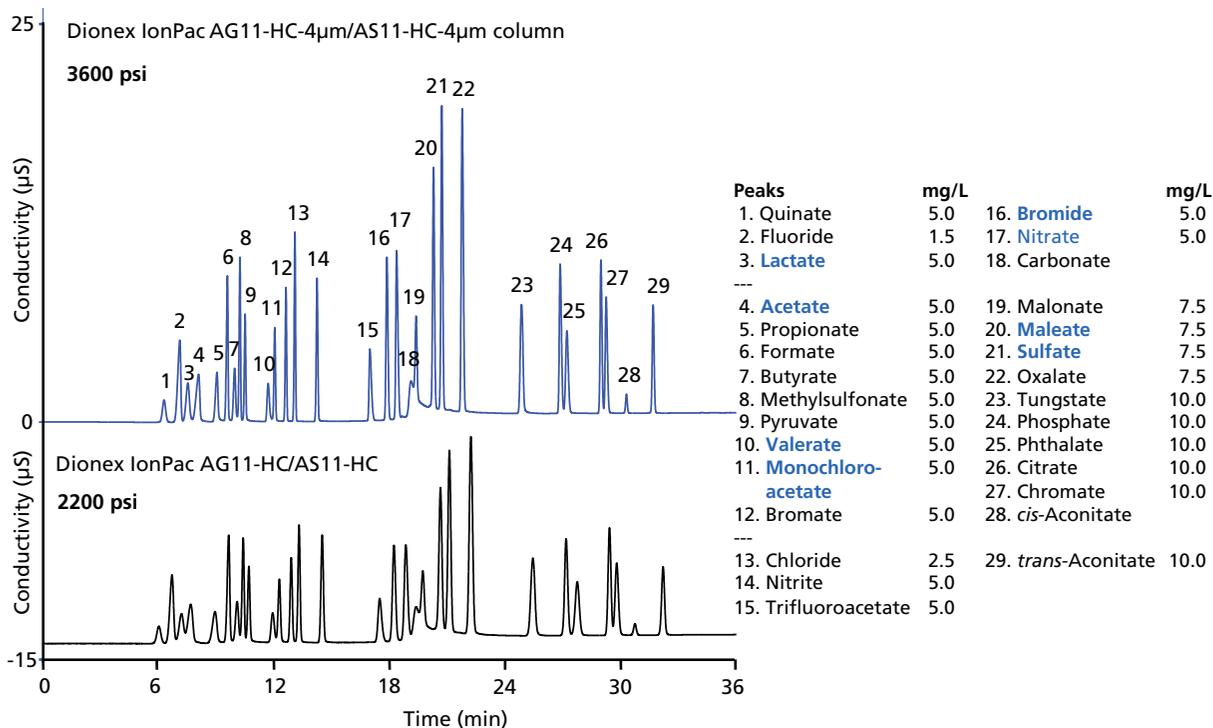


Figure 2: Separations obtained using a Dionex IonPac AS11-HC column (bottom) and a 4- μ m Dionex IonPac AS11-HC column (top). Eluent source: Thermo Scientific Dionex EGC-KOH eluent generator cartridge (capillary); gradient (potassium hydroxide): 1 mM from 0 to 5 min, 1–15 mM from 5 to 14 min, 15–30 mM from 14 to 23 min, 30–60 mM from 23 to 31 min; flow rate: 15 μ L/min; injection volume: 0.40 μ L; temperature: 30 °C; detection: suppressed conductivity, Thermo Scientific Dionex ACES 300 anion capillary electrolytic suppressor, recycle mode.

resolving power in relatively fast runs is a valuable tool.

Examples of High-Pressure IC Applications in Food and Beverage Analysis

The Thermo Scientific Dionex IonPac AS11-HC (HC = high capacity) column is a workhorse column that is useful in profiling applications. Figure 2 compares an orange juice separation using the standard packing material in the AS11-HC with a separation made using a similar

column with 4- μ m particles. The 4- μ m column yields better resolution and peak efficiency improvement for many of the peaks by as much as 50–60%.

Figure 3 illustrates a real-life example of identification of spoilage in orange juice. A 250-mm column and a flow rate of 15 μ L were used, so pressure is high at 3600 psi, but resolution is achieved between early eluted inorganic and organic peaks such as lactate and acetate, which are important in determining sample freshness.

The separations in Figure 4 are

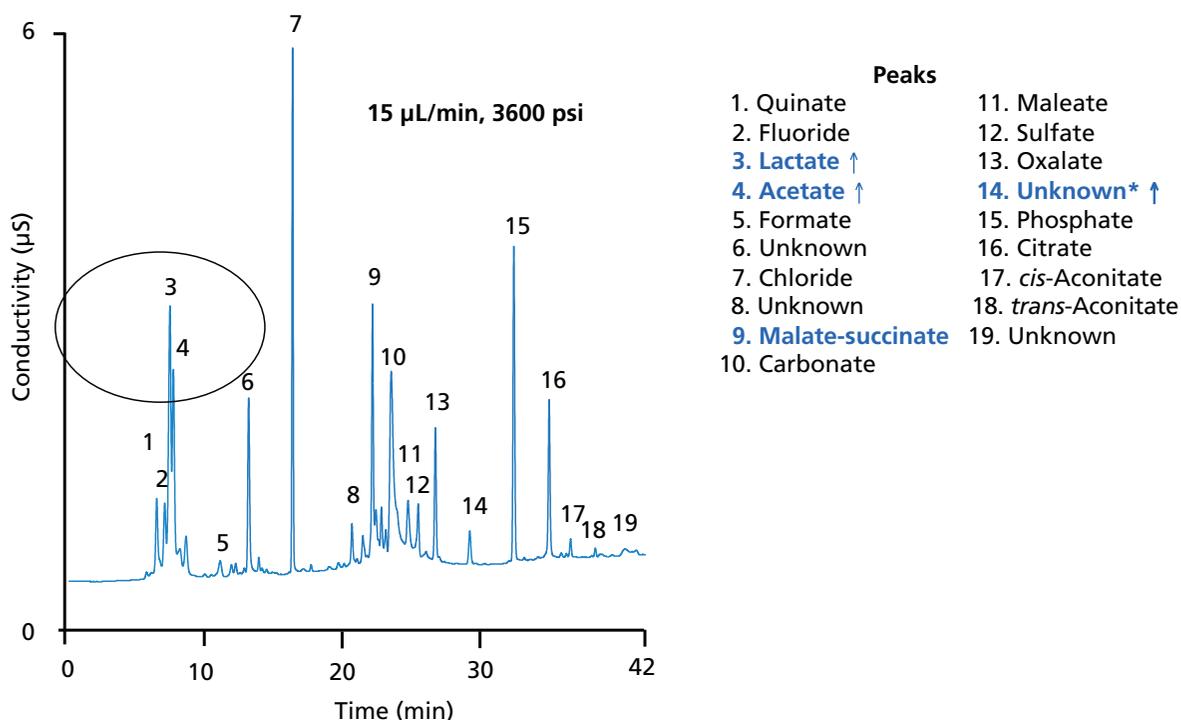


Figure 3: Spoilage identification in beverages. Column: 250 mm X 0.4 mm Dionex IonPac AS11-HC-4µm; eluent source: Dionex EGC-KOH cartridge (capillary); gradient (potassium hydroxide): 1 mM from 0 to 8 min, 1–30 mM from 8 to 28 min, 30–60 mM from 28 to 38 min, 60 mM from 38 to 42 min; flow rate: 15 µL/min; injection volume: 0.4 µL; column temperature: 30 °C; detection: suppressed conductivity, Dionex ACES 300 suppressor, recycle mode; sample preparation: 1:40 dilution with deionized water.

high-resolution fingerprints showing differences in organic profiles of cranberry and apple juice obtained by high-pressure IC. Such fingerprints are highly useful in sample identification and in determining the differences between juices.

In the production of beer, it's important to monitor inorganic and organic content in the formulation and fermentation process. Figure 5 illustrates three beer samples run by high-pressure IC. The carbonate peak is high, which is typical for a beer sample. The use of a carbonate removal device after the suppressor minimizes

carbonate response. Chloride and sulfate are monitored because they affect the hops extraction process. The organic acids, listed in blue, are the fermentation products, which are of particular interest in monitoring the process. Phosphate is monitored for pH adjustment. As can be seen in this example, high-pressure IC is a powerful tool that provides a detailed profile of the sample in just 35–40 min to determine how the fermentation process is progressing.

The Thermo Scientific Dionex CarboPac SA10 column was developed for fast analysis of common mono- and

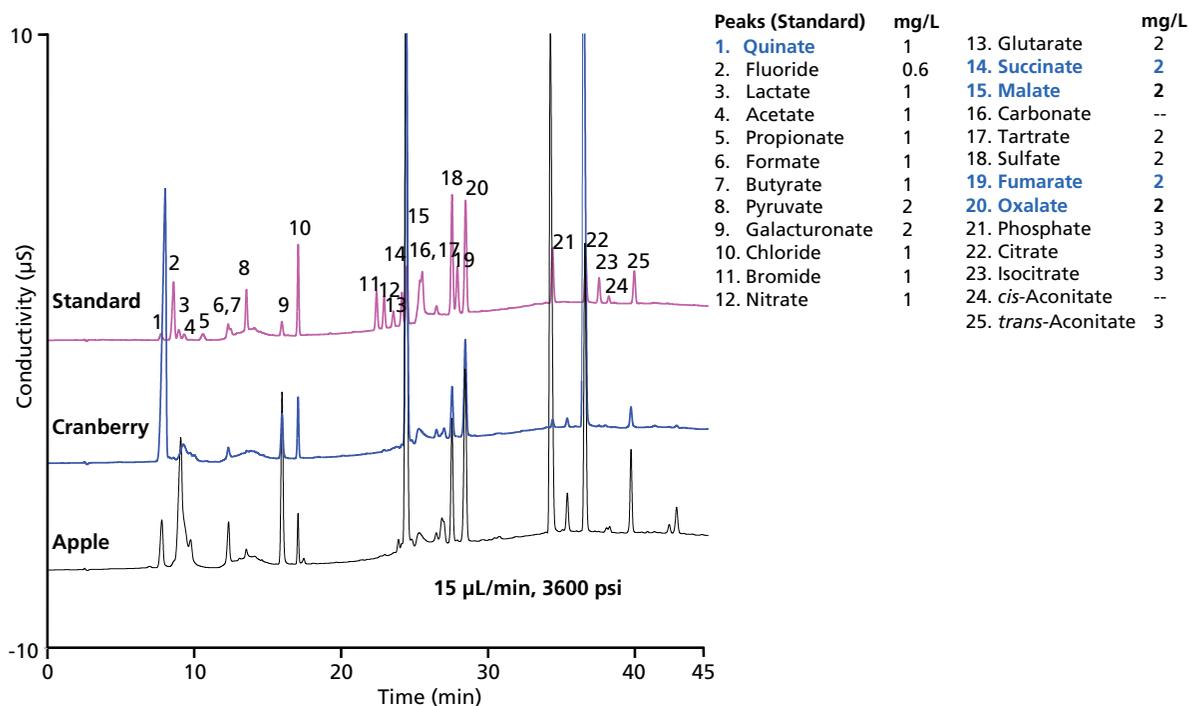


Figure 4: Characterization of beverage samples. Column: Dionex IonPac AG11-HC-4 μ m/AS11-HC-4 μ m (250 mm X 0.4 mm); gradient + 10% methanol (added to deionized water: 1 mM KOH for 8 min, 1–15 mM KOH in 10 min, 15–30 mM KOH in 10 min, 30–60 mM KOH in 10 min; eluent source: Dionex EGC-KOH cartridge (capillary); flow rate: 15 μ L/min; injection volume: 0.4 μ L; temperature: 30 $^{\circ}$ C; detection: suppressed conductivity, Dionex ACES 300 suppressor, external water mode; sample: juice, 1:50 sample dilution.

disaccharides in foods, beverages, and biofuels. To meet the challenge of achieving fast, high-capacity runs, we developed a 4- μ m version of the Dionex SA10 column with a supermacroporous substrate coated with latex nanoparticles. Figure 6 compares the separation of mono- and disaccharides on the 4- μ m Dionex SA10 column (top chromatogram) and a standard SA10 column (bottom chromatogram). As can be seen, resolution and efficiency are significantly improved on the 4- μ m column. Pulsed amperometric detection is used for direct

detection of nonderivatized sugars; this is also a useful tool for detecting amino acids and amines and other electroactive species.

The Dionex CarboPac SA10 column is very stable. Figure 7 shows an overlay of 1000 injections (every 100th run shown) without a shift in retention times or problems with capacity loss. A long column lifetime is maintained by using simple eluent cleanup steps.

Figure 8 illustrates the high capacity of the Dionex CarboPac SA10 4- μ m column in a separation of a concentrated

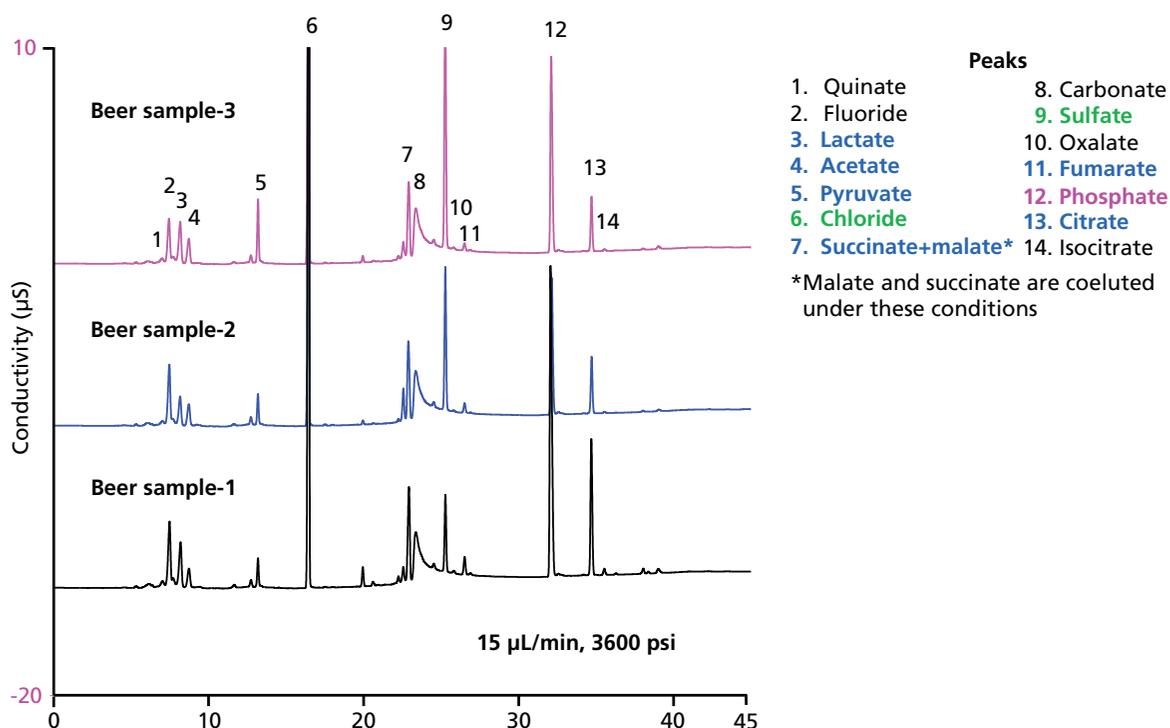


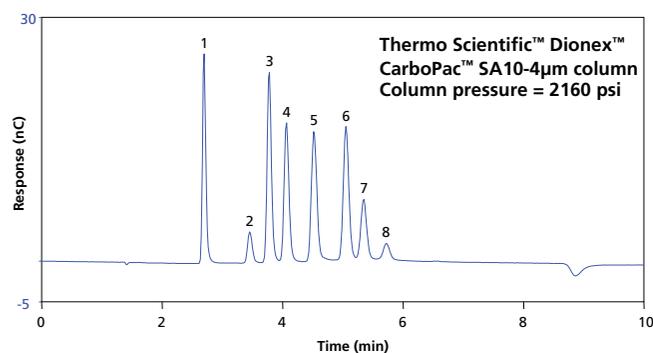
Figure 5: Beverage process control. Column: Dionex IonPac AG11-HC-4 μm /AS11-HC-4 μm , 0.4 mm; eluent source: Dionex EGC-KOH cartridge (capillary); gradient (potassium hydroxide): 1 mM for 8 min, 1–15 mM in 10 min, 15–30 mM in 10 min, 30–60 mM in 10 min; flow rate: 15 $\mu\text{L}/\text{min}$; injection volume: 0.4 μL ; temperature: 30 $^{\circ}\text{C}$; detection: suppressed conductivity, Dionex ACES 300 suppressor, Thermo Scientific Dionex CRD Carbonate Removal Device, recycle mode; samples: beer samples diluted 1:25 with deionized water.

corn stover hydrolysate with a 1:200 dilution. Excellent resolution is achieved without column overload. The detector is configured using a 15-mil (0.015-in.) gasket to increase the volume of the flow path through the electrochemical cell, which essentially desensitizes the cell and allows for running higher concentration samples.

Instrumentation for High-Pressure IC

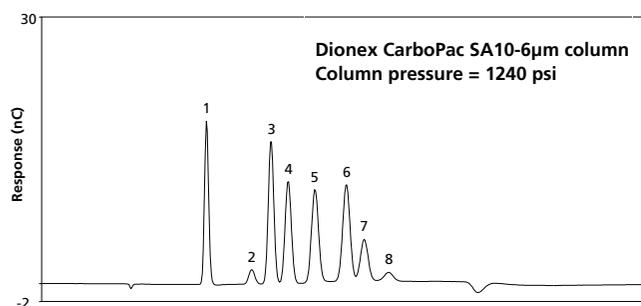
What's common to these applications is the high-pressure IC approach of

operating at high pressures and nominally higher flow rates. High pump pressures demand valves and fittings that function reliably when generating the eluent. For electrolytic eluent generation, Thermo Fisher Scientific has developed cartridges able to handle pressures up to 5000 psi, which is significant for inert, polymeric (PEEK) systems. Gradient capabilities—using Electrolytic Eluent Generation, as well as low-pressure mechanical gradients—are essential for taking full advantage of the resolving power of high-efficiency columns.



4-µm 2 mm

Number	Peak Name	Ret. Time (min)	Plates (EP)	Resolution
1	Fucose	2.7	11,602	6.13
2	Sucrose	3.5	9055	2.44
3	Arabinose	3.8	13,729	2.05
4	Galactose	4.1	12,105	2.84
5	Glucose	4.5	11,377	3.15
6	Xylose	5.1	14,128	1.73
7	Mannose	5.4	14,337	1.95
8	Fructose	5.7	13,416	n.a.



Std 2 mm

Number	Peak Name	Ret. Time (min)	Plates (EP)	Resolution
1	Fucose	2.7	7103	4.71
2	Sucrose	3.5	5391	1.84
3	Arabinose	3.8	7706	1.53
4	Galactose	4.1	6862	2.07
5	Glucose	4.5	6720	2.37
6	Xylose	5.1	7803	1.21
7	Mannose	5.4	7901	1.63
8	Fructose	5.8	7211	n.a.

Figure 6: Separation of carbohydrates using 4-µm (top) and standard (bottom) CarboPac SA10 columns.

The Thermo Scientific Dionex ICS-5000+ universal high-pressure IC system runs microbore, standard bore, and capillary flow rates. The system can be used for continuous operation at pressures as high as 5000 psi when it's configured as a Reagent-Free (RFIC) system. The system has versatile detection options, including conductivity for organic acids and inorganic anions, electrochemical for carbohydrate separations, as well as absorbance detection.

The Thermo Scientific Dionex ICS-4000 is a dedicated capillary ion chromatography system designed for ease-of-use for practical process monitoring. The instrument can be used as a walk-up system to run applications

as needed because it can be left on 24/7. With waste generation of only 15 mL/day, the system can run off 1 L of deionized water eluent over 2 months.

In addition to conductivity and electrochemical detection, a newer technology called *charge detection* is available with capillary systems. Charge detection has a different response factor than conductivity and is useful for detecting weakly ionic species. It has better linearity for weakly ionized components such as organic acids and amines. As seen in the pomegranate juice example of Figure 9, the charge detector (blue) is able to provide better response for some of the key organic acids that are important in profiling this juice.

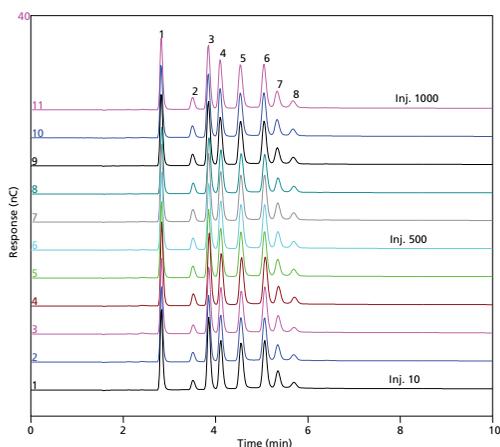


Figure 7: Separation of carbohydrates using a 4- μ m Dionex CarboPac SA10 column (1000 runs were performed, and every 100th run is shown). Column: Dionex CarboPac SA10-4 μ m, guard + analytical (250 mm X 2mm); eluent: 1 mM KOH; eluent source: Dionex EGC 500 KOH cartridge; flow rate: 0.38 mL/min; injection volume: 2.5 μ L; gasket: 2 mil; temperature: 45 °C; detection: integrated amperometry, quadruple pulse waveform working electrode: PTFE gold, disposable electrode; reference electrode: silver/silver chloride. Peaks (10 mg/L): 1 = fucose, 2 = sucrose, 3 = arabinose, 4 = galactose, 5 = glucose, 6 = xylose, 7 = mannose, 8 = fructose.

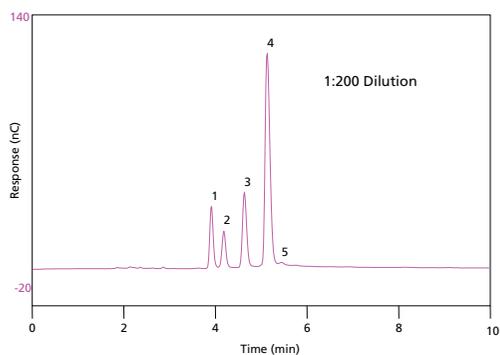


Figure 8: Separation of a corn stover sample (diluted 1:200) using a 4- μ m CarboPac SA10 column. Column: Dionex CarboPac SA10-4 μ m guard + analytical, 250 mm X 2mm; eluent : 1 mM KOH; eluent source: Dionex EGC 500 KOH cartridge; flow rate: 0.38 mL/min; injection volume: 2.5 μ L; gasket: 15 mil; temperature: 45 °C; detection: integrated amperometry, quadruple pulse waveform; working electrode: PTFE gold, disposable electrode; reference electrode: silver/silver chloride; sample: corn stover, 43 g/L, 1:200 dilution. Peaks: 1 = arabinose, 2 = galactose, 3 = glucose, 4 = xylose, 5 = fructose.

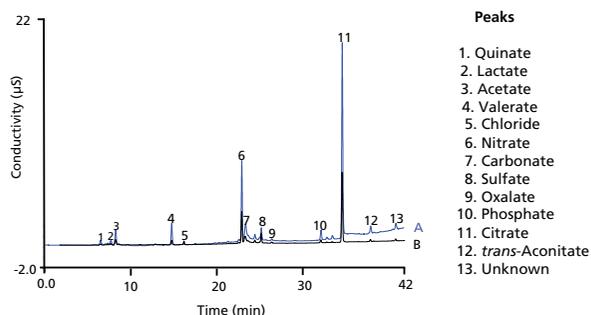


Figure 9: Analysis of pomegranate juice using charge and conductivity detection. Column: Dionex IonPac AS11-HC-4 μ m capillary (250 mm X 0.4 mm); instrument: Dionex ICS-5000 system; eluent source: EGC-EG; gradient (potassium hydroxide): 1 mM from 0 to 8 min, 1–30 mM from 8 to 28 min, 30–60 mM from 28 to 38 min, 60 mM from 38 to 42 min; flow rate: 0.015 mL/min; injection volume: 0.4 μ L; column temperature: 30 °C; detection: suppressed conductivity; suppressor: Dionex ACES 300 suppressor, AutoSuppression recycle mode; sample preparation: 1:40 dilution with deionized water. A = charge detection, B = conductivity detection.

Conclusion

High-pressure IC, with its use of smaller particle columns, provides more information, better peak efficiency, and the possibility of better speed and resolution—or finding the best balance of those. Better peak shapes improve peak integration for more accurate and reliable results. Sample throughput is increased without compromising data quality. The ability of high-pressure IC to yield high-resolution results quickly for profiling applications is particularly useful in industries such as food and beverage where answers are needed quickly in process monitoring.

Mike Doyle is the Product Manager for Ion Chromatography and Sample Preparation Products at Thermo Fisher Scientific.

Small Particles

Maximize chromatographic resolution with unique Ion Chromatography (IC) column chemistries that are designed for high efficiency separations. Now using smaller resin particles, the **Thermo Scientific™ Dionex™ IonPac™ 4 μm columns** deliver higher resolution for better peak identification and reliable quantification without sacrificing speed. The high resolution IC columns separate ionic analytes from complex sample matrices with ease. A broad range of chemistries offered in three formats – standard bore, microbore, and capillary – enable users to take control of their separations and results.

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FAST ION CHROMATOGRAPHY FOR HIGH THROUGHPUT WITH ENVIRONMENTAL SAMPLES

Peter Bodsky



Ion chromatography (IC) is following the footsteps of high performance liquid chromatography (HPLC) by moving to higher pressures in the form of fast IC and high-pressure IC. Recent innovations in columns and instruments provide powerful tools to enhance control of the analysis through increased speed, enhanced selectivity, and even modification of separations on the fly.

High-pressure IC and fast IC offer very fast analysis times, which is an attractive benefit to environmental, industrial, pharmaceutical, and biopharmaceutical laboratories that require maximum sample throughput. Analysis speed is also a growing need in monitoring process streams for wastewater, drinking water, and food processes, as well as for ultrapure water for semiconductor manufacturing. Fast analytical answers to changes in the process stream mean more control and improved troubleshooting. High-pressure IC is also useful in meeting the challenges of running complex samples and dealing with matrix issues.

High-Pressure IC Defined

A good general definition of *high-pressure IC*

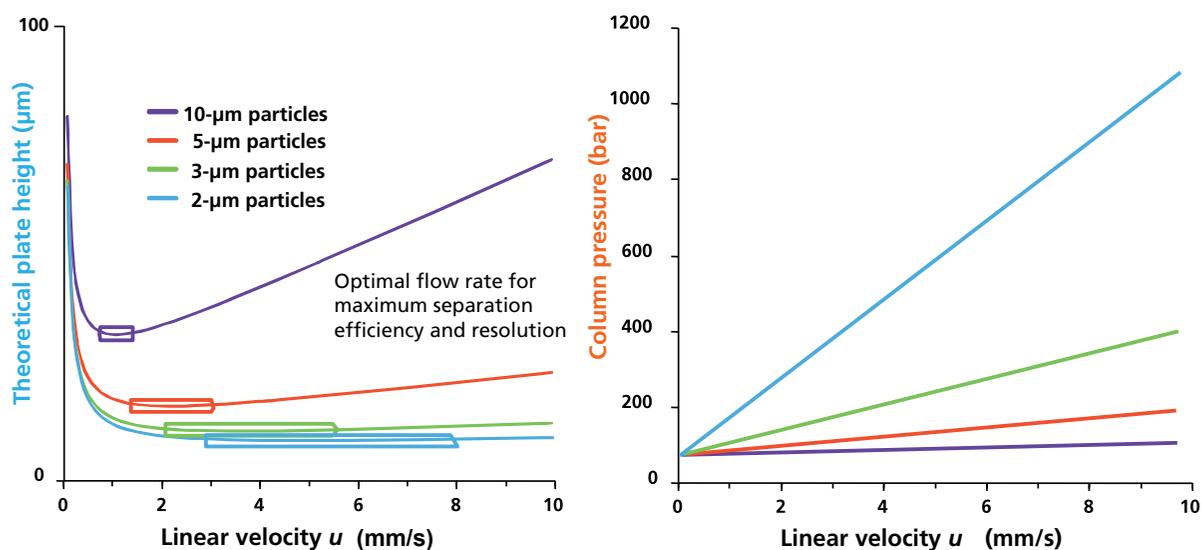


Figure 1: The influence of particle diameter on efficiency (left) and pressure (right).

is that it is the practice of using smaller-particle-size resins in combination with hardware capable of high pressure to achieve better chromatographic control.

The two key parameters controlled in high-pressure IC are speed and resolution. On standard IC systems, samples can be pushed through the column at a faster rate, of course, but resolution may erode as a result. Approaches such as using a shorter column or a faster flow rate with the same resin or increasing mobile phase strength are useful, but they offer limited success when working with complex or poorly resolved samples. Resolution can be improved using smaller-particle-size columns, but systems must be able to handle the high pressures involved to take full advantage of these columns. High-pressure IC, however, is capable of

overcoming these limitations and offering excellent separation control for both routine and complex samples.

Hardware Considerations

The greatest benefit from high-pressure IC comes from the use of decreased particle size of the columns combined with hardware that can tolerate the elevated pressures these columns produce. The pumps and electrolytic eluent generators used must operate reliably under these pressures over the lifetime of the instrument. Gradient capabilities, although not always necessary, provide extraordinary resolving characteristics, flexibility, and control.

A fundamental concept in HPLC is that decreasing column particle size increases separation efficiency. For example, reducing an 8-µm particle size resin to

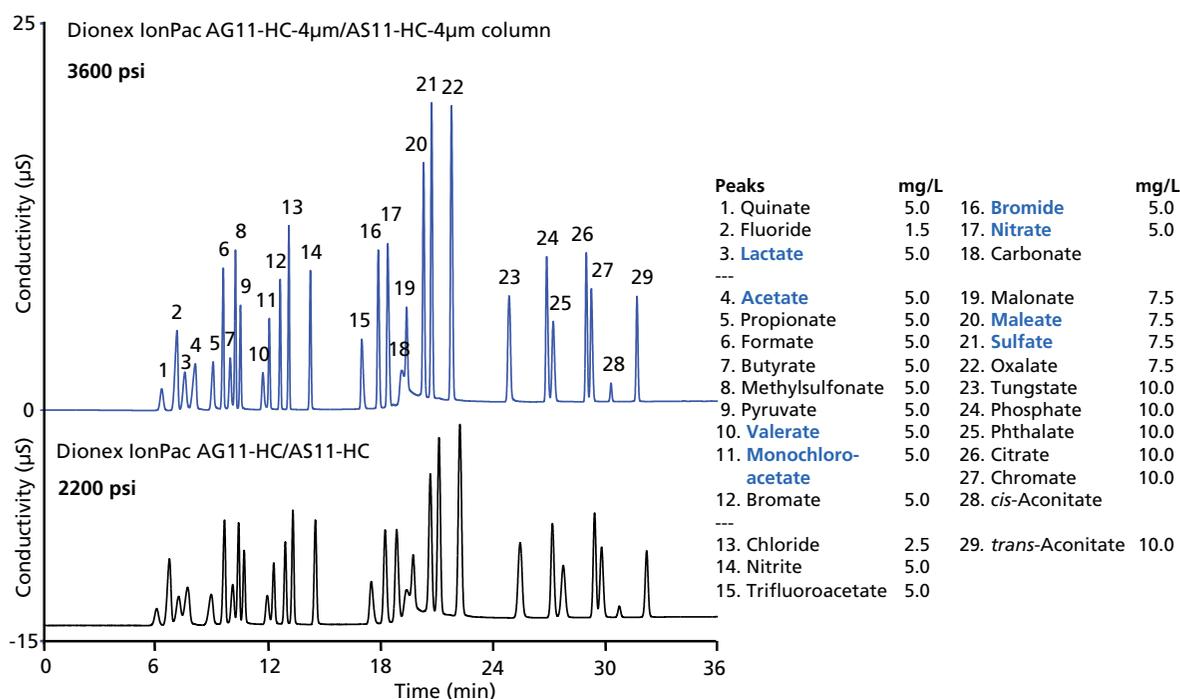


Figure 2: Separations obtained using a 4- μm Dionex IonPac AS11-HC column (top) and a standard AS11-HC column (bottom). Eluent source: Thermo Scientific Dionex EGC-KOH eluent generator cartridge (capillary); gradient (potassium hydroxide): 1 mM from 0 to 5 min, 1–15 mM from 5 to 14 min, 15–30 mM from 14 to 23 min, 30–60 mM from 23 to 31 min; flow rate: 15 $\mu\text{L}/\text{min}$; injection volume: 0.40 μL ; temperature: 30 $^{\circ}\text{C}$; detection: suppressed conductivity, Thermo Scientific Dionex ACES 300 anion capillary electrolytic suppressor, recycle mode.

4- μm doubles efficiency, although with a consequent fourfold increase in column pressure. Like HPLC, IC is moving toward column technology using smaller particles, and, now, newer instrumentation exists that can handle the pressures of these small particles, even at high flow rates. These smaller particle (4- μm) resins provide high efficiency and, when packed in 150-mm columns, offer extremely fast separation speeds. For complex samples, 4- μm particles in longer columns run at standard flow rates yield excellent resolving power.

The van Deemter equation describes several relationships in chromatography.

The left graph of Figure 1 shows that as particle size decreases, in this case, from 10 μm to 2 μm , the optimal flow rate actually increases. For maximum efficiency and resolution, smaller-particle-size columns give you the best flexibility and control with respect to flow rate.

As the 3- μm particle size line shows, as linear velocity or flow rate increases, theoretical plate height remains the same. This means that the separation can be run at even higher flow rates without degradation of the efficiency and resolution. This is the essence of fast IC and HPLC.

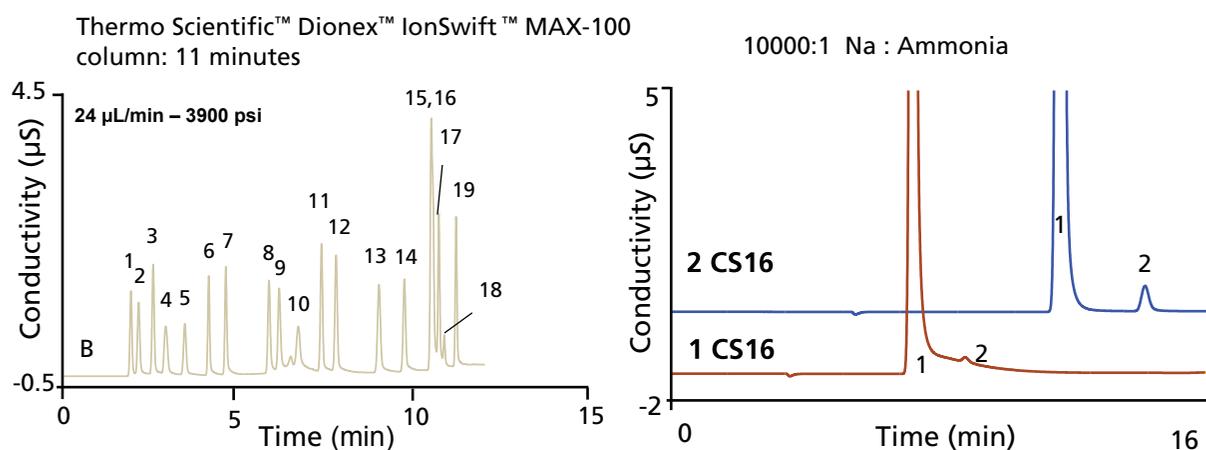


Figure 3: Separations showing the high-pressure capabilities of IC. Left: Separation of 18 ions on a Thermo Scientific Dionex IonSwift MAX-100 column. Right: Separation of a 10,000:1 mixture of sodium and ammonia using two Dionex IonPac CS16 columns (upper trace) and one CS16 column (lower trace).

The right side of Figure 1 illustrates the dramatic effect on pressure of using smaller particle sizes, and, thus, the importance of using high-pressure-compatible systems.

High-pressure IC systems with 4-µm particle-size columns deliver significant performance advantages, including more efficient peaks, improved speed and resolution, increased sample throughput without compromising data quality, and improved quality of analytical results.

High-Pressure IC Applications

Improved resolution. Figure 2 illustrates the improvement in resolution that can be achieved using 4-µm particle size capillary columns. This separation was run on the Dionex ICS-5000 high-pressure IC system under gradient conditions for the 4-µm Dionex IonPac AS11-HC column (top) and the standard Dionex IonPac AS11-

HC column (bottom). The 4-µm column produced narrower peaks, which results in improved integration and more reliable quantification. A number of critical peak pairs in this separation, such as lactate–acetate, valerate–monochloroacetate, bromide–nitrate, and maleate–sulfate, show distinctly better resolution.

High-pressure capabilities. Hardware designed to handle high pressures greatly expands the range and flexibility of separations. The separation on the left in Figure 3 was run on a high-capacity ion-exchange column (Dionex IonSwift MAX-100 anion-exchange column) at high pressure. Monolith columns contain a network of large separation pores and, thus, exhibit high permeability for fast mass transfer and provide high-speed, high-resolution performance. As shown in the figure, 18 peaks are resolved at high resolution in 15 min.

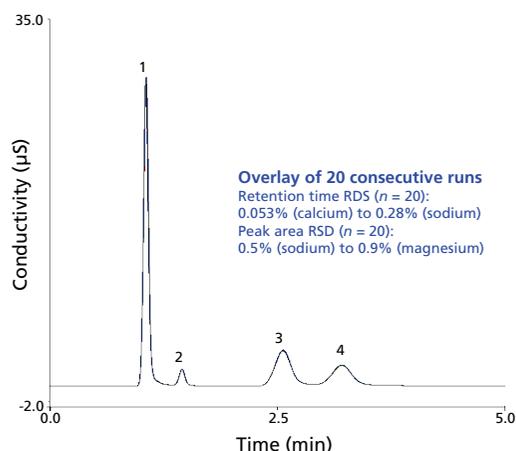


Figure 4: Rapid determination of cations in treated wastewater. Column: 150 mm X 0.4 mm Dionex IonPac CS12A-5 μ m; eluent source: Dionex EGC-MSA capillary cartridge; eluent: 28 mM MSA; flow rate: 18 μ L/min; temperature: 30 °C; suppressor: electrolytic capillary anion suppressor; sample: treated wastewater (1:10 dilution). Peaks: 1 = sodium (19.6 mg/L), 2 = potassium (0.8 mg/L), 3 = magnesium (3.5 mg/L), 4 = calcium (3.1 mg/L).

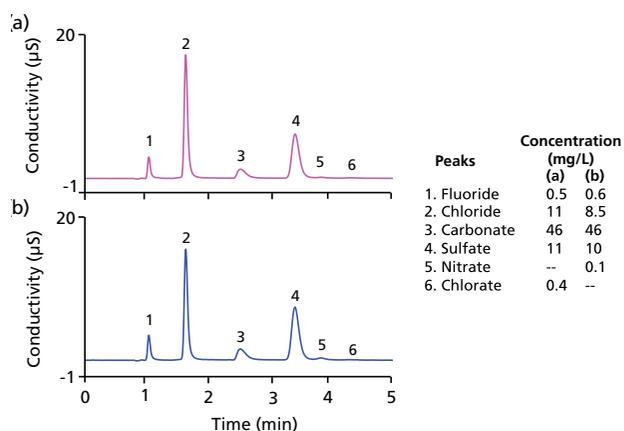


Figure 5: Analysis of municipal drinking water samples from (a) San Francisco and (b) Sunnyvale, California. Column: Dionex IonPac AG18-Fast, AS18-Fast, 2 mm; eluent source: Thermo Scientific Dionex EGC III KOH cartridge; eluent: 23 mM potassium hydroxide; column temperature: 30 °C; flow rate: 0.55 mL/min; injection volume: 5 μ L; detection: Thermo Scientific Dionex ASRS 300 Anion Self Regenerating Suppressor, 2 mm, 32 mA, recycle mode.

Reproducibility. Figure 4 shows an overlay of 20 consecutive runs of a treated wastewater sample on a CS12A 5- μ m column. Retention time relative standard deviations (RSDs) and peak area RSDs are very low, indicating that this is an extremely reproducible separation.

Speed. The Dionex IonPac AS22-Fast 4- μ m column was designed for monitoring inorganic anions in accordance with US EPA Method 300.1 (A) and 300.1. The column is used with an isocratic carbonate, bicarbonate eluent and suppressed conductivity detection. The column works well with Reagent-Free ion chromatography, or from eluent concentrates that can be manually prepared. The column is ideal for fast IC because it is designed to have sufficient

capacity to maintain resolution even in a short-column format, as shown here. When the short column is packed with smaller-particle resin, back pressures produced at higher flow rates are reduced and shorter overall run times can be achieved. Anions can be determined with high resolution even in drinking, surface, groundwater, and wastewater matrices, all in less than 5 min.

The Dionex IonPac AS18 column also now comes in a 4- μ m format. The column offers the same selectivity as the standard Dionex IonPac AS18-Fast column but the reduced particle size allows separation of common inorganic anions in significantly less time without loss of chromatographic resolution.

Convenience. Figure 5 shows a comparison of two municipal drinking

Dionex IonPac Anion-Exchange Columns

Dionex IonPac AS22-IC columns

Dionex IonPac AS22-Fast

Dionex IonPac AS19-4 μ mDionex IonPac AS18-4 μ mDionex IonPac AS18-4 μ m FastDionex IonPac AS11 HC- μ m**Dionex IonPac Cation-Exchange Columns**Dionex IonPac CS19-4 μ mDionex IonPac CS12A-5 μ m**Table I:** Dionex IonPac high-capacity, ion-exchange, and small-particle resin columns

water samples from California, one from Sunnyvale and one from San Francisco. The samples were run on a Dionex IonPac AS18-Fast 2-mm column with an eluent generation cartridge. The samples are similar in composition, as would be expected, and the run time is less than 5 min. The system was able to generate ultrapure potassium hydroxide electrolytically with only the addition of water, combining speed with ease of use.

Thermo Fisher Scientific offers three high-pressure IC systems, the Thermo Scientific Dionex Integrion HPIC system, the Dionex ICS-4000, and the Dionex ICS-5000+. The Dionex IonPac 4- μ m columns are designed for high-pressure

IC (Table I) and significantly improve separation efficiency and resolution. For high-pressure IC, analysts now have the choice of running fast separations on short 150-mm columns and high-resolution separations with standard-length 250-mm columns and finding the optimum balance between resolution and throughput.

Conclusion

High pressure IC and fast IC systems provide flexibility and a high degree of separation control, even for challenging samples. With high-pressure PEEK flow paths and the ability to operate continuously at pressures as high as 5000 psi, these reagent-free systems represent a significant improvement in IC capabilities. The systems are designed to tolerate high pressures, which allows the analyst to increase flow rate to maximize throughput while benefiting from the advantages of electrolytic eluent generation and suppression. The small 4- μ m particle size columns used with these systems allow fast run times while maintaining excellent resolution.

Peter Bodsky is the Regional Marketing Manager for the Americas for Ion Chromatography and Sample Preparation Products at Thermo Fisher Scientific.

What happens inside

Sometimes it's the people we don't see who have the biggest impact on our day-to-day lives, like the IC chromatographers who perform routine analyses that give us the peace of mind to use everyday products and resources without a second thought. The Thermo Scientific™ Dionex™ Integron™ HPIC™ system was thoughtfully developed around the analysis challenges of modern day laboratories. With a logical, flow-based plumbing layout and remote performance monitoring, it's a reliable instrument made to support you in your mission to protect what matters most.

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TRENDS IN ION CHROMATOGRAPHY: USER-FRIENDLY INSTRUMENTS, HIGHER-CAPACITY COLUMNS, AND THE MOVE TO HIGH PRESSURE

An interview with Chris Pohl

In this interview, Chris Pohl, the Vice President of Chromatography Chemistry at Thermo Fisher Scientific, talks about current trends in ion chromatography (IC).

Why should we move to high pressure in IC?

As with other modes of liquid chromatography (LC), smaller particles produce higher-efficiency columns, all other things being equal. Smaller particles require significantly higher pressures because there is an inverse square relationship between particle diameter and pressure. In other words, reducing particle diameter by a factor of two will increase pressure by a factor of four. Higher efficiency can be used to advantage in the analysis of complex mixtures that contain dozens of distinct species; such mixtures are common in foods and environmental samples. Alternatively, in the case of well-defined samples of low complexity such as drinking water or high-purity water, which don't benefit from increased column plate count, shorter columns of equivalent efficiency allow for faster analysis without sacrificing resolution.

Why is it taking so long to move to high-pressure IC, given that high pressures have been used in high performance LC (HPLC) for so long?

In IC, the preferred eluent species are hydroxide in the case of anion analysis and hydronium in the case of cation analysis. These ions produce the lowest background conductivity when operating in the suppressed mode. However, they tend to be corrosive to the metallic components commonly used to achieve the high pressures associated with ultrahigh-pressure LC (UHPLC). Such eluent species are especially problematic when exposing metallic surfaces to extremes of pH in an alternating fashion. This exposure can strip the protective passivation layer from the metal surface at high pH followed by corrosion of the metal surface upon return to low pH. As a result, nonmetallic materials are required for IC instrumentation to ensure long-term reliability with such eluent systems. Unfortunately, the tensile strength of polymeric materials commonly used in IC is much lower than that of metallic materials, which limits the maximum pressure rating of the IC system. To increase the operating pressure of the IC system it has been necessary to re-engineer most of the chromatographic components of the IC instrument so as to maximize the upper pressure limit of the system with available materials. Only recently has this combination of component design and material science

enabled a significant increase in IC operating pressure, making high-pressure IC feasible.

There is also a trend to move to higher-capacity columns for IC. Why?

High-capacity columns offer the ability to handle diverse sample matrices with analytes present at a wide range of concentrations. In IC, it is not uncommon to find analytes of interest that are eluted immediately adjacent to a ubiquitous matrix ion. When the concentration ratio of the matrix ion to the analyte ion becomes extreme, it can be difficult to quantitate the analyte in the presence of the matrix species, especially if the concentration of the matrix species exceeds the loading capacity of the analytical column. Dilution of the sample can overcome problems with sample overload but can compromise the analyte limit of detection (LOD). It is preferable to use high-capacity columns for such samples as this enables determination of analyte species without the necessity of sample dilution, providing superior analyte LODs. In the early days of ion chromatography, suppressors had limited capacity and thus IC columns typically had correspondingly low ion-exchange capacity. The development of modern continuous suppressors compatible with high mobile-phase concentrations enables the use of IC columns of much higher capacity, greatly expanding the utility of IC for the

analysis of trace analytes in the presence of large molar excesses of matrix ions.

With its new instrument, Thermo Fisher Scientific is focusing a lot on ease of use. Have IC instruments not been very user friendly to date?

Previous IC instruments have been designed so as to maximize analytical performance. Achieving high performance in IC involves a number of components not present in a conventional HPLC system, such as the suppressor, the eluent generator, the electrolytic trap column, and the high-pressure degasser. We have found that the greater operational complexity of these IC-specific components makes an IC instrument more difficult to use than an HPLC instrument, especially for those who are new to IC.

With our newest IC instrument, the Thermo Scientific Dionex Integrion HPIC system, we worked hard to make sure that all of these components are easy to use through the use of built-in plumbing diagrams, an intuitive fluidic layout, and a simple, easy-to-use tablet control panel available in 11 languages. Taken together, these features combine to make the Dionex Integrion IC system the easiest-to-use instrument we've ever made, without sacrificing performance

In addition, we have developed new a IC PEEK Viper finger-tight fitting system that guarantees a perfect zero dead volume connection every time, minimizing issues commonly seen with conventionally tubing and fittings. Viper fittings are in wide

use in UHPLC and are available in an IC-compatible format for the first time. These new fittings make small-diameter columns as easy to use as 4-mm columns.

Why are you now providing a new system for consumable identification? How does it work?

It's long been my goal to incorporate automatic consumable identification as part of the instrument. Automatic identification ensures that 100% of the consumable utilization record is tracked as part of the identification system. Identification is achieved using either radio-frequency identification (RFID) tags on consumables without electrolytic control or electronic tags embedded in the control cable for electrolytic consumables. These tags allow for keeping records of up to 20 different parameters associated with each consumable, such as number of injections, maximum temperature exposure, volume of mobile phase passed through the column, column pressure history, suppressor voltage history, and eluent generator ion count, along with device parameters such as column diameter, column type, and suppressor type. It can also be used to identify when the performance of a component has drifted outside recommended operating parameters and the component needs replacement. Because the information is stored on a tag attached to the consumable rather than being stored in software, the information stays with the consumable even if the consumable is

moved to a new instrument. Furthermore, the information stored on the consumable helps technical support personnel troubleshoot issues that customers report by providing key information about the way the consumable was operated and performance trends over the life of the consumable.

Can the new system be controlled remotely?

The instrument is available with a WiFi interface that allows for remote control of the instrument with any WiFi-enabled tablet or mobile device that has been connected to the instrument and loaded with a suitable control app. Although the tablet interface is not a requirement to operate the instrument, the tablet can either be magnetically mounted to the front of the instrument or can be detached from the instrument; in the latter case, the tablet can be used to remotely control the instrument as long as it is within WiFi communication range. The interface is compatible with mobile devices with Android, iOS, and Windows operating systems.

The new instrument includes features to help prevent user error. Can you tell us about those?

Because all of the consumables used in the instrument contain tags that are automatically identified by the instrument, the instrument can immediately identify incompatible combinations. For example, the instrument will identify

and warn about improperly configured components such as an anion-exchange column in combination with a cation suppressor or an anion eluent generator in combination with a cation-exchange column. In addition, the instrument uses a new chemical barcode standard system for improved peak identification tracking. Unlike conventional software that uses retention time windows for each analyte in the chromatogram, this new analyte tracking system uses the area percent of each peak relative to the largest peak in the chromatogram as the basis for identifying each of the peaks in the chromatogram. Even as retention time shifts over the life of the column, peak identifications remain accurate, whereas under similar conditions the use of retention time windows may result in erroneous peak identifications.

What if users still need more help?

Because control of the instrument uses a tablet interface with considerable memory capacity, it is feasible to provide much more information in the tablet interface than has been available in previous instruments. The instrument manual in its entirety is stored in tablet memory. In addition, installation guides, installation videos, and troubleshooting guides are available through the tablet-based instrument control interface to help the chromatographer each step of the way.

Chris Pohl is the Vice President of Chromatography Chemistry at Thermo Fisher Scientific.