



IonPac™ AS31 anion-exchange column

Faster separation of haloacetic acids

Benefits

- Separation of 9 haloacetic acids, bromate, and dalapon in approximately 35 minutes
- Up to 39% shorter run times for US EPA Method 557 relative to Dionex IonPac AS24 columns
- Determine HAAs in high ionic strength samples without sample treatment

Keywords

Anion-exchange chromatography, bromate, dalapon, inorganic anions, drinking water, Method 557, HAA5, HAA9

The Thermo Scientific™ Dionex™ IonPac™ AS31 column is a high-capacity, hydroxide-selective anion-exchange column designed for fast determination of nine haloacetic acids (HAAs), bromate, and dalapon in drinking water prior to detection with mass spectrometry. The 2 mm column format was specifically developed for use with IC-MS or IC-MS/MS. For added convenience and increased reproducibility, the column can be used in high-pressure ion chromatography (HPIC) systems equipped with Thermo Scientific™ Dionex™ Eluent Generators that can automatically produce potassium hydroxide eluents from deionized water.

- Column selectivity has been optimized at an operating temperature of 15 °C to ensure reproducible recovery for heat labile haloacetic acids.
- High capacity (116 µeq/column) allows injection of complex matrices without overloading the column while maintaining excellent peak shapes for lower concentration analytes.
- Requires no sample pre-treatment when determining haloacetic acids and dalapon in high-ionic strength matrices.
- Use with Thermo Scientific™ Dionex™ Haloacetic Acid Internal Standards for fast, simple, and accurate quantitation by isotope dilution.

High-efficiency particle structure

The Dionex IonPac AS31 column was developed using a unique polymer bonding technology. The stationary phase consists of a novel, hyper-branched anion-exchange condensation polymer electrostatically attached to the surface of a sulfonated wide-pore polymeric substrate. Alternating cycles of epoxy monomer and amines produce a coating which is grown directly off the substrate as illustrated in Figure 1. Resin capacity is controlled through the number of alternating coating cycles. The Dionex IonPac AS31 column uses a high-capacity resin (116 $\mu\text{eq}/\text{column}$) with optimized selectivity for HAAs, bromate, and dalapon in environmental water samples. The resin is composed of a polymeric 6 μm substrate consisting of ethylvinylbenzene cross-linked with 55% divinylbenzene.

Monitor haloacetic acids in drinking water

Haloacetic acids containing chlorine and bromine are formed during the disinfection of drinking water by chlorine. The presence of haloacetic acids in drinking water has been linked to several adverse health effects including bladder, kidney, and colorectal cancer.

The Dionex IonPac AS31 column can separate the following nine HAAs:

- Monochloroacetic acid (MCAA)
- Monobromoacetic acid (MBAA)
- Dichloroacetic acid (DCAA)
- Bromochloroacetic acid (BCAA)
- Dibromoacetic acid (DBAA)
- Trichloroacetic acid (TCAA)
- Dichlorobromoacetic acid (DCBAA)
- Dibromochloroacetic acid (DBCBA)
- Tribromoacetic acid (TBAA)

Five HAAs including MCAA, DCAA, TCAA, MBAA, and DBAA are cited in the US EPA haloacetic acid regulation. This regulation requires that the total of these five HAAs does not exceed a maximum concentration (MCL) of 60 $\mu\text{g}/\text{L}$.¹ All drinking water plants in the United States must determine the HAA levels in drinking water.

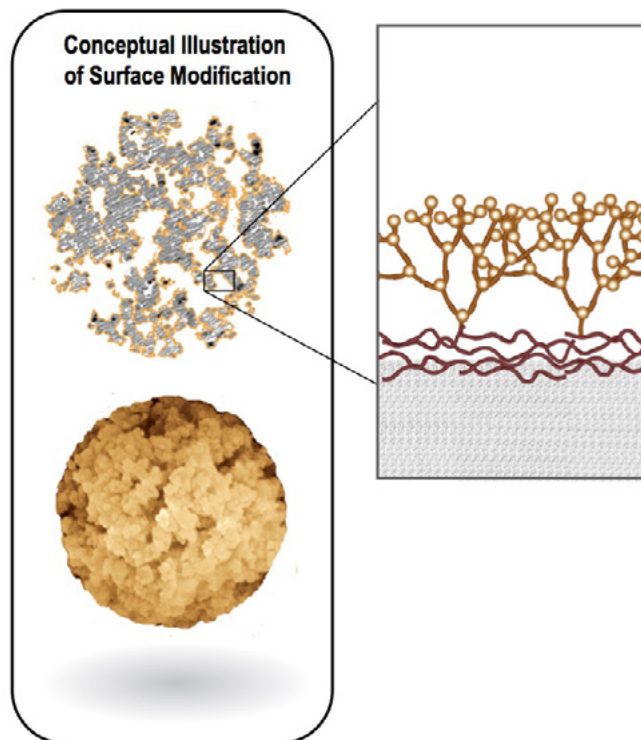
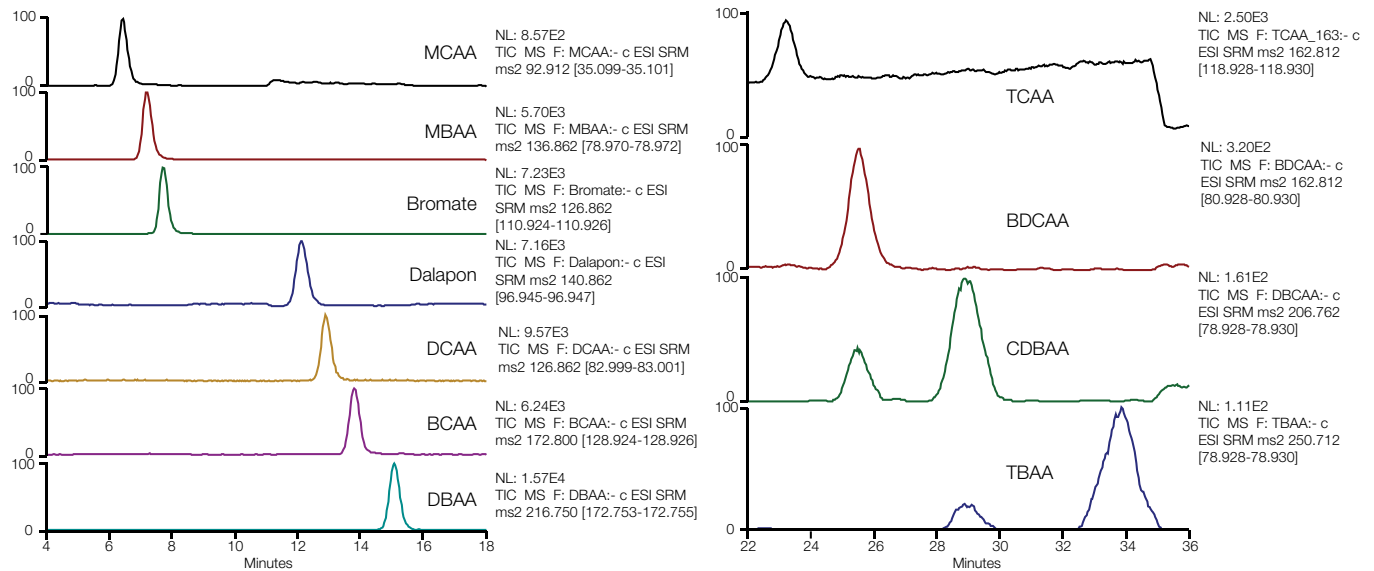
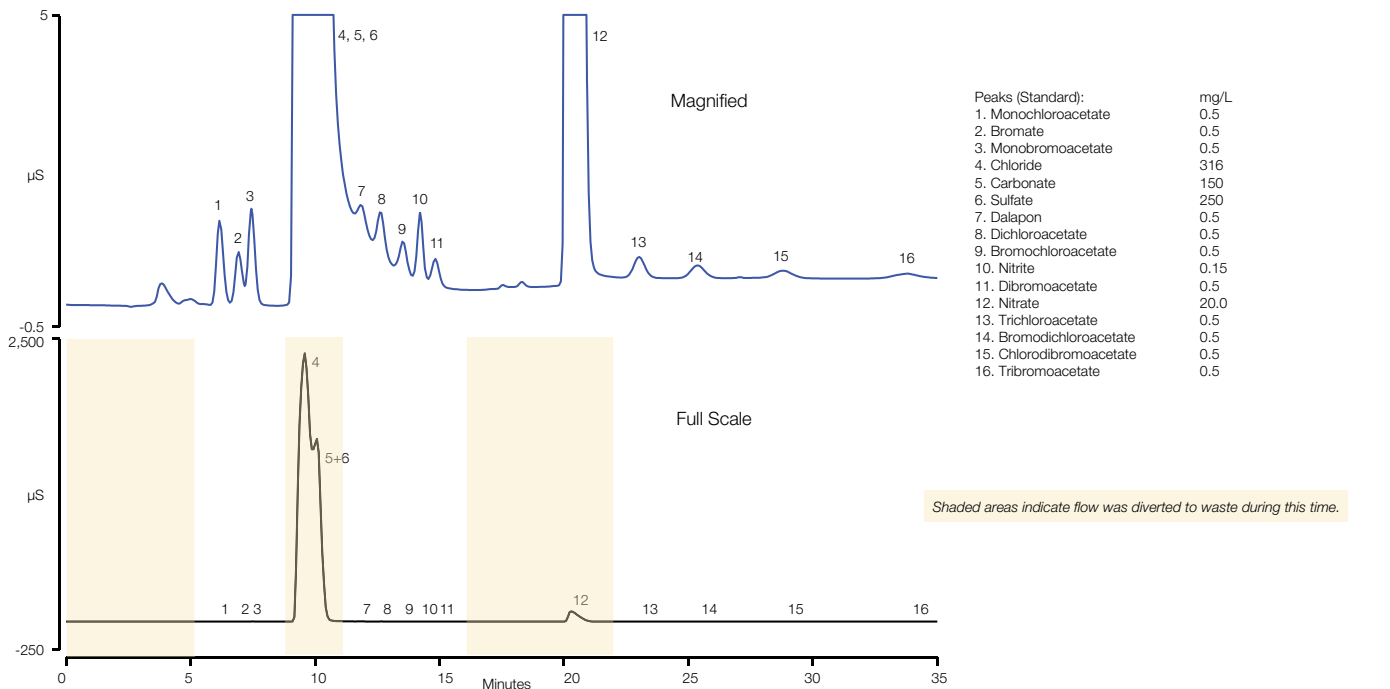


Figure 1. Structure of a Dionex IonPac AS31 column resin particle.

The Dionex IonPac AS31 column is designed for determination of haloacetic acids by IC-MS/MS in high ionic strength matrices. Low $\mu\text{g}/\text{L}$ (ppb) levels of haloacetic acids can be determined easily using MS/MS detection. Figure 2 (top) shows the determination of 0.5 mg/L of nine different haloacetic acids, bromate, and dalapon in EPA Method 557 Laboratory Synthetic Sample Matrix (LSSM) using conductivity detection and a potassium hydroxide gradient delivered by an eluent generator. Figure 2 (middle) shows more sensitive determination of nine HAAs, bromate, and dalapon at 5 $\mu\text{g}/\text{L}$ in EPA Method 557 LSSM using MS/MS detection.



Thermo Scientific™ Dionex™ ICS-6000 System and Thermo Scientific™ TSQ Fortis™ Mass Spectrometer

Column: Dionex IonPac AG31/AS31
 Eluent: KOH Gradient (see timed events)
 Suppressor: Dionex ADRS 600, 2 mm, external water, 0.3 mL/min
 Suppressor current: 64 mA
 Analytical flow rate: 0.3 mL/min
 Column temp: 15 °C
 Injection volume: 100 µL
 Detector: CD, TSQ Fortis

Time	[KOH]	Divert Valve
-0.5	Begin	17.0
0.0		17.0
5.0		17.0
7.0		17.0
8.5		
11.1		85.0
15.6		17.0
18.0		85.0
21.7		17.0
35.0		85.0
35.1		17.0
36.0	End	

TSQ Fortis Tune Parameters:

Ion source type: H-ESI (negative polarity)
 Spray voltage: 3200 V
 Cycle time: 2.3 secs
 Resolution: Q1 (FWHM) 0.7
 Q3 (FWHM) 0.7
 CID gas: 20 (temp 100 °C, no sweep gas)
 Sheath gas: 50 Arb
 Aux gas: 10 Arb
 Sweep gas: 3 Arb
 Ion transfer tube temp: 225 °C
 Vaporizer temp: 275 °C
 Make-up solvent: IPA at 0.1 mL/min

Figure 2. Determination of nine haloacetic acids, bromate, and dalapon in EPA Method 557 LSSM at 0.5 mg/L using conductivity detection (top) and 5 µg/L using MS/MS detection (middle) using the Dionex IonPac AS31 column.

Achieve faster run times

The Dionex IonPac AS31 column has been specifically designed for fast analysis. As shown in the comparison in Figure 3, the Dionex IonPac AS31 can achieve 39% faster run times compared to the Dionex IonPac AS24. Note the run time for EPA Method 557 can be reduced from 57 min to approximately 35 min. The relatively high capacity of the Dionex IonPac AS31 allows for large loop injections for trace analysis without the need for sample pre-treatment.

Extended application capabilities

The unique selectivity and high capacity of the Dionex IonPac AS31 makes it an ideal column for development of specialized applications, providing excellent separation of environmental anions including inorganic anions, oxyhalides, oxyanions, and organic acids using a potassium hydroxide eluent. Using gradient elution, these analytes are easily separated in less than 26 min as shown in Figure 4.

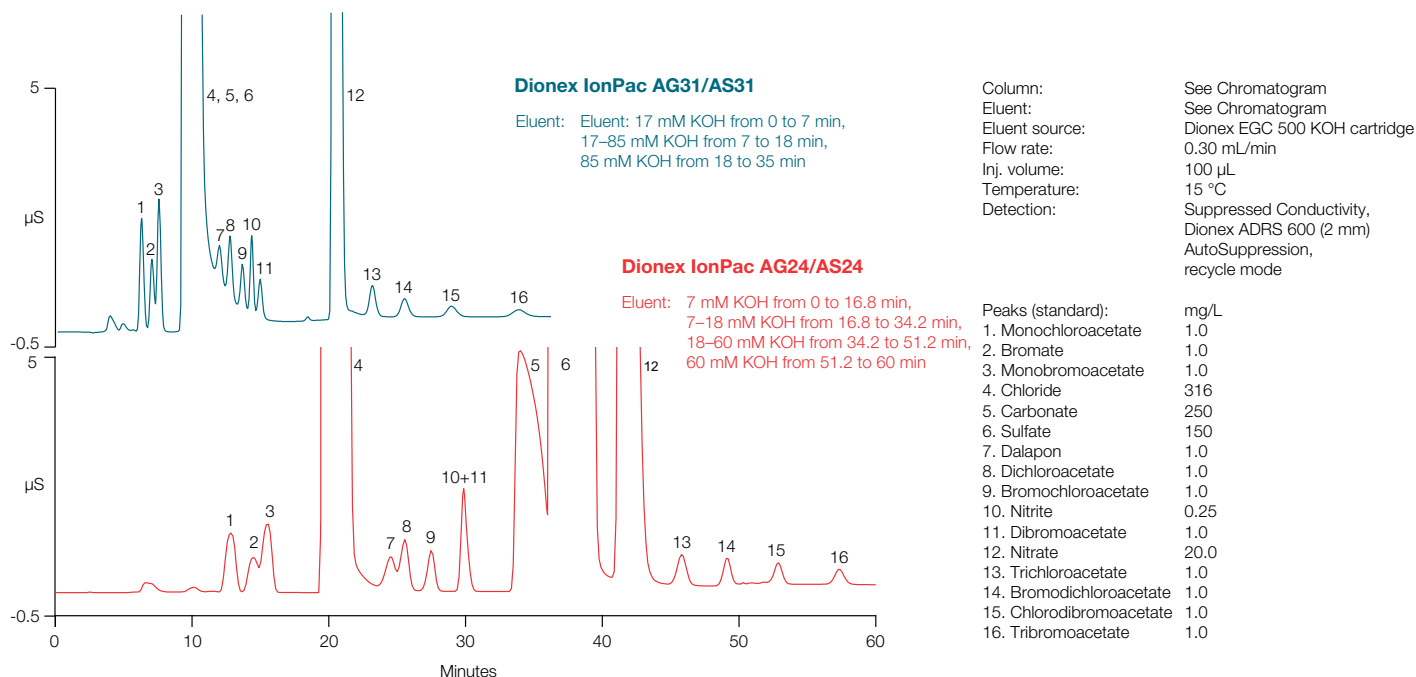


Figure 3. Comparison of the separation of haloacetic acids, dalapon, and bromate in EPA Method 557 LSSM matrix on the Dionex IonPac AS31 (2 × 250 mm) and Dionex IonPac AS24 (2 × 250 mm) columns.

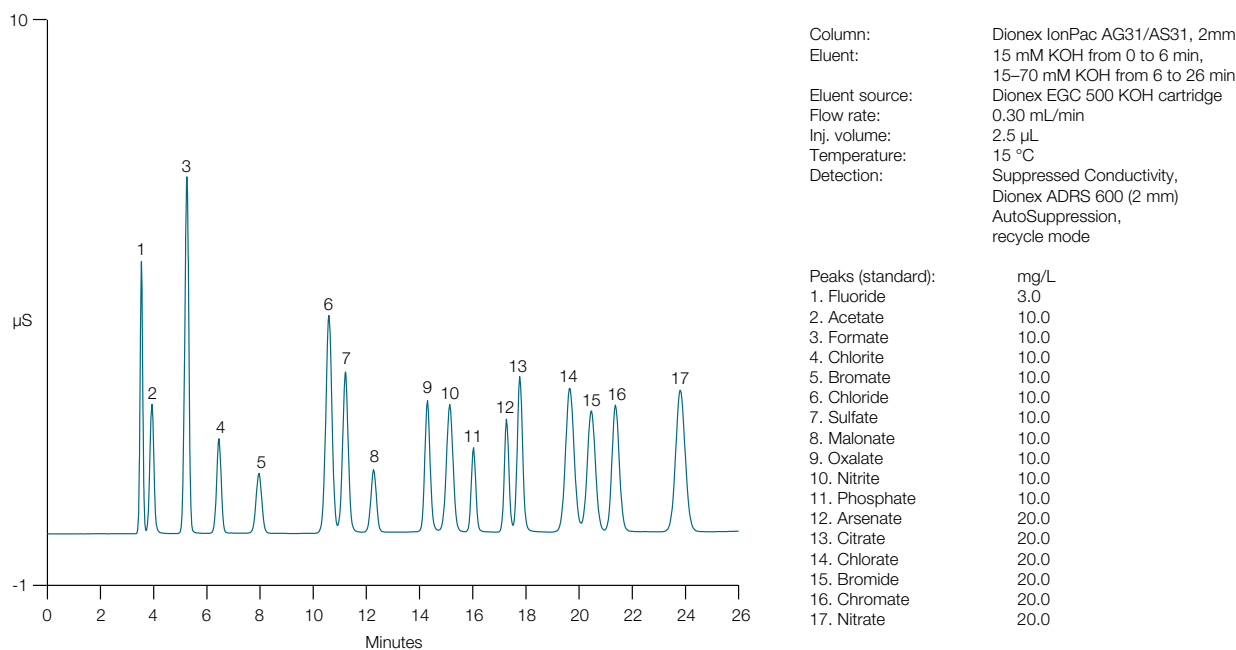


Figure 4. Separation of various anions including inorganic anions, organic acids, oxyanions, and oxyhalides on a Dionex IonPac AS31 column using a potassium hydroxide eluent delivered by an Eluent Generator.

Effect of temperature on selectivity

The Dionex IonPac AS31 column is optimized to separate nine HAAs using a reduced temperature of 15 °C to ensure reproducible recovery for haloacetic acids. Figure 5 shows the effect of temperature on column selectivity. Note that at higher temperature (30 °C), the resolution of sulfate and chloride decreases and there is a greater effect on the divalent anions (sulfate and thiosulfate) relative to the monovalent anions (fluoride, chloride, bromide, and nitrate). Because of this sensitivity to temperature changes, it is important to verify the column oven temperature and adjust as needed to obtain the optimal resolution.

System requirements

The Dionex IonPac AS31 column is recommended for use with the Thermo Scientific™ Dionex™ ICS-6000 or ICS-5000+ HPIC™ systems equipped with an eluent generator and Low Temperature DC Detector/Chromatography Compartment capable of maintaining a column temperature of 15 °C. These HPIC systems can operate continuously at up to 5000 psi to support the backpressure generated by the Dionex IonPac AS31 column under standard operating conditions. The eluent generator produces potassium hydroxide eluent from deionized water. For all systems, the use of Thermo Scientific™ Dionex™ IC PEEK Viper™ fittings (Figure 6) is recommended to achieve consistent low dead volume connections and ensure optimum chromatographic performance.

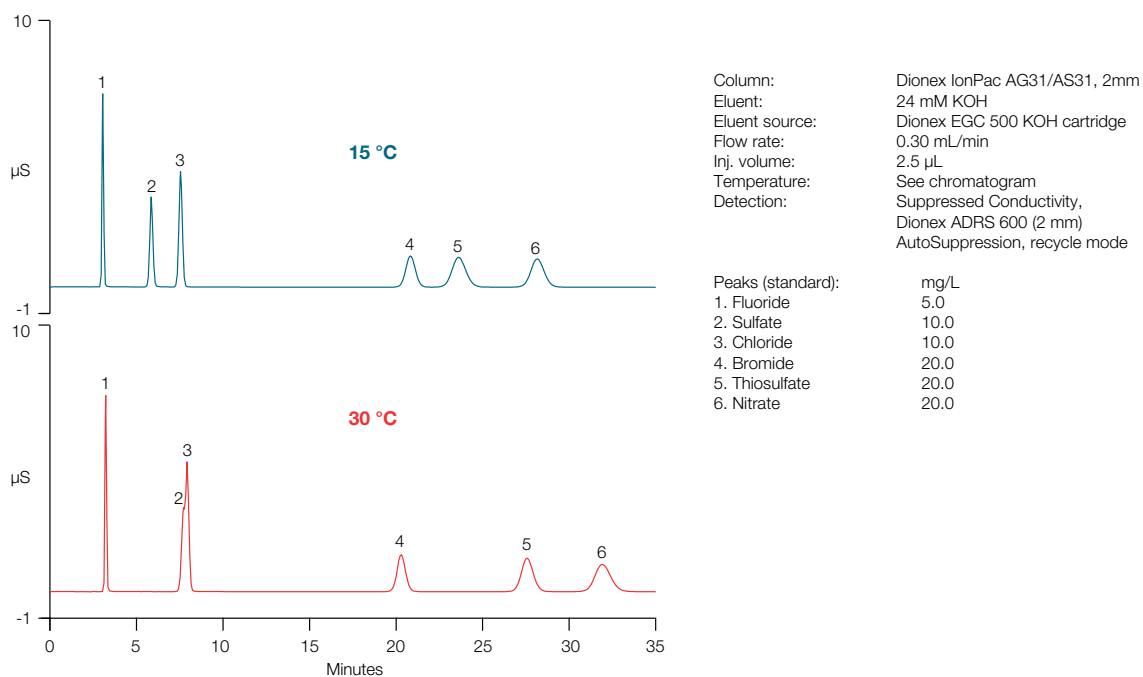


Figure 5. Effect of temperature on the Dionex IonPac AS31 column selectivity.



Figure 6. For best chromatographic performance, Dionex IC PEEK Viper fittings are recommended for use with Dionex IonPac AS31 columns.

Suppressor recommendations

For optimum ease of use and performance, Dionex IonPac AS31 analytical columns should be used with a Dionex AERS 500e Anion Electrolytically Regenerated Suppressor for External Water Mode. Alternatively, a Dionex ADRS 600 Anion Dynamically Regenerated Suppressor may be used in external water mode.

References

1. Stage 1 Disinfectants and Disinfection Byproducts Rule: A Quick Reference Guide, p. 2, US EPA, 816-F-01-010

Specifications	
Dimensions:	Dionex IonPac AS31 Analytical Column: 2 × 250 mm Dionex IonPac AG31 Guard Column: 2 × 50 mm
Maximum operating pressure:	5000 psi
Mobile phase compatibility:	pH 0–14, 100% HPLC solvents (e.g., acetonitrile, methanol, and 2-propanol)
Substrate characteristics	
Analytical columns:	Supermacroporous resin Particle Diameter: 6 µm Pore Size: 2000 Å Crosslinking (%DVB): 55%
Guard columns:	Microporous resin Particle Diameter: 11 µm Pore Size: < 10 Å Crosslinking (%DVB): 55%
Functional group:	Alkanol quaternary ammonium
Hydrophobicity:	Low
Capacity:	116 µeq (2 × 250 mm column) 1.5 µeq (2 × 50 mm column)
Column construction:	PEEK™ with 10–32 threaded ferrule-style end fittings. All components are nonmetallic.

Ordering information

To order in the US, visit thermofisher.com, call (800) 532-4752, or contact the nearest Thermo Fisher Scientific office. Outside the US, order through your local Thermo Fisher Scientific office or distributor. Refer to the following part numbers.

Description	Part Number
Analytical and guard columns	
Dionex IonPac AS31 Analytical Column (2 × 250 mm)	303147
Dionex IonPac AG31 Guard Column (2 × 50 mm)	303148
Haloacetic acid internal standards	
Dionex Monochloroacetic Acid (MCAA-2- ¹³ C) 1000 mg/L, 1 mL	069406
Dionex Monobromoacetic Acid (MBAA-1- ¹³ C) 1000 mg/L, 1 mL	069407
Dionex Dichloroacetic Acid (DCAA-2- ¹³ C) 1000 mg/L, 1 mL	069408
Dionex Trichloroacetic Acid (TCAA-2- ¹³ C) 1000 mg/L, 1 mL	069409
Inorganic anion and oxyhalide standards	
Dionex Fluoride Standard, 1000 mg/L, 100 mL	037158
Dionex Chlorite Standard, 1000 mg/L, 125 mL	303167
Dionex Bromate Standard, 1000 mg/L, 125 mL	303168
Dionex Chloride Standard, 1000 mg/L, 100 mL	037159
Dionex Nitrite Standard, 1000 mg/L, 125 mL	303169
Dionex Chlorate Standard, 1000 mg/L, 125 mL	303170
Dionex Nitrate Standard, 1000 mg/L, 100 mL	056497
Dionex Phosphate Standard, 1000 mg/L, 125 mL	303172
Dionex Sulfate Standard, 1000 mg/L, 100 mL	037160
Dionex Nine Anion Standard, 50 mL	303173
<i>Nine anion standard contains 20 mg/L fluoride; 30 mg/L chloride; 100 mg/L nitrite, bromide, nitrate, bromate, and chlorate; 150 mg/L phosphate and sulfate</i>	
Eluent generator accessories	
Dionex EGC 500 KOH Eluent Generator Cartridge	075778
Dionex CR-ATC 600 Continuously Regenerated Anion Trap Column (for use with Dionex ICS-6000 HPIC systems)	088662
Dionex CR-ATC 500 Continuously Regenerated Anion Trap Column (for use with Dionex ICS-5000+ HPIC systems)	075550
Dionex IC PEEK Viper fittings kits	
Dionex IC PEEK Viper Fittings Kit for Dionex ICS-6000 and ICS-5000+ systems with conductivity detectors	088803
Suppressors	
Dionex AERS 500e (2 mm) Anion Electrolytically Regenerated Suppressor for External Water Mode	302662
Dionex ADRS 600 (2 mm) Anion Dynamically Regenerated Suppressor	088667

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