

ENVIRONMENTAL



Environmental Analysis Using the ICS-3000 Ion Chromatography System

Ion chromatography (IC) is well established as a routine method for the analysis of ionic analytes in environmental samples. IC has been incorporated into environmental regulatory methods, worldwide, for quantifying contaminants in drinking water, wastewater, surface and ground water, rain water, soil extracts, and other environmental sample matrixes. Official methods from organizations provide detailed instructions on the application of IC methods to determine the concentration of regulated ionic contaminants. Examples of such organizations include the U.S. Environmental Protection Agency (EPA), American Society for Testing and Materials (ASTM), International Standardization Organization (ISO), Deutsches Institut für Normung (DIN), Association Française de Normalisation (AFNOR), and Japanese Institute for Standards (JIS).

The ICS-3000 delivers a new level of performance for environmental IC analysis. Equipped with either single or dual eluent pumps, ultrastable detectors (conductivity, amperometry, UV-Vis, MS) and a versatile valving system allows the ICS-3000 to execute even the most complex IC methods with ease, precision and accuracy.

The ICS-3000 can easily perform all the traditional IC methodologies using, for example, carbonate eluents. In addition, it can also perform the more advanced methodologies using Reagent-Free™ IC (RFIC™) systems. RFIC systems bring a new level of precision and accuracy, as well as outstanding ease of operation, to IC analysis.

When configured in the dual IC mode, the ICS-3000 can easily deliver simultaneous anion and cation analyses, simultaneous multi method analyses, as well as two-dimensional (2-D) methods that significantly enhance analysis sensitivity and selectivity. Furthermore, the ICS-3000 is the ideal RFIC system to couple with mass spectrometry (MS) detection, to deliver the ultimate in sensitivity and selectivity for ion analysis.



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IC Analysis Examples

The following figures illustrate the performance of the ICS-3000 for a broad range of environmental analytical applications.

Figure 1 shows the use of the ICS-3000 RFIC system with hydroxide eluent generation and IonPac® columns for the accurate and precise determination of common anions in environmental water samples.

- This combination has approval for drinking water and interim approval for wastewater compliance monitoring, using U.S. EPA Method 300.0(A) and 300.1(A).

Figure 2 shows the use of the ICS-3000 RFIC system mode with the hydroxide-selective IonPac AG19 and AS19 ion-exchange columns, for the accurate and precise determination of common anions and oxyhalides, such as bromate, chlorite, and chlorate, in environmental water samples.

- This combination is approved for compliance monitoring using U.S. EPA Method 300.0 (A and B) and 300.1 (A and B).
- Using this combination, bromate can be determined in municipal drinking water and bottled drinking water, with a detection limit (DL) of 0.3 µg/L (ppb).
- The high sensitivity performance of this method is more than adequate to do compliance monitoring of bromate in ozonated drinking water, where the maximum contaminant limit (MCL) is 10 µg/L.

Figure 3 shows the use of the ICS-3000 with a large loop injection (500 µL) for the determination of sub-ppb level bromate in drinking water.

- The high capacity of the AS19 column, combined with the good separation of bromate from chloride, and the superior stability of the ICS-3000 pump and conductivity detector, ensures excellent low-level bromate analysis, even when analyzing high TDS drinking water samples.

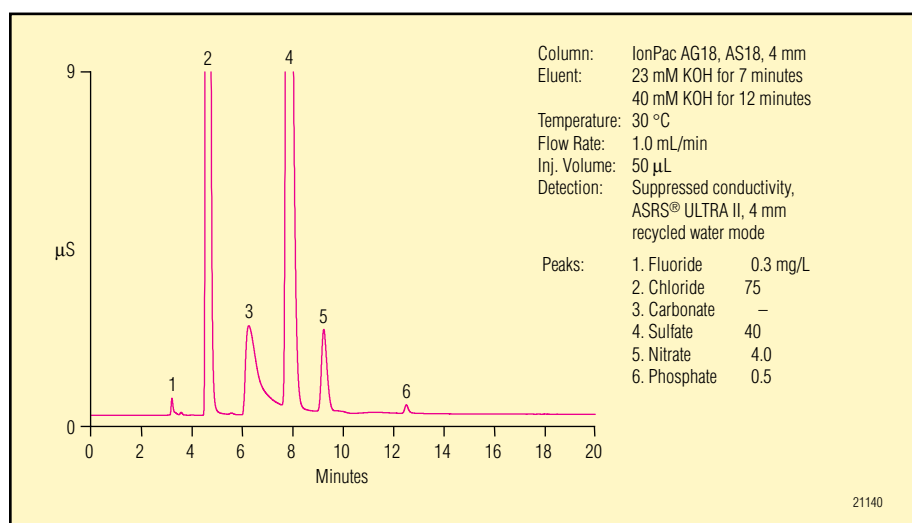


Figure 1. Determination of inorganic anions in drinking water.

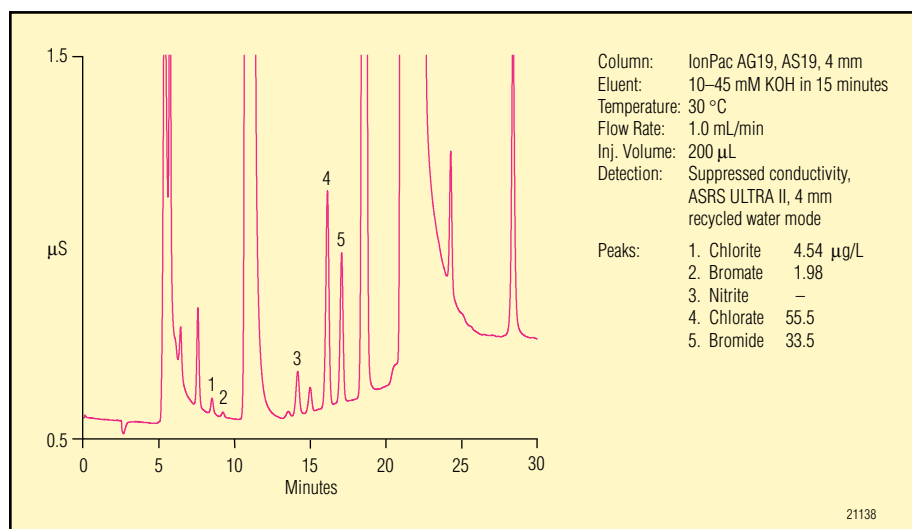


Figure 2. Determination of oxyhalides and bromide at trace-level concentrations in drinking water.

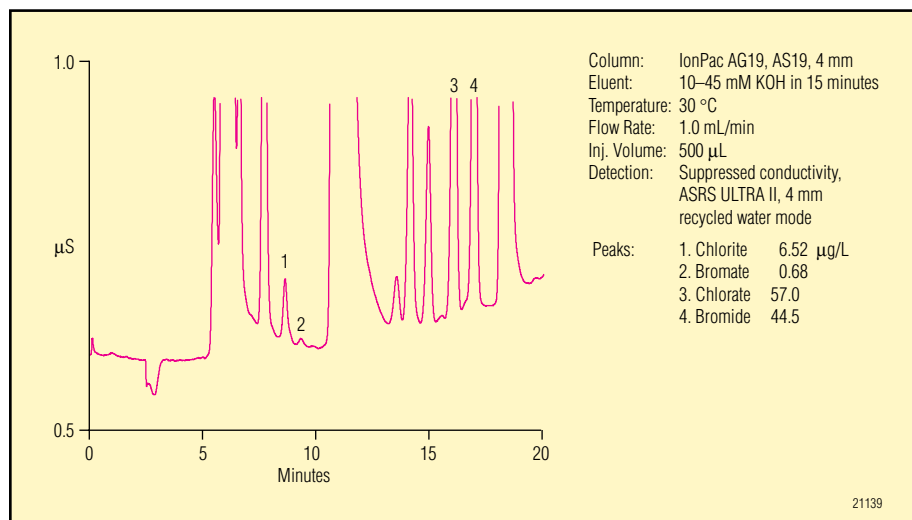


Figure 3. Determination of bromate at concentrations below 1 ppb in drinking water.

Figures 4a and 5 show the use of the ICS-3000 dual IC system to deliver greater sensitivity and selectivity for selected ion analysis (e.g., bromate) using 2-D IC. Figure 4a is a schematic diagram showing how the ICS-3000 is configured in a 2-D mode for analysis of low-level bromate in environmental water samples. Two distinct separation channels or columns are set up in the DC Detector Chromatography module of the ICS-3000 and used to automate this 2-D IC application. In this case, the first IC channel is used to separate bromate from other potentially interfering ions (e.g., chloride), using a high capacity 4-mm AS19 column, and to transfer it to a concentrator column. The concentrator column contents are then transferred to a lower capacity 2-mm AS19 column for independent separation and detection in the second IC channel. This mode of operation is best accomplished using RFIC hydroxide selective, high-capacity column chemistry to:

- Enhance sensitivity, since the large, 500 μ L sample is ultimately chromatographed using a 2-mm column.
- Enhance selectivity, since the selected ion (e.g., bromate) is chromatographed a second time with fewer matrix ions, which significantly improves peak shape and minimizes potential interferences.

The suppressed hydroxide eluent is water, which provides an ideal medium for trapping, focusing, and concentrating the ions of interest eluted from the first dimension (1-D) separation onto the concentrator column in the second dimension.

For complex applications (Figure 4a), the ICS-3000 delivers a solution: the AM Automation Manager, shown in Figure 4b. Typically, adding extra valves, plumbing and determining void volumes is cumbersome and tedious. With the AM, complex plumbing configurations, such as that used in the 2-D preconcentration for bromate (Figure 4a) are easier to implement.

Preconcentration is controlled independently with Injection Valve 1

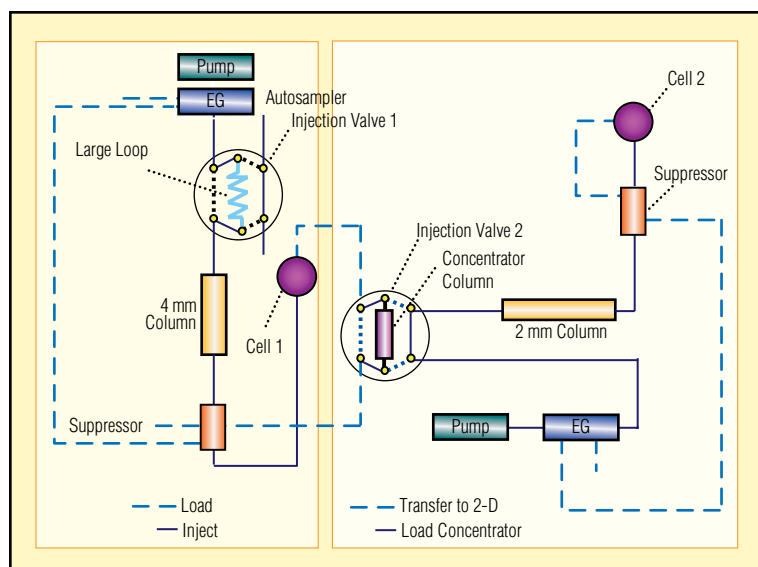


Figure 4a. Configuration of the ICS-3000 for 2-D IC determination of trace-level bromate and perchlorate in environmental water samples (see Figures 5c, 5d, and 9).



Figure 4b. The AM Automation Manager mounts inside the Detector Chromatography compartment (DC) and integrates up to two high-pressure valves, two solenoid valves, and a postcolumn heater. The entire AM mounts on a moveable slide for easy configuration for any application. All components are completely controlled through Chromeleon.

in the AM while injection is controlled by Injection Valve 2 within the DC. This allows the entire process—sample preparation, injection, separation, and analysis—to occur in the temperature-controlled DC compartment. The result is greater reproducibility for complex applications.

- By placing the AM within the DC, samples are preequilibrated for the precise temperature of the separation. This provides greater consistency from run to run.
- Control all valves with Chromeleon® software, simplifying program-

ming and eliminating the use of TTL contact closures. Each valve is individually named so that it is easy to synchronize fluidic pathways and to simplify troubleshooting.

- Use the AM as a separate injection system. With a dual-heated zone, you can run applications at different temperatures. For example, channel one can have the injection valve and column in the lower chamber at one temperature, while channel two can have the injection valve and column in the upper chamber at a different temperature. Applications with typical loop injection or preconcentra-

tion and matrix elimination can be accomplished on any channel.

Figure 5a shows the result of a 1-D separation on a 4-mm column of a 500- μ L injected sample containing 5 μ g/L bromate in a low salt matrix. Here the bromate peak is small but quantifiable.

Figure 5b shows the result of a 1-D separation from a 500- μ L injected sample also containing 5 μ g/L bromate in a high salt matrix. Here the bromate peak is not distinguishable from baseline variation.

Figure 5c shows the result of a 2-D separation of bromate from the low salt matrix that was trapped on a concentrator column and transferred to 2-D column (2-mm) for further separation and detection. The bromate peak from the secondary column is larger than from the 1-D column.

Figure 5d shows the result of a 2-D separation of bromate from the high salt matrix sample that was trapped on a concentrator column and transferred to a second column (2-mm) for further separation and detection. The bromate peak from the second column is now clearly distinguishable as compared to the 1-D column results. Furthermore, this bromate peak is the same size as that obtained from the low salt sample. Thus, this mode of operation yields greater sensitivity and minimizes interference from the high salt matrix.

This mode of operation is sometimes called heart-cutting. This technique is used to isolate unresolved solutes from one separation and place the mixture on another column that resolves the solutes. In this example, the technique is used as a form of enrichment process where the solute of interest, e.g., bromate, is non-detectable due to the presence of large amounts of chloride.

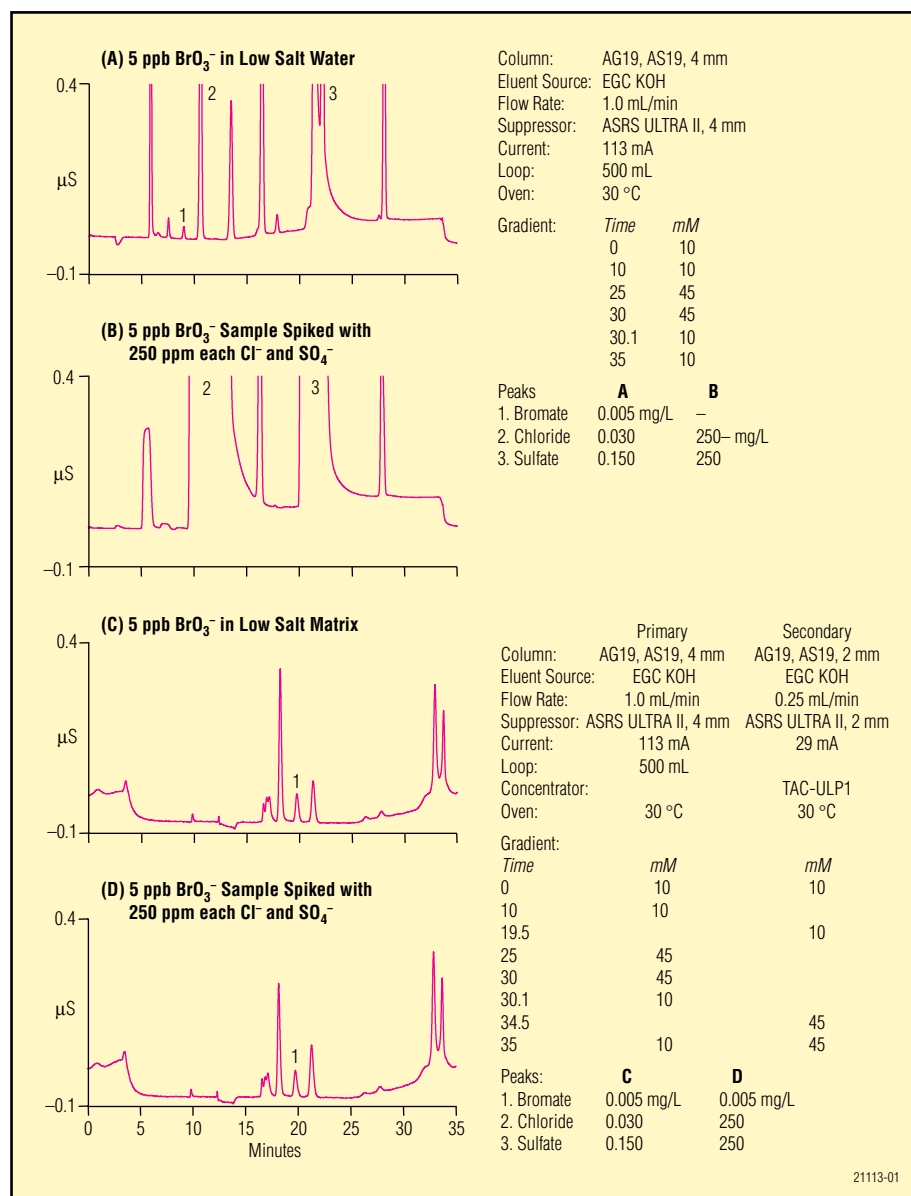


Figure 5. The ICS-3000 dual IC system delivers greater sensitivity and selectivity for selected ion analysis (e.g., bromate).

Figure 6a shows the use of the ICS-3000 in the RFIC mode with the CG16 and CS16 cation-exchange columns, for accurate and precise determination of common cations and ammonium using ASTM Method D6919-03.

- This method has been proposed by the U.S. EPA for compliance monitoring of drinking water and wastewater.

Figure 6b shows how the CS16 column gives sufficient resolution of sodium and ammonium to allow quantification of ammonium at the $\mu\text{g/L}$ level, even in the presence of high mg/L levels of sodium.

Figure 7 shows the use of the ICS-3000 dual RFIC IC system to deliver simultaneous anion and cation analyses of the same sample, using only one autosampler. In this case, the autosampler loads the injection loop of each IC channel with the same sample, so that anions and cations can be analyzed simultaneously, without cross-contamination of eluents. By providing simultaneous analysis of anions and cations, the dual ICS-3000 increases IC sample throughput without taking more benchspace than a single system.

The Chromeleon data system manages detection and quantitation and delivers a report which coordinates the anion and cation results for each sample in a convenient, easy to read format. Note that this simultaneous mode of dual channel IC operation can also be used for simultaneous small loop and large loop anion or cation analysis.

- This mode of operation is especially beneficial when analyzing samples of unknown ionic concentration levels, where some are suspected to be of low concentration and others of high concentration. For example, one IC channel with a $100\ \mu\text{L}$ loop can be calibrated from 10 ppb to 1000 ppb (1 ppm), while the other IC channel with a $10\ \mu\text{L}$ loop can be calibrated from 1 ppm to 100 ppm.

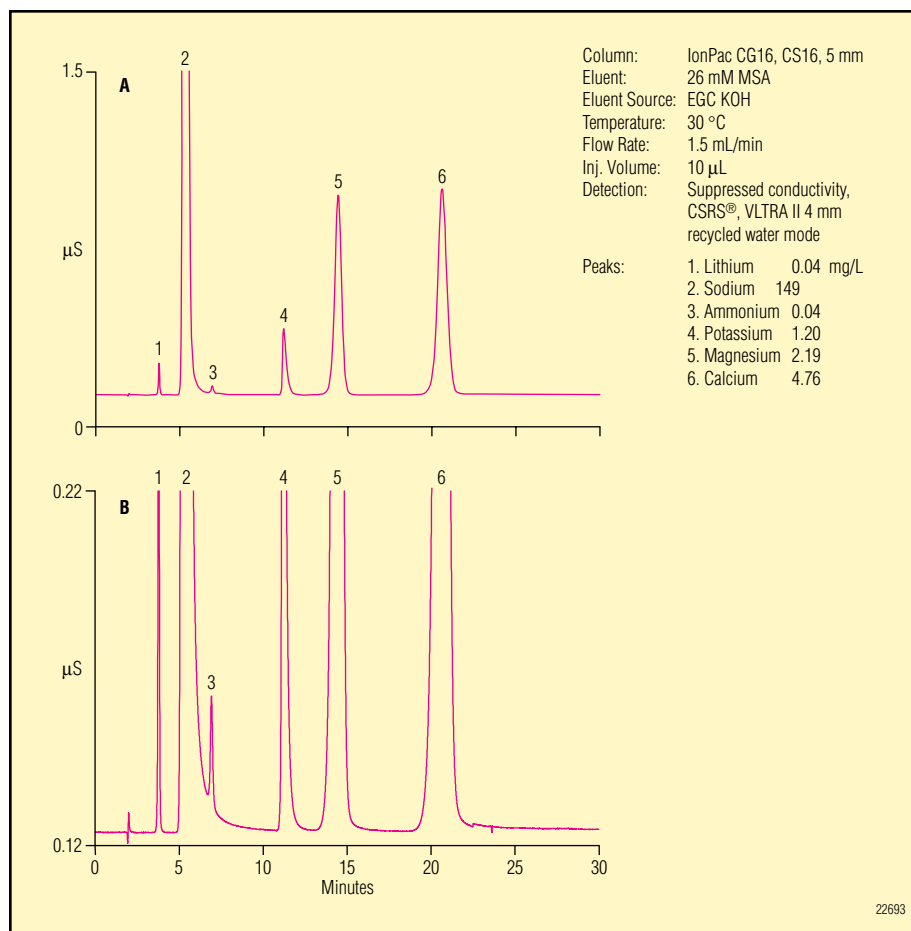


Figure 6. Determination of common alkali and alkaline earth cations and ammonium using an RFIC system.

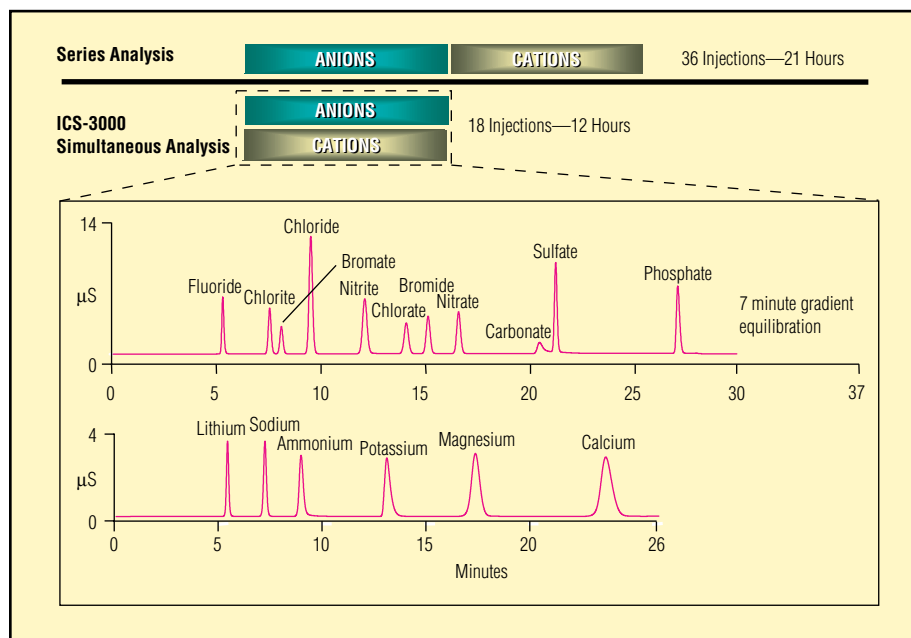


Figure 7. The AS Autosampler with simultaneous analyses provides nearly twice the throughput compared to traditional IC systems.

- Using simultaneous dual channel IC operation samples can be accurately analyzed over four orders of magnitude, i.e., 10 ppb to 100 ppm, without any advanced estimate of analyte concentrations. This virtually eliminates sample dilution and time consuming sample reruns.

Figure 8 shows the use of the ICS-3000 system for the determination of sub-ppb levels of perchlorate in drinking water by EPA Method 314.1. Typical levels of TDS in the drinking water range from 30 to 70 ppm each for chloride and sulfate, as well as residual ppm levels of carbonate. These anions can interfere with the accurate determination of ppb levels of perchlorate. Figure 8 chromatograms are shown with and without the use of the new Dionex Carbonate Removal Device (CRD). The CRD removes residual amounts of carbonate from the sample and facilitates enhanced detection and quantitation of sub-ppb perchlorate in drinking water samples. Benefits of using the ICS-3000 for perchlorate analysis by Method 314.1 include the following:

- Using this instrument/column combination, and sodium hydroxide eluent generation, 0.05 µg/L detection limits for perchlorate are achieved, even with a common anion concentration of up to 1000 mg/L, each, of chloride, bicarbonate, and sulfate.
- EPA Method 314.1 documents this method and details the use of a primary column set (AG16 and AS16) and a confirmatory column set (AG20 and AS20) to provide the

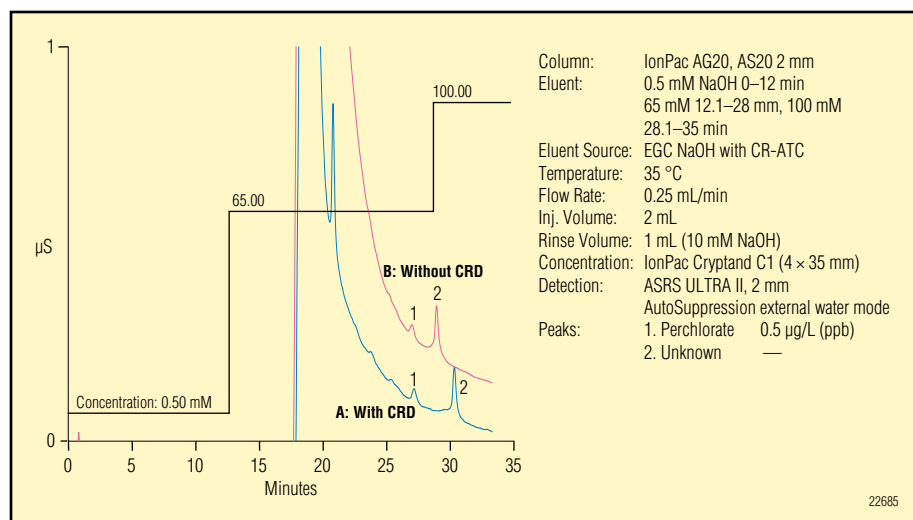


Figure 8. Chromatogram showing 0.5 ppb (µg/L) perchlorate in Sunnyvale drinking water.

- opportunity to confirm that the peak quantified is actually perchlorate.
- The ICS 3000 dual RFIC IC system allows the simultaneous determination of perchlorate, using both the primary and confirmatory columns, simultaneously from only one injection and one autosampler.
- Sequential analysis increases flexibility by using a diverter valve with the AS autosampler to deliver any sample to any channel, one at a time on a first come, first served basis. With this capability, you can introduce the same sample or two different samples to two independent eluent streams.
- Whether you choose simultaneous or sequential injection, one autosampler is shared between two independent systems.
- Combine detectors for conductivity, electrochemical, or UV detection on the same sample or different samples. Virtually any analysis combination is possible—anions/cations, anions/trace metals, etc.

As shown previously in Figure 4, the ICS-3000 can be configured to perform 2-D IC for enhanced detection sensitivity of low-level environmental contaminants such as perchlorate. Figure 9 demonstrates the effectiveness of the ICS-3000 setup in the 2-D mode with an IonPac AS20 4-mm column in the first dimension and a 2-mm AS20 column in the second dimension. Chromatogram 9A shows the analysis of 0.5 µg/L perchlorate in a simulated high TDS matrix of 1000 ppm each of chloride, carbonate, and sulfate. Figure 9B shows the same analysis in reagent water. The perchlorate peak areas in these two analyses are virtually identical. Further, interference and tailing from the other high-concentration matrix anions is significantly reduced. This demonstrates the power of 2-D IC on the ICS-3000 for detecting and quantifying sub-ppb levels of perchlorate in challenging matrices.

What the ICS-3000 Means to You

The ICS-3000 brings a new level of precision, accuracy, versatility, and ease of operation to environmental ion analysis. Dual pump and dual IC configurations add new capability to IC methodology. Heart-cutting methods, simultaneous method analysis and postcolumn reagent addition methods are easily and reliably performed using the new ICS-3000.

The robust ICS-3000 delivers a whole new level of productivity, power, and sensitivity to the environmental analysis laboratory.

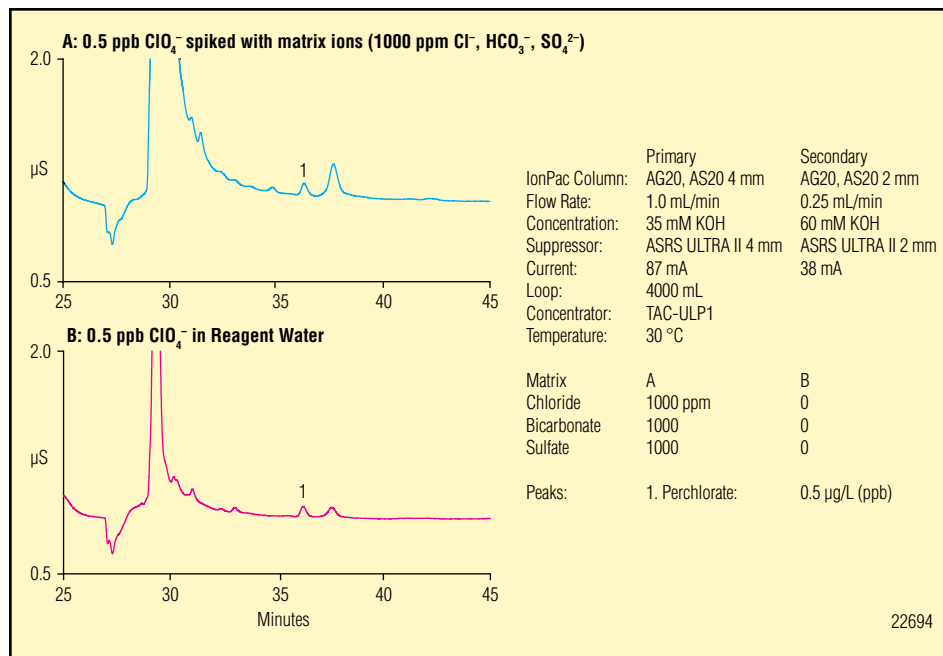


Figure 9. Two-dimensional perchlorate analysis with the IonPac AS20 column.

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