

The Determination of Trace Anions in Concentrated Phosphoric Acid

INTRODUCTION

The determination of trace anions in phosphoric acid is hampered by a large excess of phosphate ion. Chloride determination at 0.1 mg/L (ppm) in 85% (w/w) phosphoric acid represents a concentration ratio of 1:10⁶ (chloride to phosphate). Diluting the concentrated sample overcomes the problem of a large concentration of the interfering matrix ion, but lacks the required sensitivity for the contaminant ions of interest. An improved method for determining trace anions in concentrated phosphoric acid has been developed to overcome this problem.^{1,2} Trace inorganic anions are separated from the high concentration of phosphate by an ion-exclusion separation prior to an ion-exchange separation.

This Technical Note describes the theory, set up, and analytical procedure for the determination of chloride, nitrate, and sulfate at sub-mg/L (ppm) levels in 85% (w/w) phosphoric acid.

SUMMARY OF THE METHOD

An IonPac[®] ICE-AS6 ion-exclusion column is used to separate the analyte ions from an excess of phosphate matrix ions. A selected fraction from the ion-exclusion separation is “cut” and sent to a 4-mm IonPac AS11-HC anion-exchange concentrator column. The concentrated ions are then eluted onto a 2-mm IonPac AS11-HC column set, where the anions of interest are separated and detected by suppressed conductivity.

EQUIPMENT

Dionex DX-500 Ion Chromatography system consisting of:

- GP50 Gradient Pump, microbore configuration
- CD20 Conductivity Detector with a temperature-controlled conductivity cell (DS3)
- LC20 Enclosure with 2 Rheodyne valves, PEEK, rear loading

Dionex RP-1 single piston pump

Pressurizable Reservoir Chamber (P/N 37053)

(Three) 4-L Plastic bottle assemblies, 2 for external water and 1 for rinse solution (P/N 39164)

1 O-ring, Teflon encapsulated, for rinse solution bottle (P/N 43523)

2 O-rings Teflon encapsulated, for Reservoir Chamber (P/N 055703)

1 Air pressure gauge, 0–171 kPa (0–25 psi) (for external water)
305 cm (120 in.) of green PEEK tubing, diameter of 0.75 mm (0.030 in.), to connect columns and make a 200- μ L sample loop

Teflon bottles as sample containers (VWR, P/N 16071-041 or Nalge, P/N 1600-0004 for 125 mL narrow mouth bottles)

PeakNet Chromatography Workstation

Columns

IonPac AG11-HC guard column, 2 mm (P/N 52963)

IonPac AS11-HC analytical column, 2 mm (P/N 52961)

IonPac AG11-HC concentrator column, 4 mm (P/N 52962)

IonPac AG10 as trap column, 4 mm (P/N 43119)

IonPac ICE-AS6 analytical column, 9 x 250 mm (P/N 46023)

Anion Self-Regenerating Suppressor[®] (ASRS[®]),
2 mm (P/N 53947)

REAGENTS AND STANDARDS

Deionized water (DI H₂O), Type I reagent grade,
17.8 MΩ-cm resistance or better

Sodium hydroxide, 50% (w/w) aqueous solution
(Fisher Scientific)

Chloride standard 1000 mg/L, 100 mL (Dionex, P/N 37159)

Sulfate standard 1000 mg/L, 100 mL (Dionex, P/N 37160)

Nitrate standard 1000 mg/L, 100 mL (Ultra Scientific,
VWR, P/N ULICC-004)

CONDITIONS

Ion Exclusion

Column: IonPac ICE-AS6
Trap Column: IonPac AG10, 4 mm
Eluent: Deionized water
Flow Rate: 0.50 mL/min

Ion Chromatography

Analytical Column: IonPac AS11-HC, 2 mm
Guard Column: IonPac AG11-HC, 2 mm
Concentrator
Column: IonPac AG11-HC, 4 mm
Eluent: 20 mM sodium hydroxide, step to
200 mM sodium hydroxide
Flow Rate: 0.38 mL/min
Sample Volume: 200 µL
Detection: Suppressed conductivity, ASRS,
AutoSuppression® external water
mode
Suppressor
Current Setting: 300 mA
Expected System
Backpressure: 16.5 MPa (2400 psi) (with concentrator
column in line)
Expected
Background
Conductivity: 2–3 µS

PREPARATION OF SOLUTIONS AND REAGENTS

Eluent Solutions

20 mM Sodium hydroxide (IC eluent)

Dilute 1.60 g of 50% (w/w) sodium hydroxide with degassed, deionized water (having a specific resistance of 17.8 MΩ-cm or greater) to a final weight of 1000 g in the eluent bottle. Avoid the introduction of carbon dioxide from air.

200 mM Sodium hydroxide (IC eluent and AG10 trap column regeneration)

Dilute 16.00 g of 50% (w/w) sodium hydroxide with degassed, deionized water (having a specific resistance of 17.8 MΩ-cm or greater) to a final weight of 1000 g in the eluent bottle. Avoid the introduction of carbon dioxide from air.

Standard Solutions

Stock standard solution (1000 mg/L)

Use Dionex or commercially available 1000 mg/L ion standard solution.

Working standard solution (1 mg/L)

To prepare a mixed working standard solution, combine 1.00 mL of each anion stock solution with deionized water and dilute to a final volume of 1000 mL.

CALIBRATION

Prepare calibration standards at a minimum of three concentration levels by diluting the working standard. Select a range similar to the expected concentrations in the samples. The method of standard addition (adding one or more increments of a standard solution to sample aliquots of the same size) can be used to minimize the effect of the concentrated acid matrix on the measured conductivity of the analytes of interest.³

To minimize dilution of the concentrated acid sample, it is recommended that small aqueous aliquots be used for the method of standard addition. Each of these additions will have a consistent volume while the concentration for the anions of interest varies. A convenient sample size for spiked standards in 85% phosphoric acid is 20 mL (34 g) with a 0.020 mL aqueous spike. This represents a dilution of 0.1% (0.020 mL spike/20 mL sample = 0.1% dilution). To minimize introducing contamination through sample handling, the concentrated acid can be dispensed directly into the sample container, by weight, on a top-loading balance. Table 1 illustrates how aqueous standards are diluted and prepared. The following formula can be used to calculate concentrations in mg/L for dilutions:

$$\begin{aligned} &(\text{Conc. of stock solution, mg/L}) * (\text{Vol. of stock solution, mL}) = \\ &(\text{Conc. of dilute standard, mg/L}) * (\text{Vol. of dilute standard, mL}) \end{aligned}$$

Table 1 Method of standard additions for concentrated phosphoric acid

	Concentration of stock standard (mg/L)	Amount of stock to make a 10-mL final volume of working standard in deionized water (mL)	Concentration of working standard (mg/L)	Concentration of 20 mL 85% phosphoric acid when 20 μ L of working standard is added (mg/L)
Chloride	1000	0.500	50	0.050
Sulfate	10000	2.00	2000	2.0
Nitrate	1000	1.00	100	0.10

IONPAC AG10 TRAP COLUMN REGENERATION

The AG10 column must first be regenerated. Monitoring the blank will indicate when regeneration is necessary. Typically, monthly regeneration is necessary, but it will depend on the quality of the deionized water and usage rate of the instrument. Increased contamination in the water blank indicates that the AG10 needs to be regenerated. The procedure is as follows:

1. Pump 200 mM sodium hydroxide through the AG10 at 1.0 mL/min for 50 minutes.
2. Follow with a rinse of deionized water at the same flow rate for 20 minutes.

DISCUSSION OF METHOD

This method addresses the challenge of determining trace concentrations of contaminant ions such as nitrate, chloride, and sulfate in a matrix composed of a high concentration of phosphate ion. This is accomplished in two steps: an ion-exclusion (ICE) pre-separation followed by injection of a portion of the ICE separation to an ion chromatographic (IC) separation.

The ion-exclusion mechanism separates ionized species from nonionized or weakly ionized species. This occurs because of a negatively charged hydration shell on the stationary phase surface called the Donnan membrane.⁴ Figure 1 illustrates the application of the ICE mechanism to the separation of 85% concentrated phosphoric acid that was injected onto an ICE-AS6 ion-exclusion column. This chromatogram is a measurement of the unsuppressed conductivity response for the ICE separation. The strong acid ions, such as nitrate, chloride, and sulfate, are excluded and elute as a small peak at 12 minutes. The weakly ionized phosphate matrix ions are retained and elute as a large peak. This separation is not applicable to dilute phosphoric acid samples because dilute phosphoric acid is partially ionized.

A series of schematics (Figures 2–5) illustrates the operation of the chromatography hardware. The concentrated phosphoric acid sample is loaded via a pressurized

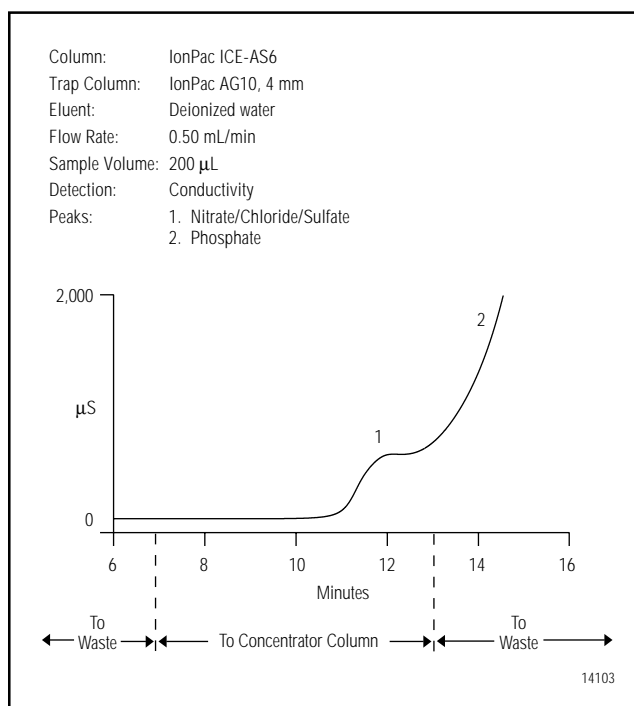


Figure 1 Ion exclusion separation of trace anions in 85% phosphoric acid.

reservoir into the 200- μ L sample loop (Figure 2). Helium at 34.5 kPa (5 psi) is used to push sample out of the sample container into the sample loop at a flow rate of 0.1 mL/min (6 mL/hr). This technique ensures that a representative sample of the concentrated phosphoric acid sample is loaded into the sample loop. It is important to pass at least 4 loop volumes through the sample loop to ensure reproducible sampling.⁵ While the 40-minute IC separation is taking place, approximately 4 mL of sample will have been pushed through the sample loop with the pressurized reservoir.

The concentrated phosphoric acid sample is then delivered with the high-purity water carrier stream to the IonPac ICE-AS6. An AG10 is placed after the RP-1 pump to act as an anion trap column for the deionized water. Any contaminants present in this water will impact the quality of the blank.

The first portion of the ICE separation from 0.0 to 7.0 minutes is sent to waste (Figure 3). Then, the concentrator column is placed in-line with the ICE column and the portion from 7.0 to 13.0 minutes is captured on the concentrator column (Figure 4). After 13.0 minutes the 4-mm AG11-HC concentrator column is placed in-line with the 2-mm AS11-HC analytical column set and the concentrated ions are separated (Figure 5). This time window should ensure the concentration of all the nitrate, chloride, and sulfate with minimal concentration of phosphate.

The IC separation utilizes the AS11-HC with an isocratic eluent of 20 mM sodium hydroxide. The high capacity of the AS11-HC allows injection of these relatively concentrated samples without overloading. Figure 6

shows a separation of the common anions with the 4-mm AS11-HC under standard conditions. The attractive feature of this separation is that phosphate elutes last.

A 2-mm microbore column was chosen because it has a 4-fold increase in mass sensitivity over the standard bore column. This facilitates faster loop loading because smaller sample amounts are required. The microbore format also offers low eluent consumption as well as less waste generation. An IonPac AG11-HC ion exchange column was used as the concentrator column in the 50 x 4 mm format instead of 50 x 2 mm because the 4-mm column had four times more capacity than the 2-mm column and lower back pressure at the microbore flow rate. No significant degradation in separation efficiency was

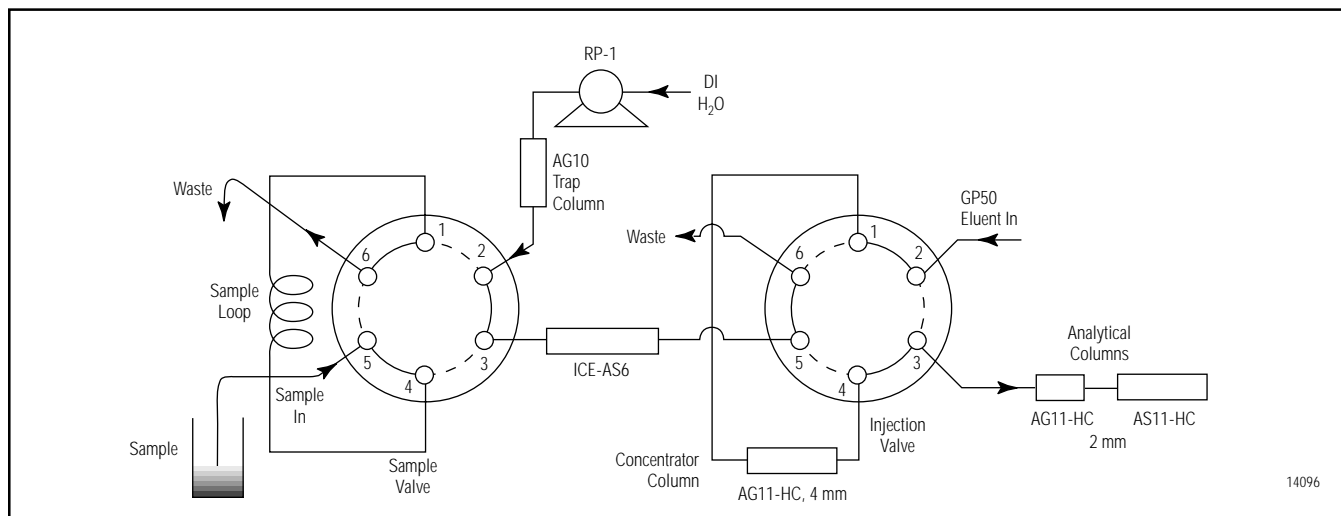


Figure 2 Loading the sample loop.

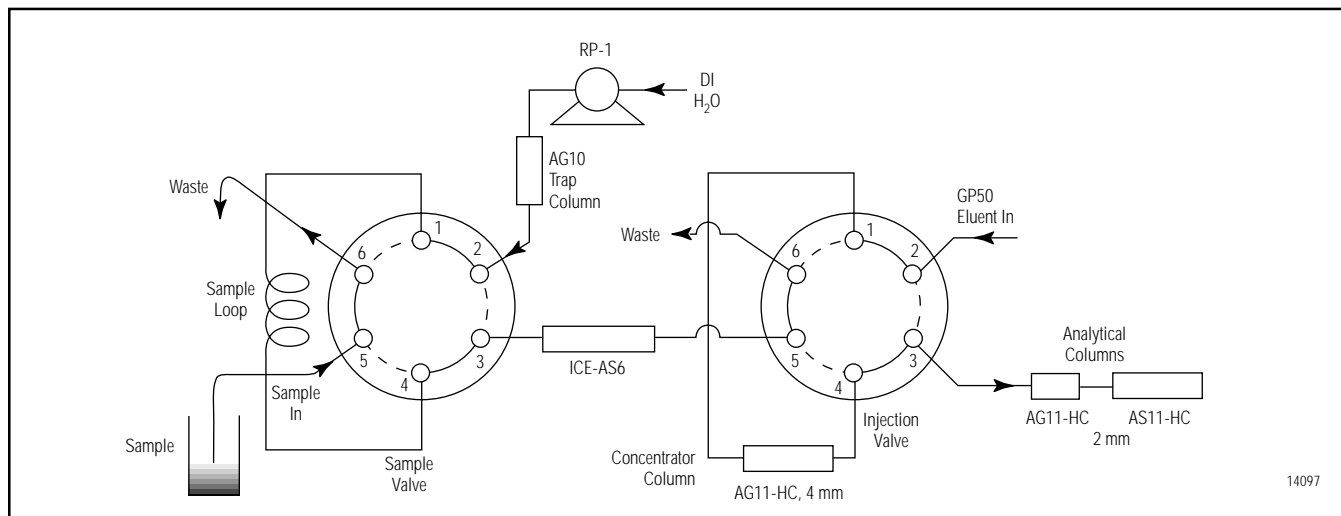


Figure 3 First portion of the ICE separation (time 0.0–7.0 min).

4 The Determination of Trace Anions in Concentrated Phosphoric Acid

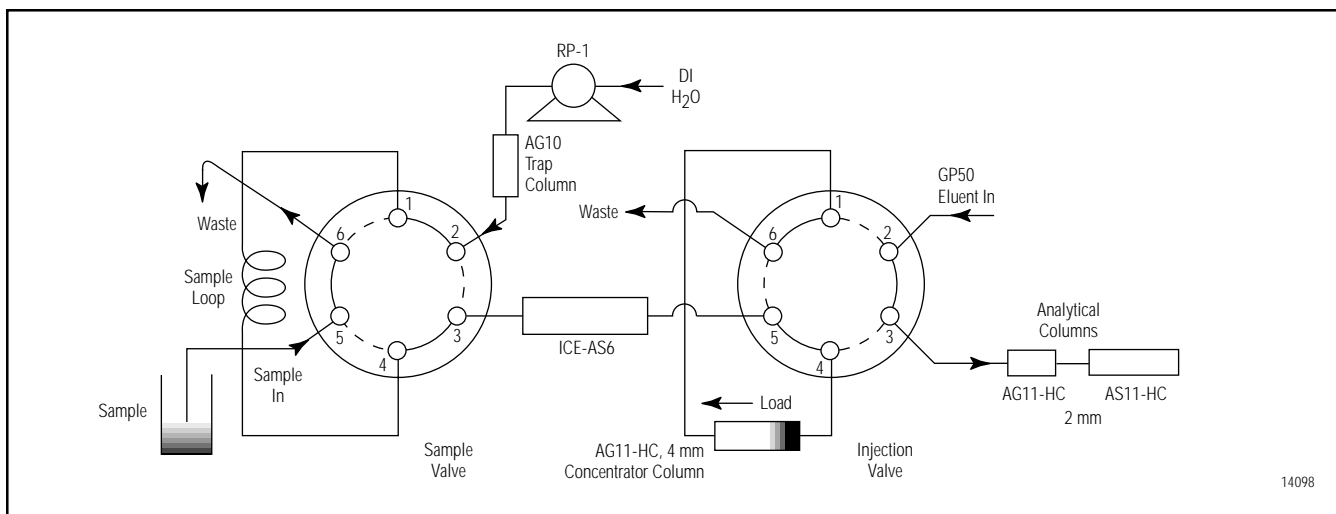


Figure 4 Concentrating the "cut" portion from the ICE separation (time 7.0–13.0 min).

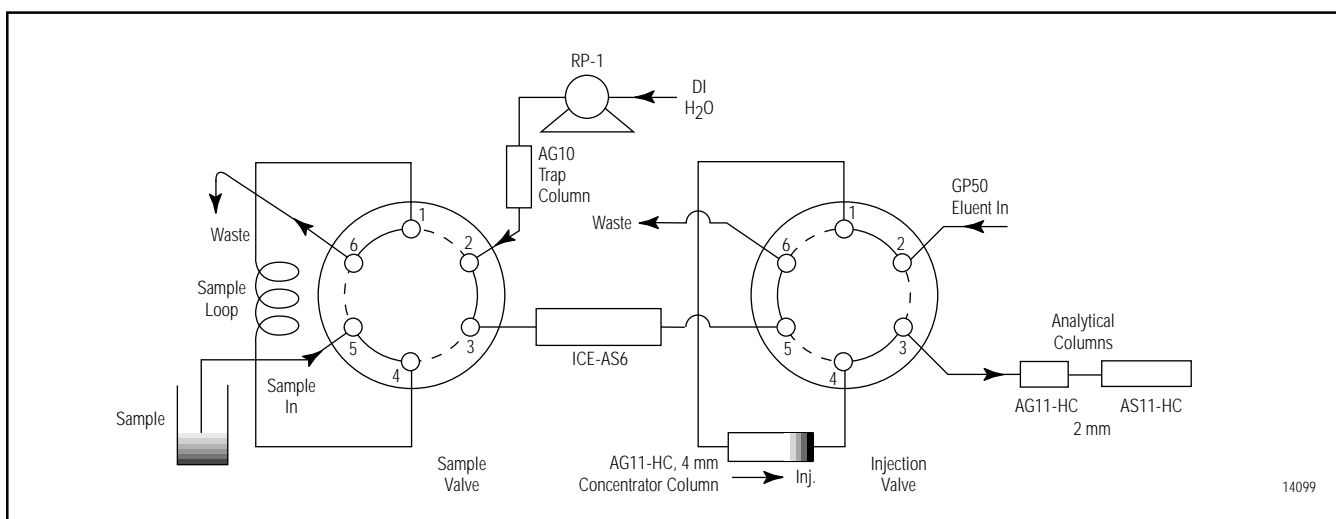


Figure 5 Separating the retained ions.

observed when coupling a 4-mm concentrator column with a 2-mm analytical column set.

For this method the AS11-HC standard conditions were modified. The eluent strength was reduced from 30 mM to 20 mM sodium hydroxide to optimize the separation of the trace anions from the excess of phosphate matrix ions. The method was run at ambient temperature.

During the IC separation, the pressurized vessel is filling the sample loop for the next analysis. The deionized water rinses the ICE-AS6 and associated tubing to ensure there is no contamination from the previous sample.

After the phosphate has eluted from the column, the eluent concentration is stepped from 20 to 200 mM sodium hydroxide for 5 minutes. This ensures that the column is

rinsed of residual phosphate. The method returns the system to equilibrate at 20 mM sodium hydroxide for the next injection. If this cleanup step is not used, phosphate will continue to build up on the analytical column and cause a gradual decrease in retention time for phosphate and the other analytes.

SYSTEM PREPARATION AND TEST

Refer to the system configuration schematics in Figures 2–5 and Table 2, which summarize the types and lengths of tubing required for system configuration. The chromatography hardware is divided into two parts: the ion-exclusion pretreatment portion with the ICE-AS6 and the IC analysis portion with the AS11-HC.

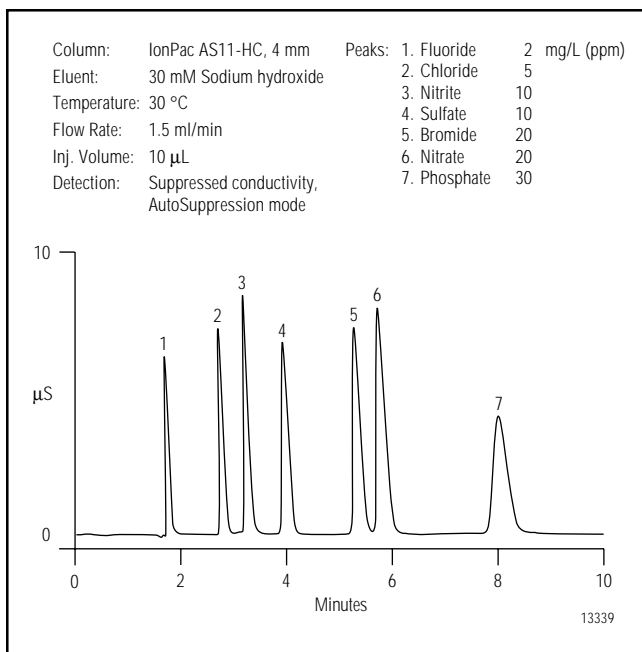


Figure 6 Isocratic separation of the common anions.

IC SYSTEM

1. Prepare the ASRS by following the Quickstart instructions (Dionex Document 031368-01) included with the Instructions and Troubleshooting Guide for the ASRS.
2. Install the 2-mm AG11-HC and AS11-HC column set by using the red 0.125-mm (0.005 in.) tubing. To minimize dead volume, use the smallest possible lengths of tubing and ensure that the ends of the tubing are smooth and level.
3. Construct a 5-µL sample loop by cutting a 9.9-cm (3.9 in.) portion of the black 0.25-mm (0.010 in.) PEEK tubing.
4. Install this loop in place of the 4-mm AG11-HC concentrator column between ports 1 and 4 of the injection valve in the IC analysis system.
5. Install the ASRS and configure it in the external water mode as described in the SRS manual.
6. Establish eluent flow through the 2-mm AG11-HC and AS11-HC analytical column set. The expected background conductivity is 2–3 µS. (Note: For trace analysis, it will take at least 5 hours for the system to achieve a stable background conductivity.)

7. Verify proper operation of the IC portion of the system by injecting a low-level ppm standard to replicate the column test chromatogram.
8. Remove the 5-µL sample loop and install the 4-mm AG11-HC concentrator column. Make sure that the arrow indicating flow on the column is pointed from port 1 to port 4 and that the tubing length connecting the outlet of this column and port 4 is as short as possible.
9. Configure the IC valve so that the 4-mm AG11-HC concentrator column is in-line with the 2-mm AG11-HC and AS11-HC analytical column set. Check for leaks. The expected system backpressure for these three columns at 0.38 mL/min is ~2,400 psi (16.5 MPa).

ION-EXCLUSION SAMPLE PRETREATMENT SYSTEM

This section describes the preparation of the pretreatment portion of the system. It is important that the same type and length of tubing as described in Table 2 be used to successfully perform this analysis. Changes in tubing length will result in a different “cut” from the ICE-AS6 column being delivered to the AS11-HC concentrator column.

Table 2 Details of tubing configuration for trace anions in phosphoric acid

Connection Points	Tubing Description	Length (cm)	Remarks
ICE exit to Port 5	Green 0.75 mm (0.030 in.)	30	
ICE input to Port 3	Green 0.75 mm (0.030 in.)	70	
Port 1 to Port 4	Green 0.75 mm (0.030 in.)	44	200-µL sample loop
4-mm AG11-HC to Port 4	Red 0.125 mm (0.005 in.)	3	Should be as short as possible
4-mm AG11-HC to Port 1	Black 0.25 mm (0.010 in.)	25	
Port 3 to analytical column	Red 0.125 mm (0.005 in.)	3	Should be as short as possible

- Cut a 44-cm (17 in.) portion of the 0.75-mm (0.030 in.) PEEK green tubing to make a 200- μ L sample loop and install this loop between port 1 and 4 of the sample valve.
- Prepare the AG10 trap column according to the directions in the section titled "IonPac AG10 Trap Column Regeneration." (Caution: Before the AG10 is installed in the system it is important that the sodium hydroxide solution used for storage or cleaning be completely rinsed away. The ICE-AS6 is not compatible with sodium hydroxide eluents.)
- The entire pathway from the RP-1 pump to port 5 of the IC valve is plumbed with the 0.75 mm (0.030 in.) PEEK green tubing. Install the AG10 at the exit port of the RP-1 pump.
- Install the ICE-AS6 column using a 70-cm piece of green tubing between the exit of the ICE-AS6 and port 5 of the injection valve. Use a 30-cm portion of green PEEK tubing between port 3 of the sample valve and the input of the ICE-AS6.
- Check to see that there is about 34.5 kPa (5 psi) of head pressure on the incoming deionized water that feeds the RP-1 pump.
- Connect the exit port of the reagent reservoir to port 5 of the sample valve with the 0.75-mm (0.030 in.) green tubing.
- Configure a waste line from port 6 of the sample valve with the green tubing.
- Connect the reagent reservoir to helium pressure of about 34.5 KPa (5 psi).
- Begin with a container filled with deionized water as a sample to rinse the sample lines of any trace contamination.
- Set a flow rate of 0.50 ± 0.02 mL/min for the RP-1 pump by adjusting the dial on the pump. This should be measured with the 4-mm AG11-HC concentrator column out of line. Measure the flow rate by collecting the waste coming out of port 6 of the IC valve. It is critical for the success of this method that the flow rate be consistent.
- Pump deionized water at 0.50 mL/min through the ICE-AS6 to waste without the 4-mm AG11-HC in line for 1 hour. This will remove the 0.4 mM heptafluorobutyric acid storage solution. Sulfate is usually found in the blank. Typical initial blank sulfate values will be ~ 200 μ g/L.
- The ICE-AS6 can be further conditioned by rinsing it with 100 mM phosphoric acid for 2 hours at 0.50 mL/min followed by a 1-hour rinse with 17.8 M Ω -cm deionized water. This will reduce the sulfate blank to 150 μ g/L or less. Continue to monitor the blank, especially when starting up the system after it has been idle for more than two days.

SYSTEM OPERATION

After all aspects of the instrumentation have been prepared, the system is ready for analysis.

- Load the PeakNet method shown in Table 3.
- Fill the 200- μ L sample loop with deionized water. Use helium gas pressure to push the deionized water sample using the reagent reservoir (Figure 2).
- Analyze a blank by loading deionized water as the sample. It may take several runs until the system has been rinsed of contamination.
- After an acceptable blank has been established, the system is ready for analysis.

Table 3 PeakNet method for the analysis of concentrated phosphoric acid

Total Time (min)	ICE Time (min)	IC Time (min)	Injection Valve	Column Valve	%A 200 mM NaOH	%B 20 mM NaOH	Figure	Comment
Init			Inject	A	0	100	2	Load the sample loop
0.00			Inject	A	0	100	2	
8.00	0.00		Inject	B	0	100	3	Begin ICE separation
15.00	7.00		Load	B	0	100	4	Send cut portion from ICE separation to AG11-HC, 4 mm
21.00	13.00	0.00	Inject	B	0	100	5	Begin IC separation. Concentrator column in-line.
45.49		24.49	Inject	A	0	100	5	
45.50		24.50	Inject	A	100	0	5	Step to 200 mM sodium hydroxide for cleaning
50.50		29.50	Inject	A	100	0	5	End 200 mM sodium hydroxide
50.51		29.51	Inject	A	0	100	5	Equilibrate at 20 mM sodium hydroxide
61.00		40.00	Inject	A	0	100	5	

A = Inject

B = Load

5. Exercise caution when handling concentrated phosphoric acid. Consult the applicable Material Safety Data Sheet (MSDS) for specific details about protective equipment, reactivity, storage, disposal, and health effects.
6. Concentrated phosphoric acid, 85% (w/w), can be loaded directly into the 200- μ L loop by the reagent delivery module. At this concentration the phosphoric acid is viscous and moves slowly to fill the sample loop. Ensure that the loop has had enough sample pass through by collecting the liquid that exits port 6 of the sample pretreatment valve. A good practice is to load at least 4 loop volumes. The method is set up so that the reagent reservoir pushes sample into the sample loop during the IC separation. With a pressure of 34.5 KPa (5 psi), 4 mL of concentrated phosphoric acid will have been passed through the sample loop in 40 minutes.
7. Ensure that the RP-1 pump is consistently delivering 0.50 ± 0.02 mL/min. Figure 7 illustrates what happens when the flow rate is faster or slower. At the slower flow rate, not enough of the sample is cut from the ICE separation, resulting in incomplete recovery of the anions of interest. At the faster flow rate, too much of the ICE separation is cut, resulting in the phosphate peak almost obscuring the analytes of interest.
8. Other factors will also affect the quality and consistency of the ICE pre-separation. Changing the cut time window specified in the method (7.0 to 13.0 minutes) will impact the amount of analyte and matrix ions that are delivered to the concentrator column. Varying the sample volume will also affect the character of the ICE separation. For example, doubling the sample volume from 200 to 400 μ L increases the sensitivity for trace determinations. Method development will be needed to ascertain the impact of any changes from the specified method.
9. Quantifying the levels of anions in phosphoric acid is best accomplished by the method of standard additions. This involves adding one or more increments of a standard solution to sample aliquots of the same size (see the Calibration section).

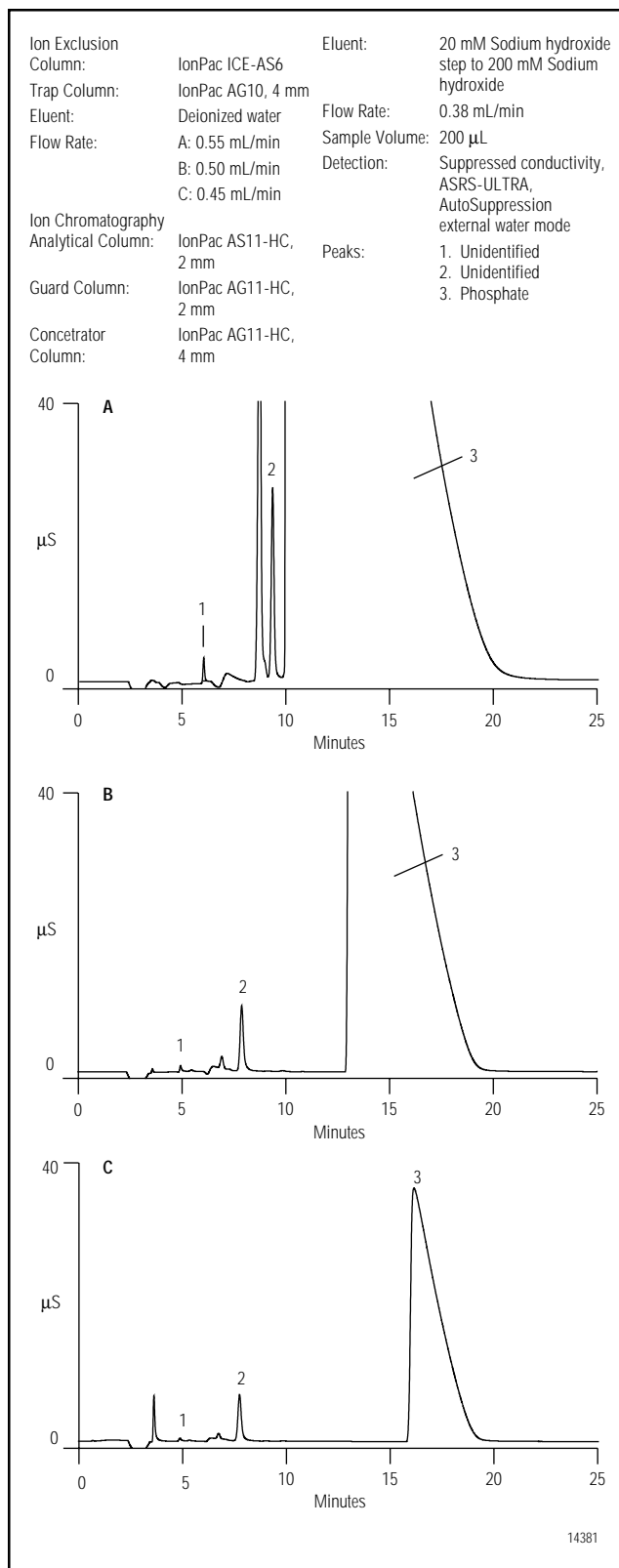


Figure 7 Effect of ICE eluent flow rate on IC separation.

RESULTS AND DISCUSSION

A representative blank is shown in Figure 8. It was found that after concentrated phosphoric acid had been in the sample loop pathway, several runs of deionized water were required to completely rinse away the high concentration of phosphate matrix ions. A blank was established after seven replicate runs yielded reproducible results. These levels, quantified based on a calibration curve for these ions in deionized water, are below the expected concentrations for high-purity grade concentrated phosphoric acid. The anion values, of this deionized water blank, were subtracted from the levels found in the concentrated phosphoric acid samples. A small amount of phosphate was detected in the blank, as carryover from previous injections. This will not significantly impact sample analysis.

A chromatogram for the analysis of 85% (w/w) phosphoric acid is shown in Figure 9. The large phosphate matrix (peak 5) is well separated from the anions of interest. Chloride determination was hampered by a dip before the peak. A feature in the PeakNet chromatography software called “void treatment” was utilized to reliably quantitate this peak. A detail of the chloride peak is shown in Figure 10. The peak that starts at 29 minutes is a result of the step to the higher eluent concentration. Any residual phosphate left in the column is eluted with this high eluent concentration.

To verify proper quantification of analytes in the phosphoric acid matrix, increasing concentrations of chloride, nitrate, and sulfate were added into 85% phosphoric acid. Spikes of 30, 100, and 300 $\mu\text{g/L}$ of chloride; 100, 300, and 1000 $\mu\text{g/L}$ of nitrate; and 300, 1000, and 3000 $\mu\text{g/L}$ of sulfate yielded coefficients of determination (r^2) values greater than 0.99.

Based on this calibration curve, a spike of 50 $\mu\text{g/L}$ chloride, 2000 $\mu\text{g/L}$ sulfate, and 100 $\mu\text{g/L}$ nitrate yielded recoveries between 84 and 111%. These values are within the SEMI (Semiconductor Equipment and Materials International) recommended guidelines of 75–125% recovery at 50% of the specified maximum limit of impurity. Table 4 summarizes the spike/recovery data.

To determine method precision, a sample of a high-purity grade 85% phosphoric acid was analyzed by this method. For $n=7$, a relative standard deviation (RSD) of less than 6% was obtained for 34 $\mu\text{g/L}$ chloride and 730 $\mu\text{g/L}$ sulfate. Nitrate levels were determined to have an average concentration of 16 $\mu\text{g/L}$ with an RSD of 21%. At these low nitrate concentrations, partial coelutions of other analytes with nitrate cause a higher variability in the nitrate peak integration.

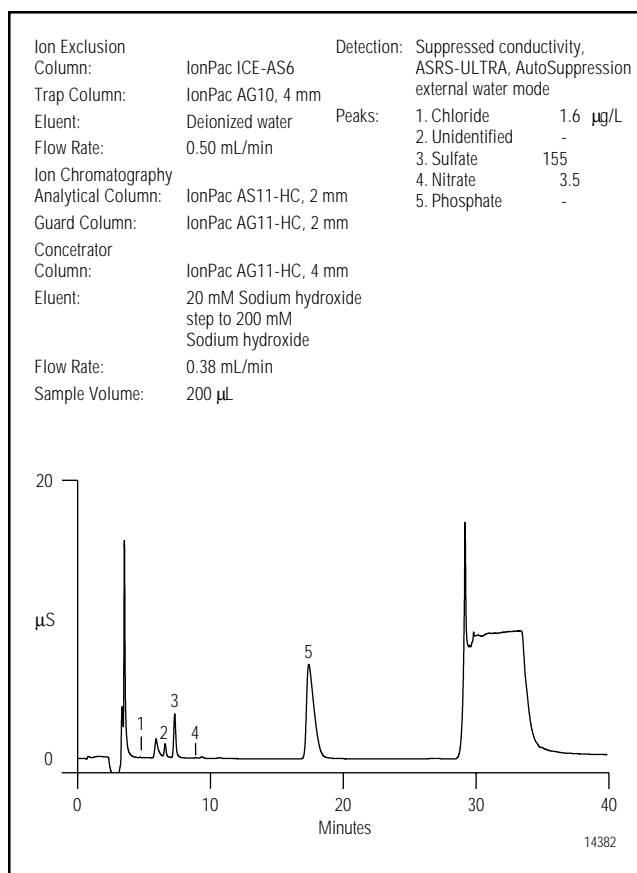


Figure 8 Representative blank.

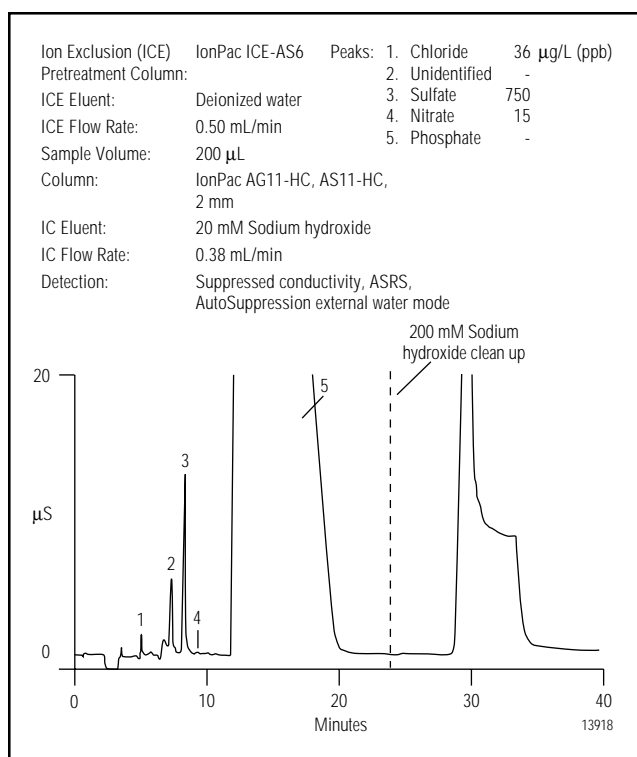


Figure 9 Determination of trace anions in high purity 85% phosphoric acid.

Method detection limits (MDLs) were calculated using the standard deviation of seven replicate injections multiplied by the Student's t-value for the 99.5% confidence level. MDLs for chloride, sulfate, and nitrate are in the low µg/L (ppb) range. The MDLs for this method are substantially below the maximum limit of impurity guidelines for phosphoric acid⁶ established by SEMI for the purest grade of phosphoric acid, as shown in Table 5.

PRECAUTIONS

Exercise caution when handling concentrated phosphoric acid. Consult the Material Safety Data Sheet (MSDS) for more specific details about protective equipment, reactivity, and health effects. Use only the highest quality deionized water for the preparation of standards and eluents. Any ionic contamination present in the deionized water will adversely affect results. Teflon containers are recommended for holding the concentrated acid samples for delivery to the sample loop. Containers should be soaked for at least 24 hours with 17.8 MΩ-cm deionized water prior to use. It is good practice to dedicate all containers for trace analysis and keep them filled with deionized water when not in use.

Method success depends on maintaining a consistent flow rate of deionized water from the RP-1. Verify that the flow rate is 0.50 ± 0.02 mL/min. If the deionized water container feeding the RP-1 pump is not pressurized to at least 34.5 KPa (5 psi), the pump may be prone to losing prime.

Do not leave concentrated phosphoric acid in the sample loop and sample inlet lines for more than 6 hours. The PEEK tubing can degrade after extended contact time with the concentrated acidic sample.

Table 4 Spike recovery of trace anions in 85% phosphoric acid

Anion	Phosphoric Acid Blank (µg/L ± SD)*	Spike (µg/L)	Found-Blank (µg/L ± SD)	Recovery (%)
Chloride	34 ± 2.1	50	51 ± 2.1	102
Sulfate	730 ± 40	2000	2220 ± 44	111
Nitrate	16 ± 3.4	100	84 ± 4.3	84

* corrected for system blank
For n = 7

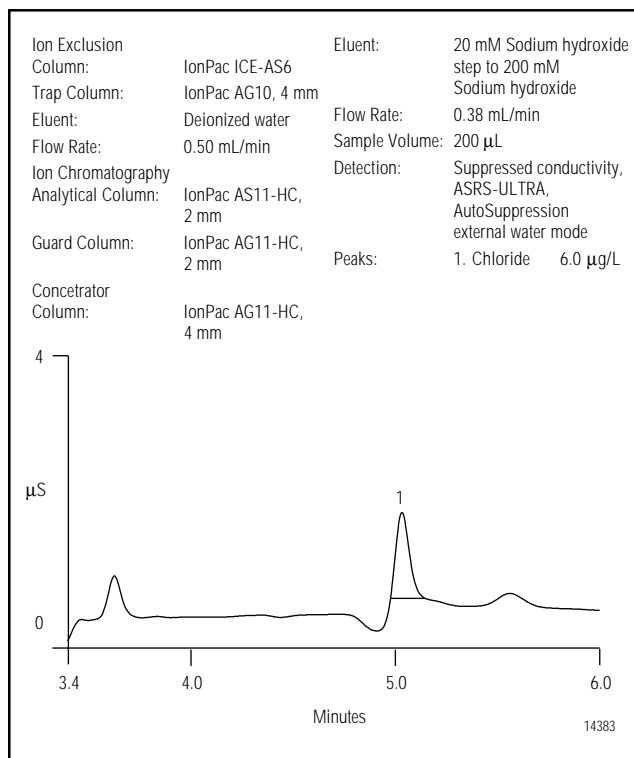


Figure 10 Detail of chloride peak.

Table 5 Method detection limits and SEMI specifications for trace anions in high purity phosphoric acid

Anion	Method Detection Limits (µg/L)	SEMI C7.11-93 Specification (µg/L)
Chloride	0.15	1000
Sulfate	31	8000
Nitrate	2.5	200

Method Detection Limit = (SD) x (t)_{99.5%} where (t)_{99.5%} is for a single-sided Student's t-test distribution for n = 7.

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