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Determination of trace anions in concentrated weak acids by ion chromatography

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Abstract

Ion-exclusion chromatography (ICE) followed by ion chromatography (IC) was used for the determination of trace anionic contaminants in concentrated weak acids. The ICE separation was used as a pretreatment step to isolate the contaminant anions of strong acids from the excess of matrix ions. Then a fraction containing the analyte ions was separated using IC with suppressed conductivity detection. Microbore–ion-exchange columns were chosen to address the increased purity requirements for use of these concentrated acids in semiconductor applications. The chromatographic conditions were optimized for determining trace chloride, sulfate, phosphate, and nitrate in concentrated 24.5% (v/v) hydrofluoric acid; trace chloride, sulfate, and nitrate in concentrated 85% (w/w) phosphoric acid and trace chloride and sulfate in concentrated 0.7% (v/v) glycolic acid. Method detection limits for the anions of interest were below 100 μg/l.

Keywords: Sample handling; Inorganic anions; Hydrofluoric acid; Phosphoric acid; Glycolic acid

1. Introduction

There is a need for a reliable method to determine trace anions in concentrated acids. Concentrated acids play an important role in the fabrication of semiconductor devices. Purity requirements for these reagents are more stringent as the feature sizes for microelectronic circuitry continue to shrink. Hydrofluoric acid (HF) is used to remove oxide layers from silicon wafer surfaces [1]. Glycolic acid is the primary component in water-soluble soldering flux that is used for tinning or solder coating electronic components and leads [2]. Phosphoric acid is used by the semiconductor industry for etching silicon nitride on wafer surfaces [3].

The large excess of acid matrix ions in these reagents hampers analysis. For example, to determine chloride at 100 μg/l in 24.5% (v/v) hydrofluoric acid represents a concentration ratio of $1:10^6$ (chloride to fluoride). Diluting the concentrated sample overcomes the problem of the large concentration of the interfering matrix ion but results in a lack of sensitivity for the contaminant ions of interest.

Siriraks et al. demonstrated method detection limits of between 0.04 to 0.08 mg/l for trace chloride, sulfate, phosphate, bromide in 0.25% hydrofluoric acid by direct injection [4]. An alternative selective matrix elimination approach was used to rinse away the high ionic strength matrix with a

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methanol–water eluent. With this method the authors demonstrated method detection limits of 25 to 50 μg/l for trace anions in 5% hydrofluoric acid. This concentration of concentrated HF represents the limit for conventional ion chromatographic analysis. However this sensitivity was not adequate for the more stringent requirements of semiconductor fabrication.

To address this need, Watanabe and Ishzaki developed a method in which an ion-exclusion chromatographic (ICE) separation was coupled to an ion chromatographic (IC) separation [5]. Using this approach allowed the separation of strongly and weakly ionized analytes and facilitated method detection limits as low as 30 μg/l in 25% hydrofluoric acid. Chen and Wu optimized the experimental conditions for hydrofluoric acid [6], and showed application to the determination of trace anions in phosphoric acid [7].

The goal of this investigation was to expand upon the previous work and document a procedure that reliably determines trace anions in concentrated weak acids. Anion-exchange columns in the microbore format were chosen to enhance sensitivity at trace levels. The following weak acids were evaluated: hydrofluoric, phosphoric, and glycolic acids. These acids were chosen because of their use in the microelectronics industry.

2. Experimental

2.1. Chromatographic system

All chromatography was performed on a Dionex (Sunnyvale, CA, USA) DX-500 ion chromatograph. The system consists of a gradient pump (GP40), a conductivity detector (CD20), a liquid chromatography module (LC20), a thermally stabilized conductivity detector (DS3) and a RP-1 single piston pump. Two Rheodyne (Cotati, CA. USA) six-port 9126-038 valves were used. A pressurizable reservoir chamber (Dionex) large enough to accommodate the sample container was maintained at 5 p.s.i. (34.5 kPa) with helium to fill the sample loop with sample. PeakNet chromatography software (Dionex) was used to acquire the data and control the instrumentation.

All columns used in this study were manufactured by Dionex. An IonPac ICE-AS6 (250×9 mm) ion-exclusion column was used for sample pretreatment to separate the analyte ions from an excess of weak acid matrix ions. The ion chromatographic separation was performed on two different high-capacity ion-exchange column sets as summarized in Table 1.

Polyether ether ketone (PEEK) tubing was used to connect all of the chromatographic hardware. The column and suppressor in the IC system were connected with 0.005 in. (0.125 mm) I.D. PEEK tubing. The lengths of the connecting tubing were kept as short as possible to minimize system void volume and to ensure efficient 2 mm column operation. Care was taken to evenly cut tubing ends to avoid introducing any unwanted void volume. Sample loops were made with 0.030 in. (0.75 mm) I.D. PEEK tubing.

Deionized water was used as the eluent for the ion exclusion separation. To prevent contamination of the sample with anionic impurities in the ICE deionized water eluent, an IonPac AG10 was used as an anion trap column. The AG10 was initially prepared for use by flushing (2 ml/min) with 200 ml of 200 m*M* sodium hydroxide followed by 100 ml of deionized water at the same flow-rate. The AG10 was periodically regenerated using this procedure. An anion self regenerating suppressor (ASRS) from Dionex was used to reduce the conductivity of the eluent [8].

2.2. Chemicals, solutions and samples

Reagent grade chemicals were used for standard and eluent preparation. Sodium hydroxide, 50% (w/w) from Fisher Scientific, (Pittsburgh, PA, USA) and a 0.5 *M* sodium carbonate eluent concentrate (Dionex) were used to prepare eluent. Deionized water with a specific resistance of 17.8 MΩ cm or greater from a Labconco (Kansas City, MO, USA) Water Pro PS water purification system was used to prepare all eluents, reagents, standards and the rinse solution. The deionized water was free of measurable levels of ionic impurities, organics and particulate matter (larger than 0.2 μm).

Samples of semiconductor grade 49% (w/w) hydrofluoric acid and 85% (w/w) phosphoric acid were kindly provided by Ashland (Columbus, OH, USA). Technical grade 70% (w/w) glycolic acid was

Table 1
Chromatographic conditions for trace anions in concentrated weak acids

	HF and glycolic acid	Phosphoric acid
Ion-exclusion sample pretreatment		
Pretreatment column	IonPac ICE-AS6 (250×9 mm)	IonPac ICE-AS6 (250×9 mm)
ICE eluent	Deionized water	Deionized water
ICE flow-rate	0.55 ml/min	0.50 ml/min
Sample volume	750 μl	200 μl
Ion chromatography analytical system		
Guard column	IonPac AG9-HC (50×2 mm)	IonPac AG11-HC (50×2 mm)
Analytical column	IonPac AS9-HC (250×2 mm)	IonPac AS11-HC (250×2 mm)
Concentrator column	IonPac AG9-HC (50×4 mm)	IonPac AG11-HC (50×4 mm)
IC eluent	8.0 m*M* Sodium carbonate 1.5 m*M* Sodium hydroxide	20 m*M* Sodium hydroxide step to 200 m*M* at 25 min.
IC flow-rate	0.25 ml/min	0.38 ml/min
Detection	Suppressed conductivity, ASRS-ULTRA, external water mode	Suppressed conductivity, ASRS-ULTRA, external water mode
ASRS current setting	100 mA	100 mA

from DuPont, Wilmington, DE, USA. Anion standards (1000 mg/l) for chloride, sulfate, phosphate and nitrate were from Dionex and Fisher Scientific. Working standards were prepared by further diluting the 1000 mg/l standards to the range expected for the anions of interest. Dilute working standards were prepared weekly. Containers presoaked with deionized water, which had a specific resistance of 17.8 MΩ cm or greater, were used to store concentrated acid samples and standards. Polyethylene containers were used to store standards, whereas PTFE containers were used for storage of the concentrated acid samples.

2.3. System operation

Trace anion analysis of concentrated weak acids was accomplished in two steps: an ICE pretreatment followed by injecting a portion of the ICE separation to an IC separation. The schematic in Fig. 1 illustrates how the chromatographic hardware was set up. This initial configuration shows the sample valve in the ''Load'' position and the injection valve in the ''Inject'' position.

The concentrated acid sample was loaded via a pressurized reservoir into a sample loop. Helium at 5 p.s.i. (34.5 kPa) was used to push sample out of the sample container into the sample loop [9]. This technique ensured that a representative sample of the concentrated acid sample was loaded into the sample loop with minimal contamination due to sample handling. At least four loop volumes were pushed through the sample loop to ensure that a representative sample had been filled [10]. Meanwhile the IC eluent was flowing through the concentrator column and analytical columns.

The concentrated acid sample was delivered from the sample loop to the IonPac ICE-AS6 with the deionized water ICE eluent by switching the sample valve to the inject position. The first portion of the ICE separation was sent to waste. Next, the concentrator column was placed in line with the ICE column and the ''cut'' portion was preconcentrated on the concentrator column by placing the injection valve in the ''Load'' position. Finally, the concentrator column was placed in line with the analytical column set and the concentrated anions were separated. The timing of the ''cut window'' was opti-

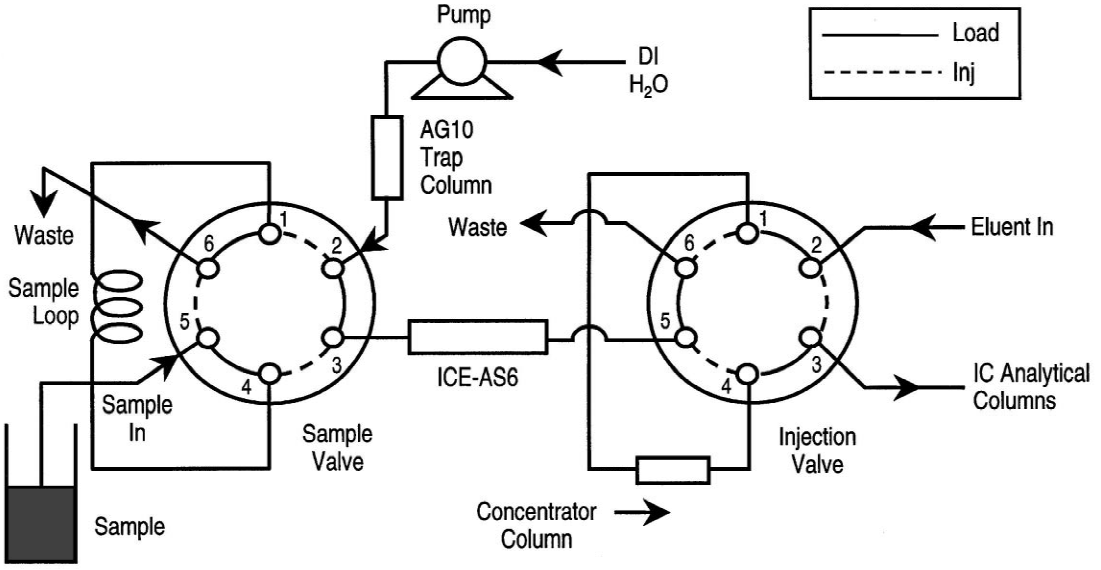

Fig. 1. Chromatographic hardware configuration for trace anion analysis in concentrated weak acids.

mized for each sample matrix to ensure that the anions of interest were preconcentrated with a minimal amount of the weak acid anions from the matrix.

While the IC separation was taking place, the deionized water stream was rinsing the ICE-AS6 and associated tubing to ensure that there was no contamination from the previous sample. Disposable chemical resistant gloves were worn at all times when handling apparatus that made contact with standard or sample.

3. Results and discussion

3.1. Ion-exclusion pretreatment

The ion-exclusion mechanism separates ionized species from nonionized or weakly ionized species. This makes it a good match for this analysis, where the matrix ions are weakly ionized and the analyte ions are strongly ionized. Fig. 2 illustrates the application of this ICE mechanism to the separation of 10 mg/l of chloride from 100 mg/l fluoride using an ICE-AS6 ion-exclusion column. This chromatogram is a measurement of the unsuppressed conductivity response for the ICE separation. Because chloride is a strong acid ion, it cannot enter the resin and elutes first as a small peak starting at approximately 9 min. The weakly ionized fluoride matrix ions are retained and thus elute later as a large peak.

To determine the ''cut window'' of the ICE separation, the ICE column and conductivity cell were configured separately without the IC system. The ''cut window'' time was established by examining these ICE chromatograms for the concentrated acid sample injected into the ICE column. The goal was to extract a portion of the ICE separation that contained a majority of the strong acid anions yet contained a minimum of the weak acid matrix anions. For the analysis of hydrofluoric a time window from 7 to 12 min was selected, whereas for glycolic acid a time window of 7 to 14 min was selected. In the case of phosphoric acid, a portion from 7 to 13 min was preconcentrated (see Fig. 3).

The ''cut window'' from the ICE separation was optimized for a given flow-rate to achieve the best subsequent IC separation. For the analysis of hydrofluoric and glycolic acid, an ICE flow-rate of 0.55 ml/min was selected, whereas for phosphoric acid, 0.50 ml/min was selected. Deviation from this flow-rate would lead to a different cut volume reaching the IC concentrator column. This could lead to a loss of recovery due to incomplete capture or poor resolution of the ions of interest due to an excess of

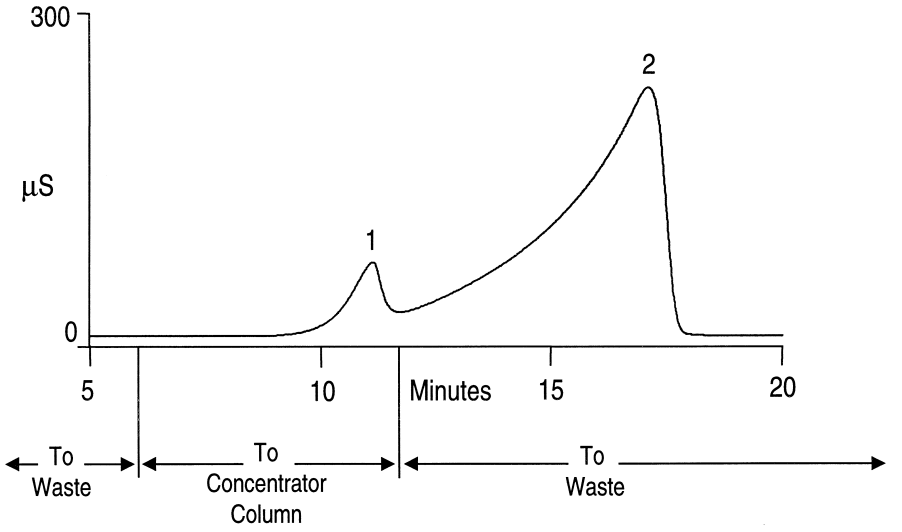

Fig. 2. Ion-exclusion separation of 10 mg/l chloride from 100 mg/l fluoride. Peaks: 1=chloride (10 mg/l); 2=fluoride (100 mg/l). Sample volume: 750 μl; analytical column IonPac ICE-AS6 (250×9 mm); trap column IonPac AG10 (50×4 mm), 4 mm; detection: conductivity; eluent: deionized water; eluent flow-rate: 0.55 ml/min.

matrix ions. It was important to maintain a consistent ICE eluent flow-rate to insure a complete capture of the anions of interest and avoid an excessive capture of the sample matrix.

3.2. Ion chromatographic separation

The deionized water eluent from the ion exclusion separation acted as a carrier stream to deliver the cut portion from the ICE separation to the IC hardware. The anions of interest were concentrated on the anion-exchange concentrator column that was off-line from the IC system. This concentrator column was then place in-line with the analytical column set where the anions of interest were separated.

The microbore format was chosen for the analytical columns and suppressor because it has several advantages [11]. There is a fourfold increase in mass

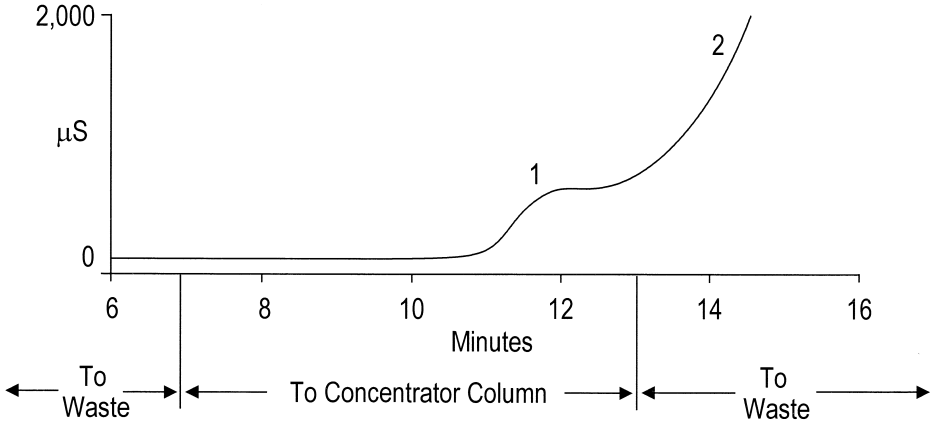

Fig. 3. Ion-exclusion separation of 85% phosphoric acid. Peaks: 1=nitrate/chloride/sulfate; 2=phosphate. Sample volume: 200 μl; analytical column IonPac ICE-AS6 (250×9 mm); trap column IonPac AG10 (50×4 mm), 4 mm; detection: conductivity; eluent: deionized water; eluent flow-rate: 0.50 ml/min.

sensitivity for the microbore (2 mm) over the standard (4 mm) format with no loss in concentration sensitivity. Thus, smaller amounts of concentrated acid sample were required. This facilitated more convenient and faster loop loading. The microbore format also offered lower eluent consumption, as well as less eluent waste.

The concentrator column in the 50×4 mm format were used instead of 50×2 mm because the 4 mm column had four times more capacity and lower back pressure than the 2 mm column. No significant degradation in separation efficiency was observed when coupling a 4 mm concentrator column with a 2 mm analytical column set.

A blank was determined by performing all the steps of the analysis with deionized water as the sample. This result was then applied as a correction to all subsequent sample measurements. The blank value established baseline anion concentrations from such sources as the sample container, tubing, eluents, and columns.

The ICE-AS6 column came packed with 0.4 m*M* heptafluorobutyric acid. The column was rinsed off-line with deionized water at 0.5 ml/min for at least 24 h to rinse away this storage solution. Fig. 4 shows a representative blank run prior to trace anion in hydrofluoric acid. The only significant contaminant present was sulfate at less than 100 μg/l. Heptafluorobutyrate was found to elute at the same retention time as sulfate in the subsequent IC separation. Thus, blank concentration values reported as sulfate could also contain heptafluorobutyrate. Performance for the analysis of phosphoric acid was similar with a sulfate blank of approximately 150 μg/l.

The IonPac AS9-HC anion-exchange column was chosen for the hydrofluoric and glycolic acid analyses because of its ability to separate the weakly retained analyte ions from the other anions of interest with a carbonate-based eluent. It was best suited for this analysis because it was able to separate the ions of interest in less than 30 min with an isocratic carbonate-based eluent [12]. Chloride and carbonate coelute with the IonPac AS9-HC when using 9 m*M* sodium carbonate under standard conditions. Increasing the eluent pH through the addition of 1.5 m*M* sodium hydroxide resulted in stronger retention of carbonate. Consequently, chloride was baseline resolved and did not coelute with carbonate.

For the analysis of phosphoric acid, an IonPac AS11-HC anion-exchange column was selected. By using 20 m*M* sodium hydroxide eluent at 0.38 ml/min it was possible to achieve good separation between the early eluting contaminant anions and the

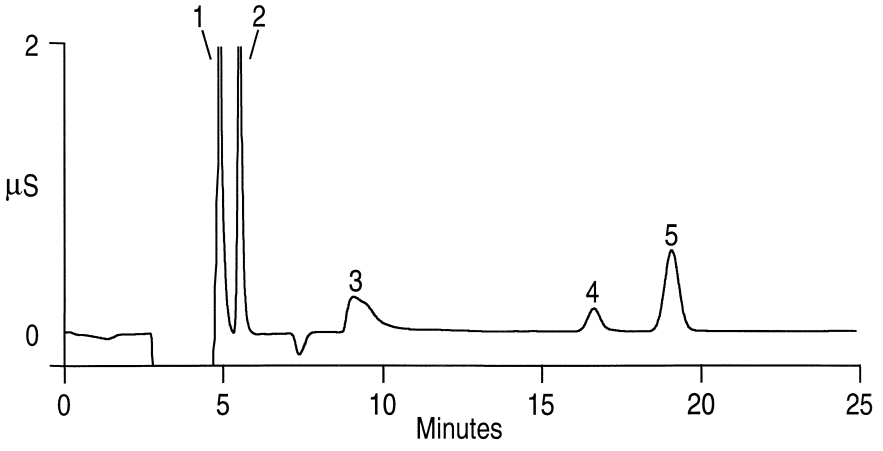

Fig. 4. Representative system blank. Peaks: 1=fluoride; 2=unidentified, 3=carbonate; 4=unidentified; 5=sulfate (32 μg/l). Sample volume: 750 μl; analytical column IonPac AG9-HC (50×2 mm), AS9-HC (250×2 mm); pretreatment column IonPac ICE-AS6 (250×9 mm); concentrator column IonPac AG9-HC (50×4 mm); trap column IonPac AG10 (50×4 mm), detection: suppressed conductivity, ASRS-ULTRA, in external water mode; ICE eluent: deionized water; IC eluent: 8.0 m*M* sodium carbonate–1.5 m*M* sodium hydroxide; ICE eluent flow-rate: 0.55 ml/min; IC eluent flow-rate: 0.25 ml/min.

large excess of phosphate matrix ions. The eluent was stepped to 200 m*M* sodium hydroxide for 5 min at the end of the run as a cleanup step.

3.3. Method performance

This method was applied to the three samples of interest: hydrofluoric acid, glycolic acid and phosphoric acid. Semiconductor grade 49% (w/w) hydrofluoric acid was diluted 1:1 to 24.5% (v/v) with deionized water. Technical grade 70% (w/w) glycolic acid was diluted to 1:100 to bring the analyte response within a reasonable range (<100 μS). Semiconductor grade 85% (w/w) phosphoric acid was injected into the instrumentation without dilution.

A chromatogram for the analysis of trace anions in semiconductor grade 24.5% (v/v) hydrofluoric acid is shown in Fig. 5. The large fluoride peak beginning at 5 min is well separated from chloride. A chromatogram for the analysis of trace anions in 0.7% (v/v) technical grade glycolic acid is shown in Fig. 6. Glycolate has similar behavior to fluoride under these chromatographic conditions because it is weakly retained and thus elutes early.

A gradient eluent on a hydroxide selective column such as the IonPac AS10 could have enhanced this separation between fluoride and chloride. However, the drawback of this approach is that the total analysis time (including sample loading, equilibration, ICE separation and IC separation) would have increased to 70 min. Thus the use of the AS9-HC for the analysis of hydrofluoric and glycolic acids represents the best compromise between separation efficiency and run time.

The method was modified for the determination of trace anions in phosphoric acid. As mentioned earlier, the AS11-HC with a hydroxide eluent was better suited for the ion chromatographic separation because phosphate elutes last and the eluent could be readily stepped up in concentration for a clean up step. When concentrated phosphoric acid is diluted the acid will partially ionize because of the nature of its ionic equilibrium [7]. This dilution renders the ICE separation for this analysis ineffective.

It was found that after concentrated phosphoric acid had been in the sample loop pathway that several runs of deionized water were required to completely rinse away the high concentration of the 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 specified concentrations for high purity grade concentrated phosphoric acid [13]. The anion values of this deionized water blank were subtracted from the

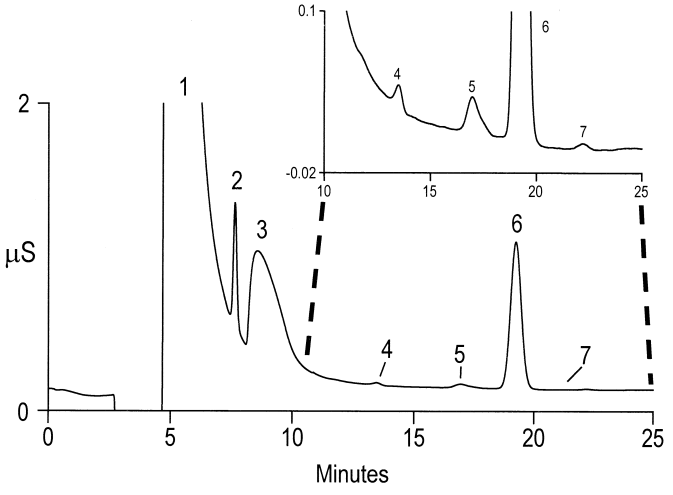

Fig. 5. Determination of trace anions in high purity 24.5% (v/v) hydrofluoric acid. Peaks: 1=fluoride; 2=chloride (7.9 μg/l); 3=carbonate; 4=nitrate (0.89 μg/l); 5=unidentified; 6=sulfate (10.1 μg/l); 7=phosphate (2.4 μg/l). Chromatographic conditions as in Fig. 4.

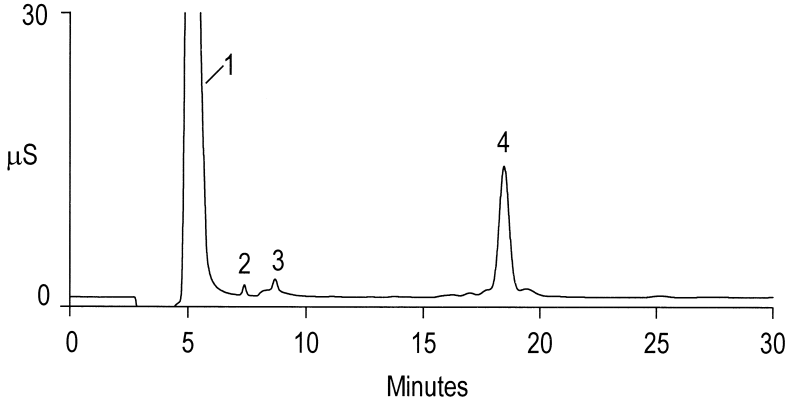

Fig. 6. Trace anions in 0.7% (v/v) glycolic acid. Peaks: 1=glycolate; 2=chloride (11 μg/l); 3=carbonate; 4=sulfate (336 μg/l). Chromatographic conditions as in Fig. 4.

levels found in the concentrated phosphoric acid samples. A small amount of phosphate is detected in the blank as carryover from previous injections.

A chromatogram for the analysis of 85% (w/w) phosphoric acid is shown in Fig. 7. The large phosphate matrix peak beginning at 12 min is well separated from the anions of interest. The peak that starts at 29 min is a result of the step to the higher eluent concentration. Any residual phosphate left in the column and flow path is eluted with this high eluent concentration. This wash step was found to be necessary for obtaining reproducible retention times.

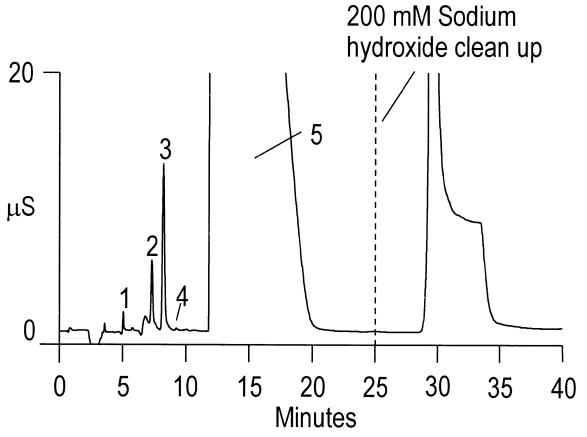

Fig. 7. Determination of trace anions in high purity 85% (w/w) phosphoric acid. Peaks: 1=chloride (36 μg/l); 2=unidentified; 3=sulfate (750 μg/l); 4=nitrate (15 μg/l); 5=phosphate. Sample volume: 200 μl; analytical column IonPac AG11-HC (50×2 mm), AS11-HC (250×2 mm); Ion exclusion pretreatment column IonPac ICE-AS6 (250×9 mm); concentrator column IonPac AG11-HC (50×4 mm); trap column IonPac AG10 (50×4 mm), detection: suppressed conductivity, ASRS, AutoSuppression, external water mode; ICE eluent: deionized water; IC eluent: 20 m*M* sodium hydroxide: ICE flow-rate: 0.50 ml/min; IC flow-rate: 0.38 ml/min.

To verify proper quantification of the ions of interest in these concentrated acid matrices increasing concentrations were added into the deionized water used to dilute the hydrofluoric and glycolic acid. In the case of phosphoric acid, a small aqueous spike was added which resulted in a negligible dilution (0.020 ml spike/20 ml sample volume= 0.1% dilution). Linearity for the anions of interest in these matrices yielded coefficients of determination (r^2) greater than 0.99 as summarized in Table 2.

Method detection limits (MDLs) were calculated using the standard deviation for seven replicate injections of the anions of interest [14]. When possible, analytes were spiked to be in the same concentration range as the estimated MDL. The standard deviation of the seven replicate injections was multiplying by the Student's *t* value for the 99.5% confidence level. These MDLs were substantially below the specified levels for the Semiconductor Equipment and Materials International (SEMI) guidelines established for the best grade of hydrofluoric acid [15] and phosphoric acid [13] as shown in Table 3. SEMI has not established guidelines for glycolic acid.

Based on these calibration curves, spikes were added into the samples to calculate recovery. These spikes were at 50% of the specified levels SEMI

Table 2
Calibration[a] results for trace anions in concentrated weak acids

	Anion	Data points	r^2	Slope ($\cdot 10^{-4}$)	Intercept	Dynamic range (μg/l)
24.5% (v/v) Hydrofluoric acid	Chloride	8	0.9994	0.935±0.05	−8.2±1.5	10–100
	Nitrate	6	0.9994	2.82±0.9	0.68±1.9	10–300
	Sulfate	8	0.9966	1.20±0.07	−49±11	30–300
	Phosphate	7	0.9997	18.2±0.3	−1.2±2.4	10–300
0.7% (v/v) Glycolic acid	Chloride	8	0.9988	1.01±0.04	−9.8±2.1	10–300
	Sulfate	8	0.9986	0.962±0.03	−402±71	300–3000
85% (w/w) Phosphoric acid	Chloride	7	0.9987	4.23±0.36	−26.0±8.0	30–300
	Sulfate	8	0.9941	5.56±0.04	−740±170	300–3000
	Nitrate	7	0.9984	10.3±0.95	0.11±19	100–1000

[a] Calibration curve based on linear regression for spiked concentrations of the analytes of interest prior to blank correction. Standard deviations are based on a 95% confidence interval.

Table 3
Method detection limits and SEMI specifications for trace anions in concentrated weak acids[a]

Anion	Hydrofluoric acid		Glycolic acid 0.7% (v/v)	Phosphoric acid	
	24.5% (v/v) MDL (μg/l)	49% (w/w) SEMI Spec. C8.3-96 (μg/l)	MDL (μg/l)	85% (w/w) MDL (μg/l)	85% (w/w) SEMI Spec.C7.11-93 (μg/l)
Chloride	0.64	200	2	0.15	1000
Nitrate	0.14	100		10	200
Sulfate	4.2	200	20	31	8000
Phosphate	2.3	100			

[a] Method detection limit=(SD)×$(t_s)_{99.5\%}$ where (t_s) is for a single sided Student's t-test distribution for $n=7$.

Table 4
Spike/recovery of trace anions in concentrated weak acids[a]

	Anion	Sample[b] (μg/l±SD)	Spike (μg/l)	Found-sample (μg/l±SD)	Recovery (%)
24.5% (v/v) Hydrofluoric acid	Chloride	8.0±0.17	50	42±0.53	84
	Nitrate	0.95±0.039	25	21.7±0.32	87
	Sulfate	10.3±1.12	50	52.9±1.59	106
	Phosphate	3.2±0.61	25	21.7±0.92	87
0.7% (v/v) Glycolic acid	Chloride	10±0.2	20	21±0.3	110
	Sulfate	340±3.0	500	558±6.7	112
85% (w/w) Phosphoric acid	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

[a] For $n=7$.
[b] Corrected for system blank.

levels where applicable. Recoveries were found to fall between the SEMI target recovery of 75–125% for seven replicate injections as shown in Table 4.

4. Conclusions

ICE followed by IC was successfully applied for the determination of trace anionic contaminants in concentrated weak acids. It was possible to directly inject 24.5% (v/v) hydrofluoric acid, 85% (w/w) phosphoric acid and 0.7% (v/v) glycolic acid for analysis without any additional sample preparation. Chloride, sulfate and other anions were determined to the low μg/l levels with acceptable precision and recovery. This represents a significant improvement over the sensitivity that was possible with conventional IC methods. These levels were substantially below the maximum limit of impurity that was established by SEMI for these high purity reagents. The results of this study indicate that this technique would be applicable as a quality control test for high-purity applications.

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