



Ion-chromatographic determination of seven common anions in electronic-grade, water-miscible solvents

L.E. Vanatta*

Air Liquide – Balazs™ Analytical Services, 13546 North Central Expressway, Dallas, TX 75243, USA

ARTICLE INFO

Article history:

Available online 18 September 2008

Keywords:

Ion chromatography
Trace anions
Acetone
Isopropyl alcohol
Methanol
N-Methyl-2-pyrrolidone
Statistical analysis

ABSTRACT

An ion-chromatographic method has been developed to determine seven common anions in electronic-grade acetone, isopropyl alcohol, methanol, and *N*-methyl-2-pyrrolidone (NMP), solvents whose cleanliness is vital to the manufacturing of semiconductor devices. The system requires only a pump, conductivity detector, and switching valve as hardware. A calibration study was conducted first, by preparing and analyzing 11 concentrations (between 0.3 and 6.0 ng/g) in deionized (DI) water. Subsequently, a spiking study was performed by spiking each solvent at each of nine concentrations (between 50 and 500 ng/g). Dilutions of 1–100 (w/w) were prepared and analyzed. For each anion, recovered concentrations were estimated using the appropriate calibration curve. For each recovery curve, uncertainties in predicted concentrations were between ± 20 and ± 150 ng/g (except for phosphate in acetone), at 95% confidence. Recoveries were between 82% and 112% for all anions in all solvents, except for nitrite and nitrate in acetone, nitrite in methanol, and fluoride in *N*-methyl-2-pyrrolidone.

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1. Introduction

Contamination control is a critical and continuous challenge in the semiconductor industry, where unwanted material can severely interfere with manufacturing processes. Many types of chemicals are used throughout the hundreds of processing steps. Included are several electronic-grade solvents, which are referenced in the SEMI Book of Standards for chemicals [1]. Even a quick glance through this volume shows that upper limits have been placed on a variety of contaminants, some of which are common inorganic anions (Cl^- , NO_3^- , SO_4^{2-} , PO_4^{3-}).

Since ion chromatography (IC) became affordable and routine, it has replaced laborious wet-chemical methods [2] for quantifying ionic species in chemicals such as solvents. Sanders [3] combined extraction pretreatment with IC to evaluate Li^+ , Na^+ , K^+ , Mg^{2+} , and Ca^{2+} in water-immiscible solvents (*n*-amyl ketone, *n*-butyl acetate, propylene glycol monomethyl ether acetate).

On the anion side, Kaiser and Wojtusik [4] used this instrument to quantify anions in IPA. Kaiser and Rohrer [5] revised and expanded the procedure; Cl^- , NO_3^- , SO_4^{2-} , and PO_4^{3-} were quantified (via standard addition) in acetone, isopropyl alcohol (IPA), and *N*-methyl-2-pyrrolidone (NMP). Most recently, researchers at Dionex have published an Application Update [6], which brings the Kaiser/Rohrer method up to date, per present-day instrumental

advances. In all three cases, this anion procedure involves in-line matrix elimination, which requires extra hardware (i.e., a concentrator column and external-water pump).

Besides involving more complex hardware, the above approach also uses the more complicated technique of standard addition. Furthermore, none of the literature investigated F^- , NO_2^- , or Br^- in these matrices, or included a thorough statistical analysis of the data that were presented. [The 1999 Final Program for the International Ion Chromatography Symposium contains an abstract by Wang et al. [7], wherein they state they have developed procedures to test for the seven common anions (F^- , Cl^- , NO_2^- , SO_4^{2-} , Br^- , NO_3^- , PO_4^{3-}) in a variety of solvents. However, no specific details are given, and the paper that was presented at the conference was never published.]

This present paper addresses the above drawbacks, providing a method that involves only a water dilution of the solvent and a minimum of ion-chromatographic hardware. All seven common anions are included (i.e., F^- , Cl^- , NO_2^- , SO_4^{2-} , Br^- , NO_3^- , and PO_4^{3-}). The protocol uses the simpler external-standard calibration technique, establishes recovery data, and is applicable for anion concentrations in the typical range of interest for solvents (i.e., between 50 and 500 ng/g, in the solvent itself).

2. Experimental

2.1. Materials

Single-anion stock standards [each at 1000 mg/L in deionized (DI) water] of fluoride, chloride, nitrite, sulfate, bromide, nitrate,

* Tel.: +1 972 995 7541.

E-mail address: lynn.vanatta@airliquide.com.

and phosphate were purchased from SPEX (Metuchen, NJ, USA); a multi-anion standard with each of the seven analytes at 10 mg/L (in DI water) was also obtained from SPEX. For dilution of standards, DI water (18 mΩ cm) was delivered by an in-house water-purification system. Electronic grades of solvents were used for the spiking studies. Acetone and isopropyl alcohol were obtained from Air Liquide America (Dallas, TX, USA). *N*-Methyl-2-pyrrolidone was from a customer-supplied sample. Methanol (MeOH) was purchased from Fisher Chemical (Fairlawn, NJ, USA).

2.2. Apparatus and columns

Unless otherwise noted, all instrument modules and supplies were from Dionex (Sunnyvale, CA, USA). A DX 500 microbore ion chromatograph was used for all analyses. The separator columns used were an IonPac AS11-HC (250 mm × 2 mm) and an IonPac AG11-HC (50 mm × 2 mm). The columns were not heated; room temperature was ~21 °C. Post-column eluent suppression was achieved using a 2 mm Anion Self-Regenerating Suppressor (ASRS-300) in the external-water mode. For the ASRS, constant equilibrium was maintained by allowing water to flow continuously through the regenerant chamber, even when the chromatograph was not in use [8]; flow rate (with ASRS current off) was approximately 15 mL/min. Suppressor current was 95 mA.

The columns and suppressor were housed in an LC20 Chromatography Enclosure with rear-loading six-port Rheodyne valve. All tubing in the chromatography paths was PEEK (polyether ether ketone) [0.005 in. (0.125 mm) I.D.].

A GP40 Gradient Pump was used to deliver deionized water to an EluGen eluent-generator cartridge (EGC-KOH), which was controlled by a Reagent Free Controller (RFC-30). The RFC-30 also supplied current to the suppressor, as well as controlled a Continuously Regenerated Anion Trap Column (CR-ATC); this latter device removed trace contaminants from electrolytically generated eluent [9]. Eluent flow rate was 0.38 mL/min. A gradient program was used, starting at 1.0 mM for 7.5 min, followed by a 15 min linear ramp to 100 mM. A 1000 μL sample loop was loaded via an AS40 Automated Sampler, using PolyVials (5 mL) and plain caps. Before use, all vials were rinsed approximately 20 times with DI water from the tap. Detection was via a CD20 Conductivity Detector at an output range of 10 μS.

Instrument control and data collection were accomplished using a personal computer and PeakNet 5.1 software. JMP 7.0 software (SAS Institute, Cary, NC, USA) was used to carry out statistical calculations.

2.3. Preparation of standards and spikes

All solutions were prepared in new high-density polyethylene (HDPE) bottles. Three sizes were used: (1) 125 mL narrow-mouth (Wheaton Science Products, Millville, NJ, USA), (2) 250 mL narrow-mouth (Wheaton) and (3) 125 mL wide-mouth (Nalge Nunc, Rochester, USA); details of the uses are given below. Vinyl gloves (Oak Technical, Stow, OH, USA) were worn at all times when handling solution-related labware. Transfer pipets (Fisher Scientific, Pittsburgh, PA, USA) were used to deliver small volumes of liquids.

For the calibration studies, the working standards (in DI water) were prepared in three stages, each time using the appropriate size of narrow-mouth bottle. First, 250 g of a 1000 ng/g stock were made from the 10 mg/L seven-anion solution. Second, 250 g of a 100 ng/g standard were prepared from the 1000 ng/g stock. Third, 100 g of each of the 11 working standards (a blank; 0.3, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0, 6.0 ng/g) were made from the 100 ng/g solution. The blank was a “standard-preparation” blank, meaning that it was

prepared exactly as were the non-zero standards, except an aliquot of DI water was added instead of any 100 ng/g solution.

For a small-scale “variation-in-*x* study,” 100 g of a seven-anion standard (at 500 ng/g) were made in DI water from the 10 mg/L SPEX solution. This new standard was used to make three different solutions, each approximately 5 ng/g: (1) dilute 0.79–80.0 g, (2) dilute 0.80–80.0 g and (3) dilute 0.81–80.0 g.

Spiking solutions were prepared (in deionized water) in narrow-mouth bottles as follows. First, 250 g of a multi-anion standard (at 100 μg/g for each anion) were made using the individual 1000 mg/L stocks. Second, 100 g of each of nine spiking solutions (blank; 5, 10, 15, 20, 25, 30, 40, 50 μg/g) were made from the 100 μg/g solution.

To allow for easy handling of the containers during the spiking process, a separate 250 mL bottle was filled with each solvent from its original, large container; because of the very low density of methanol, two such bottles were filled. Nine spikes (blank; 50, 100, 150, 200, 250, 300, 400, 500 ng/g in each anion) were prepared, using each of the four solvents and the corresponding spiking solutions; 100 g of each spike were prepared in a 125 mL wide-mouth bottle. Because of the volatility of most of the solvents, it was necessary to work quickly in preparing the spikes. Thus, the desired amount of spiking solution was delivered and the mass recorded accurately. There was no attempt to add exactly the amount of solvent necessary for a given concentration; once the approximate level was achieved, the mass was recorded accurately and the cap was placed on the bottle. From these data, the exact concentration achieved was calculated. For analysis on the ion chromatograph, each spike was diluted 1–100 (w/w) with DI water, again in a 125 mL wide-mouth bottle.

Two different analytical balances were used. Water-based standards were prepared using a Sartorius BP 301 S (Sartorius, Edgewood, NY, USA). When solvents were being weighed, a Denver Instruments M-220 (Denver Instrument, Denver, CO, USA) was employed. This second balance was located in an exhaust hood suitable for solvents. With both balances, masses were recorded to four decimal places.

3. Results and discussion

3.1. General considerations

The position of sulfate on the AS11-HC column depends on the eluent program used. Thus, this method's elution order (F^- , Cl^- , NO_2^- , SO_4^{2-} , Br^- , NO_3^- , PO_4^{3-}) of the analytes was verified by analyzing single-anions standards.

The calibration and spiking studies were conducted on each of seven separate days, over a total time span of nine days. On each day, the 11 calibration standards were analyzed first, followed by the nine diluted spikes of each of the four solvents. Each solvent's diluted spikes were run as a group. Within each group of standards and diluted spikes, the analysis order of the solutions was randomized. Also, the order in which the solvent groups were tested was varied each day. For all analyses, peak areas (PAs) were used to measure the chromatograph's response to each anion.

To dilute each of the spiked solvents, the tip of a new transfer pipet was filled with liquid; approximate volume was 1 mL. This amount resulted in a mass of between 0.8 and 1.0 g, depending on the density of the solvent.

Since all solvents except NMP are volatile, it was imperative that all spiking and dilution processes be carried out quickly. Consequently, the balance was not allowed to equilibrate totally after the addition of a solvent to a bottle had been completed. Evaluation of this procedure indicated that, at worst, the actual mass was biased by no more than ±0.01 g. To test the effect of this bias on instrumental responses, the three “variation-in-*x*” solutions (see Section

2.3) were analyzed seven times, each time as a set arranged in random order. For each anion, comparison of the PA means showed no statistically significant difference among the three, with two exceptions. For fluoride, standard 2 (exactly 5.0 ng/g) was different from both 1 (low) and 3 (high), yet 1 and 3 were not different. For nitrite, standards 2 and 3 were different, but 1 and 3 (and 1 and 2) were not. These findings indicated that a mass bias of ± 0.01 g would not skew the PAs in the spiking study.

3.2. Calibration study

Fig. 1 shows a typical chromatogram for: (a) a blank and (b) a 2.0 ng/g standard.

For each anion, the first step was to model the standard deviation of the peak areas. The goal was to see if this statistic trended with concentration, an event that would mean that weighted least squares (WLS) would be needed as the fitting technique [10]. Only bromide exhibited a trend; thus, ordinary least squares (OLS) was appropriate for all the other analytes.

A straight line was the proposed model for all seven analytes' data. When this curve was fitted to the data, all results passed the residuals [11] and lack-of-fit tests [12] except fluoride and bromide, both of which required the quadratic model.

At 95% confidence, the half-width of the prediction interval (p.i.) [13] ranged from 0.1 to 0.6 ng/g. These uncertainties were deemed acceptable. Consequently, these calibration curves were used to predict the recovered concentrations in the spiking study.

3.3. Spiking study

3.3.1. General considerations

After the predicted concentrations were estimated from the applicable calibration curve, the standard deviations of these values (by analyte and by solvent) were modeled to select the proper fitting technique. Then the predicted concentrations were plotted versus true concentration, and a straight-line model proposed in each case.

The regression-diagnostic tools used with the calibration data were applied to the recovery results. In some cases, neither a straight line nor a quadratic model was adequate. In these instances, the better model (selected by analyzing the residual pattern) was used and the prediction intervals were adjusted to account for the bias that remained [14]. Recoveries were estimated from the slope coefficient associated with the straight-line fit to the data (using the proper fitting technique).

Tables 1–3 summarize the: (1) fitting techniques and models chosen, (2) half-widths of the prediction intervals, and (3) recoveries, respectively. The following sub-sections give details relevant to each solvent. The discussions will address the: (1) effect of any inadequate models on the shape of the prediction intervals, (2)

Table 1

Summary of the chosen fitting technique and model for each analyte/solvent combination in the spiking study

	Fluoride	Chloride	Nitrite	Sulfate	Bromide	Nitrate	Phosphate
Acetone	OLS SL*	OLS SL*	WLS Quadratic*	OLS SL*	OLS SL	OLS SL*	OLS SL*
IPA	WLS SL	OLS SL*	OLS SL*	OLS SL*	OLS SL*	OLS SL	OLS SL
MeOH	OLS Quadratic	OLS SL	WLS SL	OLS SL	OLS SL	OLS SL	OLS Quadratic
NMP	OLS SL	OLS SL	OLS SL	OLS Quadratic	OLS SL	OLS SL*	OLS SL

An asterisk (*) indicates that the model was not adequate (i.e., exhibited lack of fit), but was used along with a bias-adjusted prediction interval. See Section 3.3 for details. OLS: ordinary least squares; WLS: weighted least squares; SL: straight line.

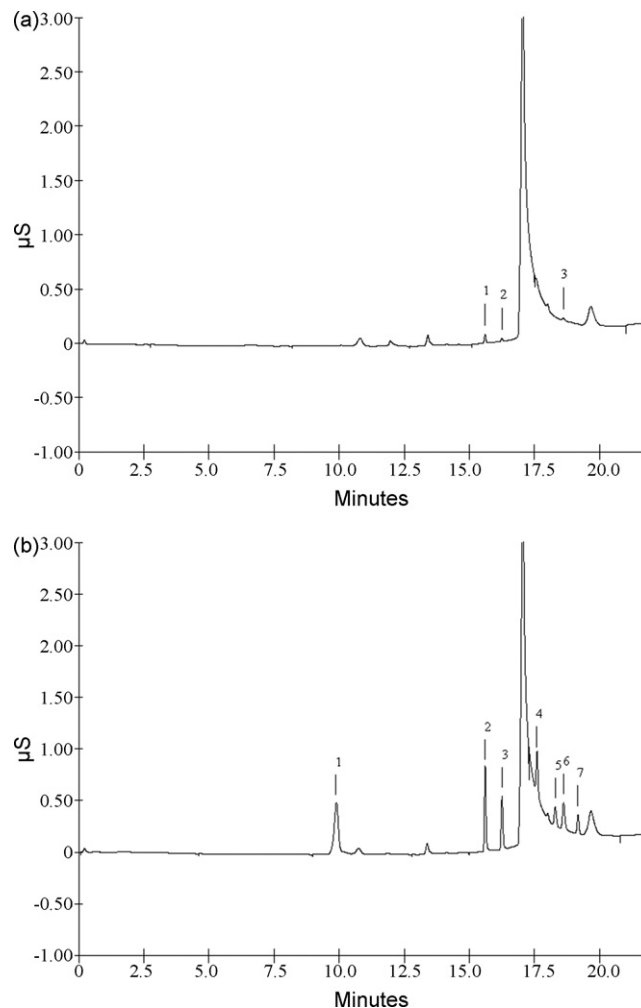


Fig. 1. (a) Chromatogram of a calibration blank. Peak identities: (1) chloride, (2) nitrite, (3) nitrate. See Section 3.2 for details. (b) Chromatogram of a calibration standard at 2.0 ng/g. Peak identities: (1) fluoride, (2) chloride, (3) nitrite, (4) sulfate, (5) bromide, (6) nitrate, (7) phosphate. See Section 3.2 for details.

effect of WLS situations on the flaring of the prediction intervals (a high degree of flaring means that the uncertainty changes markedly with concentration), (3) effect of any quadratic-model selection on the half-width of the prediction interval, and (4) existence of any recovery problems (i.e., below 80% or above 120%).

3.3.2. Acetone

Fig. 2 shows a typical chromatogram for: (a) unspiked acetone and (b) a 219 ng/g spike.

Table 2Summary of the half-widths (at 95% confidence, in \pm ng/g in the pure solvent) of the prediction intervals for each analyte/solvent combination in the spiking study

	Fluoride	Chloride	Nitrite	Sulfate	Bromide	Nitrate	Phosphate
Acetone	150*	50–80*	50–150*	150*	50	100*	200*
IPA	0–100	80–100*	20*	80*	50–100*	150	60
MeOH	10	30	100–120	30	20	130	50–90
NMP	80	100	10	30	40	100*	30

An asterisk (*) indicates that the prediction interval was adjusted to account for the bias inherent in the less-than-adequate model that was used. See Section 3.3 for details.

Table 3

Summary of the recoveries (in %) for each analyte/solvent combination in the spiking study

	Fluoride	Chloride	Nitrite	Sulfate	Bromide	Nitrate	Phosphate
Acetone	80	100	47*	94	100	157	90
IPA	91	98	99	87	97	97	87
MeOH	93*	93	42	90	94	99	82*
NMP	75	101	98	102*	104	112	101

Values are the slope coefficients for a straight-line model's being fit to the data with the appropriate fitting technique. An asterisk (*) indicates that the quadratic model was a better choice overall. See Section 3.3 for details.

Of the six analytes with inadequate models, nitrite's prediction interval was virtually unaffected by the remaining bias; chloride's interval showed a distortion only below 100 ng/g. However, between \sim 150 and \sim 300 ng/g, the prediction intervals were dis-

torted for fluoride, nitrate, sulfate, and phosphate. For these cases, the data were noisier in this concentration range; tighter data might have reduced lack-of-fit problems. The most extreme example was phosphate, whose results are shown in Fig. 3.

WLS was needed only for nitrite. However, the standard-deviation trend was mild, meaning that the prediction interval flared only slightly with concentration. This analyte also needed the quadratic model; the shape of the curve was such that the width of the p.i. (in the x-direction) depended on the concentration.

Recovery was low (\sim 47%) for nitrite and high (\sim 157%) for nitrate. While the increase in nitrate did not totally account for the loss of nitrite, it should be kept in mind that the estimates were based on the fitting of a straight line to the data. In the case of nitrite, the quadratic was a better choice; for nitrate, there was considerable bias in the mid-range of the curve. Thus, the postulation is that nitrite is partially converted to nitrate in acetone. However, the conversion was present at the beginning of the analysis and did not trend upwards as the study progressed.

3.3.3. Isopropyl alcohol

Typical chromatograms for a blank and for a 202 ng/g spike are shown in Fig. 4a and b, respectively.

Of the four analytes that had an inadequate model, the situation mattered only for bromide and chloride. In both of these cases, the p.i. flared slightly below 50 ppb. However, no non-zero data exist below this lowest spike level, meaning that the flaring was not important.

WLS was needed for fluoride; as can be seen in Fig. 5, the result was considerable flaring of the p.i. The quadratic model was not needed in any case. There were no recovery problems with this solvent.

3.3.4. Methanol

Representative chromatograms for a blank and for a 171 ng/g spike are given in Fig. 6a and b, respectively.

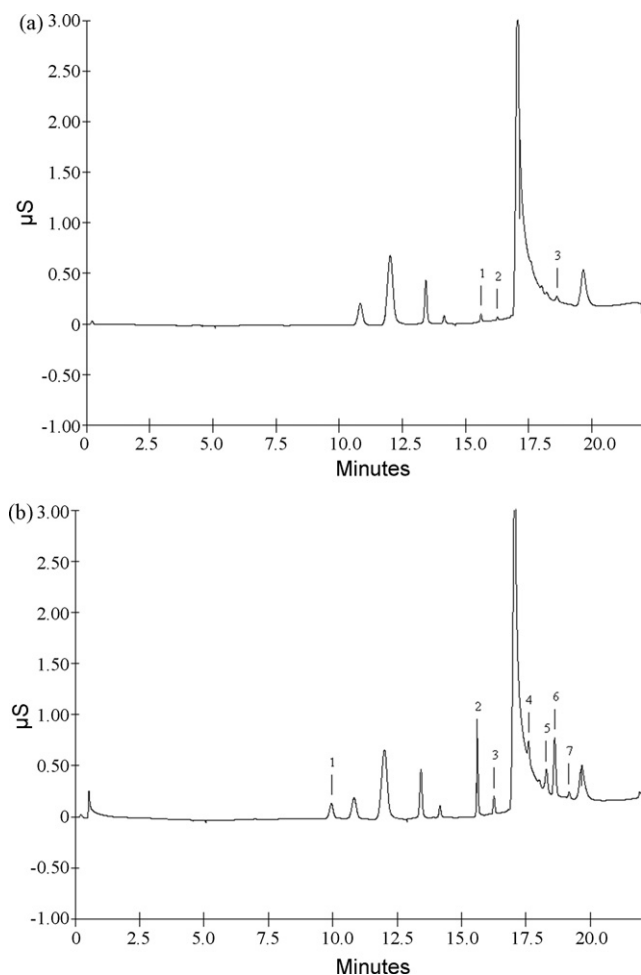


Fig. 2. (a) Chromatogram of unspiked acetone. Peak identities: (1) chloride, (2) nitrite, (3) nitrate. See Section 3.3.2 for details. (b) Chromatogram of acetone spiked at 219 ng/g. Peak identities: (1) fluoride, (2) chloride, (3) nitrite, (4) sulfate, (5) bromide, (6) nitrate, (7) phosphate. See Section 3.3.2 for details.

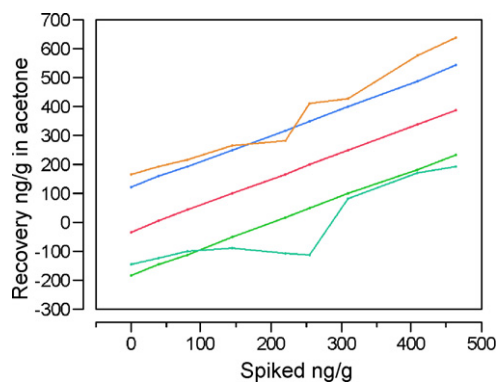


Fig. 3. Recovery curve (center line) for phosphate in acetone. Straight lines bracketing this curve are the prediction limits before bias was taken into account. Jagged lines form the prediction interval after remaining bias has been included. See Sections 3.3.1 and 3.3.2 for details.

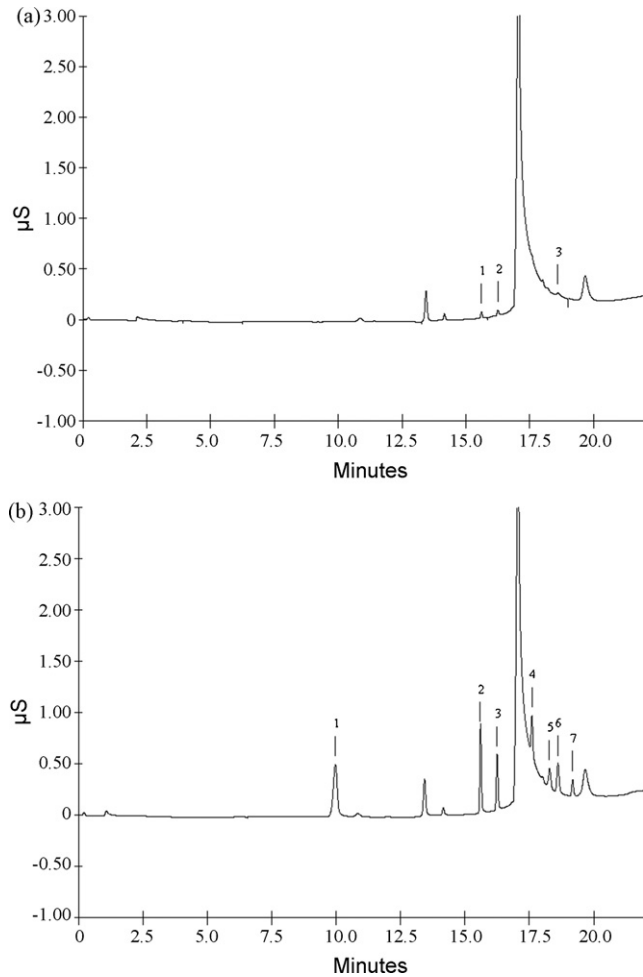


Fig. 4. (a) Chromatogram of unspiked IPA. Peak identities: (1) chloride, (2) nitrite, (3) nitrate. See Section 3.3.3 for details. (b) Chromatogram of IPA spiked at 202 ng/g. Peak identities: (1) fluoride, (2) chloride, (3) nitrite, (4) sulfate, (5) bromide, (6) nitrate, (7) phosphate. See Section 3.3.3 for details.

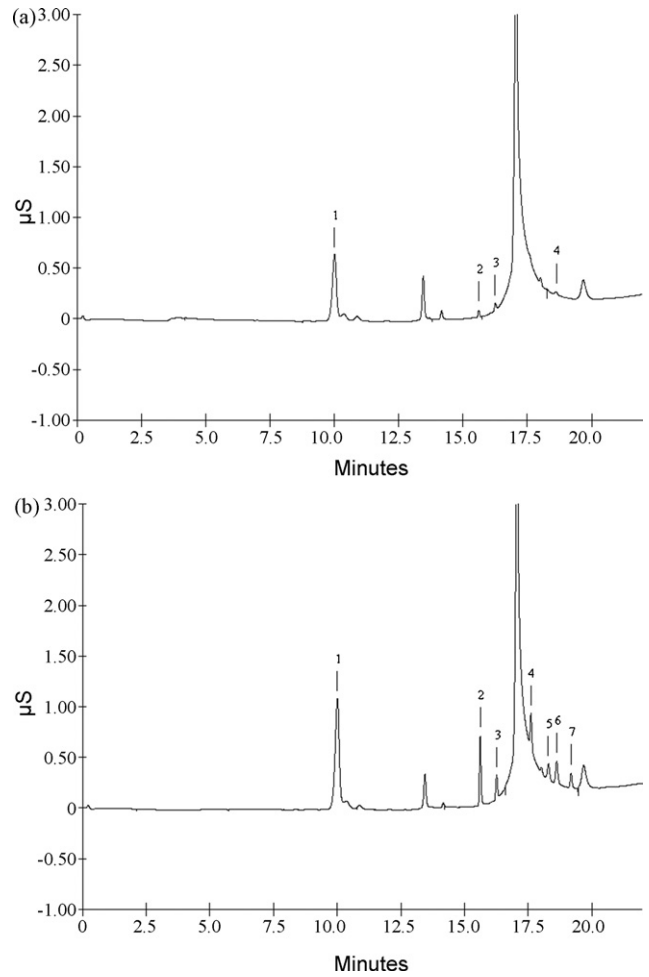


Fig. 6. (a) Chromatogram of unspiked MeOH. Peak identities: (1) fluoride, (2) chloride, (3) nitrite, (4) nitrate. See Section 3.3.4 for details. (b) Chromatogram of MeOH spiked at 171 ng/g. Peak identities: (1) fluoride, (2) chloride, (3) nitrite, (4) sulfate, (5) bromide, (6) nitrate, (7) phosphate. See Section 3.3.4 for details.

With this solvent, the highest spike level used was 250 ng/g. As mentioned in Section 2.3, two bottles (250 mL each) of MeOH were needed to prepare all nine spikes. Thus, two blanks (one from each bottle) were prepared; analysis showed that the two were not the same. All but the highest three concentrations were made from the

first bottle, so only the data from these lower-concentration spikes were used in this study.

Adequate models were found for all analytes. Because of the standard-deviation trend of the nitrite responses, the p.i. flared dramatically, meaning that the uncertainty depends on the concentration. The need for the quadratic model had little effect on

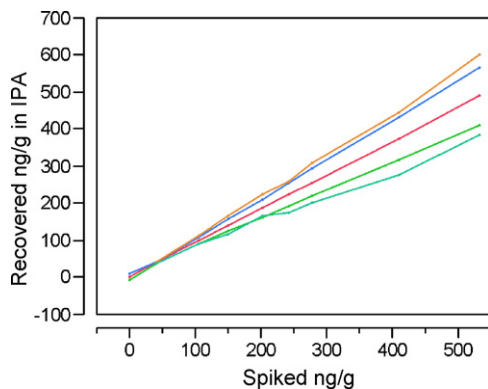


Fig. 5. Recovery curve (center line) for fluoride in IPA. Straight lines bracketing this curve are the prediction limits before bias was taken into account. Jagged lines form the prediction interval after remaining bias has been included. See Sections 3.3.1 and 3.3.3 for details.

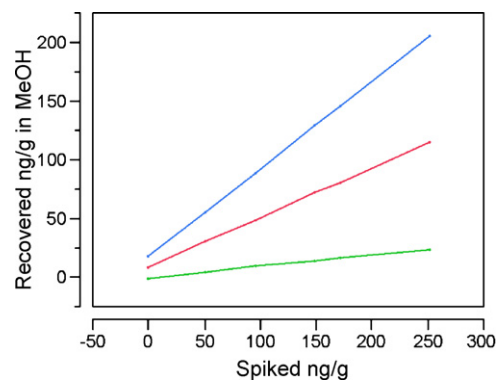


Fig. 7. Recovery curve (center line) for nitrite in MeOH. Straight lines bracketing this curve are the prediction limits. See Sections 3.3.1 and 3.3.4 for details.

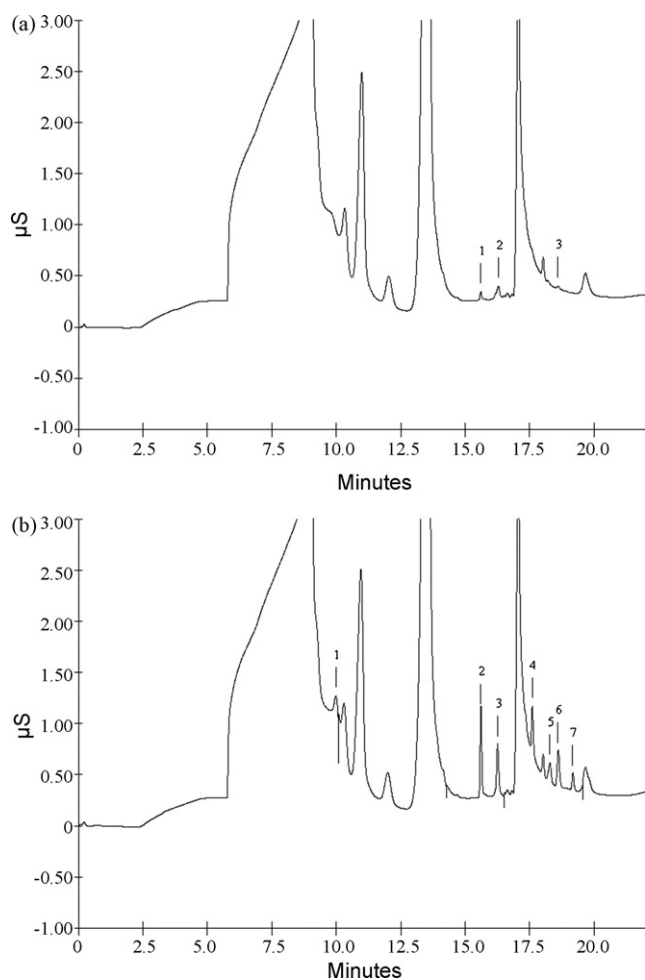


Fig. 8. (a) Chromatogram of unspiked NMP. Peak identities: (1) chloride, (2) nitrite, (3) nitrate. See Section 3.3.5 for details. (b) Chromatogram of NMP spiked at 179 ng/g. Peak identities: (1) fluoride, (2) chloride, (3) nitrite, (4) sulfate, (5) bromide, (6) nitrate, (7) phosphate. See Section 3.3.5 for details.

the half-width of fluoride's p.i., but did result in a concentration-dependent half-width for phosphate (see Table 2).

Nitrite's recovery was only ~42%; nitrate's recovery of ~99% did not account for the loss. The poor NO_2^- recovery was present from the start of the study and could not be explained. The overall results for nitrite are summed up in Fig. 7.

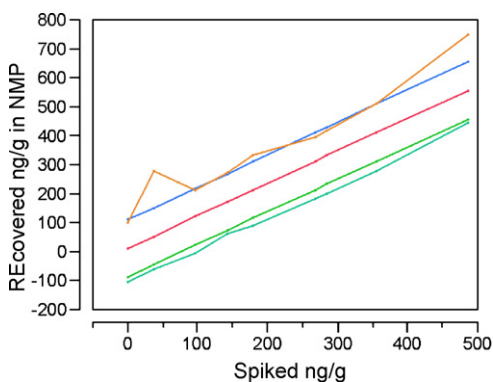


Fig. 9. Recovery curve (center line) for nitrate in NMP. Straight lines bracketing this curve are the prediction limits before bias was taken into account. Jagged lines form the prediction interval after remaining bias has been included. See Sections 3.3.1 and 3.3.5 for details.

3.3.5. *N-Methyl-2-pyrrolidone*

As can be seen in Fig. 8a and b, the front end of NMP chromatograms is quite "busy," possibly from species such as organic acids. The effect of these peaks is felt only by fluoride, which does not exhibit a discernible response for a 50 ng/g spike.

As can be seen in Fig. 9, the use of an inadequate model for nitrate has little effect on the shape of the p.i., except for spikes below 100 ng/g or above 400 ng/g. WLS was not needed for any analyte. The quadratic model had virtually no effect on the uncertainty associated with sulfate. Recoveries were adequate for all analytes except fluoride, which suffered from the interference described above (see Fig. 8) and seen in the slope of the recovery curve in Table 3.

4. Conclusions

A simple method has been developed for analyzing electronic-grade, water-miscible solvents for seven common anions. The procedure requires only a 1–100 (w/w) dilution of the solvent with water and has a total run time (from beginning of sample preparation to ending of chromatographic analysis) of 30 min. Advantages are that: (1) only a minimum of IC equipment is required, (2) dilution virtually eliminates the attack of aggressive solvents on plumbing (especially tubing) and on retention times (which did not shift during the course of the study), (3) the waste from loading the sample loop can be sent to a typical industrial drain (pure solvents generally cannot be introduced into such a system), and (4) the analysis can be conducted on any anion-exchange column (however, with NMP, the separator must be able to resolve the front-end interferences from the anions of interest).

At the 95% confidence level, the half-width of the prediction interval was at or below ± 100 ng/g for all but seven analyte/solvent pairs. Of these seven, the primary "offenders" were nitrate and nitrite. In at least the case of nitrite, the higher variabilities may be linked to poor recovery. The data from acetone suggest that some nitrite may be converted to nitrate. (Such a reaction is complete and immediate in 30% hydrogen peroxide [15]. The data from this present research indicate that such a reaction may be at least somewhat of a problem in solvents, although investigation of such a possibility was outside the scope of this work. The authors of ref. [6] also encountered nitrate-recovery problems that could not be explained; however, their recovery of ~19% was low instead of high.) The non-nitrogen-containing analytes with higher p.i. half-widths all belonged with acetone. It is possible that the cause is the high volatility of this chemical. However, since the dilution process is quite rapid, a direct-injection method probably would not eliminate this effect, since at least some increased variability would be expected just from filling autosampler vials.

The spiking range for this method is 50–500 ng/g, a span that does not go as low as that used in either ref. [5] or [6]. [In isopropyl alcohol (density = 0.78), the former citation's range was 1–10 $\mu\text{g/L}$, while the latter reference's span was 0.1–50 $\mu\text{g/L}$.] However, this current method can be used to analyze water-miscible solvents for most of the seven common anions when uncertainties (at 95% confidence) of ± 100 ng/g or less are acceptable. Except for nitrate and nitrite, the analytes in IPA, MeOH, and NMP all fall into this category. For the remaining analyte/solvent combinations, the method applies if uncertainties (again at 95% confidence) of ± 200 ng/g or less are reasonable. For any sample analyzed via this method, the equation for each applicable recovery curve can be used to correct the reported result for any recovery problems; the half-width of the prediction interval represents the overall uncertainty in the sample's reported concentration.

References

- [1] Book of SEMI Standards, 2008 Process Chemicals Volume, Semiconductor Equipment and Materials International, Mountain View, CA, USA, 2008.
- [2] Book of SEMI Standards, 1989 Process Chemicals Volume, Semiconductor Equipment and Materials International, Mountain View, CA, USA, 1989.
- [3] J.K. Sanders, J. Chromatogr. A 804 (1998) 193.
- [4] E. Kaiser, M.J. Wojtusik, J. Chromatogr. A 671 (1994) 253.
- [5] E. Kaiser, J. Rohrer, J. Chromatogr. A 858 (1999) 55.
- [6] Application Update 163, Dionex, Sunnyvale, CA, USA, 2007.
- [7] K. Wang, R. Wang, J. Kuang, S. Tan, Presented at the International Ion Chromatography Symposium, Paper 15, San Jose, CA, 1999.
- [8] L.E. Vanatta, C. Costanza, D. Brogan, Am. Lab. News 34 (March) (2002) 22.
- [9] Product Manual – Continuously Regenerated Trap Column, Doc. 031910, Rev. 01, Dionex, Sunnyvale, CA, 2003.
- [10] D. Coleman, L. Vanatta, Am. Lab. 35 (November) (2003) 40.
- [11] D. Coleman, L. Vanatta, Am. Lab. 36 (February) (2004) 64.
- [12] D. Coleman, L. Vanatta, Am. Lab. 36 (March) (2004) 46.
- [13] D. Coleman, L. Vanatta, Am. Lab. 35 (March) (2003) 60.
- [14] D. Coleman, L. Vanatta, Am. Lab. 37 (May) (2005) 37.
- [15] L. Vanatta, D. Coleman, J. Chromatogr. A 956 (2002) 23.