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Determination of sodium at low ng/l concentrations in simulated power plant waters by ion chromatography

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Abstract

The determination of low ng/l sodium in the power industry is critical in identifying and preventing corrosive conditions in many power plant components. To address this challenge, we developed an ion chromatographic method to determine sodium at ng/l concentrations in power plant samples. The ion chromatography system used on-line electrolytic eluent generation with a continuously regenerated trap column to minimize system contaminants and therefore allow low detection limits. A 10-ml sample was preconcentrated on a cation-exchange column followed by separation on a high capacity column with 20 m*M* methanesulfonic acid and detected using suppressed conductivity. Sodium response was linear from 25 to 250 ng/l (r^2=0.9990). Method performance was evaluated by analyzing synthetic samples containing ethanolamine as an additive that are typical of samples encountered in the power industry. Retention time precision for sodium was less than 0.4% (n=7) in ultrapure water and simulated sample matrices. The recovery of sodium spiked in synthetic samples at the low ng/l levels was 85–110%. System parameters were optimized to achieve method detection limits in ultrapure water to 3.2 ng/l.

Keywords: Water analysis; Sodium; Inorganic cations

1. Introduction

The build-up of impurities at the sub-μg/l level can result in their accumulation in steam generators or turbines through concentration mechanisms. These impurities can propagate stress corrosion cracking and other corrosion mechanisms in turbines, steam generator tubing, and other plant components that can eventually lead to component failures and plant shutdowns resulting in millions of dollars in lost revenue [1]. Hence, monitoring the presence of ionic impurities in cooling waters, boiler waters, feed waters, and steam condensate in both fossil fueled and nuclear power plants is critical. Corrosive ions, such as sodium, chloride, and sulfate can be minimized by continuously monitoring their respective levels and maintaining levels as low as possible. Typically, this is done by identifying the sources of these impurities.

The measurement of ultra trace levels of sodium and trending of this measurement provides valuable information for preventing corrosive conditions, unacceptable contamination levels, and other depositing conditions. The analysis of sodium can be

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applied to boiler systems to detect condenser leaks, breakthrough in cation exchangers, condensate polisher failures, steam purity for turbine use, and to ensure that there is no excess carryover from the steam process. Sodium can also be monitored in treated boiler water and condensate containing additives that are needed for pH adjustments or for oxygen control. Ammonia was replaced by morpholine as an additive in power plant waters because it is less volatile [2]. This change allowed the pH of the steam condensate from the turbine cycle to be increased to a level that significantly reduced flow-assisted corrosion. However, ammonia is still present from the thermal decomposition of morpholine. Less volatile amines with higher base strengths, such as ethanolamine, eventually replaced morpholine [3].

As the acceptable limits of ionic impurities in the power industry become more stringent, more demand is placed on the measurement technique to ensure that the water quality is within acceptable operating parameters. The accurate and reliable determination of sodium at the sub-μg/l level in the presence of ethanolamine typically in the thousands of μg/l is a challenging analytical problem. In-line sodium analyzers are not reliable at the low to sub-μg/l level and therefore have not gained the confidence of many users in the power industry [1]. Furthermore, sodium analyzers are incapable of detecting sodium at the industry levels required to prevent corrosive contamination [4]. Ion chromatography (IC) is an essential technique for addressing these challenges because IC can determine sub-μg/l ionic impurities in power plant waters [1–8].

Over the past 20 years, IC has undergone many advances that have enabled the determination of cations at the low to sub-μg/l concentrations. More recent advances have further improved trace cation analysis, even in the presence of high concentrations of other cations. The first improvement is the development of ion-exchange columns with higher exchange capacities than previous columns. The higher capacities improved the separation and resolution between many common inorganic cations and amines [9]. In addition, simple isocratic eluents in conjunction with suppressed chromatography allowed for the sensitive detection of cations at trace levels. Another advance in IC was the development of on-line electrolytic generation of high-purity methanesulfonic acid (MSA) that can be used as an eluent for cation-exchange chromatography [10]. This module simplifies and improves IC by eliminating eluent preparation errors, providing contaminant-free eluents, and allowing continuous uninterrupted system operation. In addition, it reduces the probability of introducing contaminants in a trace system. Yet another advance in IC was the improvement in eluent suppression for cation systems [11]. The use of suppressors is particularly important for determining cations at trace levels by minimizing the chromatographic baseline noise. The most recent advance in IC is the introduction of a new continuously regenerated-cation trap column (CR-CTC) [12]. The CR-CTC improves trace analysis by eliminating many cationic contaminants to significantly reduce background conductivity. Unlike conventional trap columns that require frequent off-line chemical regeneration, the CR-CTC is continuously electrolytically regenerated inline. This benefit is particularly attractive for trace analysis because it reduces downtime, which can introduce contamination. The CR-CTC also eliminates the possibility of introducing contaminates from chemicals used in offline regeneration.

In this paper, we describe an IC method that incorporates a high-capacity cation-exchange column to determine sodium in a high-ionic-strength matrix in less than 20 min. The method includes a sample preconcentration column that enables quantification of sodium at low ng/l concentrations. We evaluated method performance by analyzing synthetic samples containing high concentrations of ethanolamine that are representative of samples encountered in power plant waters.

2. Experimental

2.1. Instrumentation

A Dionex DX-600 ion chromatograph (Dionex, Sunnyvale, CA, USA) consisting of a GP50 gradient pump, an EG40 eluent generator, an LC30 chromatography oven, and an ED50A conductivity detector with a conductivity cell and DS3 stabilizer were used

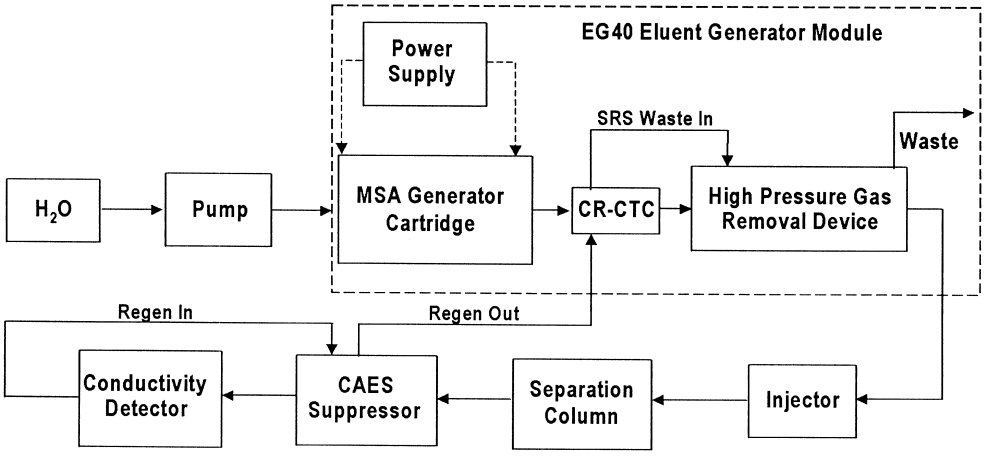

Fig. 1. Schematic diagram of the ion chromatography system.

in all experiments. The PeakNet[1] chromatography workstation was used for instrument control, data collection, and data processing. An EGC-MSA cartridge was installed in the EG40 eluent generator module to provide consistent on-line generation of high-purity MSA. To prevent ionic contaminants from entering the chromatographic pathway, a CR-CTC was installed between the EGC-MSA outlet and the injection valve. The LC30 chromatography oven was used to maintain the temperature of the guard and analytical column at 60 °C. Dionex IonPac CS16 (250×3 mm I.D.) and CG16 (50×3 mm I.D.) columns were used for all analytical separations. For preconcentration of blanks, standards, and samples, an IonPac CG16 (50×5 mm I.D.) column was used. The analytes were separated using an eluent of 20 m*M* MSA at a flow of 0.5 ml/min followed by detection with suppressed conductivity. A cation Atlas electrolytic suppressor (CAES) operated in the Autosuppression recycle mode was used as the suppressor. A schematic diagram of the IC system is shown in Fig. 1.

For sample loading, a DXP pump (Dionex) was used with a pressurizable reservoir chamber (Dionex) at a helium pressure of 5 p.s.i. (34 kPa) to enclose the samples or standards. This chamber prevents additional sample handling, isolates the samples or standards from the environment, and therefore minimizes contamination.

2.2. Reagents and samples

The water used for the preparation of rinse solutions, eluents, standards, and samples was from a point-of-use deionized water purification system. This deionized water was purified by processing the feed water through a pretreatment system with a make-up stage at a maximum resistivity of 200 kΩ cm and polishing stage with a resistivity from 1 to 10 MΩ cm. This processed water was then pumped through the point-of-use water system with four containers that used carbon adsorption in the first stage to remove organics or chlorine from the water. The other three containers contained nuclear-grade deionization cartridges that preceded UV disinfection followed by filtration with a 0.2 μm filter to obtain a resistivity of at least 18.2 MΩ cm. In addition, the system used a pump with a flow-rate of 2 l/min to continually recirculate the water in the system in order to maintain the water quality. Kyereboah-Taylor only reported trace ammonium at 25 ng/l with an almost identical water system used in our experiments (excluding the UV disinfection lamp) [13]. Similar water purification systems have reported as low as 3 ng/l sodium and 30–105 ng/l ammonium [14]. Sodium chloride salt was obtained from J.T. Baker (Phillipsburg, NJ, USA) and ethanolamine (>99.5%) was obtained from Aldrich

[1]IonPac, Autosuppression, Atlas, and PeakNet are registered trademarks of Dionex Corporation and CAES is a trademark of Dionex Corporation. Tefzel is a registered trademark of Dupont.

(Milwaukee, WI, USA). These solutions were used to prepare 1000 mg/l stock standards for sodium and ethanolamine, respectively. Standards containing less than 1 mg/l were prepared every 2–3 days and standards containing less than 1 μg/l were prepared daily and analyzed within a few hours. Working standards were prepared by performing serial dilutions of the stock solutions. Working standards of 25, 50, 100, and 250 ng/l sodium were used to prepare the calibration curve.

To simulate samples typically encountered in the power industry, synthetic samples containing ethanolamine at concentrations of 3000, 5000, and 10 000 μg/l were prepared. Each sample was spiked with 0.025 μg/l sodium. All standards and samples were prepared in 250-ml Corning polystyrene tissue culture flasks that had previously been rinsed and soaked several times prior to use.

2.3. Procedures

As with any system for trace-level ion analysis, there are many sources of potential contamination. Therefore, extreme caution should be taken throughout the entire process to eliminate or at least minimize potential contaminants. A significant number of precautions necessary for performing analyses at the ng/l levels are discussed by Vanatta [15]. However, the addition of the CR-CTC in this method is designed to effectively eliminate the system cationic contaminants. Vanatta found that the best containers were Corning polystyrene tissue-culture flask with a plug seal. We prepared these containers by rinsing each flask and lid 3–4 times with ultrapure water until overflowing. The flask was then completely filled to the top, capped tightly and allowed to soak overnight. This process was repeated until an acceptable and consistent blank was achieved. These containers were used for blanks, standards containing less than 1 mg/l sodium, and simulated matrices. Each flask was dedicated to specific concentrations and was clearly marked to distinguish them from other containers. When a flask was not in use, it was always kept full with deionized water and rinsed/soaked periodically.

On-line sample preconcentration was performed by replacing the sample loop with an IonPac CG16 (50×5 mm I.D.) column. The sample was loaded in the opposite direction of eluent flow to the analytical column. Samples were delivered by using a DXP Pump (Dionex) operating at a flow-rate of 2.0 ml/min. The effluent from the waste line was collected for a set time and the mass was obtained to determine the preconcentrated volume (assuming a density of water of ≈1 g/ml at 25 °C). The pressurized reservoir was used to house the polystyrene container. The cap of the reservoir contained a Tefzel tubing attached to the inlet and polyether ether ketone (PEEK) tubing (0.030 in. I.D.) on the outlet (1 in.=2.54 cm). The Tefzel tubing was always kept in a soaked/rinsed polystyrene container filled with deionized water, regardless of whether the pump was in use. In circumstances where samples or standards had to be exchanged from the reservoir, the cap with the Tefzel tubing attached was carefully removed and the tubing was immersed in a clean polystyrene container filled with deionized water. This container was dedicated for this use only and periodically soaked/rinsed with deionized water. In order to clean the tubing from the pressurized reservoir to the injection valve, deionized water was continuously pumped through the PEEK tubing for a period of 8 h prior to the initial sample analysis.

When a high-ionic-strength sample is preconcentrated, the sample can act as an eluent and prevent the retention of the analytes of interest on the concentrator column [16]. As with any ion-exchange resin, the resin in a concentrator column has a finite capacity (i.e., the column can retain a given amount of ions from a sample matrix). Therefore, the sample volume where the target analytes are no longer retained, defined as the breakthrough volume, must be determined. Here we determined ultra trace levels of sodium in the presence of high concentrations of ethanolamine. Because the amine is present at significantly higher concentrations than the target analyte, it was necessary to determine the breakthrough volume. The breakthough volume was determined as follows: (1) a simulated sample was prepared at the highest anticipated concentration (e.g., 0.25 μg/l sodium) containing 5000 μg/l ethanolamine; (2) increasing volumes of the sample were concentrated and the peak area of sodium was plotted versus the sample volume; (3) the breakthrough volume is the volume where the plot deviates from linearity. Using the IonPac CG16 (50×5 mm I.D.) column, the

breakthrough volume was ≈30 ml. We determined a sample preconcentration volume of 10 ml, well below the breakthrough volume, was sufficient to quantify sodium at the target concentrations.

3. Results and discussion

3.1. Optimization of system parameters

The primary objective of this work was to develop a method based on cation-exchange chromatography using preconcentration to allow for the separation and detection of low ng/l levels of sodium in the presence of high concentrations of ethanolamine. A second objective was to allow the method to be easily transferred to an on-line process chromatography system. The requirement to achieve the first objective is important for the power industry because ethanolamine is normally used as an additive to adjust the pH in the process water. However, ammonium and possibly other analytes may also be present in the matrix as breakdown products of ethanolamine. The additional analytes should be considered when developing a method because these could interfere with sodium determinations. In order to achieve the separation and detection of low ng/l sodium in high concentrations of ethanolamine, certain system parameters must be optimized, such as choice of preconcentration column, analytical column, suppressor, and column temperature.

The separation of sodium in the presence of significantly higher concentrations of ammonium or amines using a simple isocratic eluent is difficult to achieve using previous cation-exchange columns [17]. A possible solution to improve the separation of sodium and still allow for reasonable analysis times is to use a high capacity column with a moderate eluent concentration of MSA. The use of a high-capacity column should permit the desired separation of sodium and provide a significant degree of matrix independence necessary for handling high-ionic-strength matrices, such as ethanolamine-treated waters. In addition to having the appropriate capacity, a column that provides good peak efficiencies is also desirable, especially when analyzing low concentrations of the target analyte. Therefore, the initial goal in developing an optimum method for this analysis was to choose a column that provided a sufficiently high ion-exchange capacity, appropriate selectivity, and good chromatographic efficiencies for the separation of low ng/l levels of sodium in the presence of high concentrations of ethanolamine.

The column chosen for this work consisted of 55% cross-linked polymeric substrate beads grafted with carboxylic acid groups. The high ion-exchange capacity is achieved by using a smaller bead diameter (5 μm) with a high porosity (150 Å), a high density of grafted carboxylated cation-exchange sites, and a larger column format. The grafting conditions were optimized on the polymeric bead to maintain a high efficiency of the analyte peaks [18]. This resin was packed in 250×3 mm I.D. and 250×5 mm I.D. PEEK column bodies to achieve ion-exchange capacities of 3000 and 8400 μequiv./column, respectively. The separation was developed on the 3-mm I.D. column to obtain the advantages of: (1) lower eluent flow-rates, (2) improved peak shapes, and (3) reduced loading times (e.g., lower mass-to-volume ratio than the 5-mm I.D. column). The reduced flow-rate significantly decreases eluent consumption and therefore minimizes the number of eluent changes, thereby reducing the probability of introducing contaminants to the system. The lower eluent consumption also allows for longer periods of unattended operation. The above benefits provide desirable characteristics to allow the method to be easily transferred to an on-line process chromatography system.

A second consideration was to choose an appropriate concentrator column to allow for the quantitation of sodium at the target levels in typical power plant waters. The concentrator should have adequate cation-exchange capacity to quantitatively trap the sodium from the sample matrix, but not be excessively high as to cause severe band broadening of the analyte peak. In addition, the concentrator should have good selectivity for the analytes so they can be effectively removed from the column with the eluent used for the analytical separation. The concentrator should also produce a minimal void volume to avoid interfering with the integration of the target analyte. The same resin used for the analytical separation in a 50×5 mm I.D. column format provided these characteristics, and was therefore chosen as the concentrator column. The 5-mm I.D. column was chosen

over the 3-mm I.D. column as a result of the ca. three times gain in exchange capacity. This higher capacity provides the maximum loading capability while minimizing potential matrix interference.

For accurate determinations at the sub-μg/l levels, it is critical to maximize the peak response while minimizing the baseline noise to obtain an optimum signal-to-noise ratio (S/N). The choice of an appropriate suppressor is important to minimize the baseline noise. For cation-exchange chromatography the suppressor converts the analyte into its base form to increase the analyte signal, converts the eluent to water to significantly reduce the background and baseline noise, and therefore improves the S/N. To obtain the above benefits, a CAES was chosen because it typically provides low baseline noise. Baseline noise for the CAES is generally in the range of 0.2 to 0.5 nS/cm. When the suppressor was operated at the recommended current setting of 34 mA, the peak-to-peak noise measured in a 1 min representative segment of the chromatogram was less than 0.5 nS/cm. To further simplify the method, the suppressor was operated in the autosuppression recycle mode to avoid the preparation of chemical regenerates and allow the method to be easily transferred to an on-line process chromatography system.

Temperature is an additional factor to optimize the analyte response. In general, increasing column temperature can improve chromatographic efficiency, enhance resolution, and significantly reduce retention times. At elevated temperatures, the carboxylic acid groups on the resin are less ionized and therefore decrease the effective column capacity, resulting in shorter retention times and improved sensitivity [19]. The primary benefit for operating at elevated temperatures for this method was a significant improvement in peak efficiencies resulting in an improved resolution between sodium and an unknown analyte peak. Fig. 2 illustrates the separation of sodium and unknown cationic species in 5000 μg/l ethanolamine at 40 and 60 °C. The increase in temperature resulted in ≈30% increase in theoretical plates for sodium and the unknown species allowing improved resolution. An additional benefit was an improvement in the peak efficiency of ethanolamine that resulted in shorter analysis and therefore an increase in sample throughput.

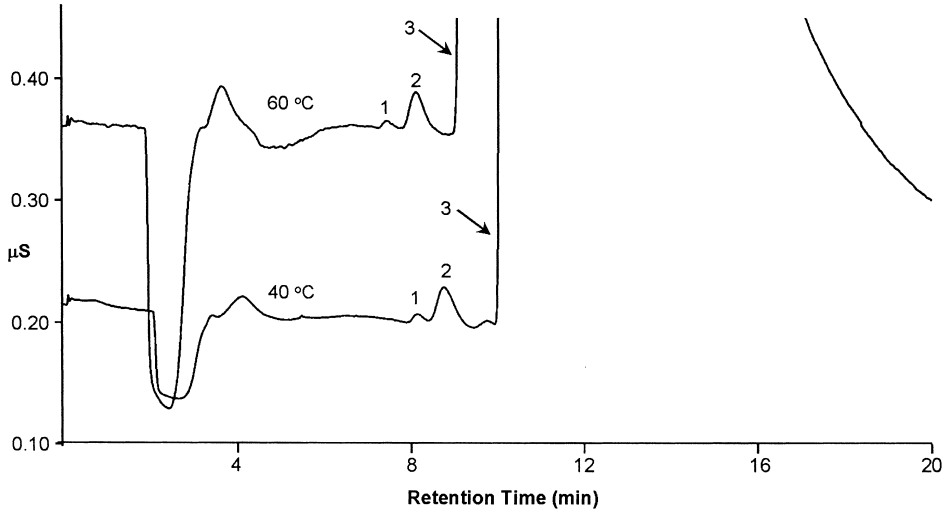

Fig. 2. Chromatograms illustrating the separation of sodium and an unknown cationic species at 40 and 60 °C in 5000 μg/l ethanolamine. Peaks: 1=sodium (~0.005 μg/l), 2=unknown, 3=ethanolamine (5000 μg/l). Sample volume: 10 ml; guard column: IonPac CG16 (50×3 mm I.D.); analytical column: IonPac CS16 (250×3 mm I.D.); concentrator column: IonPac CG16 (50×5 mm I.D.); detection: suppressed conductivity, CAES, recycle mode; eluent: 20 m*M* MSA; eluent flow-rate: 0.5 ml/min.

It is important to initially establish a system blank and ensure the stability of the blank analyses over a period of several days. For the sodium concentrations determined in this paper, it may require several weeks to obtain an acceptable blank. This time depends on the cleanliness of the laboratory, the purity of the water source, the presence of contaminates in the IC system, and other factors that can contribute to high blank levels. In this study, the blank was analyzed over a 4-day period resulting in an average sodium concentration of 4.5±0.7 ng/l (n=21). A representative chromatogram of a 25 ng/l standard is shown in Fig. 3a and its accompanying blank is shown in Fig. 3b. The chromatograms were obtained by preconcentrating 10 ml on the IonPac CG16 (50×5 mm I.D.) concentrator column fol-

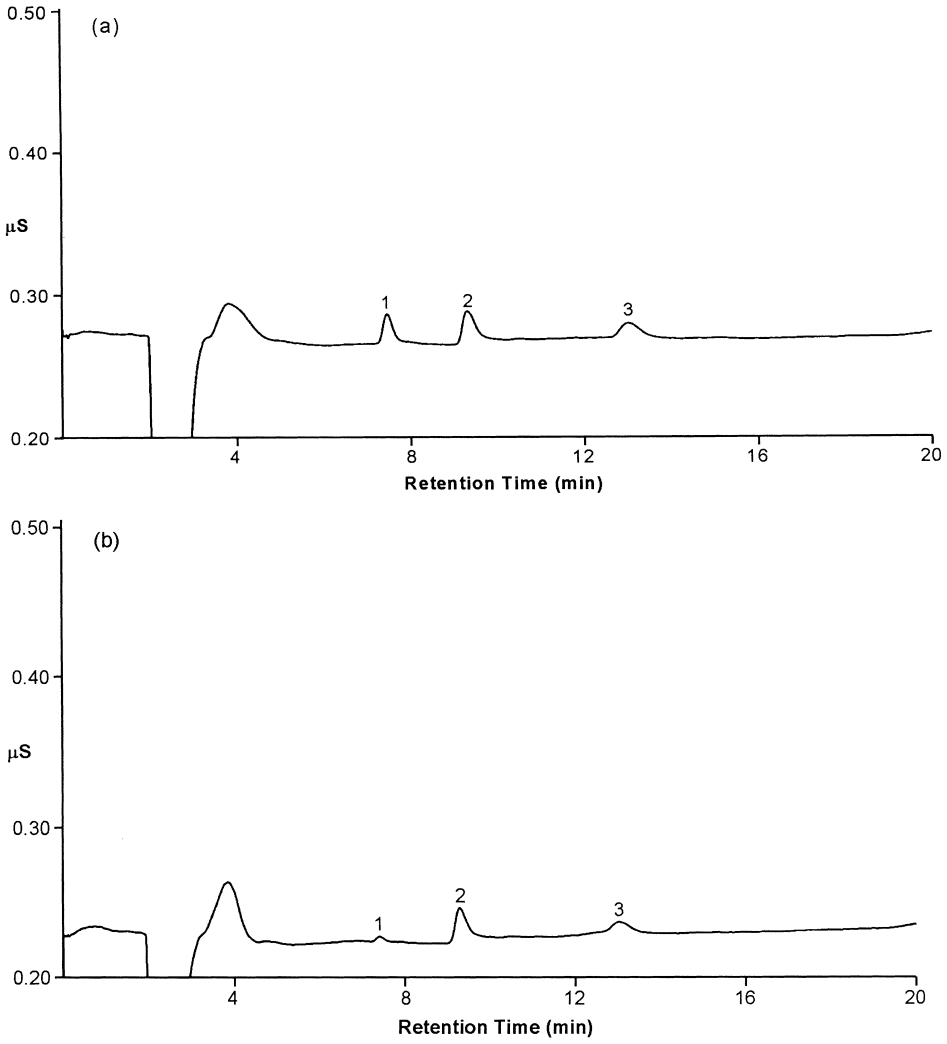

Fig. 3. (a) A representative chromatogram of deionized water spiked with 25 ng/l sodium. Peaks: 1=sodium (25 ng/l), 2=ammonium, 3=unknown. (b) A representative blank chromatogram. Peaks: 1=sodium (4.7 ng/l), 2=ammonium, 3=unknown. Experimental conditions listed in Fig. 1.

lowed by separation with 20 m*M* MSA on the IonPac CS16 (250×3 mm I.D.) analytical column. As shown in Fig. 3b, trace amounts of sodium, ammonium, and an unknown peak from the deionized water were detected.

3.2. Method performance

The calibration curve for sodium was obtained by preparing standards in deionized water. The calibration linearity and method detection limits (MDLs) for sodium are shown in Table 1. The MDL was determined by performing seven replicate injections of 25 ng/l sodium fortified in high purity reagent water. The MDL was calculated by using the standard deviation from seven replicate injections and multiplying by the Student's *t*-value for a 99% confidence level resulting in a calculated MDL of 3.20 ng/l using a 10-ml sample volume. The precision of the retention times for sodium based on the seven replicate injections yielded a relative standard deviation (RSD) of 0.33%.

The method performance was evaluated by analyzing synthetic samples containing ethanolamine concentrations in the range of 3000 to 10 000 μg/l that were designed to simulate samples encountered in the power industry. Fig. 4a shows a chromatogram of 3000 μg/l ethanolamine with no additional sodium added. The average concentration of sodium found in the matrix based on four replicate injections was 5.5 ng/l. This value is within the standard deviation of the blank and therefore indicates that no additional sodium was added upon the addition of the amine. Fig. 4b illustrates a chromatogram obtained by spiking the sample with 22 ng/l sodium. The average recovery for sodium based on seven replicate injections of the 3000 μg/l ethanolamine sample was 99%. Table 2 summarizes the data for the spiked recoveries of sodium in matrices containing 3000–10 000 μg/l ethanolamine. As shown, the average recoveries ranged from 85 to 110%. This indicates that the method performed well for samples spiked with low ng/l sodium and that the high amounts of ethanolamine do not significantly influence the separation and quantification of sodium at the target levels. In addition, the high capacity of the concentrator in combination with the high capacity of the analytical column allowed sodium to ethanolamine ratios of at least 1:400 000 with good spiked recoveries. The retention time precision data for ~0.025 μg/l sodium based on seven replicate injections ranged from 0.06% RSD for sodium in 5000 μg/l ethanolamine to 0.33% RSD for sodium in deionized water. These data indicate that the retention time precision of sodium was independent of the amount of ethanolamine present.

However, the addition of 22 ng/l sodium (Fig. 4b) to this sample, increased the response for the unknown cationic species. We believe this to be insignificant because the peak area for the unknown cationic species varied within a set of replicate unspiked sample injections, within a set of replicate spiked sample injections, and between sets of injections (e.g., between unspiked and spiked sample injections) with RSD variations ranging from 15 to 40%. Therefore, we believe the increase in response of the unknown cationic species is not a result of the sodium standard.

4. Conclusion

An IC method based on a high-capacity, carboxylated cation-exchange column, on-line elec-

Table 1
Calibration linearity and MDL for sodium prepared in deionized water

Analyte	Range[a] (ng/l)	Correlation coefficient[b] (r^2)	Calculated MDL[c] (ng/l)	MDL standard (ng/l)
Sodium	25.0–250	0.9990	3.20	25.0

[a] Calibration levels were 25, 50 100 and 250 ng/l (each standard injected in duplicate).
[b] A 10-ml volume was preconcentrated for each standard.
[c] MDL=tS where t=Student's t-test for a 99% confidence level and a standard deviation estimated with $n-1$ degrees of freedom (t=3.14 for seven replicates of the MDL standard), and S=standard deviation of the replicate analysis.

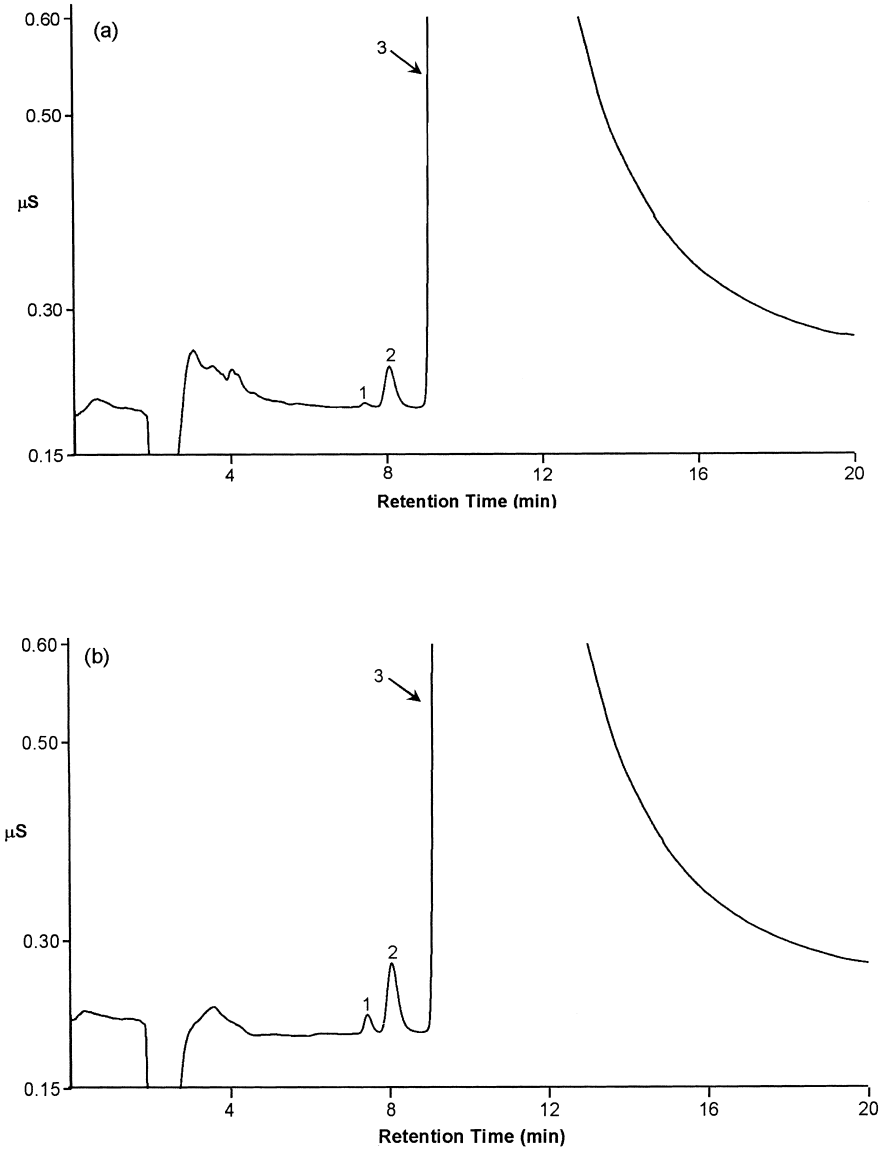

Fig. 4. (a) A representative chromatogram of 3000 μg/l ethanolamine. Peaks: 1=sodium (0.0053 μg/l), 2=unknown, 3=ethanolamine (3000 μg/l). (b) Chromatogram (a) spiked with 0.025 μg/l sodium. Peaks: 1=sodium (0.025 μg/l), 2=unknown, 3=ethanolamine (3000 μg/l). Experimental conditions listed in Fig. 1.

trolytic eluent generation, and an electrolytic suppressor was developed to determine sodium in matrices containing high concentrations of ethanolamine. The column provided good resolution and peak efficiencies at elevated temperatures even in matrices of disparate concentration ratios of sodium to ethanolamine. The method was capable of determining sodium down to low ng/l concentrations in high concentrations of ethanolamine that are typical of samples encountered in the power industry. Method detection limits at the single digit ng/l levels were achieved by optimizing several chromatographic parameters and following the necessary precautions for performing trace-level work.

Table 2
Spiked recovery data for sodium in the presence of 3000–10 000 μg/l ethanolamine using a 10-ml sample volume

Spiked sodium concentration (μg/l)	Ethanolamine concentration (μg/l)	Mean[a] sodium recovery (%)	RSD[a] (%)
0.022	3000	99.0	3.68
0.025	5000	109.7	2.78
0.027	10 000	85.0	6.89

[a] The mean recovery and relative standard deviations were calculated from seven replicate injections ($n=7$).

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