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Ion analysis by capillary zone electrophoresis with indirect injection: applications in the nuclear power industry

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Capillary zone electrophoresis (CZE) was used for the determination of trace level inorganic ions and its applicability in the power industry investigated. Detection of Cl^-, SO_4^{2-} and NO_3^- was accomplished by using a background electrolyte consisting of 7.0 mM CrO_4^{2-}, 0.5 mM TTAB and 1.0 mM $NaHCO_3$. To enhance electrokinetic injection, transient isotachophoretic conditions were employed by the addition of 100 µl of 70 mM sodium octanesulfonate to 100 ml of sample. The addition of a 20 µg l^{-1} aliquot of tungstate was used as an internal standard to limit possible electrokinetic injection biases. In addition, 150 µm extended path length capillaries were investigated for improving detection limits. Detection limits obtained *via* CZE were comparable to those given by ion chromatography (IC) with the use of the extended path capillaries. CZE results obtained were 0.38, 0.54 and 0.75 µg l^{-1} for Cl^-, SO_4^{2-} and NO_3^-, respectively, while IC results were 0.10, 0.30 and 0.25 µg l^{-1}. The advantages of using CZE as opposed to IC for the analysis of inorganic anions in power plant reactor water are discussed.

Keywords: *Ion detection; capillary zone electrophoresis; reactor cooling water; boiler feedwater; ion chromatography*

Ion analysis by capillary electrophoresis was first developed in 1967 by S. Hjerten for the separation of bismuth and copper cations.[1] Tsuda and co-workers separated copper and iron cations in 1983 and in 1987 Zare reported the separation of potassium, sodium, lithium and selected alkylamines.[2,3] Anion analysis *via* capillary electrophoresis was first reported in 1979 by Mikkers *et al.*[4] Capillary zone electrophoresis (CZE) has been used to separate inorganic ions, hydrophobic and hydrophilic organic molecules and large macromolecules, such as proteins, peptides, carbohydrates and catecholamines.[5]

CZE has grown over the last few years into a standard separation method preferred in many applications over conventional liquid chromatographic techniques because of its speed, high efficiency, low operational costs and flexibility. Reported separation efficiencies have been in excess of 500 000 theoretical plates, far beyond the 10 000–20 000 typically reported for liquid chromatography. The increased efficiency has been attributed to the elimination of chromatographic peak spreading due to diffusional contributions.[6] Unfortunately, a simultaneous analysis of cations and anions, by either ion chromatography (IC) or CZE, is not feasible in low-level analysis because of the loss of efficiency, and thus resolution.[7–9]

A detection barrier complicates the analysis of inorganic anions. Owing to the non-absorptive nature of inorganic anions, indirect UV detection is often employed.[10] In the analysis of highly mobile anions, such as chloride, sulfate and nitrate, a chromate containing background electrolyte (BGE) has been optimized.[11–14] A direct, conductimetric detection scheme, as employed in IC, is often unsuitable without suppression because of the high background conductance of the running buffer. Current research initiatives include the application of suppressed conductivity techniques and direct conductivity measurements based upon low ionic strength running buffers.[15]

Anion analyses are customarily performed with the assistance of an electroosmotic flow (EOF) modifier, such as tetradecyltrimethylammonium bromide (TTAB).[16,17] The addition of an EOF modifier produces a change in the normal direction of the EOF, cathode to anode, thus increasing the speed of the anion separation. Cation analyses are performed under normal anode to cathode EOF, often with complexing agents and BGEs such as citrate, α-hydroxyisobutyric acid (HIBA) and imidazole.[5]

In boiling water reactors (BWR), the coolant is ultra-pure water. Conductivity is frequently below 0.090 µS cm^{-1}. Reactor water chemistry is in a constant state of dynamic equilibrium, dependent on various system inputs and operating conditions. To mitigate detrimental effects, such as intergranular stress corrosion and cracking (IGSCC) and fuel cladding failures due to crud-induced localized corrosion (CILC), contaminants such as Cl^- and SO_4^{2-} must be maintained at a minimum.[18,19] Pressurized water reactors (PWR) rely upon a borated coolant in the primary or nuclear side and aminated water for corrosion inhibition in the secondary or turbine side. Amines, such as ethanolamine, 5-aminopentanol, aid in oxygen scavenging and passivation of metal surfaces in the condensate–feedwater system, inhibiting general and localized corrosion of ferrous materials and reducing pitting susceptibility of alloys.[20] Controlling the ingress of undesired components is a costly and time consuming endeavor within the power industry.

In both BWRs and PWRs, some common analytes are Cl^-, SO_4^{2-}, NO_3^-, Na^+ and various organic acids. These analytes are monitored regularly *via* ion chromatography and have become indicators of overall plant performance. In this paper we report CZE analytical methodology that uses sample electrostacking procedures in combination with the use of extended path length capillaries for trace analysis of common anions found in nuclear power plant reactor water. The CZE analytical methodology employed was compared with existing IC methodology for the analysis of trace anions in nuclear power plant reactor water. Evaluations of each technique were made with respect to detection limits, operational costs, sample throughput and overall analytical performance.

Experimental

Apparatus

Analyses were performed with a 3-D capillary electrophoresis system (Hewlett-Packard, Wilmington, DE, USA) utilizing a photodiode array detector (DAD) at 375 nm, bandwidth 40 nm.

Two polyimide-coated fused silica capillary columns (350 μm) obtained from Hewlett-Packard were employed: (*i*), an 80.5 cm × 75 μm capillary equipped with a 150 μm 'extended' path length in the detector region; and (*ii*), a 50 μm 'normal' capillary at 64.5 cm × 50 μm. The lengths from the inlet to the detector were 72.0 and 56.0 cm, respectively. Sample injection was performed electrokinetically at an applied voltage of −5 kV for 45 s. BGE replenishment was performed after each injection to minimize migration variance. Data acquisition and analysis was performed with H-P 3DCE ChemStation software. The detector time constant was set at 0.1 s and data acquisition rate was 10 points s^{-1}.

The IC instrument used in this study was a Dionex 2020I (Dionex Corporation, Sunnyvale, CA, USA) and a CDM-1 conductivity detector with A-SRS auto-suppression. The separation chemistry employed an AG12 for pre-concentration and AS12 for separation with 2.5 mM Na_2CO_3–0.5 mM $NaHCO_3$ as the eluent. The sample loading volume was 30 ml, loading being accomplished by a constant load volume apparatus. Shown in Fig. 1, the constant volume apparatus enabled consistent volumes to be preconcentrated onto the guard column (Dionex AG12). Data acquisition and analysis were performed with Dionex AI-450 V3.30 software. The data acquisition rate was 5 points s^{-1}.

Materials

The chromate electrolyte system was prepared from 100 mM sodium chromate tetrahydrate (Mallinckrodt, Paris, KY, USA: analytical grade) and 0.0056 mM sulfuric acid (J.T. Baker, Phillipsburg, NJ, USA; Ultrex grade). The electrooosmotic flow modifier was 20 mM concentrated tetradecyltrimethylammonium bromide (TTAB) obtained from Waters (Waters Corp., Milford, MA, USA). Final working electrolyte concentration employed was 7.0 mM CrO_4^{2-}, 0.5 mM TTAB and 1.0 mM $NaHCO_3$ (J.T. Baker), pH 9.1. Sodium octanesulfonate (NaOS) from Fluka (Fluka Chemika-BioChemika, Buchs, Switzerland) was employed as the terminating electrolyte by the addition of 100 μl of 70 mM NaOS to 100 ml of sample.

The stock eluent for IC was prepared by dissolving 26.5 g of anhydrous Na_2CO_3 (J.T. Baker) and 4.15 g of anhydrous $NaHCO_3$ (J.T. Baker) in 1 l of 18 MΩ water for final concentrations of 250 mM and 50 mM Na_2CO_3–$NaHCO_3$. This stock eluent solution was further diluted 1:100 for an eluent strength of 2.5 mM Na_2CO_3 and 0.5 mM $NaHCO_3$.

Standards and simulated test samples were prepared from 1000 mg l^{-1} standards obtained from J.T. Baker diluted in 18 MΩ water. Tungstate was added as an internal standard at 20 μg l^{-1}. Standards and samples simulating the secondary PWR system were prepared as the BWR standards and samples with the addition of the specified corrosion inhibitor, such as ethanolamine, at a standard concentration of 10 mg l^{-1}. Amines evaluated were ethanolamine, morpholine, 5-aminopentanol and methoxypropylamine.

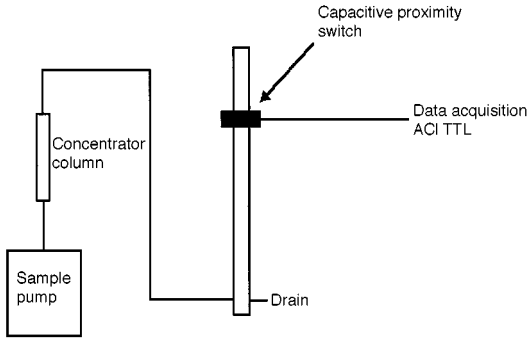

Fig. 1 Constant volume apparatus. The constant volume apparatus enabled consistent volumes to be preconcentrated onto the guard column (Dionex AG-12). When sample loading begins, the effluent stream from the guard column is diverted into an acrylic sample tube. Attached to the sample tube is a capacitive proximity switch and once the sample pump has loaded the sample to the level prescribed by the proximity switch, a signal is sent to the data system, *via* the advanced computer interface, shutting off the sample pump and beginning sample analysis. After the analysis has been completed, the data system signals the drain valve to open and re-set the system. The sample volume can be increased or decreased according to sample type by moving the position of the proximity switch. For trace analysis, *e.g.*, reactor water, the load volume was approximately 30 ml. This provides reproducible sample loading, which decreases variance and, consequently, decreases the IC LODs.

Methods

Rinsing with 1 M sodium hydroxide (NaOH) for 20 min followed by subsequent rinses with 0.1 M NaOH and demineralized water for 20 min each activated the capillary. The final rinse was performed with the operating buffer prior to use. The capillary was purged for 2 min and inlet/outlet vials replenished after each run with the BGE. Sample vials were prepared by soaking in demineralized water for a minimum of 24 h. The vials were subsequently rinsed three times each with demineralized water and sample to reduce possible contamination.

Internal standardization using tungstate (WO_4^{2-}) at 20 μg l^{-1} was employed to decrease possible bias (see Fig. 4) due to electrokinetic injection.[21,22] Analyses with recoveries of 90–110% WO_4^{2-} were considered valid, with no associated injection bias.

Calibration curves were created by plotting the normalized peak area *versus* concentration (Fig. 2). A linear regression was performed to determine slope and intercept that would allow for the quantitation of unknowns. Correlation coefficients (r^2) were calculated and used to evaluate linearity. Detection limits were calculated by determining 3σ of 30 or more data points of the 10 μg l^{-1} standards. IC calibrations were performed with 10, 20 and 40 μg l^{-1} (IC 2) and 2 and 10 μg l^{-1} (IC 3 and 4) standards. Detection limits were calculated based upon 3σ of 30 or more data points of 2 μg l^{-1} standards.

Theory

In CZE, analyte separation occurs based upon the differing electroosmotic mobilities (μ_{ep}) of the ions in the BGE when an electric field is applied through a capillary. Electroosmotic (μ_{eo}) flow reversal is essential in the analysis of anionic species, as the μ_{ep} of the analyte may exceed the μ_{eo} under normal CZE

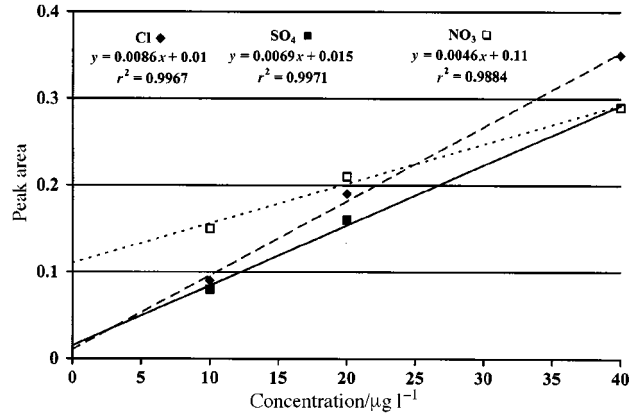

Fig. 2 Calibration curve, extended path detector. Calibration curve prepared using standards of 10, 20 and 40 μg l^{-1}. Correlation coefficients for Cl^- and SO_4^{2-} were better than 0.995, denoting linearity for analytes. NO_3^- shows deviation from linearity and possible contamination based upon intercept value.

conditions and thus the analyte will not reach the detector. Additionally, increased efficiencies are obtained when the analytes of interest migrate in the same direction as the EOF.[15,16]

The chromate BGE is used because of similar electrophoretic mobility to the three anionic contaminants studied, Cl^-, SO_4^{2-} and NO_3^-, broad UV absorption characteristics and high molar absorptivity. As the mobility differences of the co-ion and analyte increase, peak shape will become less symmetrical.[23] As the analyte mobility decreases, peaks tail; as mobility increases, analyte peaks will begin to front. Therefore, chromate satisfies the requirements established by the first and second co-ion conditions.[24,25]

Electrokinetic injection with an isotachophoretic terminating analyte is employed as a pre-concentration step in trace analysis.[9] The addition of NaOS fulfills the requirement for a terminating electrolyte by allowing the analytes of interest to 'stack' in low ionic strength samples by permitting a sufficient electric charge for ionic transfer to occur from the bulk sample solution into the capillary.[22,25] Also, the addition of WO_4^{2-} as an internal standard eliminates the reporting of inaccurate data due to electrokinetic injection bias.

In an attempt to compare the performances of the different capillaries, a number of parameters are evaluated, which include apparent mobility (μ_{app}), resolution (R_s), theoretical plates (N), capacity (k') and tailing factors (USP t).

Apparent mobility is calculated according to eqn. (1), where L_d = length to the detector, L = total capillary length, t_m = migration time and V = applied voltage.

$$\mu_{app} = \frac{L_d \times L}{t_m \times V} \quad (1)$$

USP tailing factors [eqn. (2)] are calculated to further compare the performance of the 150 and 50 μm path width capillaries in BWR and PWR matrices:

$$\text{USP } t = \frac{W_{0.05}}{2 \times t_w} \quad (2)$$

where t_w = distance between peak front and t_r at 5% peak height; units are the same as used for $W_{0.05}$ (width at 5% peak height).

Because of the velocity dependence of CZE peaks, the normalized peak areas are used for quantitation.[5]

$$A_{norm} = \frac{A}{t_m} \quad (3)$$

Results and discussion

Data analysis

According to Beer's Law, $A = \varepsilon bc$, sensitivity and detection ranges can be improved by increasing the detector path length or molar absorptivity. Chromate has a high molar absorptivity at the wavelengths employed, therefore further increases in sensitivity can be achieved by expanding the detector path length. A factor limiting the amount by which the detector can be enlarged is the increase in current and subsequent increase in Joule heating. By increasing the path length only in the detector region, no increase in current is experienced and the flow velocity decreases due to decreasing field strength. This decrease in velocity in essence stacks the analyte zones, thereby increasing the signal to noise ratio and sensitivity. Based upon detection limit calculations in the BWR matrix, a significant improvement in detection limits is realized with Cl^- and SO_4^{2-} by increasing the path length (Table 1).

Migration times within the BWR simulated matrix were extremely stable for the ionic strengths employed in both the 'normal' and 'extended' path capillaries. In both the 150 and 50 μm path capillaries, the % RSD of the migration times for all analytes was less than 0.20%. Specifically, Cl^- was 0.10 and 0.15%, SO_4^{2-} was 0.11 and 0.15% and NO_3^- was 0.12 and 0.16%. The migration times for analytes in the ETA and 5-AP matrices increased significantly, but were stable with t_m % RSDs less than 1.5. The increase in migration time can be attributed to an increase in viscosity or a change in the capillary zeta potential. Unfortunately, migration times within the morpholine and methoxypropylamine matrices were extremely unstable, hence the data was not useful.

Detection limits in the BWR matrix were higher than IC detection limits in both capillaries (Table 1). Cl^- limits of 0.38 and 0.58 μg l^{-1} were achieved in CZE, while IC was as low as 0.1 μg l^{-1}. Similar detection limits were obtained for both SO_4^{2-} and NO_3^-. Clearly, the use of the extended path length capillary yields detection limits that are lower than those obtainable with the normal capillary (50 μm), as shown in Table 1. The detection limit results shown using the normal capillary are comparable to results previously reported in which similar experimental conditions were used.[11] Compared with the BWR matrices, there was a significant decrease in signal to noise ratios in the aminated matrices, drastically reducing the sensitivity (Table 1).

Apparent mobilities (μ_{app}) were constant in the BWR matrix (Table 2), regardless of the capillary employed, as predicted by theory [see eqn. (1)]. A shift of greater than 25% was encountered in the varying matrices. Apparent mobilities for the BWR matrix were 0.989×10^{-4} to 1.06×10^{-4} cm^2 V^{-1} s^{-1} and 7.43×10^{-5} to 7.94×10^{-4} cm^2 V^{-1} s^{-1} for the amine matrices. This drastic shift in μ_{app} for the amine matrices indicates either an interaction with the capillary wall or viscosity effects.[26]

Table 1 Comparison of IC–CE detection limits—BWR and PWR matrices*

Method	Cl^-/μg l^{-1}	SO_4^{2-}/μg l^{-1}	NO_3^-/μg l^{-1}
IC BWR matrix—			
(System 2)	1.26	1.47	1.96
(System 3)	0.10	0.30	0.25
(System 4)	0.14	0.46	0.23
CZE BWR matrix—			
50 μm 'normal'	0.58	0.82	0.81
150 μm 'extended path'	0.38	0.54	0.75
PWR matrices—			
150 μm 'extended path'			
5-Aminopentanol	1.20	1.09	0.97
Ethanolamine	2.00	2.60	4.89

* Ion chromatographs were calibrated at 10, 20 and 40 μg l^{-1} (system 2) and 2 and 10 μg l^{-1} (systems 3 and 4). Capillary zone electrophoresis was calibrated at 10, 20 and 40 μg l^{-1} (system 2). Detection limits were calculated by 3 times the sample standard deviation of thirty 10 μg l^{-1} standards.

Table 2 Apparent mobilities (cm^2 V^{-1} s^{-1})*

Column/matrix	Cl^-	SO_4^{2-}	NO_3^-
50 μm/water	1.07×10^{-4}	1.04×10^{-4}	1.01×10^{-4}
150 μm/water	1.06×10^{-4}	1.02×10^{-4}	9.89×10^{-5}
150 μm/5-AP	7.94×10^{-5}	7.57×10^{-5}	7.43×10^{-5}
150 μm/ETA	7.92×10^{-5}	7.56×10^{-5}	7.43×10^{-5}

* The apparent mobilities in both BWR pure water and PWR amine matrices are shown to demonstrate effects of amines on analysis. Sample pre-treatment of aminated sample matrices is required to remove or lessen effects.

Table 3 illustrates the high separation efficiency values that are attained in CZE. In all the BWR analyses, efficiency exceeded 177 000 theoretical plates for 150 μm capillaries and 270 000 for 50 μm capillaries. Baseline resolution (R_s > 1.5) was obtained in all test matrices. USP tailing factors were less than 2 in all analyses (see Figs. 3 and 4), with NO_3^- exhibiting the poorest performance, due primarily to its high diffusion coefficient.

Multiple BWR samples were analysed under the same conditions as established for calibration and detection limit determination. As shown in Table 4, good correlation exists for analyses performed *via* IC and CZE. A notable exception is illustrated in the NO_3^- data. Generally, CZE NO_3^- data is low in comparison with values obtained *via* IC. This can be attributed to the contamination of NO_3^- found in the calibration curves. The NO_3^- contamination has been traced to organic contaminants in the source water, with subsequent UV oxidation releasing the NO_3^- ion. Unfortunately, since the detection limits are greater in CZE, samples requiring lower detection limits, such as reactor water, must be analysed *via* IC at this time.

Table 3 Comparison of chromatographic figures of merit for normal and extended path capillaries*

	USP tailing	R_s	*N*	α
50 μm 'normal'—				
Cl^-	1.10		330 571	
SO_4^{2-}	1.40	4.58	413 857	1.04
NO_3^-	1.73	3.21	272 829	1.03
150 μm 'extended path'—				
Cl^-	1.06		317 139	
SO_4^{2-}	1.35	4.46	371 434	1.04
NO_3^-	1.66	2.58	177 725	1.02
5-AP (150 μm) 'extended path'—				
Cl^-	0.94		202 795	
SO_4^{2-}	1.16	5.60	402 621	1.05
NO_3^-	1.18	1.85	199 269	1.02
ETA (150 μm) 'extended path'—				
Cl^-	1.13		264 746	
SO_4^{2-}	1.19	5.29	320 191	1.05
NO_3^-	1.40	1.90	210 673	1.02

* Tailing factors, resolution, efficiency and selectivity calculated using 10 μg l^{-1} samples.

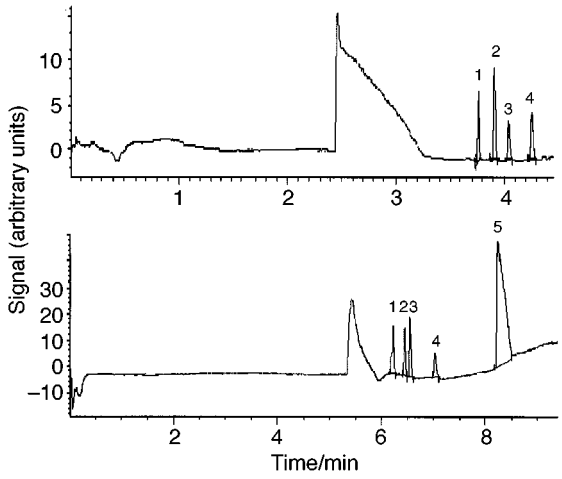

Fig. 3 Representative electropherograms, water matrix. Top: normal capillary; bottom: extended path capillary displaying analyte peaks 1, chloride, 2, sulfate, 3, nitrate, 4, tungstate and 5, carbonate. Standard concentration was 10 μg l^{-1} in pure water matrix.

Cost to benefit analysis

At the forefront of cost to benefit calculations is analysis time. Current IC analyses require a minimum of 15 min for laboratory and up to 90 min for in-line systems. The analysis *via* CZE, as illustrated by Figs. 3 and 4, can be completed within 5–7 min. A resultant increase laboratory sample throughput by a minimum factor of three can be realized over IC analyses.

Material costs are significantly decreased in CZE compared with IC. Based upon the life expectancy of columns, typical costs per year are $10–200 for CZE and $2100 for IC, assuming two concentrator, guard and separator IC column replacements. Also, self-regenerating suppressors are not required with CZE, reducing costs yet again. Assuming a flow rate of 1.3 ml min^{-1}, an IC in continual operation will require 680 l of eluent per year, compared with approximately 52 l of BGE for CZE. Mobile phase costs are reduced substantially, hence radioactive waste generation and reprocessing costs decrease.

Two major concerns within the nuclear power industry are personnel exposure and radioactive waste generation. 'As low as reasonably allowable (ALARA)' is an important fundamental principle that can be achieved with the diminished sample size of CZE. Also, capillary columns have a significantly longer life expectancy, minimizing waste generation, *i.e.*, columns and suppressors.

Finally, CZE systems can be transformed from anion to cation to organic analysis with ease, increasing the flexibility of the laboratory. Inasmuch as an IC is a dedicated system, only one type of analysis may be performed per system without expensive and time-consuming modifications.

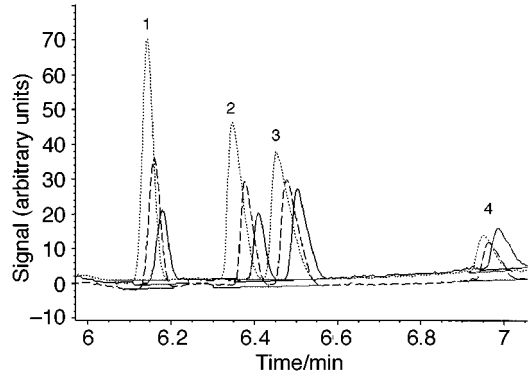

Fig. 4 Tungstate internal standard electropherogram. Calibration standards overlay with internal tungstate in BWR matrix: 10, 20 and 40 μg l^{-1} for 1, Cl^-, 2, SO_4^{2-}, 3, NO_3^- and 4, 20 μg l^{-1} WO_4^-.

Table 4 IC–CE data (μg l^{-1}) for various sampling points*

Sample	IC	CE	IC–CE
EDST-A†—			
Cl^-	1.30	1.10	1.18
SO_4^{2-}	6.10	5.80	1.05
NO_3^-	8.20	7.50	1.08
CST‡—			
Cl^-	0.50	< 0.40	—
SO_4^{2-}	5.90	6.10	0.97
NO_3^-	7.40	6.70	1.10
U1RWCU§—			
Cl^-	< 0.10	< 0.40	—
SO_4^{2-}	0.98	1.05	0.93
NO_3^-	4.30	3.20	1.34

* Comparison of results obtained from capillary zone electrophoresis (CZE), 'extended' path capillary (150 μm) and ion chromatograph (IC). † EDST-A Equipment drain sample tank A (aqueous sample). ‡ CST Condensate storage tank. § U1RWCU Unit 1 reactor water cleanup demineralizer effluent (reactor water sample point).

Conclusion

Capillary electrophoresis for the determination of inorganic ions requires extreme care in standard and sample preparation due to diminished injection volumes. Increased separation efficiency and improved selectivity allow for rapid analysis of complex samples compared with liquid chromatographic techniques. Increases in sensitivity and detection ranges, which approach limits of IC, can be realized using 'extended' path length capillary columns. Based upon cost to benefit calculations, ion analysis *via* capillary electrophoresis provides a low cost alternative to ion chromatography in most analyses performed within the nuclear power industry. Depending upon required detection limits, up to 90% of routinely performed analyses may be performed with current capillary electrophoresis systems. Since the detection limits of externally calibrated indirect CZE are factors of three to five greater than IC, increases in detection limits may be achieved through methods of standard additions for quantitation and peak area correction by internal standardization.

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