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Determination of nitrate and nitrite in Hanford defense waste by reverse-polarity capillary zone electrophoresis

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Abstract

This paper describes the first application of reverse-polarity capillary zone electrophoresis for rapid and accurate determination of nitrate and nitrite in Hanford defense waste (HDW). The method development was carried out by using synthetic Hanford waste, followed by the analysis of four real HDW samples. Hexamethonium bromide (HMB) was used as an electroosmotic flow modifier in a borate electrolyte at pH 9.2 to decrease the electroosmotic flow, enhancing the speed and resolution of the analytical determination of nitrate and nitrite in high ionic strength HDW samples. The application of this capillary zone electrophoresis method, when compared with ion chromatography for two major components of HDW, nitrate and nitrite, slightly reduced analysis time, eliminated most pre-analysis handling of the highly radioactive sample, and cut analysis wastes by more than two-orders of magnitude. The migration times of nitrate and nitrite in the real HDW and the spiked HDW samples were within a precision of less than 3% relative standard deviation. The selectivity ratio test used for peak confirmation of the spiked samples was within 96% of the real sample. Method reliability was tested by spiking the matrix with 1.0 mM nitrate and nitrite. Recoveries for these spiked samples were 93–103%. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The characterization of more than 60 million gallons of radioactive and chemical wastes stored in 177 underground tanks at the US Department of Energy Hanford Site near Richland, WA, USA has

been difficult due to the complex composition of the wastes [1,2] and high analytical costs [3] involved in the process. These wastes, known as Hanford defense waste (HDW), were accumulated during World War II and the Cold War when the mass production of plutonium for making nuclear weapons was a top priority of the USA. The tanks where the wastes are stored vary greatly in chemical and radiochemical compositions as a result of many different waste processing and treatment procedures used over the years. Tank composition has been further complicated by mixing one tank with another. The HDW

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has a very high pH and a very high ionic strength. Part or all of the liquid contents of some tanks were pumped out, resulting in a multiphase mixture of solid sludge, saltcake, slurry and supernatant liquor largely made up of various metals ions, including radionuclides. In addition, nitrate, nitrite, hydroxide, carbonate and a number of organic complexants are also present. The tanks have high nitrate and nitrite content because metal nitrates, nitric acid and sodium nitrite were major constituents used in the processing and recovery of plutonium [4–6].

Methods for determining nitrate and nitrite such as spectrophotometry [7] and the flow injection analysis cadmium reduction method [8] cannot be used for the analysis of HDW because dissolved organic matter and metal ions present in HDW interfere. Ion chromatography (IC) is the method commonly used for the analysis of nitrate and nitrite in HDW [9,10]. Some ion interferences have been reported in IC method for anions [9]. Moreover, the IC method generates high volumes of additional mixed wastes that are expensive to dispose of, resulting in increased cost of analysis per sample.

One analytical method not yet investigated for the determination of nitrate and nitrite in HDW is capillary zone electrophoresis (CZE). Several CZE analyses of nitrate and nitrite in aqueous and other samples have been reported [11–22]. Some of these CZE methods involved the use of acidic electrolytes and indirect detection based on organic acids and chromate electrolytes at pH 6–8 range [11–15]. Others involved the use of direct UV detection [16–18] and conductivity detection methods [19–22]. In the CZE of a very complex matrix like the Hanford Waste, the electrolyte and pH must be selected to insure that precipitation of the sample does not occur during the analyses. The use of acidic electrolytes requires undesirable sample preparation steps to acidify the highly basic HDW. The high ionic strength and high absorbing background of HDW also interfere with indirect detection. Although the conductivity method of detection has been shown to give lower detection limits, separation efficiency decreases [19]. A mismatch in mobilities between the background electrolyte and the analytes [21] is required for conductivity detection [22]. In addition, conductivity based commercial CE instruments are expensive.

The overall objective of this project was to develop a simple, reliable and rapid method based on reverse-polarity CZE for direct UV detection of nitrate and nitrite in HDW under basic sample conditions. Reverse-polarity CZE is a technique that should lead to waste minimization, significantly reducing the overall cost of the Hanford site characterization. Reverse-polarity CZE involves the application of negative voltage at the injection position of the CE instrument. The method can be anion specific because cations and neutral compounds migrate away from the detector and are not detected. Because analyzing HDW samples under high pH involves minimal sample preparation and sample handling, the reverse-polarity CZE method can be used for the analysis of HDW without adjusting the pH of the sample.

Specific objectives of this study were to determine whether reverse-polarity CZE could be used for the separation of nitrate and nitrite in a high ionic strength sample; to examine anions and complexants commonly found in HDW which may interfere with the analysis; and to demonstrate the method with both synthetic Hanford waste (SHW) and real HDW samples. Micro-sample sizes were used for the analyses to minimize the exposure of workers and equipment to radiation.

2. Materials and methods

2.1. Instrumentation

Two capillary electrophoresis instruments were used in this investigation. A Dionex Capillary Electrophoresis System (CES) I (Dionex, Sunnyvale, CA, USA) was used for the study involving SHW samples. The CES I system was equipped with a tunable UV–visible detector with a 190–800 nm wavelength range and a high-voltage power supply with a 0–30 kV range. Also a Waters Model Quanta 4000E capillary ion electrophoresis (water trade name: Capillary Ion Analysis) system (Waters Millipore, Milford, MA, USA), connected to a 486 personal computer for instrument control, was used for the preliminary analysis of the real HDW samples. The Waters instrument had a high voltage power supply in the 0–30 kV range. A 215 nm

wavelength filter was used for the detection of analytes.

2.2. Reagents and chemicals

Sodium nitrate analytical reagent, ACS Specifications (Mallinckrodt, St. Louis, MO, USA), and sodium nitrite Baker analyzed reagent, ACS Specifications, (J.T. Baker, Phillipsburg, NJ, USA) were dried at 110°C overnight, cooled and stored in a desiccator. Standard stock solutions containing 71.42 mM sample were made following US Environmental Protection Agency (EPA) Method 300.0 specifications [23]. Sodium borate (Matheson Coleman & Bell, Norwood, OH, USA), and hexamethonium bromide (HMB) [24] (ICN Biochemicals, Cleveland, OH, USA) were used for electrolyte preparations. The aqueous solution of hexamethonium bromide is acidic; the pH of 100 mM solution in water was 4.62. HMB partially converts to the hydroxide form on mixing with borate to maintain the pH of the electrolyte at pH 9.2. Reagent water from a 18 M Ω Nanopure UV water system (Barnstead–Thermolyne, Dubuque, IA, USA) was used for the preparation of electrolyte and all standard solutions.

2.3. Methods

2.3.1. Optimization of reverse-polarity CZE voltage and the analysis of SHW² (SHW)

The effect of negative voltage on the separation of nitrate and nitrite was examined for up to –22 kV across a 50 cm \times 50 μ m I.D. bare silica capillary column for 25 mM borate–25 mM HMB electrolyte. This study was performed by gravity injection of a mixture of 1.0 mM nitrate and 1.0 mM nitrite standards for 60 s at a 100 mm injector head height. The nitrate and nitrite were detected by direct UV measurement at 220 nm wavelength. The SHW was a simulated HDW sample matrix designed to emulate as closely as possible the composition and behavior of the major components of HDW. The SHW mixture contained 1.02 M Na₂Al₂O₄, 1.61 M NaOH, 2.59 M NaNO₃, 2.24 M NaNO₂ and 0.42 M

Na₂CO₃. This mixture was high in ionic strength, exhibited a high viscosity, and had a pH>13. For electrophoresis, the SHW sample was diluted to 0.016% (v/v). Standard addition calibration was used for quantification by adding 0.000, 0.0235, 0.118, 0.589 and 1.18 mM nitrite standards and 0.000, 0.0871, 0.436, 2.18 and 4.36 mM nitrate standards to the sample. Replicate reverse-polarity CZE runs were carried out under the optimum voltage of –20 kV.

2.3.2. Interference study of anions and complexants commonly found in Hanford defense waste tanks

The separation of nitrate and nitrite in the presence of chloride, sulfate, carbonate and phosphate anions was carried out under the electrophoretic conditions described in Section 2.3.1. Also, interference studies were conducted for several common complexing reagents. The concentrations of the complexant standards added were 10 mM oxalate, 1.8 mM nitrilotriacetic acid (NTA), 0.90 mM ethylenediaminetetraacetic acid (EDTA), and 0.90 mM *N*-hydroxyethylethylenediaminetriacetic acid (HEDTA) at sample pH of 12. For detection of nitrate and nitrite in the presence of the complexants, the detector wavelength was set at 240 nm, the absorption maxima for the complexants.

2.3.3. Analysis of real HDW samples

For the purposes of sample identification, the real HDW samples analyzed were assigned the numbers S96R000308, S96R000309, S96R000311 and S96R000312. The preliminary analysis of the real HDW samples involved a serial dilution of 100 μ l HDW to a final concentration of 0.138% (v/v) in reagent water. The real HDW samples were spiked by adding nitrate and nitrite standard solutions to a final concentration of 1.0 mM in the sample. The percent recovery of the matrix spike was used as a reliability test and the standard addition calibration used as an accuracy check of the data obtained by external calibration. Three reverse-polarity CZE replicate runs were carried out in a 50 cm \times 50 μ m I.D. capillary column. A constant current maintained at 75 μ A was applied, rather than a constant voltage, because constant current gave more reproducible

²SHW supplied by Westinghouse Hanford, Richland, WA, USA.

migration times. In addition, a 30 s rinse with 0.5 M hydroxide was carried out between runs to maintain a more reproducible migration time. Gravity injection was used for sample introduction by raising the injector to a height of 9.8 cm for 30 s sampling time.

3. Results and discussion

3.1. Optimization of reverse-polarity CZE applied voltage and the analysis of SHW

The optimization of the applied voltage for the separation of nitrate and nitrite in SHW was investigated in order to achieve a low detection limit, high separation efficiency and rapid analysis time. Fig. 1 shows the electropherogram of nitrate and nitrite standards containing 1.0 mM of each analyte under the optimal applied potential of -20 kV. As the figure indicates, the separation gave a very good peak shape, a high resolution and a short migration time. However, when voltage greater than -20 kV was applied for the reverse-polarity CZE runs, Joule heating produced a pronounced noisy background [26]. The noise limited the lowest detectable concentrations of nitrate and nitrite to $50 \mu\text{M}$. The highest achievable voltage in the 25 mM borate– 25

mM HMB electrolyte was -22 kV. When voltage greater than -22 kV was applied across the $50 \text{ cm} \times 50 \mu\text{m}$ I.D. capillary, an arc short occurred. The high power output caused the electrolyte to boil, which in turn produced micro-bubbles in the capillary, causing an arc within the high voltage circuit. Thus, optimum voltage for the separation of nitrate and nitrite in SHW was -20 kV.

For the separation of lower analyte concentrations, low voltage was required to reduce this temperature effect. Alternatively, a low ionic strength electrolyte could be used for the analysis of such low concentration samples [18]. Although greater than -20 kV voltage could be used in a low ionic strength electrolyte, the high ionic strength of the HDW samples degraded resolution under such conditions [18,25]. Because an increase in electrolyte/sample ionic strength ratio also increases the resolution, the separation of high ionic strength samples requires high ionic strength electrolyte. The separation of nitrate and nitrite in SHW was thus carried out under high ionic strength for both the sample and the electrolyte. Fig. 2 is the electropherogram of 0.016% (v/v) SHW, showing a rapid separation of nitrite and nitrate within 3 min. The standard addition method was used for quantification, and the results obtained were within 95% agreement with the amount used in

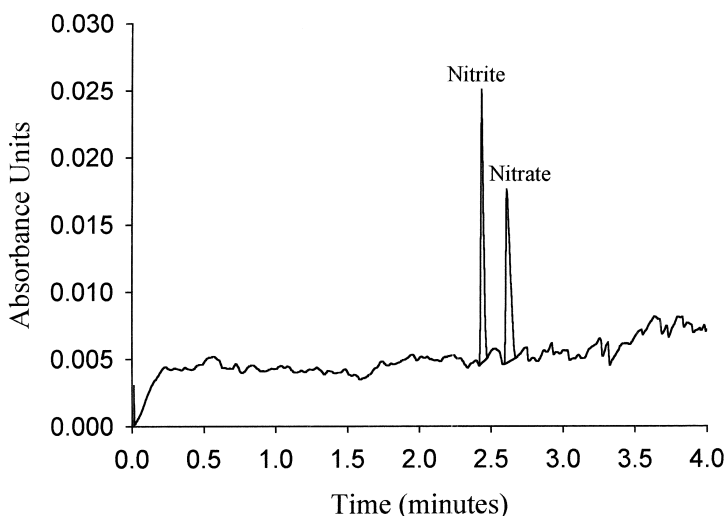


Fig. 1. An electropherogram demonstrating the rapid separation of 1.0 mM nitrate and 1.0 mM nitrite standard solution. Electrolyte: 25 mM borate, 25 mM hexamethonium bromide, pH 9.2, at 220 nm wavelength. Applied voltage: -20 kV across a bare silica capillary column of $50 \text{ cm} \times 50 \mu\text{m}$ I.D. Gravity injection time was 60 s.

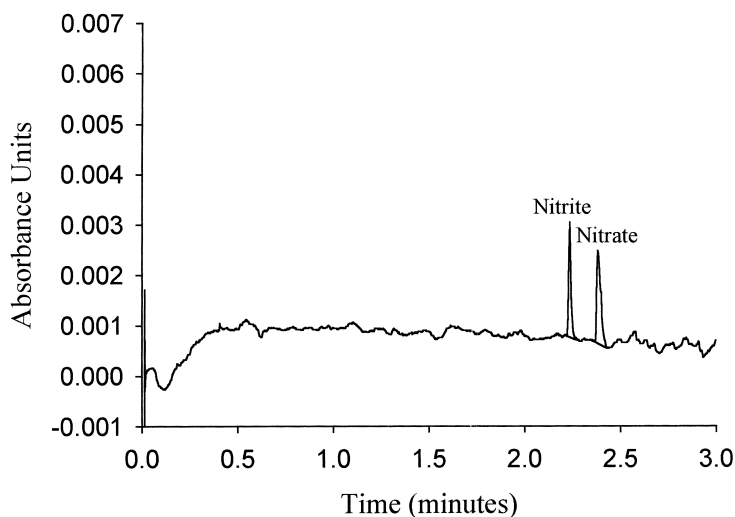


Fig. 2. The electropherogram of synthetic Hanford waste showing the separation and detection of 0.414 mM nitrate and 0.358 mM nitrite in this complex sample. Electrophoretic conditions in Fig. 1.

the preparation of the SHW (2.59 M and 2.24 M for nitrate and nitrite, respectively).

3.2. Potential interference from anions and complexants commonly found in Hanford defense waste tanks

Fig. 3 shows an electropherogram of a mixture of

nitrate, nitrite and some complexants commonly found in HDW. This figure demonstrates that the complexants did not interfere with the separation of nitrite and nitrate in the waste. The migration times of nitrate, nitrite and oxalate were under 3 min, while the migration times of NTA, EDTA and HEDTA were all under 6 min. Nitrate and nitrite displayed narrow peak shapes, while the complexants, especial-

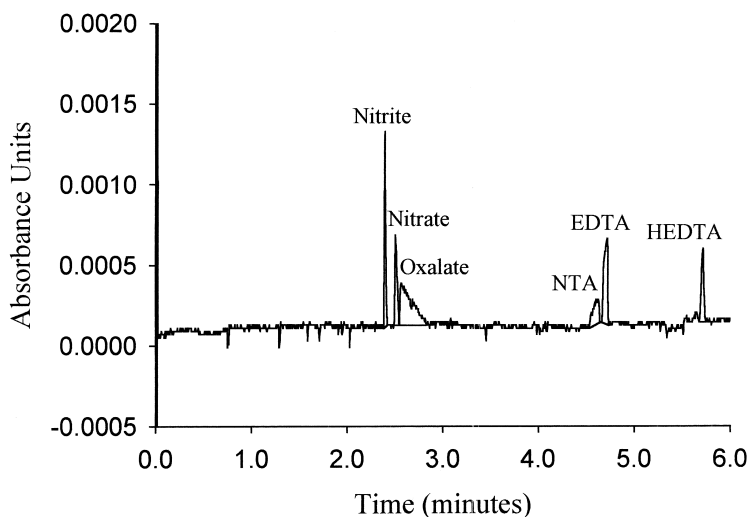


Fig. 3. The electropherogram containing a mixture of 1.5 mM nitrate, 0.70 mM nitrite, 10 mM oxalate, 1.8 mM nitrilotriacetic acid, 0.90 mM ethylenediaminetetraacetic acid and 0.90 mM *N*-hydroxyethylethylenediaminetriacetic acid. Sample pH=12, UV wavelength=240 nm. Electrophoretic conditions: electrolyte: 25 mM borate–25 mM hexamethonium bromide, pH 9.2. Applied voltage: –20 kV.

ly oxalate and NTA, showed tailing and leading edges, respectively. Sulfate, chloride, carbonate and phosphate anions were also tested at the 5 mM concentration level. None of these anions interfered with the separation and detection of nitrate and nitrite. Another major advantage of the reverse-polarity CZE over conventional CZE was that cations and neutral compounds migrate away from the detector. Accordingly, cations and neutrals were never observed and did not interfere with the method [18].

The separation of nitrate from oxalate was difficult because their equivalence conductance [27] and electrophoretic mobilities were similar [28]. In fact, an investigation reported by Wildman et al. [29] showed that nitrate co-migrated with oxalate in a separation with NICE-Pak OFM Anion-BT (Waters) electrolyte. Fig. 3 shows that with this reverse-polarity CZE method, nitrate, nitrite, oxalate and other complexants can be separated from one another. The better resolution between oxalate and nitrate in reverse-polarity CZE compared to the NICE-Pak OFM Anion-BT method can be explained in terms of the electroosmotic flow (EOF). The electroosmotic modifier used in this reverse-polarity CZE method reduced the EOF but did not reverse its normal direction, i.e., the EOF continued to flow in a direction counter to the anions. This reduction (but non-reversion) in the EOF contributed to a high separation efficiency and hence, a better resolution [18,24]. The analysis using the NICE-Pak OFM Anion-BT reversed the EOF, diminishing separation efficiency. Because of the low absorptivity of oxalate at 240 nm the oxalate concentration required in this study was about seven-times greater than that of nitrate. Fig. 3 demonstrated that even at high oxalate concentrations, better than baseline resolution was achieved.

3.3. Analysis of real HDW samples

The electropherogram of sample S96R000308 is shown in Fig. 4, demonstrating no interfering peaks within the migration times of nitrate and nitrite. Because of the high salt concentrations of the real HDW samples, constant voltage applications during the reverse-polarity CZE analysis produced electric current in the sample zone that was usually higher than the electric current in the electrolyte zone. This

caused a fluctuation in the migration times of the analytes. For this reason, a constant current of 75 μA , rather than a constant voltage, was used for the analysis. Table 1 summarizes the results obtained for the analysis of four real HDW samples, showing a concentration range of 1.03 to 1.42 M of nitrate and nitrite, respectively. The precision obtained for three replicate runs for each sample based on an external calibration curve was within 6% relative standard deviation (RSD). The standard addition calibration for nitrate and nitrite in HDW sample S96R000311 was used to check the accuracy of the method. The linearity of the calibration curve was better than $r^2=0.970$, and the extrapolated results were 1.28 M and 1.05 M for nitrite and nitrate, respectively. These results fall within 10% agreement of the 1.15 M and 1.03 M obtained by external standard calibration, as shown in Table 1.

Sample spikes were used to positively confirm the identification of nitrate and nitrite in HDW. The three replicate runs carried out for the spiked samples all produced results within 2% RSD. Peak confirmation by spiking with the standard in a high ionic strength sample may broaden peak width due to ionic strength dispersion effects in CZE [18,25]. Selectivity ratio was used to positively confirm the identification of the peaks after spiking. Selectivity ratio, S , was defined as $\Delta\mu/\mu_{av}$, where $\Delta\mu$ is the difference between the electrophoretic mobilities of two adjacent ions and μ_{av} is their average mobility. This principle of measuring the selectivity ratio was applied to the nitrate and nitrite peaks as shown in Table 2. The respective migration times of both nitrate (3.62 and 3.61 min) and nitrite (3.39 and 3.39 min) in the real and spiked HDW were within a 99% match. Similarly, the selectivity ratios (0.065 and 0.062 for the real and spiked HDW samples, respectively) showed greater than 95% agreement. The recoveries of spiked nitrate and nitrite standards were 95% or better except for sample S96R000312, which was 93% (Table 1). These high precision values and high recoveries indicate that the method is highly reliable.

4. Conclusions

The reverse-polarity CZE analysis of HDW for nitrate and nitrite was rapid, simple, precise, accur-

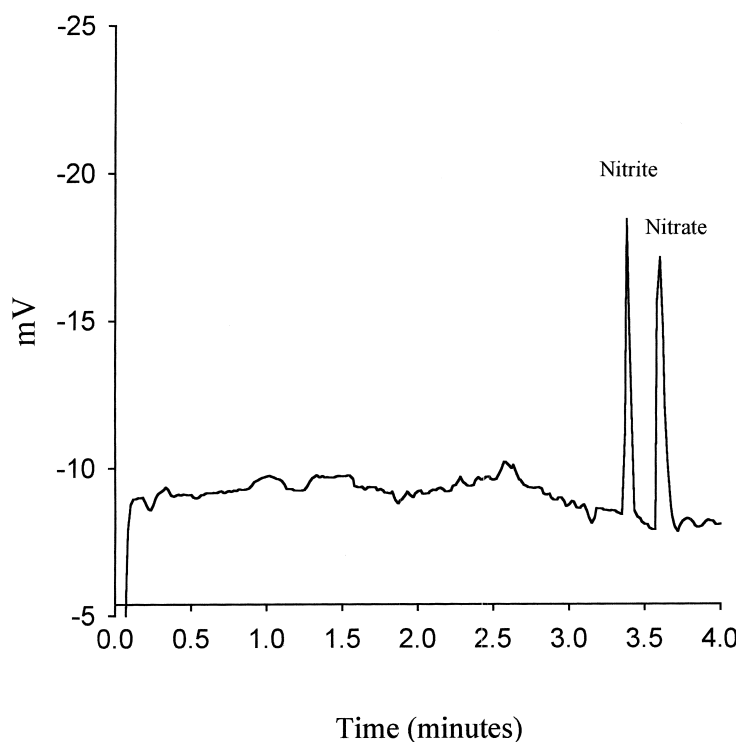


Fig. 4. The electropherogram of sample S96R000308, demonstrating the rapid separation and detection of nitrate and nitrite in a real sample. Electrophoretic conditions of 75 μ A constant current (negative polarity) across a bare silica capillary column of 50 cm \times 50 μ m I.D. Gravity injection time was 30 s.

ate, involved micro-sample sizes, and generated very little laboratory waste. Minimum sample handling was required, with only simple dilution of the sample by a factor of three-orders of magnitude without any further sample preparation or pH adjustments. This cut the overall time of analysis per sample. The high dilution factor of the sample also decreased the level

of radiation by the same factor and therefore reduced the analyst's radiation exposure. Furthermore, the method will enable a high sample throughput (>10 samples/h) in the HDW characterization because the reverse-polarity CZE method can be automated and run with minimum attention. Although the SHW samples used in reverse-polarity CZE method de-

Table 1
Results of test analysis of real Hanford defense waste

Sample	[NO ₂ ⁻] (M)	Recovery (%)	[NO ₃ ⁻] (M)	Recovery (%)
S96R000308	1.42 \pm 0.03	–	1.41 \pm 0.03	–
S96R000308 spiked	2.96 \pm 0.06	101	2.75 \pm 0.05	95
S96R000309	1.23 \pm 0.03	–	1.07 \pm 0.01	–
S96R000309 spiked	2.67 \pm 0.01	100	2.56 \pm 0.02	103
S96R000311	1.15 \pm 0.02	–	1.03 \pm 0.02	–
S96R000311 spiked	2.59 \pm 0.05	99	2.46 \pm 0.05	99
S96R000312	1.29 \pm 0.03	–	1.26 \pm 0.07	–
S96R000312 spiked	2.63 \pm 0.06	93	2.55 \pm 0.02	95

Table 2

Comparison between real HDW and spiked HDW samples for migration times and selectivity ratio, S

Sample	$t_R(\text{NO}_2)$ (min)	$t_R(\text{NO}_3)$ (min)	$S = \Delta\mu/\mu_{av}$
S96R000308	3.39±0.007	3.62±0.007	0.065±0.001
S96R000308 spiked	3.39±0.009	3.61±0.009	0.062±0.002
S96R000311	3.40±0.005	3.63±0.005	0.065±0.001
S96R000311 spiked	3.40±0.004	3.63±0.008	0.064±0.001

velopment contained higher concentrations of nitrate and nitrite than the analyzed real HDW samples, SHW samples were good surrogates for high ionic strength and high pH in HDW samples. reverse-polarity CZE appears promising as a rapid analytical method for the determination of nitrate and nitrite not only in the HDW wastes stored in the tanks, but also for other high ionic strength samples such as sea water or geothermal brine.

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