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Comparison of Ion Chromatography with Ion Selective Electrodes for the Determination of Inorganic Anions in Drinking Water Samples

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Abstract. Fluoride, chloride and nitrate anions were determined in drinking water samples using techniques of ion selective electrodes (ISE) and non-suppressed/suppressed ion chromatography (IC). Detection limit, percentage recovery and run time were evaluated for the two methods. Detection limits for ISE [0.02, 0.20 and 1.7 ppm (µg/mL) for fluoride, chloride and nitrate, respectively], were better than those for non suppressed IC (2.0, 1.0 and 2.0 ppm for fluoride, chloride and nitrate, respectively). Suppressed IC was used to measure fluoride. Statistical analysis of the data revealed no evidence of systematic difference between ISE and non suppressed IC for chloride and nitrate. Fluoride concentrations in all water samples were lower, while chloride and nitrate concentrations in some samples were higher than the maximum contaminant levels established by the United States Environmental Protection Agency.

Keywords: drinking water, nitrate, chloride, fluoride, ion selective electrode, ion chromatography

Introduction

Due to increase in population, urbanization and continued industrial growth, per capita water availability in Pakistan has decreased from 5000 m^3/annum in 1951 to 1100 m^3/annum in 2007 (WWF, 2007). The increasing gap between water demand and supply has led to severe water shortage in almost all sectors and has adversely affected the quality of drinking water; consequently, water pollution has become a serious problem in the country and most of the reported health problems are directly or indirectly related to water (PCRWR, 2008).

There are various sources of contaminants in drinking water which, when exceeding certain levels, are harmful to man. These contaminants are microorganisms, inorganic and organic chemicals and certain radioisotopes. Inorganic anions may affect the quality of water. Fluoride, chloride and nitrate have considerable importance in the quality of drinking water. Specially, the excess of nitrate and fluoride in drinking water has intense effects on human health (Meenakshi and Maheshwari, 2006; Fraser and Chilvers, 1981). Excess nitrate in drinking water could cause serious illness in infants below the age of six months. Fluoride might be the reason for different bone diseases and tenderness of bones in children (US EPA, 2009).

Fluoride, chloride and nitrate in groundwater and surface water originate from natural sources, sewage, industrial effluents, different food additives and as a result of leaching or runoff from agricultural land (WHO, 2004).

Various analytical methods have been proposed for the determination of fluoride in aqueous solutions, such as colorimetric, conductometric, complexometric and potentiometric methods (APHA, 1985). Some methods are rapid, sensitive, precise and relatively free of interferences. Traditional methods used for determination of fluoride, chloride and nitrate anions are based on colorimetric method, which due to interference by various ions, require special treatment of the samples like distillation or reduction and special analytical skills. Ion chromatography is becoming more popular for the analysis of water samples and is also recognized by the US Environmental Protection Agency (US EPA, 2009) as a method of choice for the determination of anions in water samples (Bosch *et al.*, 1995; Cheam, 1992; Pereira, 1992; Frankenberger *et al.*, 1990). Potentiometry has been widely used for quite some time due to its simplicity and prompt results. However, the selectivity is rather limited, especially if chemically similar ions are present in the sample. Recent developments in separation techniques have led to an improvement especially in the determination of fluoride in terms of selectivity and sensitivity (Weiss *et al.*, 1995; Vasconcelos *et al.*, 1994). Results of determination of bromide, chloride, fluoride, nitrate and sulphate using ion chromatography (IC) had been compared with those obtained by colorimetry for rainfall, cloud water and stream waters. According to that, there was no significant difference in chloride and nitrate measurements between the

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*Author for correspondence; E-mail: hakimsiwag@hotmail.com two methods. For fluoride, the IC method gave lower values than the colorimetry, especially for the stream waters. Since, the colorimetric method determines total fluorine, differences in the values might be expected, for example fluoride forms complexes with the available aluminium, especially in the stream water (Neal *et al.*, 2007). Statistical analysis of fluoride concentrations in rain water samples as obtained by capillary electrophoresis (CE), IC and ISE indicated that there were no systematic differences between CE and ISE, but the fluoride concentrations obtained by IC were significantly higher. The observed differences are most likely due to presence of aluminium cations (Van den Hoop *et al.*, 1996). A fully validated dual ion chromatographic method, complying with ISO 17025, has been developed at the chemical laboratory of the Athens Water Supply and Sewerage Company (EYDAP SA) for the concurrent determination of ten ions (F^-, Cl^-, NO_3^-, Br^-, PO_4^{3-}, SO_4^{2-}, Na^+, K^+, Ca^{2+} and Mg^{2+}) in surface, ground and potable water samples (Miskaki *et al.*, 2007).

The aim of the present study was to optimize a simple, selective and efficient method for simultaneous determination of chloride, fluoride and nitrate ions in drinking water samples collected from various sources by using ion chromatography and ion selective electrodes.

Materials and Methods

Reagents. High purity distilled deionized water was used throughout the work. Anion standards solutions were prepared using sodium salt of fluoride (Merck, Germany), chloride (Merck, Germany) and nitrate (BDH Chemical, England). Other chemicals were analytically pure reagents from RDH Chemicals, Germany.

Ion selective electrode. Cole-Parmer ion selective electrode chloride model EW-27502-13 (USA), Cole-Parmer combination ion selective electrode fluoride model EW-27504-14 (USA) and Cole-Parmer combination ion selective electrode nitrate model EW-27504-22 (USA) were used. The response was in mV given by OAKTON pH/mV/Ion Meter (pH 2100 series USA). Glacial acetic acid and sodium chloride were used as low level total ionic strength adjuster buffer (TISAB-2) for low level fluoride measurement by ion selective electrodes. Sodium nitrate and ammonium sulphate were used as ionic strength adjuster for chloride and nitrate, respectively.

By serial dilution, 10 ppm fluoride standard was prepared by diluting 1000 ppm standard solution. 50 mL low level TISAB-2 was added to 50 mL of the above standard solution. In a 150 mL beaker, 50 mL of distilled water and 50 mL low level TISAB-2 were added. The volume of real water samples and TISAB-2 were same as that of the standard. This solution was stirred at constant rate. The electrode tip was dipped in solution while the meter was in mV mode. Increments of 10 ppm standard solution were made after 90 second intervals to get 0.01, 0.02, 0.04, 0.06, 0.10, 0.29, 0.48 and 1.10 ppm concentrations. For nitrate and chloride, 1, 10, 50, 100, 500 ppm standard solutions were used and 2 mL of ISA (ion strength adjuster) was added to 100 mL of standard solution. Same amount of ISA was added to 100 mL of water samples. Rest of the procedure was same. Calibration curve was obtained by plotting a graph between electrode potential and concentrations from which the unknown concentrations of F^-, Cl^- and NO_3^- in water samples were calculated.

Ion chromatography. Ion chromatograph consisted of Kanauer HPLC quaternary pump Model K-1001 (Germany) with maximum operating pressure of 400 bars and flow range of 0.001-9.999 ml/min. HAMILTON PRP-X-100 polymer base reverse phase No. R-79439 (USA) anion exchange column PRP X-100 (150 mm × 4.1 mm) having 10 μm particle size with comparative guard column was used. A comparative guard column was also used. Alltech model 650 conductivity detector (USA) was used as detector. Alltech model 640 suppressor (USA) and Metrosep A supp 3 (Metrohm, Switzerland) anion exchange column (250 mm × 4.6 mm) having particle size 9 μm, packed with polystyrene/divinylbenzene copolymer were used with comparative guard column in suppressed ion chromatography. The volume of sample loop used for injection was 20 μL. 4 mM solution of *p*-hydroxy benzoic acid was used as mobile phase for non suppressed ion chromatography while 1.8 mM Na_2CO_3/1.7 mM $NaHCO_3$ solution was used as mobile phase for suppressed ion chromatography.

Optimal mobile phase and its flow rate were used for separation of F^-, Cl^- and NO_3^-, using the standard solutions. Standard solutions of varying concentrations of fluoride, chloride and nitrate were prepared from standard stock solutions. These solutions were injected into ion chromatograph. Peak areas and heights of all these solutions were measured and calibration curves for fluoride, chloride and nitrate were obtained. All water samples were filtered through 0.45 μm pore diameter membrane syringe filters and injected. The concentration of these anions in samples was determined using these calibration curves.

ICP-OES instrument. The ICP-OES instrument used in the present work is ARL 3580 model, made by Applied Research Laboratories, Switzerland. The instrument is equipped with a monochromator, a polychromator and a spark excitation source besides ICP source. Both the monochromator and the polychromator are 1 meter focal length Paschen-Runge spectrometers having 1080 groves/mm concave grating mounted in Rowland circles. The operating conditions of the ICP-OES used are given in Table 1. Metal ions were investigated in some water samples and their emission wavelengths were as follows: Al (309.271 nm), Ca (393.366 nm), Fe (261.187 nm), K (766.490 nm), Mg (279.553 nm), Mn (257.610 nm), Na (588.995 nm), Ni (221.647 nm), Pb (220.353 nm), Si (251.611 nm), Sr (407.771 nm) and Zn (213.856 nm).

Table 1. Operating conditions of ICP-OES

Generator frequency	27MHz
Incident power	1.25 kW
Out gas flow	12 L/min.
Intermediate gas flow	0.8 L/min
Carrier gas flow	1 L/min
Observation height	16 mm above the coil
Sample uptake	1-3 mL/min

Samples. Surface water and groundwater are the main water sources available to the residents of Islamabad. Drinking water samples were collected from water sources in the month of October 2008, from different sectors and nearby villages of Islamabad. The water was allowed to flow from the source for about 1-2 min in order to stabilize different parameters i.e., conductivity and pH. The collected samples were stored in pre-cleaned, sterilized polyethylene bottles of one litre capacity. The samples were cooled to 4 °C in clean and dust free environment.

Statistical analysis. A paired t-test was performed to check the validity of two methods (Miller and Miller, 1997). The formula of paired t-test is:

$$t_{cal} = \frac{\overline{x}_d \sqrt{n}}{S_d}$$

where:

S_d = standard deviation
$\overline{x}_d$ = mean of group one minus group two
n = the number of values

If t_{cal} is less than t_{tab} at a specific confidence limit then there is no significant difference between the two methods.

Results and Discussion

Optimization of mobile phase for non-suppressed IC. In order to obtain optimal separation, pH and flow rate of mobile phase was optimized. Standards containing 10 ppm, 20 ppm and 40 ppm of fluoride, chloride and nitrate, respectively, were injected at 1.0 mL/min flow rate at various pH of mobile phase. The retention was decreased by increasing the pH of the mobile phase. Variation of pH of the mobile phase led to shift in the dissociation equilibrium and thus to change the retention time. The peak height and peak area decreased, by increasing the pH of mobile phase. This might be due to the decrease in retention time. There was a shift in the base line showing incomplete separation of anions. So optimum pH was determined which was based on good resolution. The clearer picture is given in Fig. 1. Consequently optimal pH necessary for complete and in-time separation was 8.5.

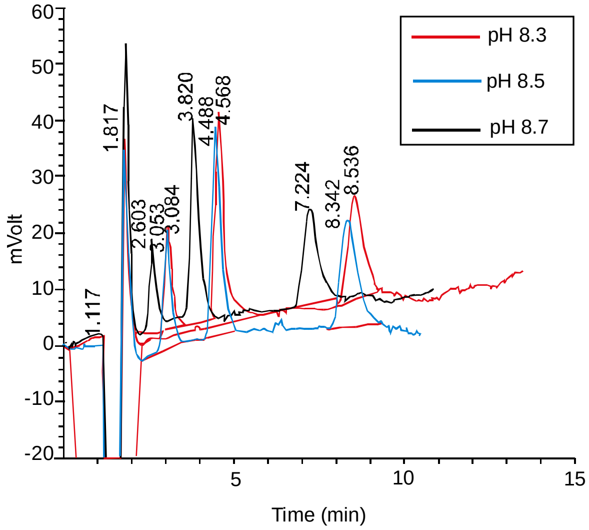

Fig. 1. Chromatogram of F^- 10 ppm, Cl^- 20 ppm and NO_3^- 40 ppm at different pH at flow rate of 1.0 mL/min by non-suppressed IC.

To see the effect of flow rate on retention time, a single standard containing 10 ppm, 20 ppm and 40 ppm of fluoride, chloride and nitrate, respectively, was injected by using mobile phase of optimal pH 8.5. The effect of flow rate was studied in the range of 0.8-1.2 mL/min. The results are shown in Fig. 2. By increasing the flow rate, the retention time decreased. The peak height and area also decreased with retention time. This was due to faster separation of anions resulting in incomplete separation of ions. The optimum flow rate was 1 mL/min.

Performance characteristics. Performance characteristics in terms of detection limit, percent recovery and total run time of the analytical response were calculated from reproducibility experiments which are shown in Table 2. The detection limits for ISE were estimated based on three times standard deviation of response plus mean response from determination of

Table 2. Performance characteristics of the applied techniques

Anions	Ion selective electrode			Non-suppressed IC			Suppressed IC		
	Detection limit (ppm)	Run time (min)	Recovery (%)	Detection limit (ppm)	Run time (min)	Recovery (%)	Detection limit (ppm)	Run time (min)	Recovery (%)
Fluoride	0.02	3	98.6	2	20	102.7	0.05	25	99.3
Chloride	0.2	3	101.5	1	20	103.2	0.05	25	102.6
Nitrate	1.7	3	109.4	2	20	98.5	0.1	25	106.5

six blank samples. The detection limit is thus the corresponding concentration of the response from calibration curve of each anion (Skoog *et al.*, 2005). The detection limit for IC is three times signal-to-noise ratio. Hence the detection limits were found by using the standard whose response was three times signal to noise. In order to evaluate the accuracy of method, percentage recovery was calculated by adding known amount of fluoride, chloride and nitrate to drinking water samples according to the following equation:

$$\text{Recovery (\%)} = \left(\frac{\text{spiked conc.} - \text{actual conc.}}{\text{conc. of standard added}}\right) \times 100$$

The total run time includes sample introduction, purging/washing time and run time, whereas, the time needed for pretreatment of the sample and to calculate the corresponding concentration were not taken into account.

Analysis of water samples. ***Measurement of pH.*** pH of all the collected samples was measured which was in the range of 6.85-8.65 (Table 3). pH of most samples was in good agreement with US EPA which is 6.5-8.5 except that of sample no.11 which was slightly higher. This sample was from Malal stream in periphery of the village of Islamabad. People living nearby this stream wash their clothes in the stream so pH may be higher due to mixing of soapy water.

Determination of anions. Determination of concentrations of fluoride, chloride and nitrate was carried out using ISE and suppressed/non-suppressed ion chromatography. The results are given in Table 3.

For the analysis of water samples, optimized non-suppressed IC conditions were used. The chromatograms obtained by injecting the samples were compared to standard chromatogram; peaks of these chromatograms were quite sharp and resolution was also very good. Some of the chromatograms of a standard and a sample are shown in Fig. 3 and 4, respectively.

Samples were analyzed by non suppressed/suppressed IC as described above. Results obtained for chloride and nitrate concentration by non suppressed IC are shown in Table 3.

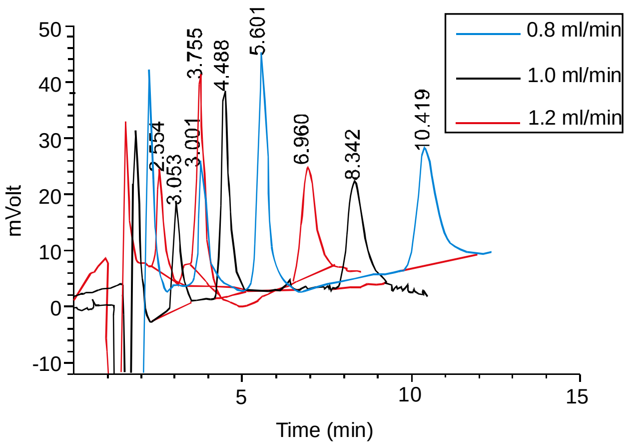

Fig. 2. Chromatogram of F$^-$ 10 ppm, Cl$^-$ 20 ppm and NO$_3^-$ 40 ppm at different flow rates at pH 8.5 by non-suppressed IC.

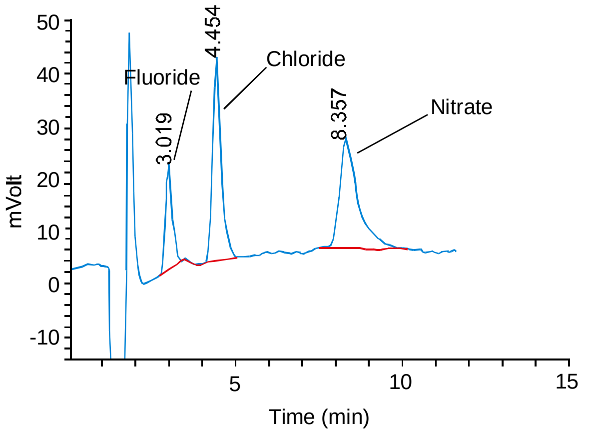

Fig. 3. Chromatogram of F$^-$ 10 ppm, Cl$^-$ 20 ppm and NO$_3^-$ 40 ppm by non-suppressed IC at flow rate of 1.0 mL/min.

Table 3. Results obtained by ion selective electrodes/pH electrode and non-suppressed ion chromatography (suppressed ion chromatography for fluoride only)

Sample	pH	Ion selective electrodes/pH electrode			Non-suppressed ion chromatography			Suppressed ion chromatography
		Fluoride (ppm)	Chloride (ppm)	Nitrate (ppm)	Fluoride (ppm)	Nitrate (ppm)	Chloride (ppm)	Fluoride (ppm)
1	7.45	0.34	92.11	90.72	nd	80.61	90.10	0.31
2	7.45	0.77	114.10	287.69	nd	222.70	118.25	0.73
3	7.30	0.15	53.93	42.31	nd	38.20	50.23	0.15
4	7.20	0.43	67.10	42.73	nd	39.10	65.53	0.41
5	7.30	0.64	128.09	226.11	nd	205.23	122.50	0.60
6	6.85	0.33	1062.07	1321.69	nd	1080.60	855.50	0.41
7	7.50	0.13	39.79	19.05	nd	15.50	37.90	0.10
8	7.35	0.79	96.14	70.59	nd	63.54	95.69	0.81
9	7.30	0.96	67.96	39.24	nd	35.42	65.21	0.92
10	7.35	0.59	30.13	38.27	nd	23.45	20.28	0.62
11	8.65	0.38	29.11	6.01	nd	5.24	22.50	0.36
12	7.05	0.23	13.02	16.39	nd	11.34	9.52	0.22
13	7.85	0.19	13.53	15.90	nd	10.59	10.15	0.20
14	8.00	0.23	12.79	19.15	nd	14.69	9.24	0.24
15	7.80	0.89	19.97	22.48	nd	16.22	19.12	0.90
16	7.25	1.08	55.81	31.94	nd	26.45	18.20	1.12
17	7.30	0.25	10.92	23.75	nd	18.65	9.10	0.24
18	7.55	0.21	8.79	28.03	nd	23.57	6.24	0.26
19	8.10	0.20	9.63	16.88	nd	13.82	8.13	0.22
20	7.55	0.07	2.92	6.82	nd	4.56	2.25	0.05
21	8.00	0.08	3.20	6.91	nd	4.89	3.10	0.06
22	7.65	0.08	251.34	379.91	nd	251.32	240.53	0.09
23	7.05	0.25	80.24	2.88	nd	2.13	78.20	0.27
24	7.15	0.08	12.34	23.52	nd	21.52	10.88	0.07

nd = not detected.

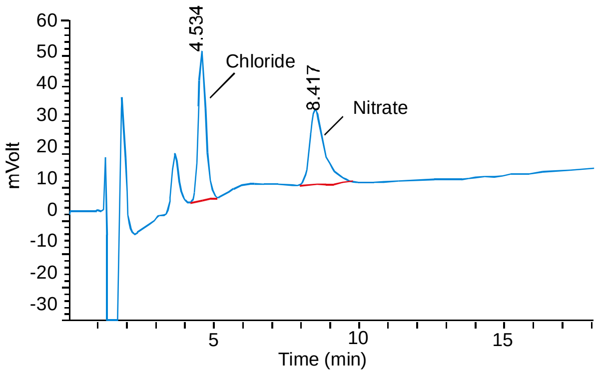

Fig. 4. Chromatogram of sample # 2 by non-suppressed IC.

Fluoride was not measured by non-suppressed ion chromatography due to its low concentration in the samples. So, suppressed IC was used. Seven anion standards were injected at the flow rate of 1 mL/min. The chromatogram is shown in Fig. 5. Quantitative determination of fluoride in the water samples was made by comparison of peak areas in the chromatograms of the samples and that of the standard; chromatogram of a sample is shown in Fig. 6. The results for fluoride analysis in water samples by suppressed IC are given in Table 3.

The concentration of fluoride in all the water samples was within the limits established by USEPA (4.0 ppm). The chloride level was also within the permissible range i.e.,

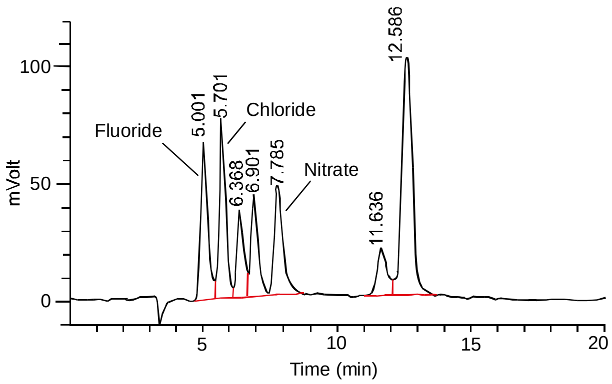

Fig. 5. Chromatogram of F$^-$ 3 ppm, Cl$^-$ 3 ppm, NO_2^- 4 ppm, Br$^-$ 4 ppm, NO_3^- 4 ppm, PO_4^{3-} 8 ppm and SO_4^{2-} 8 ppm at flow rate of 1.0 mL/min by suppressed IC.

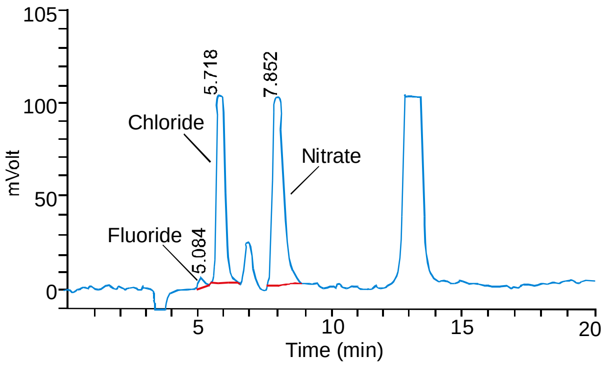

Fig. 6. Chromatogram of sample # 10 by suppressed IC at flow rate of 1.0 mL/min.

Table 4. Concentration of cations by ICP-OES

Cations	Sample 6 (ppm)	Sample 7 (ppm)	Sample 16 (ppm)
Al	nd	nd	nd
Ca	176.39	32.77	30.64
Fe	nd	nd	nd
K	5.50	8.94	1.70
Mg	328.42	17.79	37.41
Mn	nd	nd	nd
Na	481.06	61.51	185.66
Ni	nd	nd	nd
Pb	nd	nd	nd
Si	6.30	5.58	4.86
Sr	7.40	0.22	1.20
Zn	nd	nd	nd

nd = not detected.

250 ppm in all the samples except sample no. 6. In most of the samples, nitrate level was higher than US EPA standard for safe drinking water i.e. 10 ppm. Sample # 6 has the maximum level of chloride and nitrate exceeding 1000 ppm. This is the water obtained from house pump installed by boring in Nilore colony situated in the surrounding area of Islamabad. The underground water in these areas is in the narrow channels rather than in large reservoirs. So the water may be in contact with some rocks containing salts of nitrates and chlorides. Thus, metal ion analysis especially of samples 6, 7 and 17 were performed using Inductive Coupled Plasma Optical Emission Spectroscopy by conditions given in the experimental section. The results are given in the Table 4. It is clear from the results that sample 6 contained a high concentration of sodium, magnesium and calcium ions. So most probably the nitrate and chloride of these cations may exist.

The results obtained by the two methods are compared in Fig. 7-9. The correlation coefficient in each case shows good linearity in the results of the two methods from low concentration to the higher concentration.

A paired t-test was also performed to check the validity of two methods. According to this test, if t_{cal} is less than t_{tab} at a specific confidence limit then there is no significant difference between the two methods. The results are given in Table 5. The results of statistical analysis, according to student's t-test, shows that there is no significant difference between the results obtained with non-suppressed ion chromatography and ion selective electrodes for chloride and nitrate determination in water samples.

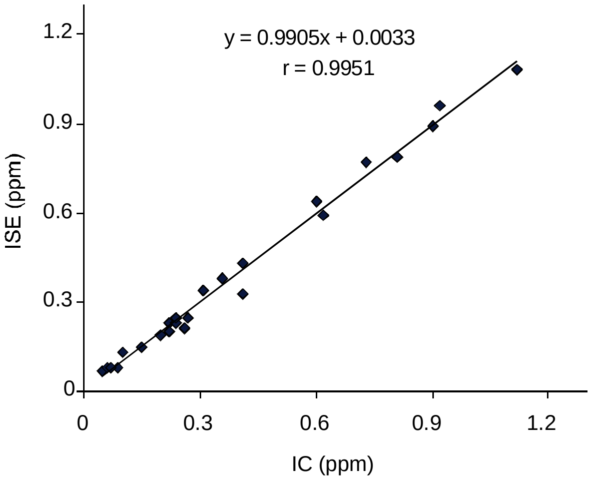

Fig. 7. Comparison of the results for the determination of fluoride in drinking water samples (n = 23) using IC (suppressed) and ISE.

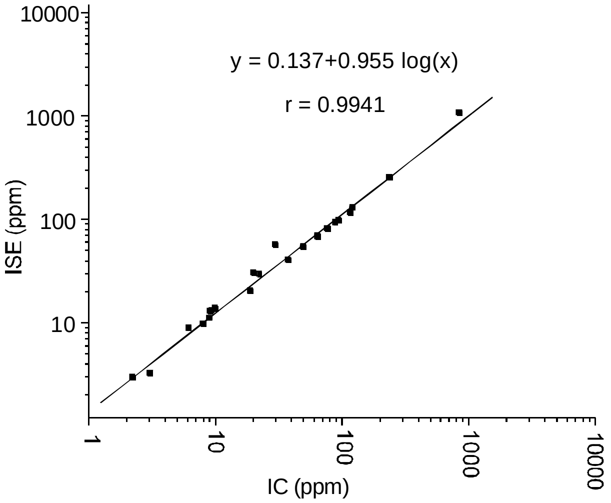

Fig. 8. Comparison of the results for the determination of chloride in drinking water samples (n = 23) using IC (non-suppressed) and ISE.

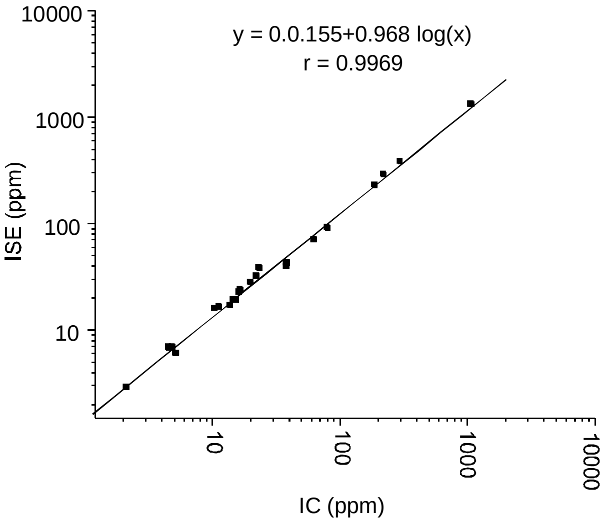

Fig. 9. Comparison of the results for the determination of nitrate in drinking water samples (n = 23) using IC (non-suppressed) and ISE.

Table 5. Statistical analysis of the correlation between chloride and nitrate concentrations by suppressed/non-suppressed IC and ISE

Anions	Intercept	Slope	r^2	t_{cal}	t_{tab}
Fluoride	0.0033 ±0.0005	0.9905 ±0.01201	0.99022	- 0.70200	2.074
Chloride	0.16042 ±0.02570	0.9637 ±0.01636	0.99398	-1.46362	2.074
Nitrate	0.15521 ±0.02604	0.9682 ±0.0166	0.99386	-2.06386	2.074

Confidence limits = ±95; two tail; n=23.

Conclusion

Both ion chromatography and ion selective electrode were employed for the determination of three anions (fluoride, chloride and nitrate) in drinking water samples. ISE is a preferred technique due to shorter analysis time and less operational cost of the equipment. Ion chromatography is sophisticated and reliable for simultaneous determination of anions in routine water analysis. This technique can be used for comparison and validation of methods.

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