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Science college/ chemical department

Determination of Anious in water samples by ion chromatography (IC)

A RESERCH

SUBMITTED TO THE DEPARTMENT OF EDUSATION AL-QADISYA UNVERSTY AS ABARTIAL FUAFILLMENT REQUIREMENT FOR Certificate THE BACHELOR OF SIENSE chemistry science

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بِسْمِ اللَّهِ الرَّحْمَٰنِ الرَّحِيمِ

﴿وَقُلِ اعْمَلُوا فَسَيَرَى اللَّهُ عَمَلَكُمْ

وَرَسُولُهُ وَالْمُؤْمِنُونَ﴾

صدق الله العلي العظيم

سورة التوبة الآية (105)

﴿ الإهـــــــــــــداء﴾

إلى من جرع الكأس فارغاً ليسقيني قطرة حب

إلى مـــــن كلّت أنامله لـــــــيقدم لنـــــا لحظة سعادة

إلى من حصد الأشواك عن دربي ليمهد لي طريق العلم

والـــــــدي العزيز

إلى مـــــــن أرضعتني الحب والحنان

إلى رمـــــــــز الحب وبلسم الشفـــــــــــــاء

إلى القلب الناصع بالبياض والدتي الحبيبة

إلى القلوب الطاهرة الرقيقة والنفوس البريئة إلى رياحين حياتي إخوتي

إلى الأجساد التي سكنت تحت تراب الوطن الحبيب المعفرة بدماء الشهادة

الآن تفتح الأشرعة وترفع المرساة لتنطلق السفينة في عرض بحر واسع مظلم هو بحر الحياة وفي هذه الظلمة لا يضيء إلا قنديل الذكريات ذكريات الأخوة البعيدة إلى الذين أحببتهم وأحبوني أصدقائي

إلى الذين بذلوا كل جهدٍ وعطاء لكي أصل إلى هذه اللحظة أساتذتي الكرام ولا سيما الدكتور الفاضل حسن محمد لعيبي

إليكم جميعاً أهدي هذا العمل

SUMMARY OF METHOD

The concentrations of $(f^-, Cl^-, No2^-, Br^-, No3^-, Bo4^{-3}, So4^{-2})$,

A small volume of sample, typically 2-3 mL, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

The main differences between Parts A and B are the separator columns and guard columns. will elicit the differences.

An extraction procedure must be performed to use this method for solids .

Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Quality Control. (1)

Introduction

The determination of common inorganic anions in drinking water is one of the most important applications of ion chromatography (IC) worldwide. The National Primary Drinking Water Standards in the United States specify a Maximum Contaminant Level (MCL) for a number of inorganic anions, including fluoride, nitrite, and nitrate. The MCLs are specified to minimize potential health effects arising from the ingestion of these anions in drinking water.1 High levels of fluoride cause skeletal and dental fluorosis, and nitrite and nitrate can cause methemoglobulinemia, which can be fatal to infants. Other common anions, such as chloride and sulfate, are considered secondary contaminants. The National Secondary Drinking Water Standards in the U.S. are guidelines regarding taste, odor, color, and certain aesthetic characteristics. Although these guidelines are not federally enforced, they are recommended to all states as reasonable goals and many states adopt their own regulations governing these contaminants.[2]

Ion chromatography has been approved for compliance monitoring of these common inorganic anions in U.S. drinking water since the mid-1980s, as described in U.S. EPA Method 300.0.3 Many other industrialized countries have similar health and environmental standards and a considerable number of regulatory IC methods have been published worldwide (e.g., in Germany, France, Italy, and Japan) for the analysis of anions in drinking water. In addition, many standards organizations (including ISO, ASTM, and AWWA)

have validated IC methods for the analysis of inorganic anions in drinking water.4,5

This application note describes the determination of inorganic anions in drinking water and other environmental waters using conditions that are consistent with those in U.S. EPA Method 300.0.3 The use of an optional column, the Thermo Scientific Dionex™ IonPac™ AS14 column, is also discussed.

Theory

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EQUIPMENT AND SUPPLIES

Balance -- Analytical, capable of accurately weighing to the nearest g. [1]

Ion chromatograph Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and detectors.

Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with a substrate the same as that in the separator column.

Anion separator column: This column produces the separation shown in Figures 1 and 2.

Anion analytical column (Method A): The separation shown in Figure 1 was generated using a Dionex AS4A column (P/N 37041). An optional column may be used if comparable resolution of peaks is obtained, and the requirements of can be met. [1]

Anion analytical column (Method B): The separation shown in Figure 2 was generated using a Dionex AS9 column (P/N 42025). An optional column may be used if comparable resolution of peaks is obtained and the requirements of can be met.

Anion suppressor device: The data presented in this method were generated using a Dionex anion micro membrane suppressor (P/N 37106).

Detector -- Conductivity cell: Approximately 1.25 µL internal volume, (Dionex, or equivalent) capable of providing data as required in Section.

The Dionex AI-450 Data Chromatography Software was used to generate all the data in the attached tables. Systems using a stripchart recorder and integrator or other computer based data system may achieve approximately the same MDL's but the user should demonstrate this by the procedure outlined in Section.

SAMPLE COLLECTION, PRESERVATION AND STORAGE

Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal. [1]

Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte Preservation Holding Time [1]

TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

TABLE 1A. CHROMATOGRAPHIC CONDITIONS AND DETECTION LIMITS IN REAGENT WATER (PART A)

Analyte	Peak	Retention Time (min)	MDL (mg/L)
Fluoride	1	1.2	0.01
Chloride	2	1.7	0.02
Nitrite-N	3	2.0	0.004
Bromide	4	2.9	0.01
Nitrate-N	5	3.2	0.002
o-Phosphate-P	6	5.4	0.003
Sulfate	7	6.9	0.02

Sample Preparation

The vial caps in the Thermo Scientific Dionex AS40 Automated Sampler contain a 20 µm filter, so no additional filtration was used in conjunction with this mode of sample introduction. If a Dionex AS40 is not used, filter all samples through appropriate 0.45 µm syringe filters, discarding the first 300 µL of the effluent. The domestic wastewater sample was treated with a C18 Sep-Pak cartridge to remove hydrophobic organic material in order to prolong column

lifetimes.6 The C18 cartridge was preconditioned with 5 mL of methanol, followed by 5 mL of deionized water. The sample (5 mL) was then passed through the cartridge, with the first 1 mL of the effluent being discarded. Aqueous soil extracts were prepared by the extraction of 3.0 g of soil in 30 mL of deionized water in an ultrasonic bath for 30 min, followed by filtration with a 0.45 µm filter.

Standard Conditions:

Columns: as specified in Detector: as specified in Pump Rate: 2.0 mL / min. Eluent: as specified in Sample Loop: 50 µL MDL calculated from data system using a y-axis selection of 1000 ns and with a stripchart recorder with an attenuator setting of 1 uMHO full scale. [1]

TABLE 1B. CHROMATOGRAPHIC CONDITIONS AND DETECTION LIMITS IN REAGENT WATER (PART B)

Analyte	Peak #*	Retention Time (min)	MDL (mg/L)
Chlorite	1	2.8	0.01
Bromate	2	3.2	0.02
Chlorate	4	7.1	0.003

Standard Conditions:

Column: as specified in Detector: as specified in Pump Rate: 1.0 mL / min. Eluent: as specified in Sample Loop: 50 µL

Attentuation - 1 y-axis - 500 ns (1)

TABLE 2A. SINGLE-OPERATOR ACCURACY AND BIAS OF STANDARD ANIONS (METHOD A)

Analyte	Sample Type	Known Conc. (mg/L)	Number of Replicates	Mean Recovery %	Standard Deviation (mg/L)
Bromide	RW	5.0	7	99	0.08
	DW	5.0	7	105	0.10
	SW	5.0	7	95	0.13
	WW	5.0	7	105	0.34
	GW	5.0	7	92	0.34
	SD	2.0	7	82	0.06
Chloride	RW	20.0	7	96	0.35
	DW	20.0	7	108	1.19
	SW	10.0	7	86	0.33
	WW	20.0	7	101	5.2
	GW	20.0	7	114	1.3
	SD	20.0	7	90	0.32
Fluoride	RW	2.0	7	91	0.05
	DW	1.0	7	92	0.06
	SW	1.0	7	73	0.05
	WW	1.0	7	87	0.07
	GW	0.4	7	95	0.07
	SD	5.0	7	101	0.35
Nitrate-N	RW	10.0	7	103	0.21
	DW	10.0	7	104	0.27
	SW	10.0	7	93	0.17
	WW	10.0	7	101	0.82
	GW	10.0	7	97	0.47
	SD	10.0	7	82	0.28
Nitrite	RW	10.0	7	97	0.14
	DW	10.0	7	121	0.25
	SW	5.0	7	92	0.14
	WW	5.0	7	91	0.50
	GW	10.0	7	96	0.35
	SD	2.0	7	98	0.08
o-Phosphate-P	RW	10.0	7	99	0.17
	DW	10.0	7	99	0.26
	SW	10.0	7	98	0.22
	WW	10.0	7	106	0.85
	GW	10.0	7	95	0.33
Sulfate	RW	20.0	7	99	0.40
	DW	50.0	7	105	3.35
	SW	40.0	7	95	1.7
	WW	40.0	7	102	6.4
	GW	40.0	7	112	3.2

TABLE 2B. SINGLE-OPERATOR ACCURACY AND BIAS OF BY-PRODUCT (PART B)

Analyte	Sample Type	Known Conc. (mg/L)	Number of Replicates	Mean Recovery %	Standard Deviation (mg/L)
RW = Reagent Water		WW = Mixed Domestic and Industrial Wastewater			
DW = Drinking Water		GW = Groundwater			
SW = Surface Water		SD = USEPA QC Solid (shale)			

TABLE 3. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR FLUORIDE

Water	Amount Added mg/L	Amount Found mg/L	St	So	Bias %
Reagent	0.26	0.25	0.08	0.11	-3.8
	0.34	0.29	0.11		-14.7
	2.12	2.12	0.07	0.12	0.0
	2.55	2.48	0.14		-2.7
	6.79	6.76	0.20	0.19	-0.4
	8.49	8.46	0.30		-0.4
Drinking	0.26	0.24	0.08	0.05	-7.7
	0.34	0.34	0.11		0.0
	2.12	2.09	0.18	0.06	-1.4
	2.55	2.55	0.16		0.0
	6.79	6.84	0.54	0.25	+0.7
	8.49	8.37	0.75		-1.4
Waste	0.26	0.25	0.15	0.06	-3.8
	0.34	0.32	0.08		-5.9
	2.12	2.13	0.22	0.15	+0.5
	2.55	2.48	0.16		-2.7
	6.79	6.65	0.41	0.20	-2.1
	8.49	8.27	0.36		-2.6

TABLE 4. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR CHLORIDE

Water	Amount Added mg/L	Amount Found mg/L	St	So	Bias %
Reagent	0.78	0.79	0.17	0.29	+1.3
	1.04	1.12	0.46		+7.7
	6.50	6.31	0.27	0.14	-2.9
	7.80	7.76	0.39		-0.5
	20.8	20.7	0.54	0.62	-0.5
	26.0	25.9	0.58		-0.4
Drinking	0.78	0.54	0.35	0.20	-30.8
	1.04	0.51	0.38		-51.0
	6.50	5.24	1.35	1.48	-19.4
	7.80	6.02	1.90		-22.8
	20.8	20.0	2.26	1.14	-3.8
	26.0	24.0	2.65		-7.7
Waste	0.78	0.43	0.32	0.39	-44.9
	1.04	0.65	0.48		-37.5
	6.50	4.59	1.82	0.83	-29.4
	7.80	5.45	2.02		-30.1
	20.8	18.3	2.41	1.57	-11.8
	26.0	23.0	2.50		-11.5

TABLE 5. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR NITRITE-NITROGEN

	Amount Added	Amount Found mg/L			Bias
Water	mg/L		St	So	%
Reagent	0.36	0.37	0.04	0.04	+2.8
	0.48	0.48	0.06		0.0
	3.00	3.18	0.12	0.06	+6.0
	3.60	3.83	0.12		+6.4
	9.60	9.84	0.36	0.26	+2.5
	12.0	12.1	0.27		+0.6
Drinking	0.36	0.30	0.13	0.03	-16.7
	0.48	0.40	0.14		-16.7
	3.00	3.02	0.23	0.12	+0.7
	3.60	3.62	0.22		+0.6
	9.60	9.59	0.44	0.28	-0.1
	12.0	11.6	0.59		-3.1
Waste	0.36	0.34	0.06	0.04	-5.6
	0.48	0.46	0.07		-4.2
	3.00	3.18	0.13	0.10	+6.0
	3.60	3.76	0.18		+4.4
	9.60	9.74	0.49	0.26	+1.5
	12.0	12.0	0.56		+0.3

TABLE 6. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR BROMIDE

Water	Amount Added mg/L	Amount Found mg/L	St	So	Bias %
Reagent	0.63	0.69	0.11	0.05	+9.5
	0.84	0.85	0.12		+1.2
	5.24	5.21	0.22	0.21	-0.6
	6.29	6.17	0.35		-1.9
	16.8	17.1	0.70	0.36	+1.6
	21.0	21.3	0.93		+1.5
Drinking	0.63	0.63	0.13	0.04	0.0
	0.84	0.81	0.13		-3.6
	5.24	5.11	0.23	0.13	-2.5
	6.29	6.18	0.30		-1.7
	16.8	17.0	0.55	0.57	+0.9
	21.0	20.9	0.65		-0.4
Waste	0.63	0.63	0.15	0.09	0.0
	0.84	0.85	0.15		+1.2
	5.24	5.23	0.36	0.11	-0.2
	6.29	6.27	0.46		-0.3
	16.8	16.6	0.69	0.43	-1.0
	21.0	21.1	0.63		+0.3

TABLE 7. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR NITRATE-NITROGEN

Water	Amount Added mg/L	Amount Found mg/L	St	So	Bias %
Reagent	0.42	0.42	0.04	0.02	0.0
	0.56	0.56	0.06		0.0
	3.51	3.34	0.15	0.08	-4.8
	4.21	4.05	0.28		-3.8
	11.2	11.1	0.47	0.34	-1.1
	14.0	14.4	0.61		+2.6
Drinking	0.42	0.46	0.08	0.03	+9.5
	0.56	0.58	0.09		+3.6
	3.51	3.45	0.27	0.10	-1.7
	4.21	4.21	0.38		0.0
	11.2	11.5	0.50	0.48	+2.3
	14.0	14.2	0.70		+1.6
Waste	0.42	0.36	0.07	0.06	-14.6
	0.56	0.40	0.16		-28.6
	3.51	3.19	0.31	0.07	-9.1
	4.21	3.84	0.28		-8.8
	11.2	10.9	0.35	0.51	-3.0
	14.0	14.1	0.74		+0.4

TABLE 8. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR ORTHO-PHOSPHATE

Water	Amount Added mg/L	Amount Found mg/L	St	So	Bias %
Reagent	0.69	0.69	0.06	0.06	0.0
	0.92	0.98	0.15		+6.5
	5.77	5.72	0.36	0.18	-0.9
	6.92	6.78	0.42		-2.0
	18.4	18.8	1.04	0.63	+2.1
	23.1	23.2	0.35		+2.4
Drinking	0.69	0.70	0.17	0.17	+1.4
	0.92	0.96	0.20		+4.3
	5.77	5.43	0.52	0.40	-5.9
	6.92	6.29	0.72		-9.1
	18.4	18.0	0.68	0.59	-2.2
	23.1	22.6	1.07		-2.0
Waste	0.69	0.64	0.26	0.09	-7.2
	0.92	0.82	0.28		-10.9
	5.77	5.18	0.66	0.34	-10.2
	6.92	6.24	0.74		-9.8
	18.4	17.6	2.08	1.27	-4.1
	23.1	22.4	0.87		-3.0

Water	Amount Added mg/L	Amount Found mg/L	St	So	Bias %
Reagent	2.85	2.83	0.32	0.52	-0.7
	3.80	3.83	0.92		+0.8
	23.8	24.0	1.67	0.68	+0.8
	28.5	28.5	1.56		-0.1
	76.0	76.8	3.42	2.33	+1.1

	95.0	95.7	3.59		+0.7
Drinking	2.85	1.12	0.37	0.41	-60.7
	3.80	2.26	0.97		-40.3
	23.8	21.8	1.26	0.51	-8.4
	28.5	25.9	2.48		-9.1
	76.0	74.5	4.63	2.70	-2.0
	95.0	92.3	5.19		-2.8
Waste	2.85	1.89	0.37	0.24	-33.7
	3.80	2.10	1.25		-44.7
	23.8	20.3	3.19	0.58	-14.7
	28.5	24.5	3.24		-14.0
	76.0	71.4	5.65	3.39	-6.1
	95.0	90.3	6.80		-5.0

TABLE 9. MULTIPLE LABORATORY (n=19) DETERMINATION OF BIAS FOR SULFAT

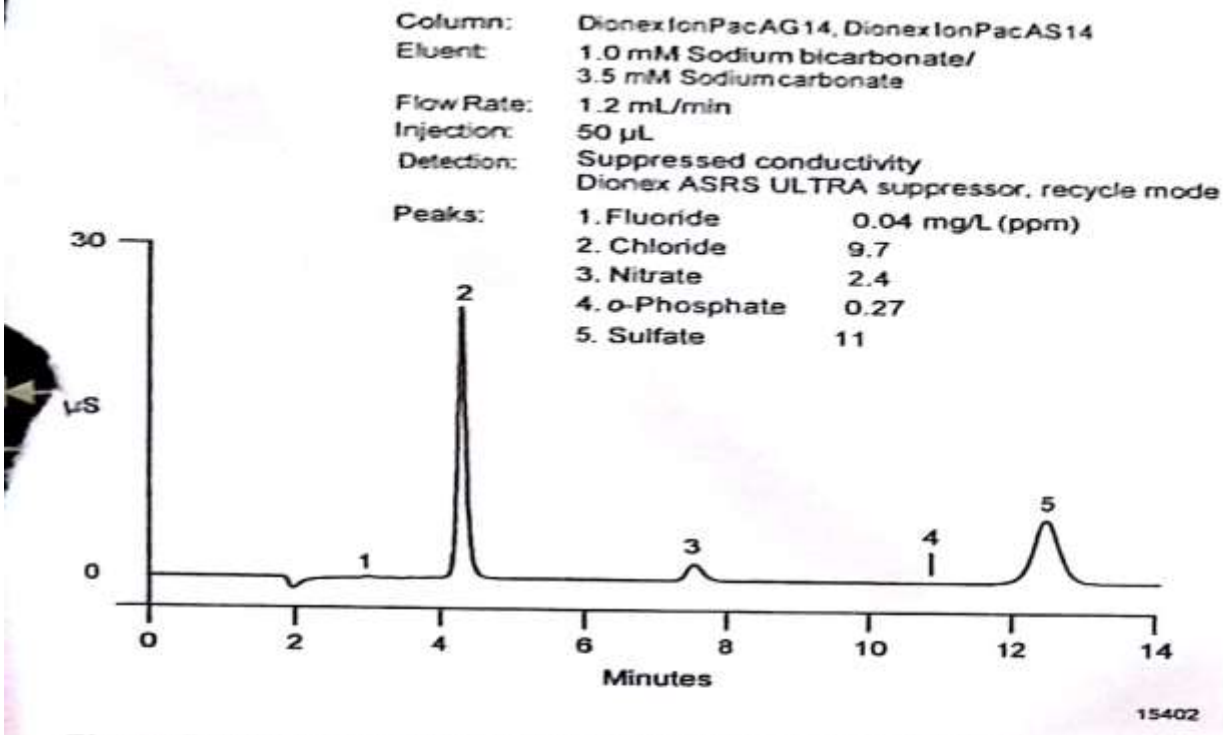

Figure 5. Determination of inorganic anions in drinking water using a Dionex IonPac AS14 column.

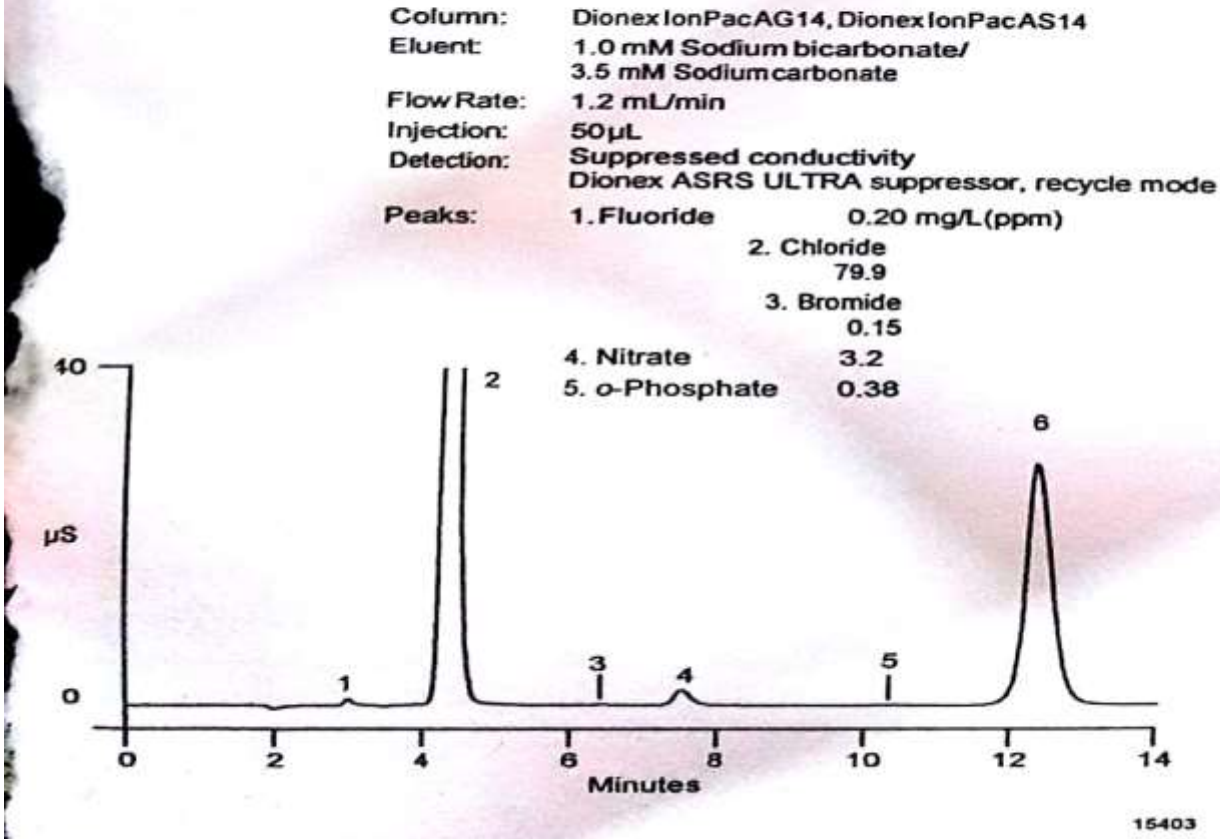

Figure 6. Determination of anions in raw water using a Dionex IonPac AS14 column.

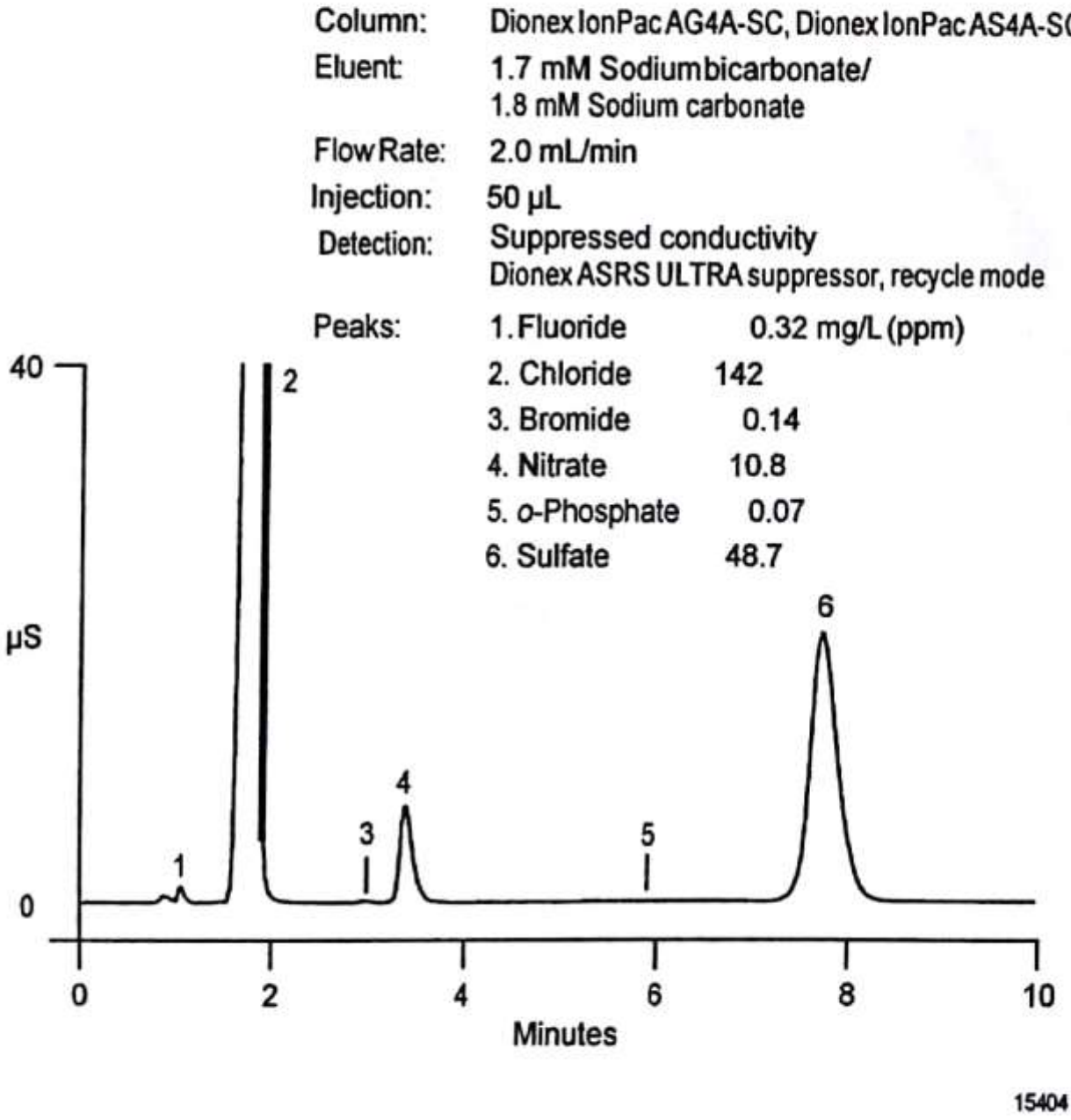

Figure 3. Determination of anions in surface water using a Dionex IonPac AS4A-SC column.

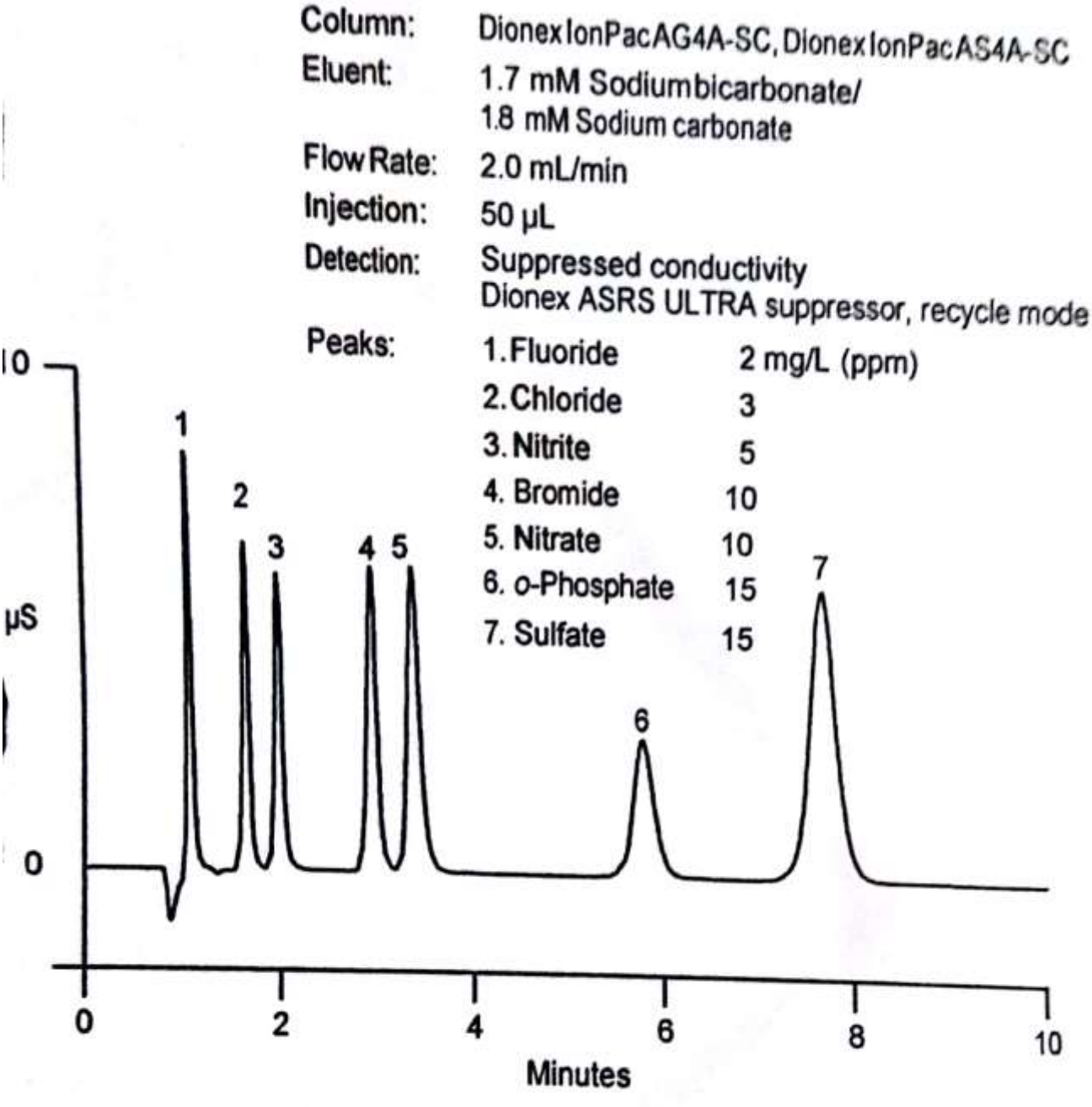

Figure 1. Separation of a low-ppm inorganic anion standard using a Dionex IonPac AS4A-SC column.

RESULTS AND DISCUSSION

The most inorganic anions and cations are measured routinely in a wide variety of water samples such as tap water, underground water, and bottled mineral water. Therefore, a simple, suitably and fast method can determine all major anions and cations in natural water samples would be very useful for routine analysis (Wang et al, 2012). This study was aimed to determination of 5 inorganic anions (fluoride, chloride, bromide, nitrate, and sulphate) and 5 inorganic cations (sodium, ammonium, magnesium, potassium, and calcium) in drinking water samples from Talkha territory and some of its villages.

The selection of chromatographic column and mobile phase (eluent(

According to the literature (Cidu et al, 2011; Zhu et al, 2006), the IonPac AS22 and CS16 columns have a high capacity ion exchange could be efficiently separating anions and cations, respectively Fig. 1A and B. These efficient separations were achieved within 20 min. Fluoride, chloride, bromide, nitrate and sulphate Fig. 1A were good separated from their retention times. Also, from Fig. 1B it could be seen that lithium, sodium, ammonium, magnesium, potassium and calcium were simply identified from their retention times because they always have a good peak shape in less than 20 min.

The selection of an eluent is important to the success separation of anions and cations using ion chromatography (IC). A high eluent concentration should be used to obtain short analysis time (Wang et al, 2012). However, a low eluent concentration was necessary for a satisfactory resolution. To achieve a better separation of the weakly and strongly retained anions, 4.5mmol/L Na_2CO_3 and 1.0mmol/L $NaHCO_3$ was chosen as the eluent. By this eluent, all the 5 anions could be well separated and determined within 20 min in one injection and the flow rate was kept at 1.0 ml/min. At first, increasing methanesulfonic acid concentration decreased retention time of the cations. However, when its concentration was 42mmol/L,

References

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