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# Analysis of anions in water samples from the Holyland (Israel and Jordan) by using Ion Chromatography

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BY

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Research Project

Submitted in  
partial fulfillment of the requirements for the  
Degree of Master of Science,  
With a major in Analytical Chemistry.

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University Park, IL 60484-0975

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**Abstract:**

Ion chromatography is a form of liquid chromatography, which measures concentration of ionic species by separating them based on their interaction with resins. Ion chromatography is mainly useful for the separation and analysis of ions (cations or anions) in water. Analysis of anions in water is of greater importance because water is very important in daily life, especially in the Holy Land, where water is the very essence of life. If variety of chemicals, such as organic and inorganic salts enter into the water system, they make water toxic and poisonous to drink. In this project water samples from different sources in Israel and Jordan were collected and analysed to determine the anion types and their concentration levels using Ion chromatography(IC).

**Introduction:**

The purpose of this study is to analyse the anions present in different water samples. Generally a variety of chemicals from industry, materials like oil, detergents float and spoil the appearance of a water body, as well as being toxic. Chemicals can enter water ways from an industrial site and also movement of rain water over land picks up pollutants such as fertilizers, insecticides and make water toxic. Thus water is being toxic through different ways. In this project we are mainly concerned with separation and analysis of anions in the water samples but not the pollutants.

The separation and analysis of the anions present in different water samples were done by using Ion chromatography (IC). IC is the high performance form of Ion-exchange chromatography. In ion chromatography an ion exchanger is used as a stationary phase and eluent for the determination of anions is a dilute solution of sodium bicarbonate and sodium carbonate. The principle of IC operation is that the stationary phase displays ionic functional group that interact with analyte ions of opposite charge. This type of chromatography is further divided into the following two categories:

- Cation Exchange Chromatography: This technique retains positively charged cations as the stationary phase displays a negatively charged functional group.

- Anion Exchange Chromatography: This technique retains negatively charged anions as the stationary phase displays a positively charged functional group.

IC is of great importance because in ion chromatography with suppressed conductivity detector, the separator column effluent passes through a suppressor column which chemically reduces the eluent background conductance, while at the same time increasing the electrical conductance of analyte ions.

### Experimental:

#### A) Reagents and Standards:

- ACS grade anion standards  
Product Number: 056933  
Lot Number : 43-24 AS

Name of the anion	CAS number
Sodium fluoride	7681-49-4
Sodium chloride	7647-14-5
Sodium nitrate	7631-99-4
Sodium nitrite	7632-00-0
Sodium bromide	7647-15-6
Potassium phosphate	7778-77-0
Disodium sulfate	7757-82-6

- Deionised water, Different water samples-DS, JD, SG, MO, STD.
- 3.5 mM sodium carbonate, 1.00 mM sodium bicarbonate was used to prepare 1000 ml (10ml + 990 ml D.I water) of mobile phase. 2L of Mobile phase was used.

## **B)Instrumentation:**

ICS- 1000 Ion chromatography system configured for anion suppression without gradient pump, conductivity detector and manual injector in a sequence mode.

### ➤ **Conditions:**

- Column: Ion pac AS14 analytical 4\*250 mm

Ion pac AG14 analytical Guard 4\*50 mm

- Eluent: 3.5mM Na<sub>2</sub>CO<sub>3</sub>

1.0mM NaHCO<sub>3</sub>

- Run time: 16 min

- Sample volume: 20μL.

## **C)Method:**

The software used was chromaleon and before the injection of sample prime was carried out to check the prime out of mobile phase. The following procedure was used to carry out prime

1. Chromaleon was selected.
2. Windows-(ICS-1000 system- GSU-DDRB9Y61) screen appears and check whether the ICS-1000 box was checked or not.
3. Pump setting was selected.
4. Option prime was selected then a message indicating to open the valve was displayed. Then valve was opened and option ok was selected.
5. Flow rate was checked and it should be 3ml/min, pump pressure was absent and also it was made sure that flow to drainage bottle is regular
6. Purge was continued for 5 min.
7. Then the option off was selected to stop priming and followed the following conditions to inject the sample
8. The valve was closed and pump setting were checked. Then the flow through column was checked and the flow started at 1.20 ml/min (Pressure 1385 psi).

9. Check was done to make sure that the suppressor was in off mode and the detector setting was checked and make up to auto zero and close option was selected.
10. A series of steps were carried out. First the file option was selected then browser , sequence and holy land water sample (Deionized water, DS water, JD,SG, MO, Standard) was selected.
11. Then the sample was injected.

**Results and Discussion:**

- The retention time of various analytes and their relative concentrations in sample of Deionized water was found to be

Sample number	Peak number	Retention time(min)	Identified Anion present	Rel.Area (%)	Concentration (mg/L)
1	2	1.743	Fluoride	52.17	48.30
2	3	3.160	Bromide	45.24	442.22
3	4	4.200	Nitrite	2.55	16.79

- The retention time of various analytes and their relative concentrations in sample of DS water was found to be

S.No	Peak number	Retention time(min)	Identified Anion present	Rel.Area (%)	Concentration (mg/L)
1	11	1.83	Fluoride	44.97	41.64
2	15	3.34	Bromide	7.08	69.21
3	18	4.657	Nitrite	45.68	300.92
4	19	5.54	Nitrate	2.21	21.14

- The retention time of various analytes and their relative concentrations in sample of SG water was found to be

S.No	Peak number	Retention time(min)	Identified Anion present	Rel.Area (%)	Concentration (mg/L)
1	10	1.84	Fluoride	10.75	9.96
2	13	3.23	Bromide	48.33	472.43
3	14	3.47	Nitrite	35.19	231.82
4	34	6.68	Phosphate	3.90	50.09

- The retention time of various analytes and their relative concentrations in sample of JD water was found to be

S.No	Peak number	Retention time(min)	Identified Anion present	Rel.Area (%)	Concentration (mg/L)
1	8	3.30	Bromide	72.89	712.51
2	13	6.67	Phosphate	10.71	137.54

- The retention time of various analytes and their relative concentrations in sample of MO water was found to be

S.No	Peak number	Retention time(min)	Identified Anion present	Rel.Area (%)	Concentration (mg/L)
1	28	3.27	Bromide	53.83	526.20
2	37	6.69	Phosphate	13.30	170.80

- The retention time of various analytes and their relative concentrations in sample of Standards was found to be

S.No	Peak number	Retention time(min)	Identified Anion present	Rel.Area (%)	Concentration (mg/L)
1	17	1.84	Fluoride	8.92	8.26
2	18	2.457	Chloride	3.90	24.35
3	20	3.157	Bromide	13.58	131.46
4	21	3.637	Nitrite	16.70	110.01
5	24	4.287	Nitrate	11.17	106.88
6	25	4.777	Phosphate	12.13	156.8
7	32	6.593	Sulfate	27.66	157.65

## **Discussion:**

The data showed that different anions are present in different samples of water with variable concentration. In the sample of **Deionized water** the anions present are fluoride, bromide and nitrite. The high concentration of bromide which may lead to irritations of skin, mucous membrane and tissues. The seriousness of poisoning caused by bromine depends on the amount, route, and length of time of exposure, as well as the age and preexisting medical condition of the person exposed.

In the **DS (Dead Sea)** water sample, the anions present are fluoride, bromide, nitrate and nitrite. The high concentration of **nitrite** may cause a disease known as “blue baby syndrome” or methemoglobinemia; where the body converts nitrate to nitrite. This is a blood disorder that impairs the ability of the blood supply to carry oxygen throughout the body. It primarily affects infants less than 6 months of age.

In the sample of **SG (Sea of Galilee)** water the anions present are fluoride, bromide, nitrite and phosphate. The high concentration of bromide may lead to irritations of skin, mucous membrane and tissues. The seriousness of poisoning caused by bromine depends on the amount, route, and length of time of exposure, as well as the age and preexisting medical condition of the person exposed.

In the sample of **JD (Jordan)** water the anions present are bromide and phosphate. The high concentration of bromide may lead to irritations of skin, mucous membrane and tissues. The seriousness of poisoning caused by bromine depends on the amount, route, and length of time of exposure, as well as the age and preexisting medical condition of the person exposed.

In the sample of **MO (Moses)** water the major anions present are bromide and phosphate, indicating highest concentration of bromide.

Finally the (STD) standard sample, contains the seven anions with little variation in concentration.

## **Conclusion:**

The separation of anions in different samples of water was achieved effectively by Ion Chromatography. The concentrations of various anions present in the samples were determined successfully. The results indicate that all the water samples have high concentrations of bromine.

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**Appendix:** Chromatograms.