



Determination of anions in 96% sodium chloride by Ion Chromatography

Keywords: Ion Chromatograph, Shine, Anion

Abstract. Through this article, we want to show how to determine other ions in high concentration salt samples.

Purpose

Confirm that this method is applicable to the determination of anion content in the sample and the feasibility of this laboratory using this method to determine the anion content in the sample.

Instruments and equipment

Ion chromatograph: CIC-D160
Qingdao Shenghan Chromatography Technology Co., Ltd

Ultra pure water machine: UPT-I-20L
Sichuan youpu Chaochun Technology Co., Ltd



Requirements

Reagents

All reagents used are superior grade pure or better, Purchase certified standard solutions F^- , Br^- , NO_3^- , SO_4^{2-} , BrO_3^{2-} , ClO_3^- standard solutions (1000 mg / L)

Deionized Water

When preparing standard samples manually or diluting real samples, please use ASTM filtration and deionization requirements that meet the specifications listed in the table 1.

Table 1: Deionized water specification.

Specification	
Ions Resistivity	$\geq 18.25 M\Omega \cdot cm$
Organics-TOC	<10ppb
Iron/Transition Metals	<1ppb
Pyrogens	<0.03Eu/mL
Particulates (>0.2 μm)	<1unit/mL
Colloids-Silica	<10ppb
Bacteria	<1cfu/mL

Sample preparation

After the sample is moderately diluted, as shown in Table 2, it passes through the activated C18 pretreatment column, and then passes through 0.22 μm filter with filter membrane, discard the 2ml

sample that comes out first, and inject the remaining sample for analysis.

(Activation method of C18 pretreatment column: pass 5 ml of methanol through C18, place it for 10 minutes, rinse it with 5 ml of deionized water, and the activation is completed)

Table 2: Sample dilution table.

Sample	Weight (g)	Solvent	Dilution volume(mL)
1#	0.2024	Deionized water	100
2#	0.2084	Deionized water	100

Chromatographic conditions

Guard column: IonPac AG11HC
 Column: IonPac AS11HC
 Injection volume: 25 µL
 Run time: 40 min
 Flow rate: 1.0 mL/min
 Column oven temperature: 30°C
 Conductivity cell temperature: 35°C
 Suppressor: SHY-A-6
 Suppressor current: 75 mA
 Conductivity detector: SHD-6

Table 3: Gradient conditions.

Time	Concentration
0-15 min	5 mM KOH
15.1-35 min	5-20 mM KOH
35.1-40 min	5 mM KOH

Standard chromatogram, As shown in Figure 1.

Calibration curve

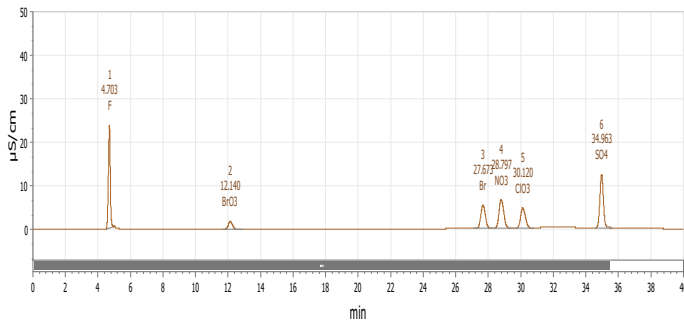


Figure 1. Standard chromatogram

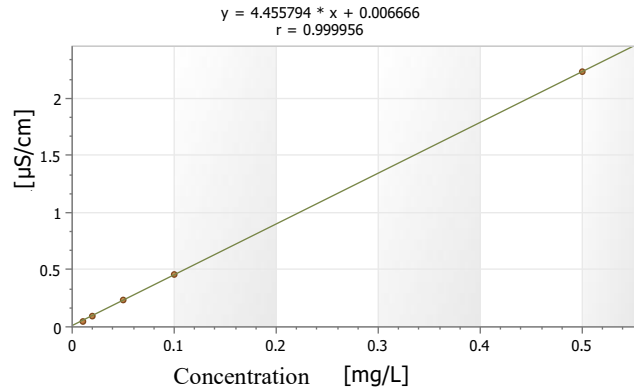


Figure 2. Fluorine ion linearity

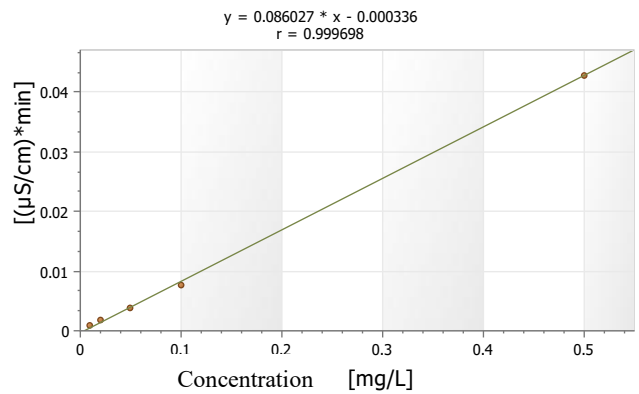


Figure 3. Bromate ion linearity

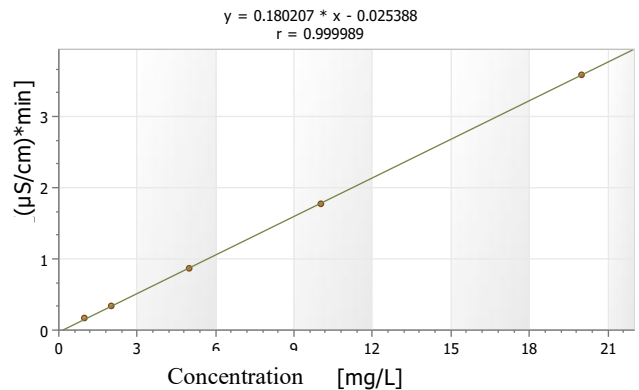


Figure 4. Bromine ion linearity

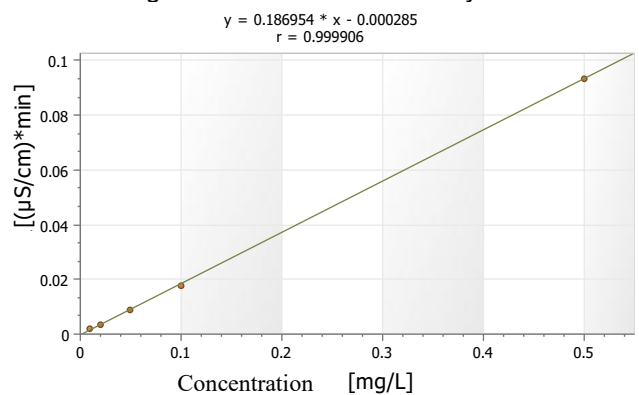


Figure 5. Nitric acid ion linearity

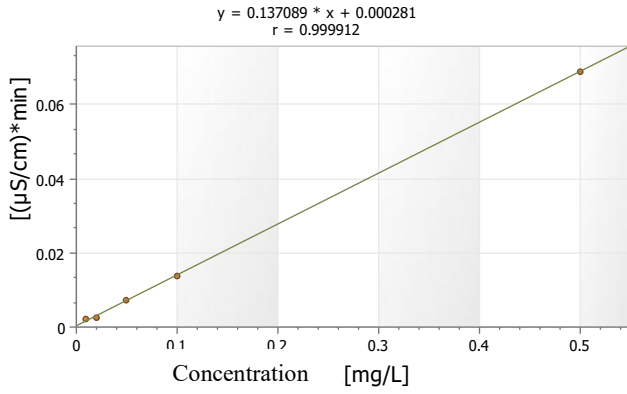


Figure 6. Chloride ion linearity

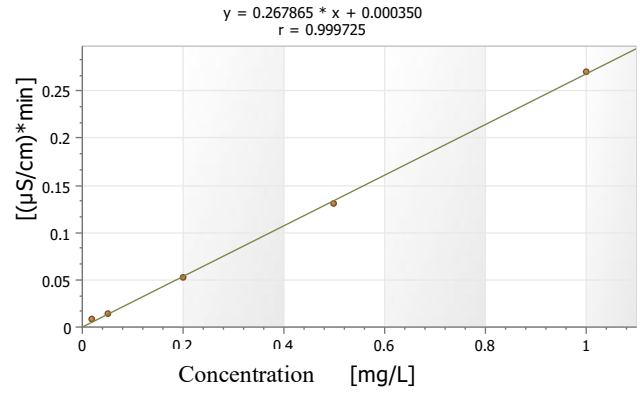


Figure 7. Sulfate ion linearity

Blank control

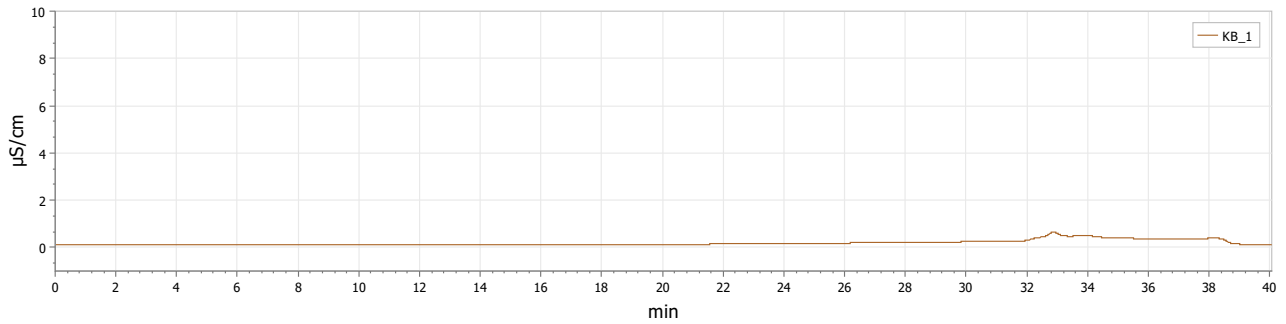


Figure 8. Chromatogram of blank sample

Sample detection

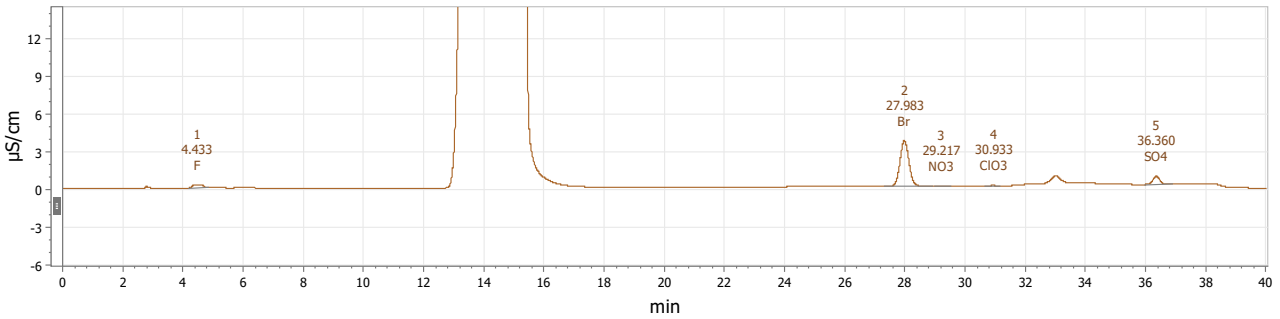


Figure 9. Chromatogram of 1# sample

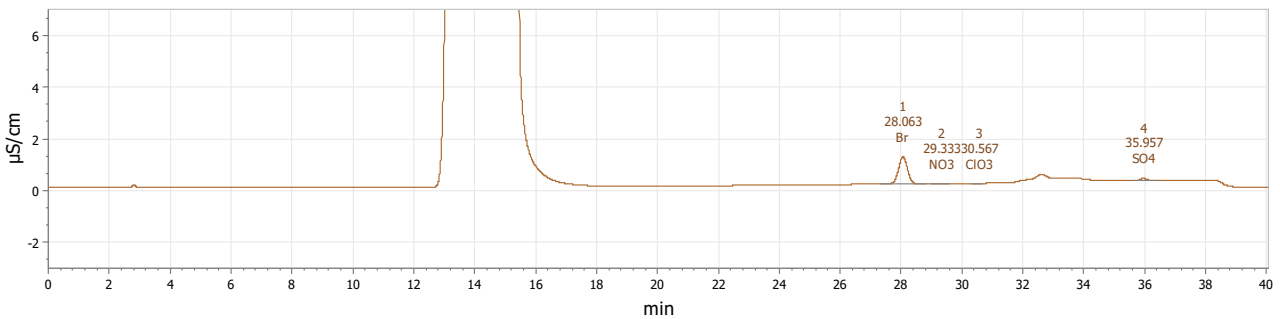


Figure 10. Chromatogram of 2# sample

Results and calculations

Sample	Concentration (mg/kg)					
	F ⁻	BrO ₃ ²⁻	Br ⁻	NO ₃ ⁻	ClO ₃ ⁻	SO ₄ ²⁻
1#	0.05330	ND	3471	6.155	17.41	321.8
2#	ND	ND	1031	2.323	4.042	44.38

Remarks: ① ND indicates that the result is not detected or lower than the detection limit; ② Blank has been deducted from the test results; ③ There will be differences in the test results between different methods and laboratories; ④ Because the separation effect of fluorine ion and Impurity peak ion in sample 1# is not good, the quantitative method of fluorine ion in sample 1# adopts peak height quantitative method.

Feasibility analysis and conclusion

Through the above experiments, it is proved that the detection method has good separation and is suitable for the determination of the content of the components to be measured in the sample.