

AN 51

Method for Determination of Anions in Sodium Hydroxide

INTRODUCTION

This method is used to determine the levels of anionic impurities in a 50% sodium hydroxide solution. Anions which can be determined by this method include chloride, nitrite, sulfate, phosphate, chlorate, and nitrate.

DISCUSSION OF METHOD

The IonPac AS9 can be used for the determination of anions in a caustic solution when used in conjunction with an OnGuard-H Sample Pretreatment Cartridge.

chlorate. Monitoring the chlorate concentration insures the process is working correctly.

RECOMMENDED EQUIPMENT

- Single-channel Ion Chromatograph with conductivity detector
- IonPac AS9 (P/N 42025)
- IonPac AG9 (P/N 42024)
- Anion MicroMembrane Suppressor (AMMS) (P/N 38019)
- 25 μ L Sample Loop
- Nylon or high-density polyethylene bottles (such as Nalgene 2010 or 2002) for storing samples with minimal carbon dioxide absorption
- 100 mL polypropylene volumetric flasks (such as Nalgene 4100) for preparing samples
- Standard laboratory apparatus (analytical balance, pipettes, etc.)
- OnGuard-H Sample Pretreatment Cartridges (P/N 39596)

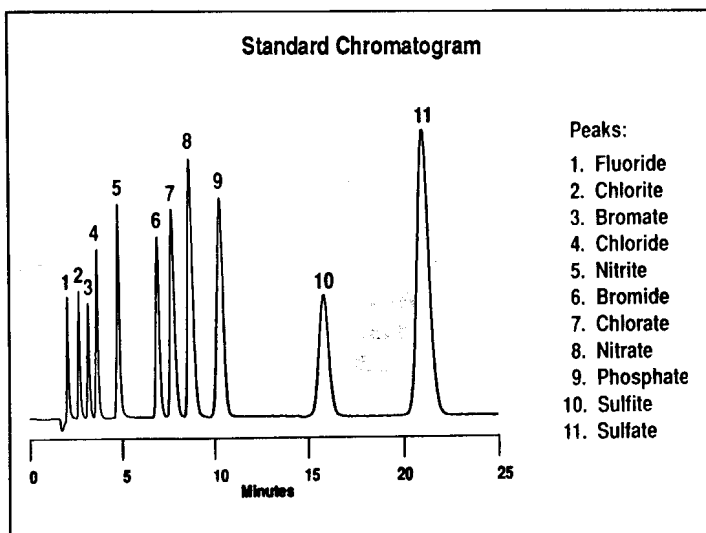


Figure 1

Caustic manufacturers need an accurate method to monitor the concentration of chlorate and other anions in their chlor-alkali processes. In chlor-alkali processes, chlorine and sodium hydroxide are produced by the electrolysis of a saturated brine solution. A diaphragm is employed to separate the chlorine generated at the anode and the caustic soda produced at the cathode. Without the diaphragm to isolate the solutions, the chlorine and caustic would react and eventually produce sodium

SOLUTIONS AND REAGENTS

18 megohm-cm deionized water
Anion Suppressor Regenerant Concentrate 0.50 N
sulfuric acid (P/N 37164 - 4 pack)
Reagent sodium salts of the analytes for preparing standards
AS4A Eluant Concentrate (P/N 39513)

Now sold under the
Thermo Scientific brand

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PREPARATION OF REAGENTS

Eluant and Regenerant Preparation

Step 1. Eluant: 1.8 mM Na₂CO₃/1.7 mM NaHCO₃

① Pipette 10.0 mL of AS4A eluant concentrate into 1000 mL volumetric flask.

② Use deionized water (18 megohm-cm) to dilute the eluant to a final volume of 1000 mL.

Caution: Do not use hydroxide to adjust the pH of the eluant to affect the selectivity of the system. Using hydroxide in the eluant may cause irreversible damage to the IonPac AS9 Separator Column.

Step 2. Regenerant: 20 mN sulfuric acid

Add 20 mL of anion micromembrane concentrate to 400 mL of deionized water. Mix and dilute to a final volume of 500 mL with deionized water.

STANDARDS PREPARATION

Stock Standards

Prepare 1000 ppm standards of each of the following analytes by dissolving the corresponding mass of the salt in 1.000 liter of deionized water:

NaF	2.210 g
NaClO ₂ ·2H ₂ O	2.409 g
NaBrO ₃	1.180 g
NaCl	1.648 g
NaNO ₂	1.500 g
NaBr	1.288 g
NaClO ₃	1.276 g
NaNO ₃	1.371 g
KH ₂ PO ₄	1.433 g
NaSO ₃	1.574 g*
K ₂ SO ₄	1.814 g

Mixed Standards

A convenient mixed standard that gives comparable responses for the different analytes can be prepared by diluting the following amounts of the individual standards together to 1000 mL. Appropriate mixed standards may be prepared from the 1000 ppm standards above.

Volume of Stock Standard	Final Mixed Std. Conc.
1 mL stock	1.0 ppm F ⁻
5 mL stock	5.0 ppm ClO ₂ ⁻
5 mL stock	5.0 ppm Cl ⁻
6 mL stock	6.0 ppm NO ₂ ⁻
10 mL stock	10.0 ppm Br ⁻
15 mL stock	15.0 ppm ClO ₃ ⁻
15 mL stock	15.0 ppm NO ₃ ⁻
20 mL stock	20.0 ppm HPO ₄ ²⁻
25 mL stock	25.0 ppm SO ₄ ²⁻

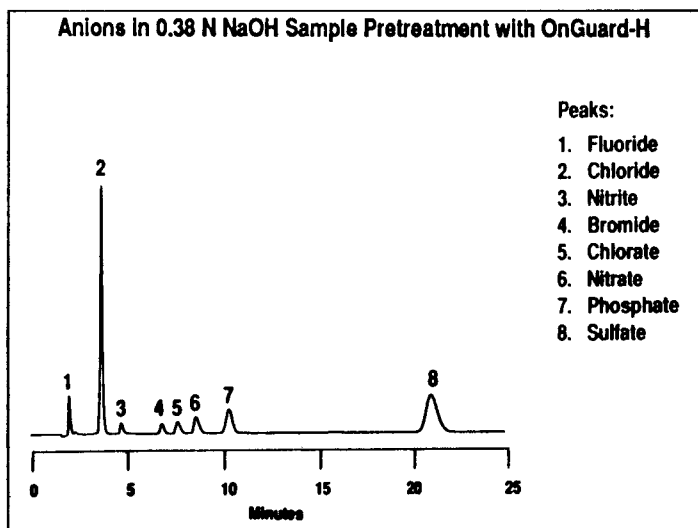


Figure 2

* Dissolve NaSO₃ in 500 mL of DI Water and 145 mL of reagent grade glycerol. Dilute to a Final volume of 1 Liter with DI Water.

SAMPLE PRETREATMENT

The 50% w/v NaOH (19.1 N) samples first must be diluted at least 1:50 (0.38 N or less). This will ensure that the capacity of the OnGuard-H cartridge (1.8 – 2.0 meq/cartridge) is not exceeded before an adequate amount of sample has been flushed through it.

Use a 5 mL syringe to provide the necessary pressure on the OnGuard-H cartridges in manual pretreatment of the sample. Flush the cartridge with 10 mL of deionized water before using a cartridge for sample pretreatment. Push the sample through the cartridge at a flow rate of 4 mL/min. to maximize loading on the cartridge bed. Discard the first 3 mL of sample pushed through the cartridge. Use the next 1 mL of sample to load the sample loop. Inject the sample onto the IonPac AS9 for the determination of the anions in the caustic (Figure 2).

Note: Without proper sample pretreatment it is not possible to obtain accurate results and the chromatography is compromised (Figure 3).

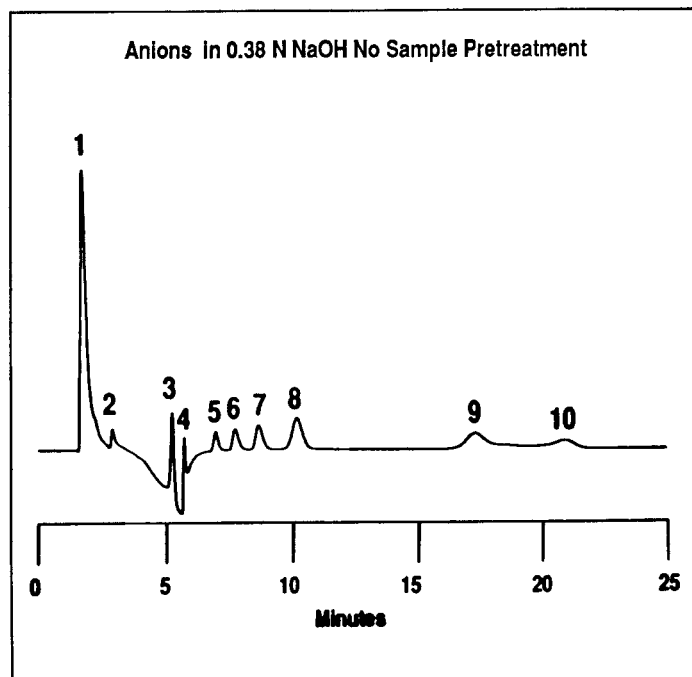


Figure 3 -Note- Anion Concentrations should be as in Figure 2.

CONDITIONS

Sample Loop Volume:	25 μ L
Guard Column:	IonPac AG9
Separator Column:	IonPac AS9
Eluant:	1.8 mM Na_2CO_3 , 1.7 mM NaHCO_3
Eluant Flow Rate:	1.0 mL/min.
Suppressor:	Anion MicroMembrane Suppressor (AMMS)
Regenerant:	20 mN H_2SO_4
Regenerant Flow Rate:	3–5 mL/min.
Expected Background Conductivity:	12–16 μ S
Expected System Operating Backpressure:	300-600 psi

Dionex Corporation
1228 Titan Way
P.O. Box 3603
Sunnyvale, CA
94088-3603
(408) 737-0700

Dionex Corporation
Salt Lake City Technical Center
1515 West 2200 South, Suite A
Salt Lake City, UT
84119-1484
(801) 972-9292

Dionex U.S. Regional Offices
Sunnyvale, CA (408) 737-8522
Westmont, IL (630) 789-3660
Houston, TX (281) 847-5652
Atlanta, GA (770) 432-8100
Marlton, NJ (856) 596-06009

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* Designed, developed, and manufactured under an NSAI registered ISO 9001 Quality System.

