Determination of Trace Anions in Concentrated Caustics Using Single-Pass Neutralization Pretreatment and IC

Aaron Rose[1] and Paul Voelker[2]
[1]Thermo Fisher Scientific, Bannockburn, IL, USA
[2]Thermo Fisher Scientific, Sunnyvale, CA, USA

Introduction

Caustic soda (sodium hydroxide) formed from the electrolysis of brine is used as feedstock in pulp and paper, plastics, detergents, textiles, and other industries. Monitoring the formation of contaminants and byproducts during salt electrolysis enables purity objectives to be achieved in a safe and efficient manner.

Ion chromatography (IC) with suppressed conductivity detection is the preferred method for determining ionic species in aqueous media, especially at ppm and ppb concentrations. Yet, analysis of anions by IC in strong bases (or acids) is a challenge. Direct injections of concentrated base samples overload the column, resulting in poor chromatography and quantification, while dilution sacrifices sensitivity for the anion of interest.

Autoneutralization, introduced by Dionex (now part of the Thermo Scientific brand) in the mid '90s, eliminated the need to dilute the concentrated base, but involved a tedious two-step process requiring either a double-pass or park-and-neutralize step. With the introduction of the new Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor, base neutralization can now be performed with a single pass.

Experimental

Sample Preparation Conditions

Sample:	Diaphragm caustic 1:500 dilution with DI water
Eluent:	DI water with Thermo Scientific™ Dionex™ IonPac™ ATC-HC 500 Anion Trap Column (9 × 75 mm)
Flow Rate:	1 mL/min
Injection Volume:	25 µL
Temperature:	30 °C
Neutralization:	Dionex AERS 500 suppressor, 4 mm (P/N 082540)
Concentrator Column:	Dionex IonPac UTAC-XLP1

Analytical Conditions

Column:	Dionex IonPac AS19 Analytical (2 × 250 mm)
Eluent:	10 mM potassium hydroxide (KOH) 0–12 min; 10–45 mM KOH 12–25 min
Eluent Source:	Thermo Scientific Dionex EGC 500 KOH Eluent Generator Cartridge
Flow Rate:	0.5 mL/min
Temperature:	30 °C
Detection:	Suppressed conductivity, Dionex AERS 500 suppressor, 2 mm (P/N 082541), AutoSuppression, recycle mode

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Results

Figure 1 depicts the two-valve configuration for single-pass neutralization with either the Thermo Scientific™ Dionex™ ICS-5000⁺ HPIC™ system, capable of high-pressure IC, or the Dionex ICS-2100 IC system.

- Valve 1 employs external water regeneration for the Dionex AERS 500 suppressor by pump (Thermo Scientific Dionex AXP Auxiliary Pump for a Dionex ICS-2100 IC system or Thermo Scientific Dionex DP1 Dual Pump for a Dionex ICS-5000⁺ HPIC system) with a Thermo Scientific Dionex ATC-HC trap column and valve switch that pushes a 25 µL loop of caustic sample through a 4 mm Dionex AERS 500 suppressor (the neutralizer mode) and then on to Valve 2.
- Valve 2 employs a KOH eluent and valve switch to elute the neutralized sample off the Dionex IonPac UTAC-XLP1 column onto a Dionex IonPac AS19 column and through a 2 mm Dionex AERS suppressor run in recycle mode.

New Optimized Single Pass Neutralization

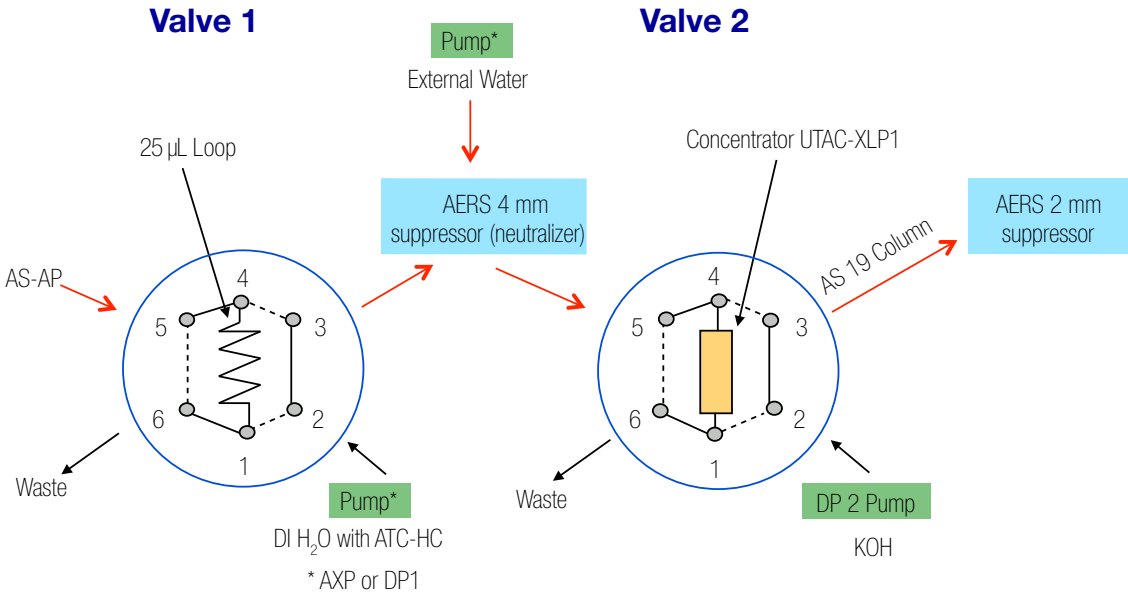

Figure 1. Single-pass pretreatment and IC analysis configuration.

Traditional Double Pass Neutralization

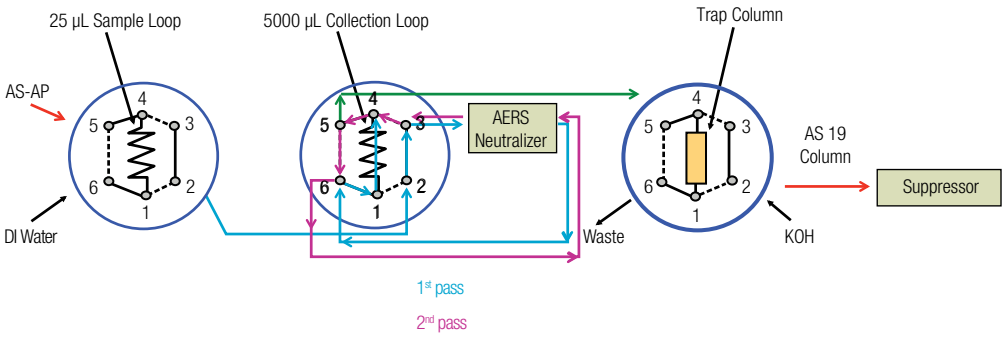

Figure 2. Double-pass neutralization and IC analysis configuration.

Figure 2 depicts the now outdated configuration for double-pass neutralization using either the Dionex ICS-5000+ HPIC system or the Dionex ICS-2100 IC system.

- Here the caustic sample is first loaded into a 25 µL sample loop. The sample is then switched in line and the concentrated base from the sample loop is directed to the Thermo Scientific™ Dionex™ ASRN™ Anion Self-Regenerating Neutralizer. There the sample is partially neutralized, then transferred to the 5000 µL loop.
- The recycle valve is then actuated to direct the sample through the Dionex ASRN neutralizer a second time to complete the neutralization. Following the double-pass neutralization process, the sample anions can be focused onto a concentrator column before eluting them onto an analytical column.

Conclusion

The single-pass neutralization pretreatment with the new Dionex AERS 500 suppressor (operated in neutralizer mode) provides a more efficient alternative to the double-pass neutralization method for the analysis of anions in concentrated caustics. Results from the single-pass method with IC satisfy purity objectives in chlor-alkali processes by measuring trace levels of chlorate, chloride, and sulfate in a faster, more effective manner (Figure 3).

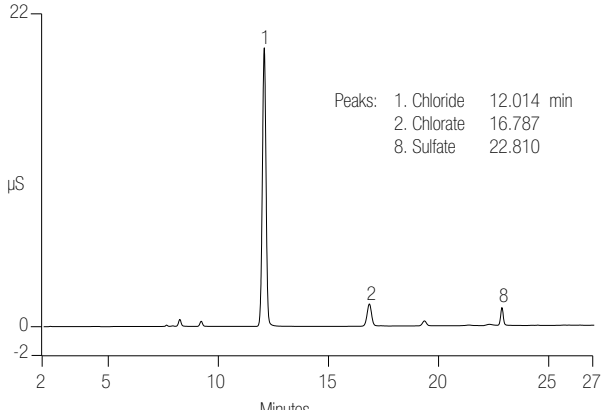

Figure 3. Single-pass neutralization and IC analysis.

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