

Application Note AN-O-043

Carbonate impurities in caustic soda

Robust IC analysis of carbonate in sodium hydroxide

Sodium hydroxide (NaOH, also known as caustic soda or soda lye) is a strong, harmful base made of solid white crystals. It is used in many industries (e.g., paper manufacturing, petrochemistry, soap manufacturing [1]) with an annual production of about 60 million tons worldwide [2]. Caustic soda is very hygroscopic and also absorbs carbon dioxide (CO_2) from the atmosphere. This increases the carbonate content while reducing the hydroxide concentration of the product. Therefore, it should be stored under both water-free and CO_2-free conditions, with minimal atmospheric contact. Depending on the application and its purity requirements, caustic soda as a raw product must fulfill the respective quality criteria.

Quality control analysis comprises the determination of contaminants such as alkalinity, trace metals, anionic impurities (e.g., nitrate and sulfate), as well as carbonate. The analysis of carbonate by ion chromatography (IC) is quite tricky. Suppressed anion IC with hydroxide eluents does not work as suppression converts carbonate back to carbonic acid (H_2CO_3) with almost no sensitivity. In this Application Note carbonate determination in NaOH is demonstrated by applying ion-exclusion chromatography with conductivity detection after inverse suppression – a way for automated, reliable, and secure quantification.

SAMPLE AND SAMPLE PREPARATION

Two 50% sodium hydroxide solutions are analyzed for their carbonate content (Figure 1). As 50% NaOH is too highly concentrated to be directly injected into the IC, a 1:20 dilution with degassed ultrapure water is applied. To avoid extra CO_2 adsorption, the final sample is kept in a closed bottle. Depending on the quality of the original sample, Metrohm Inline Ultrafiltration is a suitable choice for automated sample preparation.

EXPERIMENTAL

The analysis is performed on a Compact IC Flex system, fully automated with a 858 Professional Sample Processor and the Metrohm intelligent Partial Loop Injection Technique (MiPT) (Figure 2). Carbonate (as carbonic acid) is separated from other analytes in the sample on the Metrosep Organic Acids - 250/7.8 analytical column with a sulfuric acid eluent (Figure 1). The column is operated at 30 °C, providing thermal stability for reproducible results.

With direct conductivity detection, carbonate (as carbonic acid) cannot be quantified. Thus, after separation, inverse suppression is applied prior to conductivity detection. In contrast to classical anion suppression, inverse suppression replaces the H^+ ion with Li^+. This step neutralizes the sulfuric acid eluent and converts weak acids into their salts. In this way, the eluent background conductivity is reduced, and the weak acids are dissociated. This improves the conductivity response of e.g., organic acids or carbonic acid.

A

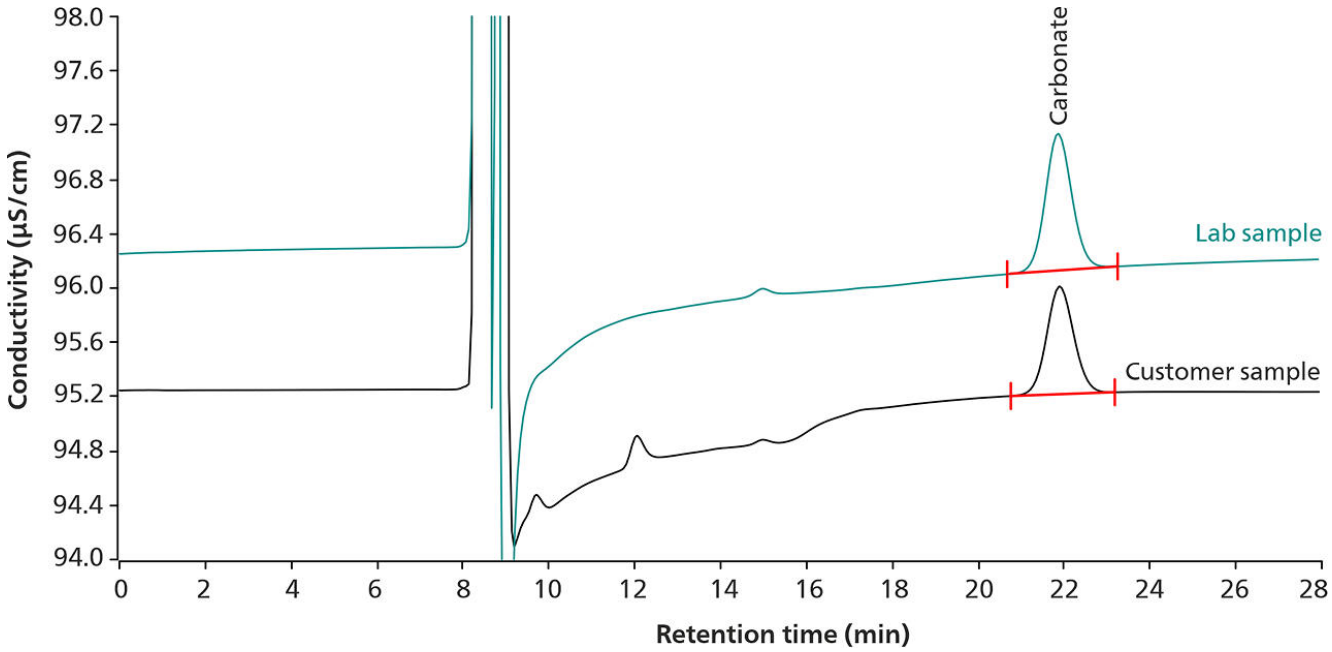


Figure 1. (A) Chromatogram overlay of carbonate in two 50% NaOH samples (lab sample: 1018 mg/kg carbonate, customer sample: 816 mg/kg carbonate) analyzed by a Compact IC 930 Compact IC Flex. A 20 µL volume of the diluted samples is injected applying the Metrohm intelligent Partial Loop Injection Technique (MiPT).

B

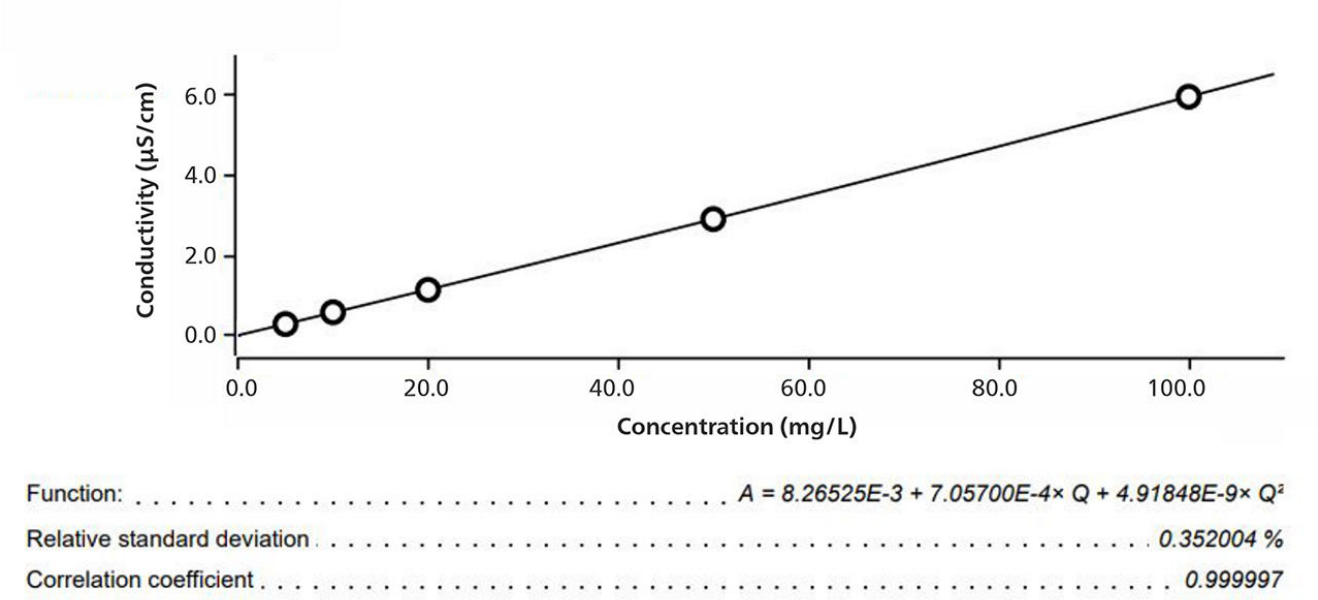


Figure 1. (B) The calibration (5–100 mg/L) is performed from a single standard using MiPT.



Figure 2. Compact instrumentation to quantify carbonate in sodium hydroxide: Compact IC Flex with a Dosino for MiPT and a 858 Professional Sample Processor.

RESULTS

The calibration ranged from 5 to 100 mg/L for carbonate (Figure 1). The calibration curve is built by injecting different volumes of a single standard solution as a fully automated process with MiPT (Figure 1).

Two sample bottles of 50% NaOH were tested in this application example. The first was supplied externally, and the second bottle was sourced from the testing laboratory. Table 1 shows the concentration of carbonate in sodium hydroxide from these two samples.

Table 1. Concentration of carbonate in two samples of 50% sodium hydroxide as measured by ion-exclusion chromatography.

	Carbonate (mg/kg)
Customer sample	816
Lab sample	1018

SUMMARY

The quality control of sodium hydroxide for carbonate content is possible with ion-exclusion chromatography. To use the universal maintenance-free detection technique of conductivity, inverse suppression with Li^+ is a crucial step. This is easily performed with the Metrohm Suppressor Module (MSM). The freedom of usable regeneration solutions and 100% pH stability allows full flexibility of the MSM for a variety of challenging applications.
Automated IC delivers fast and reliable results. The Metrohm intelligent Partial Loop Injection Technique and optional Inline Ultrafiltration save additional time, reduce labor costs, and make this an efficient and accurate choice for quality control measurements.

REFERENCES

1. NIOSH. *Sodium Hydroxide, The National Institute for Occupational Safety and Health (NIOSH).* https://www.cdc.gov/niosh/topics/sodium-hydroxide/ (accessed 2022-05-13).
2. Vargel, C. Chapter E.4 - Inorganic Bases. In *Corrosion of Aluminium*; Vargel, C., Ed.; Elsevier: Amsterdam, 2004; pp 385–393.

Internal reference: AW IC CH6-1220-122014

CONTACT

Metrohm Central Africa
2 Almond Street, Accra,
Ghana
0000 Accra
info@metrohm.com.gh

CONFIGURATION



Metrosep Organic Acids - 250/7.8

The Metrosep Organic Acids - 250/7.8 is a polymer-based cation exchanger column. It is the high-performance column for the determination of organic acids and for the solution of difficult and complex separation problems. In addition, carbonate (with inverse suppression), fluoride (hydrofluoric acid) and phosphate (phosphoric acid) can be determined along with organic acids. In comparison with the Hamilton PRP-X300 - 250/4.0, the Metrosep Organic Acids - 250/7.8 has greater capacity and enhanced selectivity.

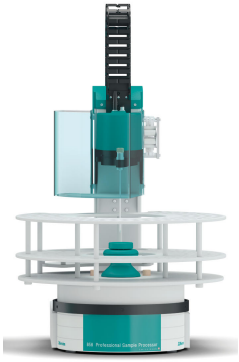


930 Compact IC Flex Oven/ChS/PP/Deg

The 930 Compact IC Flex Oven/ChS/PP/Deg is the intelligent Compact IC instrument with **column oven**, **chemical suppression** and a **peristaltic pump** for suppressor regeneration and built-in **degasser**. The instrument can be used with any separation and detection methods.

Typical areas of application:

- Anion determinations with chemical suppression and conductivity detection
- Organic acids with ion-exclusion chromatography and inverse suppression



858 Professional Sample Processor – Pump

The 858 Professional Sample Processor – Pump processes samples from 500 µL to 500 mL. The sample transfer takes place either with the installed bidirectional two-channel peristaltic pump or with an 800 Dosino.



IC equipment: MiPT

Accessory set for assembling a Dosino for Partial-Loop-Injection.



800 Dosino (cable: 1.5 m)

The 800 Dosino is a drive with write/read hardware for intelligent dosing units. With permanently attached cable (length 1.5 m).