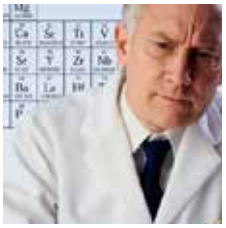
HORIBA Scientific

JOBIN YVON Technology

ICP-OES

Analysis of 10% NaOH and 10% NaCl



Application Note

Chemicals 01

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Introduction

NaOH and NaCl samples at 100 g/L were presented to this laboratory for trace determination, where analyte levels were thought to be less than 100 ppm. This document gives a selection of the best wavelengths for analysis in these matrices as well as an estimation of the detection limits.

Production of salt is mainly done by solar evaporation of seawater or inland brines (solar salt) and by mining rock salt deposits (rock salt called Halite). Annual production is exceeds 250 million metric tons and is increasing, due to the growing demand from the Chinese chemical industry. Since salt's value increases with purity, industrial production is focussed on making the purest sodium chloride possible.

Principle

Technique used

Elemental analysis of solutions of 100 g/L NaOH and 100 g/L NaCl was undertaken by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

Limits of detection estimation

The limits of detection are calculated using the following formula:

$$\mathbf{LOD = k \times BEC \times RSD_0}$$

Where:

LOD = limits of detection,
k = 3 for the normal 3-sigma values,
BEC = Background equivalent concentration, RSD_0 = relative standard deviation of the blank.

To calculate the LOD, a calibration curve is constructed using two points, 0 ppm and 5 ppm, or some concentration where the calibration is linear; this gives the BEC. The RSD_0 is evaluated by running the blank ten times.

Sample preparation

Pellets of NaCl and NaOH were dissolved in deionized water (10 g in 100 mL).

Instrument specification

The work was done on a Ultima Expert. The specifications of this instrument are listed below.

Table 1: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny-Turner
Focal length	1 m
Thermoregulation	Yes
Variable resolution	Yes
Nitrogen purge	Yes
Grating number of grooves	2400 gr/mm
1st order resolution	0.010 nm
2nd order resolution	0.005 nm
Order	2nd order

Table 2: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state, crystal controlled
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of pump flow	By computer
Cooling	Air

Operating conditions

The operating conditions are listed in table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1200 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer gas flowrate	0.6 L/min
Nebulizer flowrate	3.4 bars (51 psi)
Sample uptake	1 mL/min
Type of nebulizer	Cross flow
Type of spray chamber	Scott
Argon humidifier	Yes
Injector tube diameter	3.0 mm

Wavelength selection and analytical conditions

The line with the highest sensitivity was used for analysis of all analytes, as there were no problems with interferences. The analysis conditions were the same for all elements except the alkalis.

Table 4: Definition of slits

Element	Slits (µm)	Analysis mode	Integration time (sec)
All elements	20 x 15	Direct peak	8
Alkali elements	20 x 15	Gaussian	1

The use of the argon humidifier, a cross flow nebulizer and the large internal diameter (ID) of the injector tube enabled trouble free analysis, even with the high dissolved salts. The larger ID injector tube also ensures a minimization of interferences. Due to the high dissolved salts an initial conditioning of the spray chamber is advisable for maximum stability, it is also imperative to use matched standards or standard addition because of the viscosity of the solutions.

Discussion

Limits of Detection

The limits of detection have been calculated using the formula presented before. They are calculated in µg/L. The less sensitive Ca 317.933 nm line was chosen because of the high concentrations present in the solutions. The Ca 393.366 nm and Ca 396.847 nm lines are also suitable where lower detection limits are required.

Table 5: Limits of detection

Element	Wavelength (nm)	LOD in 10% NaOH	LOD in 10% NaCl
Al	396.152	5	5
As	189.042	5	2
B	208.959	1	1
Ba	455.403	0.1	0.1
Be	313.042	0.5	1
Ca	317.933	2	2
Cd	228.802	0.2	0.2
Co	228.616	0.2	0.2
Cr	267.716	0.5	0.5
Cu	324.754	1.3	1
Fe	259.940	0.3	0.3
K	766.490	5	5
Li	670.784	0.5	0.1
Mg	279.553	0.05	0.1
Mn	257.610	0.1	0.1
Mo	202.030	0.5	0.5
Ni	231.604	1	1
P	178.229	10	10
Pb	220.353	5	5
S	180.676	5	5
Sb	206.833	5	5
Se	196.090	5	10
Sr	407.771	0.1	0.1
Ti	334.941	0.3	0.4
Tl	190.864	5	10
V	292.402	2.5	2
Zn	213.856	0.2	0.5

Results

For the analysis of these samples, the standard addition method was used. The results are summarized in table 6 below.

Table 6: Results obtained on real samples

Element	Unit	Samples		
		NaCl 100 g/l	NaOH 100 g/l	NaOH 300 g/l
Al	µg/L	12	6	31
As	µg/L	2	7	16
B	µg/L	38	14	27
Ba	µg/L	1	13	32
Be	µg/L	1.2	< 0.5	< 0.5
Ca	mg/L	0.79	0.02	78.2
Cd	µg/L	0.4	1	< 0.2
Co	µg/L	< 0.2	1	1.4
Cr	µg/L	< 0.2	3	16
Cu	µg/L	6	2	3
Fe	µg/L	136	62	154
K	mg/L	19.2	2.7	3.7
Li	µg/L	4	54	56
Mg	µg/L	190	6	13
Mn	µg/L	0.1	2	3
Mo	µg/L	< 0.5	< 0.5	< 0.5
Ni	µg/L	< 1	64	38
P	mg/L	0.025	0.32	0.06
Pb	µg/L	< 5	< 5	< 5
S	mg/L	4.54	0.32	0.96
Sb	µg/L	< 5	< 5	< 8
Se	µg/L	< 10	< 5	< 10
Sr	µg/L	21	192	12
Ti	µg/L	1	< 0.3	1.7
Tl	µg/L	< 10	< 5	< 10
V	µg/L	< 1	< 2	< 2
Zn	µg/L	7	15.7	42

Summary

Dilution is undesirable when trying to achieve the lowest detection limits. The results presented in this Application Note show that the HORIBA spectrometers perform the analysis very well with high dissolved solids, eliminating the need for dilution and thus achieving superior detection limits.

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