

SODIUM CHLORITE

New specifications prepared at the 68th JECFA (2007) and published in FAO JECFA Monographs 4 (2007). An ADI of 0.03 mg/kg bw for chlorite was established at the 68th JECFA (2007).

SYNONYMS

Chlorous acid sodium salt

DEFINITION

Sodium chlorite is manufactured by first reducing, chemically or electrochemically, sodium chlorate, in the presence of hydrochloric acid, to produce chlorine dioxide. The chlorine dioxide is then reduced with hydrogen peroxide in a sodium hydroxide solution to yield a solution containing 30 to 50 percent sodium chlorite, which can be dried to give a solid with ca. 80% sodium chlorite.

Alternatively, chlorine dioxide may be obtained by reacting together sodium chlorate, hydrogen peroxide, and sulfuric acid. The chlorine dioxide is then reduced with hydrogen peroxide in sodium hydroxide solution to yield a solution of sodium chlorite, which can be neutralized with sulfuric acid. The resulting solution may be dried to a solid and the sodium chlorite content may be adjusted to ca. 80% by the addition of sodium chloride, sodium sulfate, or sodium carbonate.

Sodium chlorite is marketed as a solid or an aqueous solution.

C.A.S. number

7758-19-2

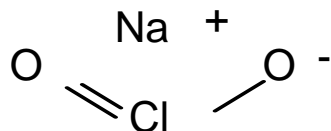
Chemical formula

NaClO₂

Formula weight

90.44

Structural Formula



Assay

79-86%

DESCRIPTION

White crystalline powder, solutions can be colourless to greenish yellow

FUNCTIONAL USES

Antimicrobial agent (for use in antimicrobial washing solutions)

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Soluble in water, sparingly soluble in polar solvents and insoluble in non-polar solvents.

Chlorite Add 2 g of sample into a 250-ml Erlenmeyer flask and add 50 g of deionized water to dissolve. Add 10 ml of 1.0N hydrochloric acid and swirl flask until a yellow colour appears. Add 2 g potassium iodide and swirl to dissolve. The solution turns brown and a grey precipitate forms.

Sodium (Vol. 4) Passes test

PURITY

Loss on drying (Vol.4) Not more than 6% (105°, 24 h, use 5 g of sample)

Sodium carbonate Not more than 8% on the dried basis
See description under TESTS

Sodium hydroxide Not more than 3% on the dried basis
See description under TESTS

Sodium sulfate Not more than 5% on the dried basis
See description under TESTS

Sodium chlorate Not more than 4% on the dried basis
See description under TESTS

Sodium chloride Not more than 19% on the dried basis
See description under TESTS

Lead (Vol. 4) Not more than 5 mg/kg on the dried basis
Determine using an AAS/ICP-AES technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Vol. 4 (under "General Methods, Metallic Impurities").

TESTS

PURITY TESTS

Sodium chlorate and Sodium sulfate Determination by Ion Chromatography

Apparatus

Ion Chromatograph with anion suppressor, autosampler, conductivity detector and a data station (Dionex or equiv.)

Analytical Column: [Dionex AS9-HC, 2 mm or 4 mm or equiv.]

Guard column: [Dionex AG9-HC, 2 mm or 4mm or equiv.]

Mobile phase: 9 mM Na₂CO₃ at flow rate of 0.4 ml/min

Reagents

Deionized water: Ultra high quality (UHQ) passing through 0.20 µm filter, Milli Q or equiv.

Mobile phase [sodium carbonate solution (9mM)]: Dissolve 1.91 g Na₂CO₃ in deionized water and dilute to 2 liters.

Chlorate (ClO₃⁻) stock standard solution (1000 mg/l): Transfer 0.1275 g of sodium chlorate, accurately weighed, into a 100-ml volumetric flask. Dissolve the sample in deionized water, make up to volume and mix.

Sulfate (SO₄²⁻) stock standard solution (1000 mg/l): Transfer 0.1814 g of potassium sulfate, accurately weighed, into a 100-ml volumetric flask. Dissolve the sample in deionized water, make up to volume, and mix.

Ethylenediamine (EDA) preservation solution (100 mg/ml): Dissolve 2.5 g of EDA in 25 ml of deionized water. Prepare fresh monthly.
Surrogate solution: Transfer 0.065 g of potassium dichloroacetate into a 100-ml volumetric flask, dissolve in deionized water, make up to volume, and mix. (prepare fresh every 3 months)

Working standard solutions for standard curve: Prepare a series of 5 standards covering the entire calibration range by diluting the stock chlorate and sulfate standards with deionized water in suitable volumetric flasks. Add enough EDA preservation solution to the chlorate working standards to obtain a final concentration of 50 mg/l, in order to normalize any bias from the presence of EDA in analysis samples.

Sample solutions: Accurately weigh about 2.0 g of solid sample, dissolve in deionized water, quantitatively transfer into a 100-ml volumetric flask, make up to volume, and mix. Prepare sample solution fresh prior to analysis. If analysis is delayed, refrigerate solution. Draw solution into a 10-ml plastic syringe, attach a 0.45 µm filter and filter into an auto sampler vial.

Procedure

Construction of standard curve: Set up the ion chromatograph, purge the column with the mobile phase and check the base line stability. Inject 10 µl each of working standards for analysis of sulfate and 50 µl each for analysis of chlorate using the 2 mm column (inject 50 and 200 µl samples, respectively, when using 4 mm column). Construct standard curves for sulfate and chlorate and check curves for linearity ($R^2 > 0.99$). Verify calibration curve acceptability each day prior to analysis by injecting a middle standard as a sample and confirm acceptance (error shall not be more than 5%). If the error is more than 5%, repeat construction of standard curve.

System calibration: Transfer a 10.0 ml aliquot of sample solution and 20 µl of the surrogate solution to a 20 ml disposable plastic beaker and mix. Draw the solution into a 10-ml plastic syringe, attach a 0.45 µm filter and filter the solution into an auto-sampler vial. Inject the solution [10 µl for determination of sulfate and 50 µl for determination of chlorate using a 2 mm column (inject 50 and 200 µL samples, respectively, when using 4 mm column)]. Record retention times and peak areas. Calculate the surrogate recovery from the surrogate concentration obtained and surrogate concentration fortified. The percent surrogate recovery shall fall between 90-115%. If the

surrogate recovery falls outside the 90-115% window, an analysis error is evident and the analysis should be repeated.

Inject sample solutions. If the analyte response exceeds the calibration range then dilute and inject again. Obtain the concentration of sulfate and chlorate from the respective standard curves and calculate the sulfate and chlorate content in the samples from the weight of sample and concentration of analyte in the injected solution.

Sodium chloride

Determination by potentiometric titration with silver nitrate solution

Apparatus

pH meter capable of reading millivolts (mV) and equipped with a chloride-specific-ion electrode or silver/silver chloride electrodes

Procedure

Electrode calibration: Connect the electrodes to the pH meter and set the meter to read mV. Transfer about 2 g of sample into a beaker and dissolve in water. Place the electrodes in the solution. Titrate with 0.1N silver nitrate (0.5 ml increments), recording the volume of titrant added along with the mV reading after each incremental addition.

Plot the titer value (ml) on the X-axis and mV on the Y-axis.

Determine the mV reading at the inflection point of the titration curve and use this point as the endpoint in subsequent titrations. Repeat the endpoint determination on a regular basis.

Accurately weigh about 1 g of solid sample and dissolve in about 100 ml of deionized water in a beaker. Add 2 drops of phenolphthalein indicator and nitric acid (1:3 by volume) drop-wise until the phenolphthalein endpoint is reached (alternatively, test with pH test paper). Place the electrodes in sample solution. Titrate with 0.1N silver nitrate solution until the endpoint determined in the electrode calibration is reached. Record the titer value in ml (T).

Calculation:

$$\text{NaCl, \% (w / w)} = \frac{T \times N \times 5.845}{W}$$

where:

58.45 is the formula weight of sodium chloride,

N is the normality of silver nitrate solution,

W is the weight of sample (g), and

$$\text{the factor 5.845 is } \frac{58.45 \times 100}{1000}$$

METHOD OF ASSAY Determination of sodium chlorite content by an iodometric method

Accurately weigh about 4.0 g of solid sample (W), dissolve in deionized water, quantitatively transfer into a 100-ml volumetric flask, make up to volume and mix. Pipet 50 ml of into a 500-ml volumetric flask, dilute to volume and mix to obtain the test sample solution.

Pipette 25 ml of sample solution into a 500 ml narrow-mouth Erlenmeyer flask, add 100 ml of deionized water and 10 ml 0.5N Hydrochloric acid. Add about 2 g of potassium iodide, stopper the flask, mix and keep in a dark place for 2 min. Titrate the liberated iodine with 0.1N Sodium thiosulfate until most of the iodine colour has disappeared. Add 2 ml Starch TS and titrate until the blue colour is discharged. Record the titer value in ml (T_1). Conduct a blank determination and record the titer value in ml (T_2).

Calculation

$$\text{NaClO}_2\%(\text{w} / \text{w}) = \frac{(T_1 - T_2) \times N \times 90.44}{W}$$

where:

22.61 is the equivalent weight of sodium chlorite,
N is the normality of sodium thiosulfate,
W is the weight of sample (g), and

the factor 90.44 is $\frac{22.61 \times 100 \times 500 \times 100}{1000 \times 50 \times 25}$