

British Standard Methods of testing

Water used in industry

Part 123. Sodium

Méthodes d'essai de l'eau à usage industriel
Partie 123. SodiumPrüfverfahren für Wasser für industrielle Zwecke
Teil 123. Natrium

IMPORTANT NOTE. It is essential that this Part be read in conjunction with the information in Part 100 of this standard, 'Foreword, scope and general requirements', which is published separately.

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Compliance with a British Standard does not of itself confer immunity from legal obligations.

0. Introduction

Parts 123, 124 and 125 of this standard together supersede BS 2690 : Part 10 : 1970, which is withdrawn.

This Part supersedes clause 2 of BS 2690 : Part 10 : 1970, and includes a third, potentiometric method.

1. Scope

The methods described are for the determination of sodium in industrial waters, using a flame photometric method, a flame spectrophotometric method and a potentiometric method. The flame photometric method is suitable for samples containing high sodium concentrations, while the more sensitive flame spectrophotometric and potentiometric methods are suitable for the determination of

sodium ions in feedwater, steam condensate and other similar pure waters.

2. Flame photometric method

2.1 Range. 0.2 mg to 5 mg of sodium (expressed as Na) per litre, depending on the instrument used.

2.2 Principle. A flame photometer, fitted with a selective filter for sodium determination, measures photoelectrically the intensity of colour imparted to the flame of a Meker-type burner when the sample is introduced into the flame under carefully standardized conditions. The intensity of colour is proportional to the sodium content. Air for

combustion draws the sample through a fine atomizing jet into a chamber where it is mixed with the combustible gas, the surplus liquid passing to drain. The resulting mixture is passed to the burner where it is ignited. The light emitted by this flame is passed through a filter transmitting light in the region of 590 nm and then to a photoelectric cell, the output of which is measured as a galvanometer deflection.

2.3 Interferences. The method is applicable to the determination of sodium concentrations within the range of the particular instrument used. In many cases, interferences by other elements may be ignored, provided that a filter as described in 2.2 is used. Potassium, calcium and magnesium are the most common interfering elements. If it is necessary to allow for these, the instrument shall be standardized using sodium solutions containing the appropriate amounts of the interfering elements.

Alternatively, interference may be avoided by 'spiking' or loading the sample with measured amounts of the element to be determined and extrapolating the estimated concentrations back to the point of zero loading. If calcium is the only significant interfering ion, the 'blanketing' technique described in 2.8 may be used.

2.4 Reagents

2.4.1 Reference water (low in sodium). Treat water by passing it through a mixed bed deionizing unit containing strong cation and anion resins. Prepare a fresh stock daily and store in a closed polyethylene container.

2.4.2 Standard sodium solutions

2.4.2.1 Stock solution. Dissolve 0.254 g of sodium chloride, dried to constant mass at 250 °C to 350 °C, in water and dilute to the mark in a 100 mL one-mark volumetric flask. 1 mL $\equiv$ 100 μ g sodium.

2.4.2.2 Working solution. Pipette 50.0 mL of the sodium stock solution (2.4.2.1) into a 1000 mL one-mark volumetric flask and dilute to the mark with the reference water (2.4.1). After thorough mixing, transfer immediately to a polyethylene container. 1 mL $\equiv$ 5 μ g sodium.

2.5 Apparatus

2.5.1 Flame photometer, with selective filter for sodium determination.

NOTE. Suitable narrow-band optical filters are commercially available and will minimize interference.

2.6 Calibration. Set up the instrument in accordance with the manufacturer's instructions. Set the zero using the reference water (2.4.1) and set the maximum deflection (D) using the sodium working solution (2.4.2.2). The sodium content of the reference water (2.4.1) can be ignored in comparison with the sodium content corresponding to the range of the instrument. Two intermediate settings should also be checked, using the sodium working solution (2.4.2.2) suitably diluted with the reference water (2.4.1), in order to confirm the calibration and the linearity of the scale. If a significant deviation from linearity is found, prepare a calibration graph. Check both the zero and the full scale readings periodically while the instrument is in use.

NOTE. The instrument should be protected from direct sunlight when in use.

2.7 Procedure. Replace the working solution by the sample and continue to atomize until a constant galvanometer deflection is obtained, taking care not to alter the burner settings. If the reading is off the scale, dilute the

sample with the reference water (2.4.1) until a suitable reading is obtained. Do not use the instrument beyond the maximum limit specified by the manufacturer.

2.8 Blanketing effect. If there is interference from calcium, this can be overcome by employing the 'blanketing' effect of aluminium. Dissolve 87.9 g of hydrated aluminium potassium sulphate, $\text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$, in water containing 50 mL of hydrochloric acid solution, $c(\text{HCl}) = 5 \text{ mol/L}^*$, and dilute to the mark in a 1000 mL one-mark volumetric flask. 1 mL $\equiv$ 5 mg aluminium. To a known volume of sample add a measured volume of the solution so that the ratio of aluminium to calcium is of the order 4:1. Atomize the solution and measure the emission intensity in the normal manner. Apply a correction for the change in volume of the sample on addition of the aluminium solution. Carry out a blank determination on the aluminium solution.

2.9 Calculation. The sodium content, expressed in milligrams of Na per litre, is given by:

$$5 \times \frac{d}{D}$$

where

d is the galvanometer deflection for the sample;

D is the galvanometer deflection for the sodium working solution (2.4.2.2).

If a calibration graph has been prepared, read the sodium concentration directly from the graph.

3. Flame spectrophotometric method

3.1 Range. Up to 0.1 mg of sodium (expressed as Na) per litre, the lower limit being governed by the instrument used, the laboratory conditions and the sodium content of the reference water (3.4.1).

3.2 Principle. A flame spectrophotometer measures photoelectrically the intensity of colour imparted to the flame of a Meker-type burner when the sample is introduced into the flame under carefully standardized conditions. The intensity of colour is proportional to the sodium content. The sample is atomized by compressed air and the mixture of air and sample in the form of fine mist is fed to the burner where it is mixed with combustible gas. The mixture is ignited. Radiation from the flame is passed through a monochromator to a photo-multiplier detector. After amplification the resulting signal may be displayed either as a galvanometer deflection or as a pen trace on a recorder.

3.3 Interferences. This method is applicable to the determination of trace amounts of sodium in industrial water having low solids content, such as steam condensate and deionized water. In such cases, interferences caused by other elements are usually negligible. If there is any doubt about the interference of other elements with the determination, the instrument shall be standardized using sodium solutions containing appropriate amounts of the interfering elements. Care shall be taken to avoid contamination of the flame by airborne dust. The air supply to the burner, as well as the combustion gas, shall always be filtered.

NOTE. Rubber gloves should be worn when handling samples of low sodium content.

*Previously 5N.

3.4 Reagents

3.4.1 Reference water (low in sodium). Circulate water several times through a mixed bed deionizing unit containing strong cation and anion resins, and finally through a unit containing strong cation resin only, all resins being in a freshly regenerated state. Prepare a fresh stock daily and store the water in a closed polyethylene or stainless steel container. By this means it is possible to prepare water containing as little as 2 µg of sodium per litre. Use the water to set an arbitrary zero for the instrument.

3.4.2 Standard sodium solutions

3.4.2.1 Stock solution. Dissolve 0.254 g of sodium chloride, dried to constant mass at 250 °C to 350 °C, in the reference water (3.4.1) and dilute to the mark in a 1000 mL one-mark volumetric flask. Pipette 250 mL of this solution into a 250 mL one-mark volumetric flask and dilute to the mark. Store in a polyethylene container. 1 mL ≡ 10 µg sodium.

3.4.2.2 Working solution. Take a clean polyethylene bottle which has been washed well with the reference water (3.4.1) until the washings show no increase in sodium content, and then well drained. Weigh into this 198 g of the reference water (3.4.1). Pipette into this exactly 2.0 mL of the sodium stock solution (3.4.2.1). A glass pipette may be used, but only if it has been washed as described for the bottle. 1 mL ≡ 0.1 µg sodium.

NOTE. The stated sodium content of the working solution ignores the sodium content of the reference water.

3.5 Apparatus

3.5.1 Flame spectrophotometer, with polyethylene or stainless steel vessels.

NOTE 1. The glass holders supplied with most flame spectrophotometers are not suitable for high purity waters and should be replaced by polyethylene or stainless steel vessels.

NOTE 2. The instrument should be protected from direct sunlight when in use and should be installed in an air-conditioned room separate from the general laboratory.

3.6 Calibration. Set up the flame spectrophotometer in accordance with the manufacturer's instructions. Use a propane or acetylene flame with the appropriate burner. The gas shall be filtered before passing to the burner. A filter (pore size 0.8 µm)* held on a sintered polyethylene disc in a suitable container shall be used. Air for combustion shall be supplied from a cylinder or from an air compressor and shall be filtered in the same manner as the gas.

Light the flame, allow a period of time for the instrument to stabilize and set the wavelength to about 590 nm. Atomize the sodium working solution (3.4.2.2) and rotate the wavelength control slowly and carefully in the vicinity of 590 nm. In this manner the setting for the maximum galvanometer reading may be ascertained. Check that this reading corresponds to the correct wavelength setting for sodium by atomizing the reference water (3.4.1). The galvanometer reading should decrease. Atomize the sodium working solution again and set the scale reading dial to 100 divisions. Replace the sodium working solution with the reference water (3.4.1) and continue to atomize until the burner is free from the sodium solution. Adjust the instrument to zero. Check the linearity of the instrument by carrying out at least two intermediate

calibrations using the sodium working solution (3.4.2.2), suitably diluted with the reference water (3.4.1). If a significant deviation from linearity is found, prepare a calibration graph.

3.7 Procedure. Replace the reference water with the sample and continue to atomize until a constant galvanometer deflection is obtained, taking care not to alter the burner settings.

3.8 Calculation. The sodium content, expressed in milligrams of Na per litre, is given by:

$$d \times \frac{0.1}{100}$$

where

d is the galvanometer deflection for the sample.

If a calibration graph has been prepared, read the sodium concentration directly from the graph.

4. Potentiometric method

4.1 Range. From 0.1 µg to 2300 µg of sodium (expressed as Na) per litre.

4.2 Sample. A minimum sample of 250 mL is required.

4.3 Principle. The potential of a sodium-responsive glass electrode immersed in the sample is measured and compared with the potential obtained when a standard solution is used. The measurement is made in a continuous flow-cell, with ammonia vapour introduced.

4.4 Interferences. Interferences are as shown in table 1.

Table 1. Potentiometric method: interference levels

Impurity	Concentration of impurity	Interference at sodium concentrations of	
		1.4 µg/L	24.4 µg/L
	µg/L	µg of Na/L	
Calcium (Ca ²⁺)	1000	2.3	5.0
Magnesium (Mg ²⁺)	1000		
Sulphate (SO ₄ ²⁻)	4000		
Chloride (Cl ⁻)	2000	0.5	—
Iron (Fe ³⁺)	2000		
Hydrazine	1000	1.6	3.0
Cyclohexylamine	1000		
Morpholine	1000		
<i>n</i> -octadecylamine	10 000	14.9	—

4.5 Reagents

4.5.1 Ammonia solution, *c*(NH₃) = 12 mol/L†. Dilute concentrated ammonia solution, 35 % (*m/m*), 2 + 1.

4.5.2 Water. Water of very low sodium content shall be used for preparing standard sodium solutions and for washing the apparatus. In many cases water with an extremely low sodium content (< 1 µg/L) may be obtained by condensing the high-pressure steam produced in a power station. Water normally containing less than 5 µg/L may be prepared conveniently in the laboratory by passing distilled water through a strongly functional mixed-bed ion-exchange unit, and storing in a clean polyethylene container.

*For information as to the availability of suitable filters, apply to Enquiry Section (London), British Standards Institution, enclosing stamped addressed envelope for reply.

†Previously 12N.