British Standard

BRITISH STANDARD

BS 2690-115: 1983

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APRIL 1989

Methods of testing

Water used in industry —

Part 115: Cyclohexylamine: spectrophotometric method

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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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

This British Standard, having been prepared under the direction of the Environment and Pollution Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 May 1983

The following BSI references relate to the work on this standard:
Committee reference EPC/37
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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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0 Introduction

BS 2690-115, BS 2690-116 and BS 2690-117 together supersede BS 2690-8:1969. This Part is a revision of clause **2**.

1 Scope

The method described is for the determination of cyclohexylamine in industrial waters by a spectrophotometric method.

2 Range

Up to 250 µg of cyclohexylamine in a test portion not exceeding 50 mL.

3 Principle

Cyclohexyla mine reacts with sodium 1,2-naphthoquinone-4-sulphonate to give a yellow compound which is extracted into chloroform and determined colorimetrically.

NOTE The method is susceptible to a number of experimental parameters. These include the temperature during, and the time allowed for, the reaction and the intensity of daylight while the solutions are being treated prior to setting aside in the dark. Great care should be taken to ensure that, as far as possible, standards and blanks are exposed to the same influences as samples.

4 Interferences

Significant interference by ammonia may result when some batches of naphthoquinone-sulphonate reagent are used. In such circumstances ammonia reacts similarly to cyclohexylamine and its effect is approximately proportional to its concentration. A preliminary check, as described in Appendix A, shall be conducted on each fresh batch of napthoquinone-sulphonate reagent to determine the effect of ammonia.

Under the conditions described in the method, the presence of 50 µg of octadecylamine, hydrazine, ferric (Fe^{3+}) and ferrous (Fe^{2+}) iron, zinc (Zn^{2+}), nickel (Ni^{2+}), copper (Cu^{2+}), aluminium (Al^{3+}), chromium (Cr^{3+}), potassium (K^{+}), calcium (Ca^{2+}) and magnesium (Mg^{2+}) and 500 µg of chloride (Cl^{-}), sulphate ($SO_4{}^{2-}$) and phosphate ($PO_4{}^{3-}$) individually causes no significant interference.

5 Sample collection

If the temperature of the water being sampled is above 40 °C, the sampling line shall contain a stainless steel cooling coil capable of reducing the temperature of the sample to below 40 °C. Collect the sample in a glass bottle, stopper, and, before analysis, cool the sample to within 5 °C of room temperature.

6 Reagents

6.1 *Ammonia-free water.* A satisfactory ammonia-free water can usually be obtained by shaking 5 L of water with 10 g of a strong cation exchange resin in the hydrogen form, or by passing water through a column of such resin.

It may also be prepared by distilling tap water in an all-glass apparatus after adding 1 mL of 0.5 g/L sulphuric acid solution.

6.2 *Naphthoquinone-sulphonate solution.* Dissolve 0.175 g of sodium 1,2-naphthoquinone-4-sulphonate in water and dilute with water to the mark in a 100 mL one-mark volumetric flask. Prepare freshly each day, and store in the dark when not in use. A new calibration graph shall be constructed for each batch of reagent.

6.3 *Phosphate buffer solution.* Add 35.0 g of anhydrous disodium hydrogen orthophosphate (Na_2HPO_4) to about 400 mL of water, and stir until all the solid is dissolved. Add 30.0 mL of 40 g/L sodium hydroxide solution. Transfer to a 500 mL one-mark volumetric flask and dilute to the mark with water. Stored in a polyethylene bottle, this solution is stable for at least 3 weeks.

6.4 *Chloroform*

WARNING. Chloroform is harmful by inhalation. Avoid breathing its vapour and contact with eyes. It is also a suspected carcinogen.

6.5 *Cyclohexylamine standard solutions*

6.5.1 *Cyclohexylamine stock solution.* Using a safety pipette introduce 5.00 mL of cyclohexylamine into a 1 000 mL one-mark volumetric flask and dilute to the mark with water. Store in a stoppered glass bottle for not longer than 3 weeks.

6.5.2 *Cyclohexylamine working solution.* Pipette 5.00 mL of the cyclohexylamine stock solution (**6.5.1**) into a 1 000 mL one-mark volumetric flask and dilute to the mark with water (1 mL = 21.7 µg of cyclohexylamine). Prepare this solution immediately before use.

7 Preparation of calibration graph

Add 0, 1.0, 2.5, 5.0, 7.5, 10.0 and 12.5 mL of the cyclohexylamine working solution (**6.5.2**) to a series of 100 mL conical flasks. This will correspond to 0, 21.7, 54.3, 108.5, 162.8, 217.0 and 271.3 µg of cyclohexylamine in a 100 mL flask. In each case make up to a total volume of 50.0 mL with the ammonia-free water (**6.1**). Treat the contents of each flask as follows.

Add 1.00 mL of the phosphate buffer solution (**6.3**) followed immediately by 10.0 mL of the naphthoquinone-sulphonate solution (**6.2**). Allow the mixture to stand for 2 h in the dark, and then transfer quantitatively to a 250 mL separating funnel, rinsing the flask with 5 mL of the ammonia-free water (**6.1**). Add 20.0 mL of the chloroform (**6.4**) to the separating funnel, swirl the contents of the funnel gently for a few seconds, cautiously releasing the pressure generated, and then shake the funnel vigorously for 1 min. Set the funnel aside in the dark (or otherwise shield from light) to allow phase separation and, when complete, rinse the bore of the separating funnel stopcock by running 3 mL to 5 mL of the chloroform extract to waste. Then pass a further 2 mL to 3 mL of the chloroform extract to waste through a fast, medium grade filter paper[1)] held in a small glass filter funnel, and finally a further portion of the extract through the filter into a dry 10 mm spectrophotometer cell. Measure the absorbance of the solution at a known temperature between 20 °C and 25 °C in a spectrophotometer at the wavelength corresponding to maximum absorption (approximately 449 nm, but the exact wavelength shall be checked for each spectrophotometer), using 10 mm cells. Use chloroform in the compensating cell.

Deduct the reading for the blank from those for the standard solutions and plot a calibration graph of absorbance against the number of micrograms of cyclohexylamine.

The absorbance given by 250 μg of cyclohexylamine in the total volume of test solution is approximately 0.5.

NOTE The graph should be linear to at least 250 μg.

8 Procedure

Measure a suitable volume of the sample (containing less than 250 μg of cyclohexylamine) into a 100 mL conical flask and, if necessary, dilute to 50 mL with the ammonia-free water (**6.1**). Add 50 mL of the ammonia-free water to a second flask to serve as a blank. Treat the contents of each flask as follows.

Add 1.00 mL of the phosphate buffer solution (**6.3**) followed immediately by 10.0 mL of the naphthoquinone-sulphonate solution (**6.2**). Allow the mixture to stand for 2 h in the dark, and then transfer quantitatively to a 250 mL separating funnel, rinsing the flask with 5 mL of the ammonia-free water (**6.1**). Add 20.0 mL of the chloroform (**6.4**) to the separating funnel, swirl the contents of the funnel gently for a few seconds, cautiously releasing the pressure generated, and then shake it vigorously for 1 min. Set the funnel aside in the dark (or otherwise shield from light) to allow phase separation and, when complete, rinse the bore of the separating funnel stopcock by running 3 mL to 5 mL of the chloroform extract to waste. Then pass a further 2 mL to 3 mL of the chloroform extract to waste through a fast, medium grade filter paper[3)] held in a small glass filter funnel, and finally a further portion of the extract through the filter into a dry 10 mm spectrophotometer cell.

Measure the absorbances of the sample and blank at a temperature within 1 °C of that at which the calibration graph was prepared, using the wavelength used for the calibration graph and 10 mm cells. Use chloroform in the compensating cell.

9 Calculation

Deduct the reading obtained for the blank from that for the sample and read off the cyclohexylamine content, in micrograms, from the calibration graph. The concentration, in milligrams per litre, of cyclohexylamine is given by

$$\frac{m}{V}$$

where

m is the mass of cyclohexylamine from the calibration graph (in μg).

V is the volume of the sample (in mL).

[1)] Whatman No. 41 paper is suitable.

Appendix A Interference by ammonia

Test each batch of solid naphthoquinone-sulphonate reagent for the effect of ammonia, as follows.

Place 2.5 mL of the cyclohexylamine working solution (**6.5.2**) in each of four 100 mL flasks. Make additions of standard ammonium chloride solution such that, when made up to a total volume of 50 mL with the ammonia-free water (**6.1**), the solutions contain 0, 0.1, 0.2 and 0.5 mg of ammonia per litre. Determine the cyclohexylamine content of each solution as described in clause **8**; it should remain constant at 54.3 μg. If a significant error is shown up by this test, determine the ammonia concentration in the sample by the method described in BS 2690-7, and carry out the following procedure.

To a series of standard cyclohexylamine solutions, prepared as described in clause **7**, make additions of a standard solution of ammonium chloride such that the solutions contain the determined amount of ammonia. Determine the apparent cyclohexylamine content of each flask, as described in clause **8**, and calculate the effect of ammonia. From these results the true cyclohexylamine content may be determined.

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Publications referred to

BS 2690, *Methods of testing water used in industry.*
BS 2690-7, *Nitrite, nitrate and ammonia (free, saline and albuminoid)*[2)].
BS 2690-116, *Morpholine: spectrophotometric method*[3)].
BS 2690-117, *Long-chain fatty amines: spectrophotometric method*[3)].

[2)] Currently being revised. Will be published as Part 114.
[3)] Referred to in the introduction only.

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London
W4 4AL