British Standard

BRITISH STANDARD

BS 2690-117: 1983

Methods of testing

Water used in industry —

Part 117: Long-chain fatty amines: 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 and 2, 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 **4**.

1 Scope

The method described is for the determination of long-chain fatty amines in industrial waters by a spectrophotometric method.

2 Range

From 5 µg to 200 µg of long-chain fatty amine, expressed as octadecylamine, in a test portion not exceeding 100 mL.

3 Principle

The primary, secondary and tertiary forms of long-chain fatty amines react with methyl orange at pH 3 to 4 to form yellow complexes which are extracted into dichloromethane and determined colorimetrically.

The calibration graph is prepared with the fatty amine that is added to the steam plant under investigation. As octadecylamine is the most commonly added fatty amine, it is used in the following method.

4 Interferences

Cyclohexylamine, morpholine and ammonia do not interfere. Detergents interfere seriously and shall not be used for cleaning any glassware used in the test.

5 Reagents

5.1 *Buffer solution*. Dissolve 125 g of potassium chloride and 70 g of sodium acetate trihydrate in 500 mL of water.

Add 300 mL of glacial acetic acid and dilute to 1 L with water. The pH value is 3.4.

5.2 *Dichloromethane*

WARNING. Dichloromethane is harmful by inhalation. Avoid breathing its vapour and contact with skin and eyes.

5.3 *Propan-2-ol*

5.4 *Methyl orange solution*. Dissolve 100 mg of methyl orange in 200 mL of water and add 50 mL of industrial methylated spirits (see **3.8** of BS 2690-100:1978).

5.5 *Octadecylamine standard solutions*

5.5.1 *Octadecylamine stock solution*. Weigh 0.500 g of octadecylamine into a 250 mL beaker, add 50 mL of glacial acetic acid and stir to dissolve. Transfer the solution to a 500 mL one-mark volumetric flask and dilute to the mark with water. Mix well.

5.5.2 *Octadecylamine working solution*. Dilute 10.0 mL of the octadecylamine stock solution (**5.5.1**) with water to the mark in a 1 000 mL one-mark volumetric flask (1 mL ≡ 10 µg of octadecylamine). Prepare this solution fresh before each calibration.

6 Treatment of apparatus

Long-chain fatty amines tend to be absorbed on to glass surfaces and therefore all glassware apart from the optical cells shall be silicone-coated. This can be done by first cleaning the glassware with chromic acid and then rinsing the clean and dry surface of the glassware with a proprietary silicone emulsion, in accordance with the manufacturer's instructions. The coating will usually last for about 6 months and the glassware shall be re-treated when a normal water meniscus appears.

7 Sampling

If the temperature of the water being sampled is above 30 °C, the sampling line shall contain a small, permanently fixed, stainless steel cooling coil capable of reducing the temperature of the sample to about 25 °C to 30 °C. The sampling line shall be kept as short as possible and the sample allowed to run to waste for at least 30 min before collection. Run the sample into a stoppered 250 mL silicone-coated glass bottle and analyse immediately.

8 Preparation of calibration graph

Add 0, 1.0, 2.0, 5.0, 10.0, 15.0 and 20.0 mL of the octadecylamine working solution (**5.5.2**) to a series of 150 mL separating funnels. This will correspond to 0, 10, 20, 50, 100, 150 and 200 µg of octadecylamine in a separating funnel. Treat the contents of each funnel as follows.

Dilute to 100 mL with water, add 4.0 mL of the buffer solution (**5.1**), 2.0 mL of the methyl orange solution (**5.4**) and 20.0 mL of the dichloromethane (**5.2**), stopper and shake for 5 min. Invert the funnel and dry the stem with a spill of filter paper extending to the open tap. Close the stopcock and allow the funnel to stand upright for 3 min. Run the dichloromethane layer into a dry 50 mL separating funnel. Add 0.5 mL of the propan-2-ol (**5.3**) to the separated dichloromethane layer to remove any turbidity and swirl gently. Run off the dichloromethane into a 10 mm cell. Immediately measure the absorbance of the dichloromethane solution at a known temperature between 20 °C and 25 °C in a spectrophotometer at the wavelength corresponding to maximum absorption (approximately 430 nm, but the exact wavelength shall be checked for each spectrophotometer), using 10 mm cells. Use dichloromethane 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 octadecylamine.

The absorbance given by 200 µg of octadecylamine in the total volume of test solution is approximately 1.2.

9 Procedure

Measure a suitable volume of the sample (containing less than 200 µg of octadecylamine) into a 150 mL separating funnel and, if necessary, dilute to 100 mL with water. Add 100 mL of water to a second separating funnel to act as a blank. Treat the contents of each funnel as follows.

To each funnel add 4.0 mL of the buffer solution (**5.1**), 2.0 mL of the methyl orange solution (**5.4**) and 20.0 mL of the dichloromethane (**5.2**), stopper and shake for 5 min. Invert the funnel and dry the stem with a spill of filter paper extending to the open tap. Close the tap and allow the funnel to stand upright for 3 min. Run the dichloromethane layer into a dry 50 mL separating funnel. Add 0.5 mL of the propan-2-ol (**5.3**) to the separated dichloromethane layer to remove any turbidity and swirl gently. Run off the dichloromethane into a 10 mm cell. Immediately measure the absorbance of the dichloromethane solution at the wavelength used for the calibration graph and at a temperature within 1 °C of that at which the calibration graph was prepared in the spectrophotometer. Use dichloromethane in the compensating cell.

10 Calculation

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

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

where

m is the mass of octadecylamine from the calibration graph (in µg);

V is the volume of sample (in mL).

Publications referred to

BS 2690, *Methods of testing water used in industry.*
BS 2690-115, *Cyclohexylamine: spectrophotometric method*[1)].
BS 2690-116, *Morpholine: spectrophotometric method*[1)].

[1)] Referred to in the introduction only.

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