

AMINES METHOD 2

Using Methyl Orange

INTRODUCTION

This test is based on a method developed by Silverstein¹ for the colorimetric determination of traces of amines in water. It is very simple and also has the advantage of being more sensitive than some alternative methods^{2,3} it is suitable for the determination of octadecylamine, which is used as a corrosion inhibitor in water treatment.

PRINCIPLE OF THE METHOD

Certain primary, secondary and tertiary amines react with Methyl Orange at a pH of 3-4 to form a yellow complex. This complex is soluble in organic liquids, such as methyl chloroform, which can be used to extract the complex from the water. The colour intensity of the complex solution is a direct measure of the total amine concentration, and this is determined by comparing the colour with a series of Lovibond permanent colour glass standards.

REAGENTS REQUIRED

1. **Methyl Orange Indicator.** Dissolve 0.04g. of Methyl Orange in 100ml. of hot deionised water, or in 20ml. of propan-2-ol followed by 80ml. of deionised water. Cool, and filter if necessary.
2. **Amine Buffer Solution.** Dissolve 125g. of potassium chloride (KCl) and 70g. of sodium acetate trihydrate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) in 500ml. of deionised water. Add 300ml. of glacial acetic acid (CH_3COOH) and make up to 1 litre with deionised water.
3. **1, 1, 1-Trichlorethane.** (methyl chloroform) (CCl_3CH_3).
4. Reagents 2 and 3 should be of analytical reagent grade.

THE STANDARD LOVIBOND COMPARATOR DISC 3/64

This disc covers the range 0 - 2mg./l. of octadecylamine in 5 steps as follows:-
0, 0.25, 0.5, 1.0, 2.0mg./l.

METHOD

If the water to be sampled is above ambient temperature the sample should be collected through a small stainless-steel cooler.

1. Run the sample into one of two stoppered 50ml. cylinders which have been silicone treated, (Note 2). When a representative sample has been collected, reject excess sample down to the 50ml. mark.
2. To the other 50ml. cylinder add 50ml. deionised water.
3. To both cylinders add 2ml. of amine buffer solution (reagent 2), 1ml. of methyl orange indicator (reagent 1) and 10ml. of 1, 1, 1-trichloromethane (reagent 3).
4. Shake both cylinders for 5 minutes.
5. Allow to stand for 3 minutes and then carefully decant off as much as possible of the upper aqueous layer from both cylinders.

6. Transfer the remaining liquid in each cylinder into a 13.5mm. /10ml. moulded cell, making sure the 1, 1, 1-trichloromethane layer falls to the bottom of the tube and ignoring any aqueous solution which may overflow.
7. Dry the outside of the cells with a clean cloth. Place the cell containing the treated sample in the right hand compartment and that containing the deionised water blank in the left hand compartment of the Comparator.
8. Match against the disc by holding the Comparator against a standard source of white light, such as the Lovibond Daylight 2000 Unit or, failing this, North daylight (not fluorescent lighting). Rotate the disc until a colour match is obtained.
9. The figure displayed in the bottom right hand aperture of the Comparator is the Amine concentration as octadecylamine in mg. /l.

NOTES

1. The adaption of this test to the comparator is due to the "Alfloc" Water Treatment Service of I.C.I. Ltd., to whom acknowledgement is made.
2. The 50ml. stoppered cylinders can be silicone coated by completely filling the cylinder with a 2% aqueous solution of I.C.I. Silicone Emulsion M.4600 and stoppering the cylinder. After 15 minutes drain out the solution and dry the cylinders at a temperature of about 180°C. The coating will last about 6 months. Retreatment is necessary when a normal water meniscus reappears.

REFERENCES

1. R.M. Silverstein, *Anal. Chem.*, 1963, **35**, 154
2. K.B. Coates, *Corrosion Tech*, 1960, **7**, 46
3. A.S. Pearce, *Chem. and Ind.*, 1961, 825
4. Brit. Standards Inst., B.S. 4445: 1969, "*Schedule of tests for gasification and reforming plants using hydrocarbon feedstocks*", London, 1969.

REVISION HISTORY

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