Lovibond®

CHLORINE METHOD 1

Using Neutral *o*-Tolidine

PRINCIPLE OF THE METHOD

The procedure consists in the treatment of the chlorine-containing solution with neutral *o*-tolidine, in the presence of stabilising agents, for the production of a blue coloration. Reaction with chloramine is activated by the addition of potassium iodide. The only interfering substance is manganese, and a method is given for making due allowance for this. Unlike the acid o-tolidine method, nitrites in this method are without effect. **This method is no longer used in the U.K. as *o*-tolidine is a known carcinogen**.

REAGENTS REQUIRED

1. **Buffer Stabilising Solution**. Dissolve 4g. sodium hexametaphosphate ("Calgon") in about 80ml. deionised water, add 0.4ml. "Teepol 610" (Shell Chemicals Limited), mix, and make up to 100ml. with deionised water.

   Note - If subjected to low temperatures, this solution may become turbid, but may be clarified by warming. Cool afterwards to room temperature for use. Alternatively, the inclusion of 10% v/v acetone in the reagent will inhibit precipitation and not influence the reaction.

2. **Neutral o-Tolidine Solution 0.1%**. Place 1g. *o*-tolidine (specially purified Reagent quality) in a mortar with 5ml. of 20% v/v HCI. Grind to a thin paste. Dissolve in about 500ml. deionised water, and then dilute with more deionised water to 1 litre. Store in an amber bottle. The water must be chlorine free.
   ***o*-tolidine is carcinogenic, and must be handled with all due precautions.**

3. **Potassium Iodide (KI)**. Crystals (analytical reagent grade).

THE STANDARD LOVIBOND COMPARATOR DISCS 3/25A AND 3/25B

3/25A covers the range 0.1 to 1.0mg./litre chlorine in steps of 0.1, omitting 0.9. The disc is used with 40mm. cells calibrated at 20ml.

3/25B covers the range 0.25 to 5.0mg./litre chlorine in steps 0.25, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0. The disc is used with 13.5mm./10ml. cells.

The chlorine values obtained by the use of these discs are identical with those obtained by the F.A.S. titration method of Palin.

METHOD

Disc 3/25A

1. Measure 1.0ml. of reagent 1 into the 40mm. cell, followed by 1.0ml. of reagent 2.

2. Add the water sample up to 20ml, mix and immediately place in the right hand compartment of the comparator, so that it comes behind the centre of the disc.

3. Place a similar cell, containing the water sample, only into the left hand compartment.

4. Hold the comparator facing a standard source of white light such as the Lovibond Daylight 2000 unit, or failing this North daylight, and rotate the disc until the nearest match is obtained.

5. The figure displayed in the indicator window is the concentration of free chlorine present in the sample in mg. /l. The matching must be made immediately after mixing the solutions.

Disc 3/25B

1. Measure 0.5ml. of reagent 1 into the 13.5mm. cell, followed by 0.5ml. of reagent 2.

2. Add the water sample up to the 10ml. mark, mix, place at once in the right hand compartment of the comparator, so that it comes behind the centre of the disc.

3. In the left hand compartment place a similar cell filled with the water sample. Match at once as above. The figure displayed is the concentration, in mg./l., of free chlorine as before.

DETERMINATION OF CHLORAMINES BY THESE DISCS

Monochloramine may be determined as follows: - continue the above test by adding a small crystal of potassium iodide (analytical reagent grade) to the right hand cell and mix well. Evaluate the colour as previously, and deduct from this reading first obtained as above. The result represents monochloramine in mg. /litre chlorine.

Dichloramine is not usually present in significant quantities if the pH of the water is above 7. If it is required to estimate it, proceed as follows: - To the solution which has already been treated with reagents 1 and 2 and potassium iodide (see above) add 0.5ml.* of 5% w/v sulphuric acid (analytical reagent grade) and mix. Then add 0.5ml.* of 5% w/v sodium bicarbonate solution and again mix.

Evaluate this colour as previously, multiply the result by 1.1 to allow for the volume change, and subtract from this reading the result previously obtained with potassium iodide. This reading is the mg./l. chlorine as dichloramine.

Trichloramine. If nitrogen trichloride is present in the sample, which would be indicated by its distinct odour, it should be removed by extraction with one tenth its volume of carbon tetrachloride (analytical reagent grade).

NOTES

1. The only interfering substance likely to be present in water is oxidized manganese. Its effect can be allowed for by developing the manganese colour in the "blank" as follows: - Instead of using a "blank" cell, containing the sample only, in the left hand compartment place 0.5ml.* of reagent 1 in a clean cell, add a crystal of potassium iodide and 1 drop* of a 0.5% solution of sodium arsenite ($NaAsO_2$). Make up to the 10 ml* mark with sample, and mix. Add 0.5ml.* of reagent 2 and mix. Place this cell in the left hand compartment as a blank: thus the colour due to manganese will have developed equally in both fields, and cancels out.
   *For 40mm. cells, double these figures.

2. All glassware used must be very thoroughly rinsed after making a test; this is particularly important, as only a trace of potassium iodide will cause a chloramine colour to develop.
   The colour produced is reasonably stable at temperatures up to 80°F. Above this temperature, fading will occur and increase proportionately rapidly, but if the reading is taken immediately after mixing the reagents and sample, no significant error will be introduced.

3. If the solution becomes turbid on adding reagent 1 and 2, change the order to
   (a) Reagent 2 (b) Sample (c) Reagent 1

REVISION HISTORY

Date	Change Note	Issue
02/01/03	36/460	2
18/04/05	CA243	3
12/01/06	JC14	3