

BRITISH STANDARD 1427 : 1962

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**ROUTINE CONTROL
METHODS OF TESTING
WATER USED
IN INDUSTRY**

BRITISH STANDARDS INSTITUTION

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B.S. 1427 : 1962

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B.S. 1427 : 1962

THIS BRITISH STANDARD, having been endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 5th September, 1962.

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First revision, September, 1962.

The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

This standard makes reference to the following British Standards:

- B.S. 593. Laboratory thermometers.
- B.S. 604. Graduated measuring cylinders.
- B.S. 612. Nessler cylinders.
- B.S. 700. Graduated pipettes and one-mark cylindrical pipettes.
- B.S. 718. Density hydrometers and specific gravity hydrometers.
- B.S. 846. Burettes and bulb burettes.
- B.S. 1328. Methods of sampling water used in industry.
- B.S. 1583. One-mark pipettes.
- B.S. 1792. One-mark volumetric flasks.
- B.S. 2586. Glass electrodes.
- B.S. 2690. Methods of testing water used in industry.
- B.S. 3145. Laboratory potentiometric pH meters.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following B.S.I. references relate to the work on this standard:
Committee references C/37, C/37/1, C/37/1/1.
Draft for comment AB(C)5786

CONTENTS

	Page
Co-operating organizations	4
Foreword	5
Scope	5
Notes applicable to all tests	5

METHODS OF TEST

1. Alkalinities	7
2. Aluminium	9
3. Ammonia (free and saline)	11
4. Calcium	12
5. Chloride	13
6. Residual chlorine (free and total)	14
7. Colour	15
8. Dissolved oxygen	15
9. Dissolved solids	20
10. Electrical conductivity	21
11. Free carbon dioxide	22
12. Hardness	23
13. Hydrazine	25
14. Iron	26
15. Magnesium	27
16. Nitrite	27
17. pH value	29
18. Phosphates	34
19. Silica	35
20. Specific gravity	37
21. Sulphate	39
22. Sulphite	40
23. Fluoride	41

APPENDICES

A. List of reagents	42
B. Basic apparatus	53

B.S. 1427 : 1962

CO-OPERATING ORGANIZATIONS

The Technical Committee which has been working under the supervision of the Chemical Divisional Council is responsible for the preparation of this British Standard and consists of representatives from the following Government departments and scientific and industrial organizations:

Admiralty

Association of British Chemical Manufacturers

Association of Shell Boiler Makers

British Iron & Steel Federation

British Launderers Research Association

British Railways, The British Transport Commission

D.S.I.R.—Laboratory of the Government Chemist

Electricity Council, the Generating Board and the Area

Boards in England and Wales

Gas Council

Institute of Fuel

Institute of Marine Engineers

Institute of Petroleum

Institution of Chemical Engineers

Institution of Gas Engineers

Institution of Mechanical Engineers

Institution of Municipal Engineers

Lloyd's Register of Shipping

London County Council

Ministry of Labour (Factory Inspectorate)

National Coal Board

National Physical Laboratory (D.S.I.R.)

Royal Institute of Chemistry

Society for Analytical Chemistry

Society of Laundry Engineers & Allied Trades Limited

War Office

Water Tube Boilermakers' Association

Water treatment specialists

BRITISH STANDARD

ROUTINE CONTROL METHODS OF TESTING WATER USED IN INDUSTRY

FOREWORD

These tests have been drawn up as part of a series of British Standards dealing with water, the preparation of which was authorized by the Chemical Divisional Council. The purpose of this standard is to ensure a degree of uniformity in the methods of testing and in the manner of expressing the results.

This standard covers only the tests for which no laboratory is required and the apparatus required is that which can be conveniently set up and used near the point of sampling. The tests are of a comparatively simple nature and are suitable for use by unqualified personnel after simple training. They are not necessarily intended for use in cases of arbitration or for purposes of exhaustive investigation. Additional tests, many of which require a laboratory, can be found in B.S. 2690*. Details of reagents required are given in Appendix A along with reference numbers which are used for cross-reference through all the methods. Most of these cannot be made up without a laboratory, but are commercially available. Details of basic apparatus required are given in Appendix B.

The standard originally published in 1949 only covered waters used in steam generation. The present revision, which includes the use of calibrated disks and improvements in the layout, has made the methods applicable to industrial waters in general.

The residual chlorine test has been amended to avoid the use of o-tolidine, and the nitrate test to avoid the use of 1-naphthylamine, since the carcinogenic properties of these reagents have been well established, and their use is governed by the Carcinogenic Substances Regulations 1967. (Statutory Instrument 1967 No. 879.)

*As amended
Feb. 1968*

SCOPE

This British Standard which is complementary to B.S. 2690 describes a selection of routine control methods of testing water used in industry employing portable equipment but it neither indicates which test shall be used in any given case nor implies the necessity to carry out all of them, since the tests required will depend upon local conditions.

* B.S. 2690, 'Methods of testing water used in industry'.

B.S. 1427 : 1962

NOTES APPLICABLE TO ALL TESTS

a. All reagents used are to be of recognized analytical reagent quality. Distilled water or deionized water* is to be used wherever water is required. The distilled water is to have a conductivity of less than 5 micromhos per centimetre at 20°C.

b. It is recommended that wherever possible apparatus in accordance with British Standards should be used. Appendix B gives a list of the appropriate British Standards at present available.

c. In order to achieve portability of apparatus, colour comparison by means of a disk† is given in addition to a standard series method. The accuracy of the disks may be checked against the standard series solutions of the appropriate test. It is pointed out that the disks are intended for use with North or diffused daylight and any other illumination may lead to errors.

d. It is important that a representative sample be obtained for analysis, otherwise the results of the tests will be valueless. Reference should be made to B.S. 1328, 'Methods of sampling water used in industry'.

e. Except where otherwise stated the test is to be made on clear filtered portions of the sample.

f. For tests of the unfiltered sample, as much as possible of any suspended matter is to be allowed to settle before removal of the portion required.

g. All apparatus is to be cleaned and washed with water before use and any volumetric glassware is to be rinsed with a small portion of the liquid to be measured.

h. Some of the reagents used are corrosive and some of the waters tested may be bacteriologically unsafe; in the interests of safety, therefore, pipettes shall not be filled by sucking with the mouth. The necessary suction may be obtained by a rubber ball type pipette filler, or a glass plunger.

j. Methods of preparing the indicators and standard volumetric solutions are described in Appendix A. Most of these solutions are available commercially and all apparatus is available through normal laboratory suppliers; if difficulties are encountered applications should be made to the British Standards Institution, 2 Park Street, London, W.1.

k. Industrial methylated spirits is specified in various tests. It is pointed out that the use of this material is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230)‡.

l. For each test a range has been given, quoted as an absolute amount in milligrammes (mg) or microgrammes (μg or γ). This range has also been expressed as a concentration in parts per million by weight (p.p.m.) based upon the volume of sample taken for the test.

* Water treated with the correct grades of exchange resins will have a conductivity of less than 1 micromho per centimetre at 20°C.

† Suitable disks and apparatus are obtainable from The Tintometer Ltd., Salisbury.

‡ Ethanol may be substituted for industrial methylated spirits if desired.

The range for the concentration may be extended by taking a smaller volume of sample and diluting to the specified volume.

m. Except where otherwise stated, measurement of the portion of the sample required for a test is to be carried out by means of a measuring cylinder.

Except where otherwise stated, measurement of reagents is to be carried out by means of a burette or a one-mark pipette.

METHODS OF TEST

1. ALKALINITIES

Range: 0–50 mg in terms of CaCO_3
0–500 p.p.m. in terms of CaCO_3

The alkalinity of a water is determined by titration with a standard solution of an acid to end points with pH values at 8.3 and 4.5. The end points are usually detected with phenolphthalein (pH 8.3) and methyl orange (pH 4.5) but if the water is highly coloured, indicators are not suitable and the potentiometric method is advisable.

Organic matter, which if present usually imparts a brown colour to the sample, will interfere with the indicator method of determining the alkalinity, especially the methyl orange alkalinity.

In the case of waters containing organic matter and hydroxide the alkalinity to methyl orange is unreliable and therefore the alkalinity to phenolphthalein and the alkalinity to phenolphthalein after addition of barium chloride should be determined. The latter test is a measure of the caustic alkalinity and in conjunction with the alkalinity to phenolphthalein test will allow the carbonate alkalinity to be calculated. The following table may be used as an indication of the amount of bicarbonate, carbonate and hydroxide present in a water which has not been subjected to phosphate treatment. The results are expressed in terms of p.p.m. CaCO_3 .

Result of titration	Hydroxide	Carbonate	Bicarbonate	Total alkalinity
$P = \text{Nil}$	Nil	Nil	M	M
$2P < M$	Nil	$2P$	$M - 2P$	M
$2P = M$	Nil	$2P$	Nil	M
$2P > M$	$2P - M$	$2(M - P)$	Nil	M
$P = M$	M	Nil	Nil	M
$P > P(\text{BaCl}_2)$	$P(\text{BaCl}_2)$	$2[P - P(\text{BaCl}_2)]$	Nil	$2P - P(\text{BaCl}_2)$
$P(\text{BaCl}_2) = \text{Nil}$	Nil	$2P$	*	*

* No correlation.

P = Alkalinity to phenolphthalein or to pH 8.3.

M = Alkalinity to methyl orange or to pH 4.5.

$P(\text{BaCl}_2)$ = Alkalinity to phenolphthalein after the addition of barium chloride or to pH 8.3 after the addition of barium chloride.

B.S. 1427 : 1962

If a boiler water has been subjected to phosphate treatment and the phosphate is present as the trisodium salt, part of the alkalinity to phenolphthalein will be due to the alkalinity combined with the phosphate (PO_4). If the PO_4 content is expressed in p.p.m. as such (see Phosphate) the effect on the P reading will be equal to $\text{PO}_4/2$ and this must be subtracted from P reading before using the table given above. The alkalinity to phenolphthalein after addition of barium chloride is not affected by the presence of the phosphate.

Special apparatus (potentiometric method only)

A pH meter with electrode assembly

A suitable titration assembly having a stirring mechanism and a burette.

Reagents

<i>Barium chloride</i> , 10 per cent w/v.	Reagent No. 15.
<i>Methyl orange indicator solution</i> .	Reagent No. 44.
<i>Phenolphthalein indicator solution</i> .	Reagent No. 50.
<i>Sodium sulphate crystals</i> .	Reagent No. 76.
<i>Sulphuric acid</i> , 0.02N.	Reagent No. 88.

Procedure. a. Indicator method.

(i) Alkalinity to phenolphthalein (P)

Measure 100 ml of the sample and transfer to a porcelain basin. Add 1 ml of phenolphthalein indicator and if the sample turns pink run in the 0.02N acid, slowly stirring at the same time with a glass rod until the colour just disappears. Record the number of millilitres of 0.02N acid used.

Alkalinity to phenolphthalein (P) =

$$\frac{\text{ml of 0.02N acid} \times 1000}{\text{ml of sample}} \text{ p.p.m. in terms of CaCO}_3$$

(ii) Alkalinity to methyl orange (M)

To the solution treated as described above, add 3 drops of methyl orange indicator. Titrate slowly with 0.02N acid, stirring continuously until the colour shows the first change from yellow to orange. Record the total number of millilitres of acid used, i.e. including those used in the titration to phenolphthalein.

Alkalinity to methyl orange (M) =

$$\frac{\text{ml of 0.02N acid} \times 1000}{\text{ml of sample}} \text{ p.p.m. in terms of CaCO}_3$$

NOTE. Any difficulty experienced in detecting the end point of the methyl orange titration may be reduced by placing a second 100 ml sample of the water with the same amount of indicator in a similar container alongside that in which the titration is being carried out.

(iii) *Alkalinity to phenolphthalein after the addition of barium chloride*
[P(BaCl₂)]

Measure 100 ml of the sample and transfer to a porcelain basin. Add 1 ml of phenolphthalein indicator followed by a crystal of sodium sulphate and 10 ml of barium chloride. Stir well for two minutes and then titrate with 0.02N acid until the pink colour just disappears. Disregard any reappearance of the pink colour. Record the number of millilitres of 0.02N acid used.

Alkalinity to phenolphthalein after addition of barium chloride [P(BaCl₂)] =

$$\frac{\text{ml } 0.02\text{N acid} \times 1000}{\text{ml of sample}} \text{ p.p.m. in terms of CaCO}_3$$

NOTE. If the sample is so highly coloured that the indicator end points cannot be detected and a pH meter is not available one of the following procedures can be used to obtain an approximate result:

- A. Dilute the sample with water, until an end point can be detected.
- B. Into the depressions on a porcelain spotting tile, put drops of the indicator used in the test. Titrate in the ordinary way, stopping the flow of 0.02N acid at 1 ml intervals, and transfer a drop of the titrated sample by means of the stirring rod to one of the indicator test spots. A stage will be reached when one of the spots on the tile shows a distinct change in colour. Now repeat the test, running the acid down to within 2 ml of the first approximate end point, stirring continuously, then complete the test adding the acid in 0.2 ml stages.

b. Potentiometric method. The procedure is exactly the same as for the indicator method except that the addition of the indicator is omitted and the electrode assembly is immersed in the sample. The burette is read when the pH meter reads 8.3 and 4.5.

2. ALUMINIUM

Range: 0.004 mg Al
0.4 p.p.m. Al

In this method the colour changes of the prepared sample are compared with colours of standard solutions. The final colours are from yellow through brown to violet.

If the sample contains more than 0.2 p.p.m. of manganese (as Mn) or more than 0.4 p.p.m. of iron (as Fe), interference will occur and the aluminium content of the water should be determined by the method given in B.S. 2690*.

With more than 0.2 p.p.m. of manganese the colour of the haematoxylin is bleached within two minutes of its addition, i.e. before the addition of the acetic acid, and a golden yellow colour develops which is similar to that produced after adding acetic acid to the standard containing no aluminium.

* B.S. 2690, 'Methods of testing water used in industry'.

B.S. 1427 : 1962

With more than 0.4 p.p.m. of iron 'off' shades usually greenish in colour are obtained.

Reagents

<i>Acetic acid</i> , 30 per cent v/v.	Reagent No. 2.
<i>Aluminium standard solution</i> .	Reagent No. 5.
<i>Ammonium carbonate solution</i> .	Reagent No. 11.
<i>Haematoxylin solution</i> .	Reagent No. 31.

Procedure. Prepare a working solution of aluminium by diluting 10 ml of the standard aluminium solution to 100 ml with water in a one-mark graduated flask.

$$1 \text{ ml} \equiv 5 \mu\text{g Al}$$

a. Standard series method. Into nine numbered Nessler cylinders, measure 0, 1, 2, 3, 4, 5, 6, 7 and 8 ml of the working solution and add sufficient water to each cylinder to make the volume up to 50 ml. Into a further three numbered Nessler cylinders pipette 10, 25 and 50 ml of the sample and where necessary make up the volumes to 50 ml with water.

Add to each cylinder in turn by means of pipettes 2 ml of the ammonium carbonate solution, mix, then add 1 ml of the haematoxylin solution and mix. Allow to stand for exactly ten minutes and then add 2 ml of the acetic acid solution to each cylinder and mix. Allow to stand for exactly five minutes after which compare the colours of the samples with the standards.

$$\text{Aluminium} = \frac{\text{ml of working solution} \times 5}{\text{ml of sample taken}} \text{ p.p.m. Al}$$

b. Calibrated disk method. Into three numbered Nessler cylinders pipette 10, 25 and 50 ml of the sample and make up the volumes to 50 ml with water. These cylinders will serve as blanks.

Into a further three numbered Nessler cylinders pipette 10, 25 and 50 ml of the sample and where necessary make up the volume to 50 ml with water. Treat these three cylinders as described in *a*. When the final colours have developed pour the contents of the cylinders in turn into a 13.5 mm glass cell and place in the appropriate compartment of a comparator and the corresponding blank into a 13.5 mm cell and place in the other compartment. Match the colours using the appropriate disk.

$$\text{Aluminium} = \frac{\text{Disk reading in p.p.m.} \times 50}{\text{ml of sample}} \text{ p.p.m. Al}$$

3. AMMONIA (FREE AND SALINE)

Range: 0–100 $\mu\text{g NH}_3$

0–2 p.p.m. NH_3

This method gives the total of free and saline ammonia in a water and should be carried out in an ammonia-free atmosphere. Where the ammonia is low, it is necessary to concentrate the ammonia by distillation, and reference should be made to B.S. 2690*. Hydrazine interferes with this test.

The method depends upon the reddish brown colour which ammonia in solution produces with Nessler's reagent. The determination should be made as soon as possible after sampling.

Reagents

<i>Ammonia standard solution.</i>	Reagent No. 10.
<i>Cupric sulphate solution.</i>	Reagent No. 26.
<i>Nessler solution.</i>	Reagent No. 47.
<i>Potassium sodium tartrate solution.</i>	Reagent No. 66.
<i>Sodium hydroxide, 50 per cent w/v.</i>	Reagent No. 72.
<i>Water, ammonia free. Use throughout.</i>	Reagent No. 94.
<i>Zinc sulphate solution.</i>	Reagent No. 95.

Procedure. Prepare a working solution of ammonia by diluting 10 ml of the ammonia standard solution to 1 litre with water in a one-mark graduated flask.

1 ml $\equiv$ 10 $\mu\text{g NH}_3$

When the sample requires clarification, add 1 ml of cupric sulphate solution to 100 ml of the sample in a measuring cylinder; mix thoroughly, add 1 ml of sodium hydroxide solution, again mix, and allow to settle. If settling is not satisfactory, repeat, adding the sodium hydroxide first, followed by the cupric sulphate.

If the sample contains hydrogen sulphide, substitute 1 ml of zinc sulphate solution for the cupric sulphate in the above procedure.

a. Standard series method. Into eight numbered Nessler cylinders measure 0, 0.2, 0.4, 0.6, 0.8, 1.2, 1.6, and 2.0 ml of the working solution and add sufficient water to each cylinder to make up the volume to 50 ml.

Into three similar cylinders put 10, 25, and 50 ml of the sample, clarified where necessary, make up the volumes to the mark with water, and add 1 ml of potassium sodium tartrate to prevent turbidity. Add 2.0 ml of Nessler solution to each of the eleven cylinders and mix. Allow them to stand for ten minutes for the colour to develop, then compare the samples with the standards.

* B.S. 2690, 'Methods of testing water used in industry'.

B.S. 1427 : 1962

$$\text{Ammonia} = \frac{\text{ml of ammonium chloride solution} \times 10}{\text{ml of sample}} \text{ p.p.m. NH}_3$$

b. Calibrated disk method. Fill two Nessler cylinders to the 50 ml mark with clear sample and to each add 1 ml of potassium sodium tartrate to prevent turbidity developing.

To one of the cylinders add 2 ml of Nessler's solution. Allow to stand for ten minutes for the colour to develop, then place in the appropriate compartment of the Nessleriser and the untreated sample in the other compartment. Match the colour produced against the appropriate disk.

$$\text{Ammonia} = \frac{\text{Disk reading in } \mu\text{g}}{\text{ml of sample}} \text{ p.p.m. NH}_3$$

4. CALCIUM

Range: 0-25 mg in terms of CaCO_3

0-250 p.p.m. in terms of CaCO_3

Calcium is determined by titrating the sample at the correct pH value with the disodium salt of diaminoethanetetra-acetic acid (EDTA) in the presence of Solochrome Dark Blue indicator. EDTA forms stable colourless complexes with calcium ions. Solochrome Dark Blue is an indicator for calcium ions. In their presence an alkaline solution of the indicator has a red colour. On the addition of EDTA to the red coloured solution, the calcium is converted to calcium diaminoethanetetra-acetic acid complex and, when this process is complete, the colour of the indicator reverts to blue.

Triethanolamine solution is added to suppress the effects of ferric iron and manganese which may be present in some industrial waters. Coloured samples should have a preliminary treatment for the removal of colour as described under the determination of hardness.

Reagents

Diaminoethanetetra-acetic acid, disodium salt (EDTA), 0.02N.

Sodium hydroxide, 4N.

Solochrome Dark Blue indicator.

Sulphuric or hydrochloric acid, 0.02N.

Triethanolamine, 20 per cent v/v.

Reagent No. 28.

Reagent No. 73.

Reagent No. 79.

Reagent No. 88 or 36.

Reagent No. 93.

Procedure

a. For concentrations between 10 p.p.m. and 250 p.p.m. in terms of CaCO_3 .

Measure 100 ml of the sample into a porcelain basin, add 0.02N acid to neutralize the sample, in quantity equivalent to the alkalinity to methyl orange (M). Stir well. Add 1 ml of 4N sodium hydroxide and allow to stand for five minutes. Now add 5 ml of triethanolamine and about 0.05 g of the Solochrome Dark Blue indicator. Titrate the solution with 0.02N EDTA until all trace of red colour has disappeared. Record the number of millilitres of EDTA used.

$$\text{Calcium} = \frac{\text{ml 0.02N EDTA} \times 1000}{\text{ml of sample}} \text{ p.p.m. in terms of CaCO}_3$$

b. For concentrations less than 10 p.p.m. in terms of CaCO₃. Measure 500 ml of the sample into a white porcelain basin, add 0.02N hydrochloric acid to neutralize the sample, this quantity of acid to be equivalent to the alkalinity to methyl orange (M). Stir well. Add 5 ml of 4N sodium hydroxide and allow to stand for five minutes. Now add 5 ml of triethanolamine and about 0.2 g of the Solochrome Dark Blue indicator. Titrate the solution with 0.02N EDTA until all trace of red colour has disappeared. Record the number of millilitres of EDTA used.

$$\text{Calcium} = \frac{\text{ml 0.02N EDTA} \times 1000}{\text{ml of sample}} \text{ p.p.m. in terms of CaCO}_3$$

5. CHLORIDE

Range: 0-50 mg Cl

0-500 p.p.m. Cl

Chloride is determined by titrating a neutral or slightly alkaline solution with silver nitrate solution in the presence of chromate indicator. White silver chloride is precipitated. The end point is indicated by the formation of red silver chromate. Sulphite interferes and may be removed by the addition of hydrogen peroxide.

Reagents

Calcium carbonate.

Hydrogen peroxide, 10 vol.

Methyl orange indicator solution.

Potassium chromate indicator solution.

Silver nitrate, N/35.5.

Sodium carbonate, 0.02N.

Sulphuric acid, 0.02N.

Reagent No. 22.

Reagent No. 37.

Reagent No. 44.

Reagent No. 58.

Reagent No. 68.

Reagent No. 70.

Reagent No. 88.

B.S. 1427 : 1962

Procedure. Measure 100 ml of the sample into a porcelain basin. If sulphite is present, add 2 ml of hydrogen peroxide 10 vol. Add two drops of methyl orange.

(i) If the colour is yellow add 0.02N sulphuric acid until the colour is orange.

(ii) If the colour is pink add 0.02N sodium carbonate until the colour is orange.

In either case it is an advantage to add about 0.1 g of calcium carbonate after neutralization, stirring thoroughly.

Then add 0.5 ml of potassium chromate indicator which will impart a yellow colour to the water. Add N/35.5 silver nitrate, stirring well during the addition, until the yellow colour just changes to a reddish brown tinge.

Record the number of ml N/35.5 silver nitrate used.

$$\text{Chloride} = \frac{\text{ml of N/35.5 silver nitrate} \times 1000}{\text{ml of sample}} \text{ p.p.m. Cl}$$

To convert p.p.m. Cl to p.p.m. NaCl multiply by 1.65.

6. RESIDUAL CHLORINE (FREE AND TOTAL)

*As amended
Feb. 1968*

Range: 0-0.08 mg Cl (liquid reagents)

0-0.04 mg Cl (tablets)

0-8 p.p.m. Cl (liquid reagents)

0-4 p.p.m. Cl (tablets)

Free chlorine in water reacts almost instantaneously with *NN*-diethyl-*p*-phenylene-diamine (DPD) to give a red colour. The rest of the chlorine, however, reacts much more slowly, and may be conveniently determined, following the determination of free chlorine by the addition of potassium iodide. The colours produced are compared with a series of standard colours on a calibrated disk.

Reagents

DPD tablets No. 1

Reagent No. 29a

DPD tablets No. 3

Reagent No. 29b

DPD solution

Reagent No. 29c

Phosphate buffer solution

Reagent No. 54b

Potassium iodide crystals

Reagent No. 64

Procedure

a. Free chlorine; liquid reagents. Fit the appropriate disk, with screen into the comparator. Place in the left-hand compartment, behind the colour standards of the disk, a 13.5 mm cell or test tube containing the sample only. Rinse a

similar cell or tube with the sample and leave about 1 ml in it. Add 2.5 ml of DPD solution and 2.5 ml of buffer solution. Add sample up to the 10 ml mark. Hold the comparator at eye level against a white background and at once match any red colour which has appeared. Multiply the disk reading by 2 and record the result as p.p.m. free residual chlorine.

b. Total chlorine; liquid reagents. Add about 0.5 g of potassium iodide to the right-hand cell, mix and allow to stand for 2 minutes. Again match the colour and multiply the disk reading by 2 and record the result as p.p.m. total residual chlorine.

c. Free chlorine; tablets. Fit the appropriate disk, with screen, into the comparator. Place in the left-hand compartment, behind the colour standards of the disk, a 13.5 mm cell or test tube containing the sample only. Rinse a similar cell or tube with the sample and leave in it enough sample to cover the tablet when added. Drop into the prepared cell or tube a DPD tablet No. 1. Allow the tablet to disintegrate or crush with a clean glass rod, and add the sample up to the 10 ml mark. Mix to dissolve rapidly the remains of the tablet, and place the cell in the right-hand compartment of the comparator. Hold the comparator at eye level against a white background and at once match any red colour which has appeared.

Record the result as p.p.m. free residual chlorine.

d. Total chlorine; tablets. Add one DPD tablet No. 3 to the right-hand cell, mix and allow to stand for 2 minutes. Again match the colour.

Record the result as p.p.m. total residual chlorine.

NOTE 1. It is very important to wash out the cell after the total chlorine determination 10 or 12 times with water to ensure that no potassium iodide or DPD tablet No. 3 remains. This would otherwise completely invalidate the next free residual chlorine determination to be carried out.

NOTE 2. It is very important not to handle the tablets. They may be conveniently dispensed by shaking a tablet into the bottle lid, and then into the cell or tube.

NOTE 3. It is recommended that tablets should be used in this tests as the DPD solution needs preparing at least once every two weeks and the use of solutions results in a very larger dilution of the sample.

7. COLOUR

When viewed by transmitted light through a depth of several feet, pure water exhibits a light blue colour. Frequently, however, water, though clear, appears under these conditions to be greenish or yellowish because of the presence of coloured substances.

No quantitative method for the determination of colour or suspended solids is recommended. A description of the colour of the sample, together with an indication of the amount and appearance of any suspended matter, should be given, e.g. slightly turbid, turbid, flocculent, etc. The colour cannot be judged accurately if the water contains suspended matter.

B.S. 1427 : 1962

8. DISSOLVED OXYGEN

METHOD 1

Range: 0.025 mg O₂—saturation0.05 p.p.m. O₂—saturation

Manganous hydroxide is precipitated in the sample by adding manganous sulphate and potassium hydroxide. The dissolved oxygen oxidizes part of the manganous hydroxide to manganic hydroxide. Upon acidification in the presence of potassium iodide, the hydroxides dissolve, the manganic hydroxide liberating free iodine in an amount equivalent to the oxygen originally dissolved in the sample. The free iodine is titrated with a standard sodium thiosulphate solution using starch as an internal indicator.

The method is liable to error in the presence of sulphite, hydrazine, organic matter and oxidizing agents, e.g. chlorine. The preliminary test indicates whether the method is liable to error, if so, reference should be made to B.S. 2690*.

Sampling is carried out under a continually renewed seal of the water being sampled and the reagents are added through this seal, thus avoiding contact with air.

Special apparatus. 500 ml glass stoppered bottle with sloping shoulders. The ground glass stopper should be attached to the neck of the bottle by means of a piece of string.

A vessel not less than 75 mm wider than the above bottle and fitted with an overflow so that the mouth of the bottle is at least 50 mm below the surface of the water.

Reagents

<i>Alkaline potassium iodide solution.</i>	Reagent No. 4.
<i>Manganous sulphate solution.</i>	Reagent No. 42.
<i>Potassium iodide crystals.</i>	Reagent No. 64.
<i>Potassium permanganate solution.</i>	Reagent No. 65.
<i>Sodium thiosulphate, 0.025N.</i>	Reagent No. 77.
<i>Starch indicator solution. Freshly prepared.</i>	Reagent No. 81.
<i>Sulphuric acid (1 : 1).</i>	Reagent No. 85.

Procedure

Preliminary test. Collect about 250 ml of the sample in a beaker. Add 1 ml of sulphuric acid by means of a safety pipette, mix and add 0.1 ml of potassium permanganate solution. Mix and allow to stand for five minutes. If the pink colour remains proceed as below. If the sample becomes colourless the method is liable to error.

* B.S. 2690, 'Methods of testing water used in industry'.