

ALKALINITY (ALKAPHOT)

TEST FOR TOTAL ALKALINITY IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 500 mg/l CaCO₃

Natural and treated waters may contain a variety of dissolved alkaline substances such as carbonates, bicarbonates, hydroxides and, to a lesser extent, borates, phosphates and silicates. In water at neutral pH the alkalinity derives mainly from the presence of bicarbonates.

Total alkalinity is an important test in determining the aggressiveness or scale forming tendency of the water. If the total alkalinity is low the water may be aggressive and cause corrosion to pipe work and structures; if the total alkalinity is high the water may more readily promote scale formation. Alkalinity control is therefore an important part of many water treatment programmes.

The Palintest Alkaphot test uses a colorimetric method and covers the total alkalinity range 0 - 500 mg/l CaCO₃. The test is particularly suitable for checking natural and drinking waters, swimming pool water, boiler water, etc.

Method

The Palintest Alkaphot test is based on a unique colorimetric method and uses a single tablet reagent. The test is simply carried out by adding a tablet to a sample of the water. Under the conditions of the test, a distinctive range of colours from yellow, through green, to blue is produced over the alkalinity range 0 - 500 mg/l CaCO₃. The colour produced in the test is indicative of the alkalinity of the water and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Alkaphot Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Alkaphot tablet, crush and mix until all of the particles have dissolved.
- 3 Stand for one minute then remix.
- 4 Select Phot 2 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l CaCO_3 .

Note: To convert Total Alkalinity as CaCO_3 to Total Alkalinity as HCO_3^- multiply result by 1.22.

Photometer Method

ALUMINIUM

TEST FOR TOTAL ALUMINIUM IN NATURAL AND TREATED WATERS

AUTOMATIC WAVELENGTH SELECTION

0 – 0.5 mg/l

Aluminium sulphate is widely used as a coagulant in drinking water treatment. The determination of aluminium (residual alum) is usually required for the control of alum coagulation and filtration processes at water works.

Aluminium salts are found in natural waters; levels are reported to be increasing particularly in areas affected by acid rain. High aluminium levels can be toxic to fish and aquatic life. Aluminium determination is necessary therefore for environmental control and for testing water used for fish farms, etc.

The Palintest Aluminium test provides a simple method of measuring aluminium levels in natural and drinking waters over the range 0 - 0.5 mg/l.

Method

Aluminium reacts with Eriochrome Cyanine R indicator in slightly acid solution to produce a pink-red coloured complex. The presence of ascorbic acid eliminates interference from iron and manganese. In the Palintest Aluminium method the necessary reagents are incorporated into two test tablets. The test is simply carried out by adding one of each tablet to a sample of the water. The first tablet acidifies the sample to bring any colloidal aluminium into solution and the second tablet buffers the solution to provide the correct conditions for the test.

The intensity of the colour produced in the test is proportional to the aluminium concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Aluminium No 1 Tablets

Palintest Aluminium No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Photometer Round Test Tubes, 10 ml glass (PT 595)

Sample Collection

Aluminium is readily absorbed on to the surfaces of sample containers, particularly glass containers. To avoid loss of aluminium, collect samples in plastic bottles and test as soon as possible after collection. Sample bottles should be acid-rinsed and thoroughly washed out with deionised water before re-use.

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Aluminium No 1 tablet, crush and mix to dissolve.
- 3 Add one Aluminium No 2 tablet, crush and mix gently to dissolve. Avoid vigorous agitation.
- 4 Stand for five minutes to allow full colour development.
- 5 Select Phot 3 on Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l Al.

Interferences

The presence of polyphosphate or fluoride can lead to low aluminium readings. Polyphosphate is unlikely to be present in significant quantities in normal water samples. Fluoride will only be significant for control samples from water works where fluoridation is practised. In such cases samples should preferably be taken before the final fluoridation stage.

For samples taken after fluoridation such as those from water distribution systems, or for samples containing natural fluoride, the aluminium concentration should be corrected. To obtain the corrected aluminium concentration multiply the calibration chart value by the factor $(1 + 0.4 F)$ where F is the Fluoride concentration as mg/l F. The fluoride concentration should be determined separately by normal test procedure.

AMMONIA

TEST FOR AMMONIA IN NATURAL, DRINKING AND WASTE WATERS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 1.0 mg/l N

Ammonia occurs as a breakdown product of nitrogenous material in natural waters. It is also found in domestic effluents and certain industrial waste waters. Ammonia is harmful to fish and other forms of aquatic life, and the ammonia level must be carefully controlled in water used for fish farms and aquariums. Ammonia tests are routinely applied for pollution control on effluents and waste waters, and for the monitoring of drinking water supplies.

The Palintest Ammonia Test provides a simple method of measuring ammonia (ammoniacal nitrogen) over the range 0 - 1.0 mg/l N.

Method

The Palintest Ammonia test is based on an indophenol method. Ammonia reacts with alkaline salicylate in the presence of chlorine to form a green-blue indophenol complex. Catalysts are incorporated to ensure complete and rapid colour development. The reagents are provided in the form of two tablets for maximum convenience. The test is simply carried out by adding one of each tablet to a sample of the water.

The intensity of the colour produced in the test is proportional to the ammonia concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Ammonia No 1 Tablets

Palintest Ammonia No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Instructions

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Ammonia No 1 tablet and one Ammonia No 2 tablet, crush and mix to dissolve.
- 3 Stand for ten minutes to allow colour development.
- 4 Select Phot 4 on Photometer to measure Ammonia mg/l N **or** select Phot 62 on Photometer to measure Ammonium mg/l NH_4 .
- 5 Take Photometer reading in usual manner (see Photometer instructions).

Sea Water Samples

Palintest Ammonia Conditioning Reagent is required when testing sea water or brackish water samples to prevent precipitation of salts. The reagent is supplied in a special 'spoon pack' to aid measuring out the powder.

Fill the test tube with sample to the 10 ml mark, and add one level spoonful of conditioning reagent. Mix to dissolve reagent then continue the test as described in the above test instructions. If turbidity still forms in the test, repeat using two level spoonfuls of conditioning reagent.

Notes

- 1 At low temperatures the rate of colour development in the test may be slower. If the sample temperature is below 20°C allow 15 minutes for the colour to develop.
 - 2 Ammonia concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-
 - To convert from N to NH_4 multiply by 1.3.
 - To convert from N to NH_3 multiply by 1.2.
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MEASUREMENT OF BROMINE IN THE PRESENCE OF CHLORINE

It is possible to determine bromine in the presence of chlorine, and to separate between bromine and chlorine residuals. This instruction sheet gives the test procedures for these determinations.

Reagents and Equipment

Palintest DPD No 1 Tablets

Palintest DPD No 3 Tablets

Palintest Glycine Tablets

Equipment as per Instruction Sheet PHOT.5.1.AUTO

Test Procedure - Total Bromine (in the Presence of Chlorine)

- 1 Fill a test tube with sample to the 10 ml mark. Add one DPD Glycine tablet, crush and mix to dissolve.
- 2 Take a second clean test tube and add a few drops of solution from the first tube.
- 3 Add and then crush the DPD No 1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 4 Add the treated 10 ml test solution, mix and seal the tube with the cap.
- 5 Gently invert the tube to remove any bubbles from the inner walls of the tube.
- 6 Select Phot 5 on the photometer.
- 7 Take the test reading using the photometer.
- 8 The result obtained represents the total bromine residual as mg/l Br₂ (Result A).

For most purposes the test can be terminated at this stage. However if it is desired to measure free and combined chlorine, proceed as indicated in the following section :-

Test Procedure - Free and Combined Chlorine (in the Presence of Bromine)

- 1 Rinse the test tube with sample leaving a few drops in the tube.
- 2 Add and then crush the DPD No 1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 3 Add the 10 ml test solution, mix and seal the tube with the cap.
- 4 Gently invert the tube to remove any bubbles from the inner walls of the tube.
- 5 Select Phot 5 on the photometer.
- 6 Take the test reading immediately using the photometer.
- 7 The result obtained represents total bromine plus free chlorine as mg/l Br₂ (Result B).
- 8 Continue the test by adding one DPD No 3 tablet. Crush and mix to dissolve.
- 9 Allow the tube to stand for two minutes and then take the reading on Phot 5 using the photometer.
- 10 The result obtained represents total bromine plus free chlorine plus combined chlorine as mg/l Br₂ (Result C).

Calculation of Results

The various residuals can be calculated from the above results as follows :-

$$\text{Total Bromine (as Br}_2\text{)} = \text{Result A}$$

$$\text{Free Chlorine (as Cl}_2\text{)} = (\text{Result B} - \text{Result A}) \times 0.44$$

$$\text{Combined Chlorine (as Cl}_2\text{)} = (\text{Result C} - \text{Result B}) \times 0.44$$

$$\text{Total Chlorine (as Cl}_2\text{)} = (\text{Result C} - \text{Result A}) \times 0.44$$

BROMINE

TEST FOR FREE, COMBINED AND TOTAL BROMINE IN WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 10.0 mg/l

Bromine and bromine-release compounds are used for the disinfection of swimming pool water, and in many other water treatment systems. Accurate measurement of the bromine residual is an essential aspect of control of these processes.

The bromine level can be expressed in terms of the free bromine, combined bromine or total bromine residuals. However free and combined bromine are both considered powerful disinfectants and it is not normally necessary to differentiate between these two forms. For the majority of applications therefore the measurement of the total residual is sufficient.

The Palintest DPD bromine method provides a simple means of measuring bromine residuals over the range 0 - 10.0 mg/l. A supplementary procedure can be used to differentiate between free and combined bromine if desired.

Method

The Palintest bromine test uses the DPD method now internationally recognised as the standard method of testing for disinfectant residuals. In the DPD method the reagents are provided in tablet form for maximum convenience and simplicity of use.

Bromine reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. The intensity of the colour is proportional to the total bromine concentration and is measured using a Palintest Photometer.

For the separate determination of free and combined bromine, a supplementary procedure using sodium nitrite is used. The nitrite destroys the free bromine in the sample and the colour produced in the DPD test then corresponds to the combined bromine only. The free bromine content is thus obtained by difference between the total bromine and combined bromine results.

Reagents and Equipment

Palintest DPD No 1 Clear Tablets

Palintest DPD Nitrite Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Bromine Residuals

The photometer is programmed for both total and free bromine. Use program Phot 5 Total Bromine, then select the 'Follow On' option on screen to continue test for program Phot 6 Free Bromine. The Free Bromine residual is calculated automatically.

Test Procedure

- 1 Rinse test tube with sample leaving a few drops in the tube.
- 2 Add and then crush the DPD #1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 3 Add the 10ml test solution, mix and seal the tube with the cap.
- 4 Gently invert the tube to remove any bubbles from the inner walls of the tube.
- 5 Select Phot 5 on the Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 Result displayed is Total Bromine as mg/l Br.

For most purposes the test can be terminated at this stage. If it is desired to measure free and combined bromine, select 'Follow On' from screen options and proceed as indicated in the following section.

Test Procedure - Free and Combined Bromine

- 1 Fill test tube with sample to the 10 ml mark. Add one DPD Nitrite tablet, crush and mix to dissolve.
- 2 Take a second clean test tube and add a few drops of the solution from the first tube.
- 3 Add and then crush the DPD #1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 4 Add the remaining 10ml of test solution, mix and seal the tube with the cap
- 5 Take Photometer reading in usual manner.
- 6 The Photometer carries out the necessary calculation and displays the Free Bromine residual as mg/l Br.

Note

In systems containing both chlorine and bromine it is possible to differentiate between the chlorine and bromine residuals using a supplementary procedure involving Palintest DPD Glycine tablets. Details of this procedure are given on a separate instruction sheet.

CHLORINE/ CHLORAMINES (DPD)

**TEST FOR FREE CHLORINE,
MONOCHLORAMINE AND
DICHLORAMINE IN WATER**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 5.0 mg/l

Chlorine and chlorine release compounds are widely used for the disinfection of water. When dissolved in water chlorine forms hypochlorous acid and hypochlorite ions. Chlorine remaining in the water in this form is known as the free chlorine residual.

Chlorine does however react with ammonia and nitrogen-based species to form chloramines. These compounds are poor disinfectants and can also impart a characteristic taste or odour to the water. It is important therefore in certain applications to be able to distinguish between chlorine residual present as free chlorine and as chloramines.

The Palintest DPD Chlorine/Chloramines method provides a simple means of measuring free chlorine ($\text{HOCl}/\text{HOCl}^{\cdot}$), monochloramine (NH_2Cl) and dichloramine (NHCl_2).

Method

The Palintest Chlorine/Chloramines test uses the DPD method. This method is internationally recognised as the standard method of testing for chlorine and other residuals. In the Palintest method the reagents are provided in tablet form for maximum convenience and simplicity of use.

Free chlorine reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. The intensity of the colour is proportional to the free chlorine concentration. Addition of a trace amount of potassium iodide induces further reaction with any monochloramine present. The increase in colour intensity is therefore proportional to the monochloramine concentration. Subsequent addition of excess potassium iodide causes dichloramine to react in a similar manner. The increase in colour intensity is now proportional to the dichloramine concentration.

In this way it is possible to differentiate between free chlorine, monochloramine and dichloramine residuals present in the sample. The colour intensities at each stage of the test are measured using a Palintest Photometer.

Reagents and Equipment

Palintest DPD No 1 Tablets

Palintest DPD No 2 Tablets

Palintest DPD No 3 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Chlorine Residuals

The photometer is programmed for free chlorine and for the chloramine stages. Use program Phot 71 Free Chlorine then select 'Follow On' from screen options to continue test for program 72 Monochloramine and again for program 73 Dichloramine.

Test Procedure

- 1 Rinse test tube with sample leaving a few drops in the tube.
 - 2 Add and then crush the DPD No 1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
 - 3 Add the 10 ml test solution, mix and seal the tube with the cap.
 - 4 Gently invert the tube to remove any bubbles from the inner walls of the tube.
 - 5 Select Phot 7 on photometer.
 - 6 Take photometer reading in usual manner - see photometer instructions.
 - 7 The result represents the free chlorine residual as mg/l Cl₂.
 - 8 To measure monochloramine, continue the test on the same test sample. Select 'Follow On' from screen options to continue the test program.
 - 9 Add one DPD No 2 tablet, crush and mix to dissolve.
 - 10 Gently invert the tube to remove any bubbles from the inner walls of the tube.
 - 11 Take the photometer reading. The result displayed is the mono-chloramine concentration as mg/l Cl₂.
 - 12 To measure dichloramine, continue the test on the same test portion. Select 'Follow On' option from screen options to continue the test program.
 - 13 Add one DPD No 3 tablet, crush and mix to dissolve. Stand for two minutes to allow full colour development.
 - 14 Take the photometer reading. The photometer displays the dichloramine concentration as mg/l Cl₂.
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PERSULPHATE

TEST FOR ACTIVITY OF PERSULPHATE IN WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 5.0 mg/l

Chlorine Equivalent

Chlorine and chlorine-release compounds are widely used for the disinfection of swimming pools. Activated persulphate chemicals are used for shock dosing pools to regenerate free chlorine.

The Persulphate activity can be expressed in terms of its chlorine equivalent. The Palintest DPD Method provides a simple means of measuring the persulphate activity over the range 0 - 5 mg/l chlorine equivalent and separating it from chlorine residuals.

Method

The Palintest method uses the DPD method developed by Dr A T Palin for chlorine testing. The reagents are provided in tablet form for maximum convenience and simplicity of use.

Free chlorine reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. The intensity of the colour is proportional to the free chlorine concentration. Addition of DPD Oxystop reagent prevents reaction of potassium monopersulphate shock dosing chemicals, and addition of potassium iodide induces a further reaction with any combined chlorine present. The colour intensity is now proportional to the total chlorine concentration. The increase in colour represents the combined chlorine concentration.

Using a fresh portion of sample, DPD in buffered solution in the presence of iodide produces a pink coloration. The intensity of the colour is proportional to the total chlorine plus the persulphate activity.

The colour intensities are measured using a Palintest Photometer. The difference between the total chlorine reading and the total chlorine plus persulphate reading is a measure of persulphate in the pool.

Reagents and Equipment

Palintest DPD No 1 Clear Tablets

Palintest DPD Oxystop Tablets

Palintest DPD No 3 Clear Tablets

Palintest DPD No 4 Clear Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Instructions

- 1 Rinse test tube with sample leaving a few drops of sample in the tube.
- 2 Add and then crush the DPD No 1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 3 Add the 10 ml test solution, mix and seal the tube with the cap.
- 4 Gently invert the tube to remove any bubbles from the inner walls of the tube.
- 5 Select Phot 7 on the Photometer.
- 6 Take photometer reading in the usual manner.
- 7 The result represents the free chlorine residual as milligrams per litre. (Reading A).
- 8 Continue the test on the same test portion. Select 'Follow-On' from screen options to continue the test program.
- 9 Add one DPD Oxystop tablet, crush and mix to dissolve. Stand for one minute before proceeding.
- 10 Add one DPD No 3 tablet, crush and mix to dissolve.
- 11 Stand for two minutes to allow full colour development.
- 12 Take photometer reading. The result represents the total chlorine residual as milligrams per litre. (Reading B).
- 13 The combined chlorine residual is obtained by subtracting the free chlorine residual result from the chlorine residual result. (Reading B - Reading A).
- 14 Rinse a second test tube with sample leaving a few drops of sample in the tube.
- 15 Add one DPD No 4 tablet, crush tablet and then fill the test tube with sample to the 10 ml mark. Mix to dissolve tablet.
- 16 Stand for two minutes to allow full colour development.
- 17 Take photometer reading. The result represents total chlorine residual plus persulphate activity expressed as milligrams per litre chlorine. (Reading C).
- 18 If Reading C is greater than Reading B, then persulphate is present. The measurement of persulphate activity is obtained by subtracting Reading B from Reading C.

$$\text{Persulphate Activity} = \text{Reading C} - \text{Reading B mg/l Cl}$$

Note

A too high chlorine level (above 10 mg/l) can cause bleaching of the pink coloration formed in the DPD test and give a false negative or lower than expected result. If a colourless or pale pink test solution is obtained when a high level chlorine may be present, check for the possibility of bleaching by repeating the test on a sample diluted with chlorine-free water.

Photometer Method

CHLORINE DIOXIDE

**AUTOMATIC
WAVELENGTH
SELECTION**

**TEST FOR CHLORINE DIOXIDE
AND OTHER RESIDUALS IN WATER**

0 – 25.0 mg/l as Cl
0 – 10.0 mg/l as ClO₂

Chlorine dioxide is used for the disinfection of water in a variety of different applications. Chlorine dioxide is normally generated by reacting chlorine with sodium chlorite solution in specially designed plant and equipment. Water treated with chlorine dioxide may therefore also contain amounts of chlorine and chlorite. For the control of such water treatment systems it is necessary to determine and differentiate between these different residual species.

The Palintest Chlorine Dioxide method provides a precise method of determining chlorine dioxide in treated water. Supplementary procedures provide for the determination of free and combined chlorine and chlorite.

Method

Chlorine dioxide reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. Chlorine reacts in a similar manner. Glycine is used to prevent the reaction with chlorine so as to give specific determination of chlorine dioxide.

In the supplementary part of the test the glycine is omitted and it is then possible, by differences, to measure the free chlorine content. Subsequent addition of potassium iodide induces a further reaction with any combined chlorine present. Continuation of the test using an acidification and neutralisation procedure produces a further reaction and in this way the chlorite concentration can be determined.

The colour intensities at each stage of the test are measured using a Palintest Photometer and the concentration of each individual component are obtained by a simple calculation. It is normal practice to express the concentration of each component in terms of the equivalent chlorine concentration.

Reagents and Equipment

Palintest DPD No 1 Tablets
Palintest DPD No 3 Tablets
Palintest DPD Glycine Tablets
Palintest DPD Acidifying Tablets
Palintest DPD Neutralising Tablets
Palintest Automatic Wavelength Selection Photometer
Palintest Round Test Tubes, 10 ml glass (PT 595)

Test Procedure - Chlorine Dioxide

- 1 Rinse a clean test tube with sample, then fill with sample to the 10 ml mark. Add one Glycine tablet, crush and mix to dissolve.
- 2 Decant a few drops of Glycine treated sample into a second clean test tube.
- 3 Add and then crush the DPD #1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 4 Add the remaining treated sample of the first test tube to the second test tube, mix and seal the tube with the cap.
- 5 Gently invert the tube to remove any bubbles from the inner walls of the tube
- 6 Select Phot 7 on photometer.
- 7 Take photometer reading [**Result G**]
- 8 Multiply **Result G** by 5 to obtain the chlorine dioxide residual in terms of mg/l Chlorine.

To obtain the chlorine dioxide residual as mg/l ClO_2 , multiply **Result G** by 1.9.

Test Procedure - Free and Combined Chlorine, and Chlorite

- 1 Rinse a test tube with sample leaving a few drops.
- 2 Add and then crush the DPD #1 tablet in the few drops of the water sample until the tablet is thoroughly crushed.
- 3 Add the 10ml test solution, mix and seal the tube with the cap.
- 4 Gently invert the tube to remove any bubbles from the inner walls of the tube
- 5 Take the photometer reading on Phot 7 in usual manner (**Result A**).
- 6 Continue the test by adding one DPD No 3 tablet. Crush tablet, mix to dissolve and then stand for two minutes.
- 7 Take photometer reading (**Result C**).
- 8 Continue the test by adding one DPD Acidifying tablet. Crush tablet, mix to dissolve and then stand for two minutes.
- 9 Add one DPD Neutralising tablet, crush and mix to dissolve.
- 10 Take the photometer reading (**Result D**).

The results of the tests, in terms of mg/l chlorine, are calculated from the observed results as follows :-

Chlorine Dioxide	= 5G
Free Chlorine	= A - G
Combined Chlorine	= C - A
Chlorite	= D - (C + 4G)
Total Oxidising Capacity	= D

CHLORINE (DPD)

TEST FOR FREE, COMBINED AND TOTAL CHLORINE IN WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 5.0 mg/l

Chlorine and chlorine-release compounds are widely used for the disinfection of drinking water and swimming pools, for the control of micro-biological growth in cooling water, and in many other water treatment systems. Accurate measurement of the chlorine residual is an essential aspect of the control of these chlorination processes.

The chlorine level can be expressed in terms of the free chlorine, combined chlorine or total chlorine residuals. For the majority of applications measurement of the free chlorine residual is the most important. The Palintest DPD chlorine method provides a simple means of measuring free, combined and total chlorine residuals over the range 0 - 5 mg/l.

It is recommended that if any shock treatment compounds are known to have been used in the treatment of the water to be tested, that a DPD Oxystop tablet be included in the test procedure as outlined below.

Method

This Palintest chlorine test uses the DPD method developed by Dr A T Palin and now internationally recognised as the standard method of testing for chlorine and other disinfectant residuals. In the Palintest DPD method the reagents are provided in tablet form for maximum convenience and simplicity of use.

Free chlorine reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. The intensity of the colour is proportional to the free chlorine concentration. Subsequent addition of excess potassium iodide induces a further reaction with any combined chlorine present. The colour intensity is now proportional to the total chlorine concentration; the increase in intensity represents the combined chlorine concentration. In this way it is possible to differentiate between free and combined chlorine present in the sample. The colour intensities are measured using a Palintest Photometer.

The DPD Oxystop tablet is added after measurement for free chlorine but before the DPD No 3 tablet. It prevents the reaction between shock treatment chemicals and potassium iodide which would give a positive response.

Reagents and Equipment

Palintest DPD No 1 Tablets

Palintest DPD Oxystop Tablets (Optional)

Palintest DPD No 3 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Chlorine Residuals

The photometer is programmed for both free and total chlorine. Use program Phot 7 Free Chlorine, then select the 'Follow On' option on screen to continue test for program Phot 8 Total Chlorine.

Test Instructions

- 1 Rinse test tube with sample leaving a few drops of sample in the tube.
- 2 Crush the DPD No 1 tablet in two or three drops of the water sample until the tablet is thoroughly dissolved.
- 3 Add the 10 ml test solution, mix and seal the tube with the cap.
- 4 Gently invert the tube to remove any bubbles from the inner walls of the tube
- 5 Select Phot 7 on Photometer.
- 6 Take photometer reading in usual manner - see Photometer instructions.
- 7 The result represents the **free chlorine** residual in milligrams per litre (mg/l).
Stop the test at this stage if only free chlorine determination is required.
- 8 If it is desired to measure combined or total chlorine residual, continue the test on the same test portion. Select 'Follow On' from the screen options to continue the test program.
- 9 If shock treatment chemicals are present in the pool, add one DPD Oxystop tablet, crush and mix to dissolve. Stand for one minute before proceeding.
- 10 Add one DPD No 3 tablet, crush and mix to dissolve.
- 11 Stand for two minutes to allow full colour development.
- 12 Take photometer reading in the usual manner.
- 13 The result represents the **total chlorine** residual as milligrams per litre(mg/l).
- 14 The **combined chlorine** residual is obtained by subtracting the free chlorine residual result from the total chlorine residual result :-
ie Combined Chlorine = Total Chlorine - Free Chlorine

Notes

A too high chlorine level (above 10 mg/l) can cause bleaching of the pink coloration formed in the DPD test and give a false negative or lower than expected result. If a colourless or pale pink test solution is obtained then a high level of chlorine may be present, check for the possibility of bleaching by repeating the test on a sample diluted with chlorine-free water.

CHLORINE HR

TEST FOR HIGH LEVELS OF CHLORINE IN DISINFECTING AND STERILISING SOLUTIONS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 250 mg/l

Chlorine and chlorine release compounds are widely used for disinfection or sterilisation of water distribution systems and pipe work, plant and equipment in food processing and pharmaceutical factories, and similar applications. The chlorine levels used in these applications are higher than those normally applied for the simple disinfection of water. Accurate measurement of the chlorine level is necessary to ensure it is sufficient for the intended use. The Palintest Chlorine HR test provides a simple means of measuring the total chlorine over the range 0 - 250 mg/l.

Method

The Palintest Chlorine HR test is based on an iodine release method. Chlorine reacts with potassium iodide in acid solution to release iodine which is brown in colour. The reagents for the test are provided in the form of two tablets for maximum convenience and simplicity of use.

The intensity of the colour produced is proportional to the chlorine concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Acidifying GP Tablets
Palintest Chlorine HR Tablets
Palintest Auto Wavelength Photometer
Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Acidifying GP tablet and one Chlorine HR tablet. Crush tablets and mix to dissolve. Allow any undissolved particles to settle.
- 3 Select Phot 9 on Photometer.
- 4 Take Photometer reading in usual manner (see Photometer instructions).
- 5 The result is displayed as mg/l Cl.

Note

For precise determination of lower levels of chlorine, up to 5 mg/l, the Palintest Chlorine (DPD) method should be used.

COPPER (COPPERCOL)

**TEST FOR FREE, CHELATED AND
TOTAL COPPER IN NATURAL
AND TREATED WATERS**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 5.0 mg/l

Copper occurs naturally in many waters and may also result from corrosion of pipes and fittings. The presence of copper in drinking water can give rise to discolouration or an astringent taste.

Chelated copper compounds are extensively used as algicides in swimming pool water, home aquariums and other waters. Electrolytic devices which generate copper and silver ions are used in the purification of swimming pool water.

The Palintest Coppercol method provides a simple means of measuring copper in natural and treated waters over the range 0 - 5 mg/l. The test is particularly useful since it can be used to measure specifically the concentrations of free and chelated copper present in the water.

Method

In the Palintest Coppercol! method copper salts are reduced to the cuprous form and then reacted with a 2,2 Biquinoline-4,4-dicarboxylic salt to form a purple coloured complex. This provides a measure of the free copper ions present in the sample. In the second stage of the test, a decomplexing agent is introduced and this induces a further reaction with any chelated copper compounds which might be present.

The reagents are provided in tablet form and the test is simply carried out by adding tablets to a sample of the water. The intensity of colour produced in the test is proportional to the copper concentrations and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Coppercol No 1 Tablets

Palintest Coppercol No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Copper Residuals

The direct-reading photometer is programmed for both free and total copper. Use program **Phot 10** Free Copper, then select the 'Follow On' option on screen to continue test for program **Phot 11** Total Copper.

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
 - 2 Add one Coppercol No 1 tablet, crush and mix to dissolve.
 - 3 Gently invert the tube to remove any bubbles from the inner walls of the tube.
 - 4 Select Phot 10 on Photometer.
 - 5 Take Photometer reading in usual manner - see Photometer instructions.
 - 6 The result represents the free copper concentration as mg/l Cu. Stop the test at this stage if only free copper determination is required.
 - 7 If it is desired to measure chelated or total copper continue the test on the same test portion. Select the 'Follow On' from screen options to continue the test program.
 - 8 Add one Coppercol No 2 tablet, crush and mix to dissolve.
 - 9 Gently invert the tube to remove any bubbles from the inner walls of the tube.
 - 10 Take Photometer reading.
 - 11 The result represents the **Total Copper** concentration as mg/l Cu.
 - 12 The **Chelated Copper** concentration is obtained by subtracting the free copper concentration from the total copper concentration :-
ie Chelated Copper = Total Copper - Free Copper
-

CALCIUM HARDNESS (CALCICOL)

TEST FOR CALCIUM HARDNESS IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 500 mg/l CaCO_3

Calcium hardness is caused by the presence of calcium ions in the water. Calcium salts can be readily precipitated from water and high levels of calcium hardness tend to promote scale formation in water systems. Calcium hardness is an important control test in industrial water systems such as boilers and steam raising plant; and for swimming pool waters.

The Palintest Calcicol test provides a simple method of determining calcium hardness over the range 0 - 500 mg/l CaCO_3 .

Method

The Palintest Calcium Hardness test is based on the Calcicol indicator reagent method. Calcium ions react specifically with Calcicol indicator in alkaline solution to give an orange coloration. The reagent itself gives a violet colour in solution. Thus at different calcium levels a distinctive range of colours from violet to orange is produced.

The reagents for the method are provided in the form of two tablets. The test is carried out simply by adding one of each tablet to a sample of the water. The colour produced is indicative of the calcium hardness and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Calcicol No 1 Tablets

Palintest Calcicol No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Fill the test tube with sample to the 10 ml mark.
- 3 Add one Calcicol No 1 tablet, crush and mix to dissolve.
- 4 Add one Calcicol No 2 tablet, crush and mix to dissolve.
- 5 Stand for two minutes to allow full colour development.
- 6 Select Phot 12 on the Photometer for result as mg/l CaCO_3 , or Phot 60 for result as mg/l Ca.
- 7 Take Photometer reading in the usual manner (see Photometer instructions).

Interferences

- 1 Magnesium hardness (up to 200 mg/l as CaCO_3) does not interfere with the test.
- 2 Iron at levels above 10 mg/l may cause low results. Zinc above 5 mg/l may cause high results.
- 3 The pH required in the test is closely controlled by a buffer mixture included in the tablet formulation. However, to avoid exceeding the buffer capacity, strongly acid or alkaline samples should be adjusted to within the pH range 4 to 10, prior to the start of the test.

Notes

- 1 The expression of hardness results sometimes causes confusion. It is normal practice to express the results of hardness tests as mg/l CaCO_3 (calcium carbonate). This is merely a convention to allow the comparison of different results and does not necessarily indicate that the hardness is present in the water in this form.
Results may also be expressed as mg/l Ca. To convert mg/l CaCO_3 to mg/l Ca multiply by 0.4.
 - 2 Magnesium hardness may be determined using the Palintest Magnecol method (see PHOT.21), or by taking the difference between the Total Hardness (PHOT.15) and Calcium Hardness test results.
-

CYANURIC ACID

Photometer Method

TEST FOR CYANURIC ACID IN SWIMMING POOL WATER

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 200 mg/l

Cyanuric acid is extensively used as a chlorine stabiliser in swimming pool water. Cyanuric acid itself may be added to the water when the pool is first filled, or may be introduced gradually through the use of chloroisocyanurate based chlorine donors. Swimming pool water treatment instructions generally recommend a cyanuric acid level within the range 30 - 200 mg/l. In some countries a lower maximum level is recommended. The Palintest Cyanuric Acid test provides a simple method of measuring cyanuric acid level over the range 0 - 200 mg/l.

Method

The Palintest Cyanuric Acid test is based on a single tablet reagent containing melamine and a buffer. Cyanuric acid reacts with melamine in buffered solution to form an insoluble complex. At the cyanuric acid levels encountered in pool water, this is observed as turbidity in the test sample. The degree of turbidity is proportional to the cyanuric acid concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Cyanuric Acid Tablets
Palintest Automatic Wavelength Selection Photometer
Round Test Tubes, 10 ml glass (PT 595)
Palintest Dilution Tube (PT 512)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Cyanuric Acid tablet and allow to disintegrate for at least two minutes. A cloudy solution indicates the presence of cyanuric acid.
- 3 Crush any remaining undissolved tablet and mix to ensure uniformity.
- 4 Select Phot 13 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l.

Note

The range of the test is 0 - 200 mg/l. However when a test result of 100 mg/l or over is obtained, the following dilution technique is recommended in order to obtain a more precise result.

- 1 Take a sample of pool water in a Palintest Dilution Tube (PT 512), filling to the x10 mark.
 - 2 Make up to the 'Deionised Water' mark with deionised water, or tap water, and mix.
 - 3 Fill a round test tube with diluted sample to the 10 ml mark. Test as per the earlier test procedure.
 - 4 Multiply the displayed result by 10 to obtain the cyanuric acid concentration.
-

FLUORIDE

TEST FOR FLUORIDE IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 1.5 mg/l

Fluoride occurs naturally in some ground waters and is often introduced into drinking water for the prevention of tooth decay. Excessive amounts of fluoride are however objectionable and can cause tooth discolouration.

The Palintest Fluoride test provides a simple method of monitoring fluorides in natural waters, and for the control of fluoridation plant at water works.

Method

Zirconyl Chloride and Eriochrome Cyanine R are reacted in acid solution to form a red coloured complex. This colour is destroyed by fluoride ions to give the pale yellow colour of the Eriochrome Cyanine. Differing amounts of fluoride thus produce a range of colours from red to yellow.

The particular advantage of this method is that it is substantially free from interferences which normally beset chemical methods of fluoride testing. In particular interference from aluminium and iron is eliminated by making the solution alkaline in the first stage of the test procedure. This breaks down any aluminium-fluoride and iron-fluoride complexes which may be present in the water. Interference from calcium and sulphates should not be significant at the levels normally encountered in natural and drinking waters.

In the Palintest Fluoride test two tablet reagents are used. The test is simply carried out by adding one of each tablet to a sample of the water. The colour produced in the test is indicative of the fluoride concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Fluoride No 1 Tablets

Palintest Fluoride No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Fluoride No 1 tablet, crush and mix to dissolve.
- 3 Add one Fluoride No 2 tablet, crush and mix to dissolve.
- 4 Stand for five minutes to allow full colour development.
- 5 Select Phot 14 on Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l F.

HARDNESS (HARDICOL)

TEST FOR HARDNESS IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 500 mg/l CaCO₃

Water hardness is caused by the presence of calcium and magnesium salts. High levels of hardness prevent the formation of lather with soap, and can cause scaling in water systems - particularly boilers, heat exchangers and steam generating plant. Hardness is an important control test in a wide variety of applications.

The Palintest Hardness test provides a simple method of checking water hardness over the range 0 - 500 mg/l CaCO₃.

Method

The Palintest Hardicol test is based on a unique colorimetric method. The reagents are provided in tablet form and the test is carried out simply by adding the appropriate tablets to a sample of the water.

Under the controlled conditions of the test calcium and magnesium ions react with Hardicol indicator to produce a purple coloration. The intensity of the colour is proportional to the total hardness of the water and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Hardicol No 1 Tablets

Palintest Hardicol No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Fill test tube with sample to the 10 ml mark.
- 3 Add one Hardicol No 1 tablet, crush and mix to dissolve.
- 4 Add one Hardicol No 2 tablet, crush and mix to dissolve. Ensure all particles are completely dissolved.
- 5 Stand for two minutes to allow full colour development.
- 6 Select Phot 15 on the Photometer.
- 7 Take Photometer reading in the usual manner (see Photometer instructions).
- 8 The Total Hardness result is displayed as mg/l CaCO_3 .

Interferences

- 1 Unusually high levels of iron (above 10 mg/l) will cause low results for total hardness.
- 2 The pH required in the test is closely controlled by a buffer mixture included in the tablet formulation. However, to avoid exceeding the buffer capacity strongly acid or alkaline samples should be adjusted to within the pH range 4 to 10, prior to the start of the test.

Notes

- 1 The expression of hardness results sometimes causes confusion. It is normal practice to express the result of hardness tests as mg/l CaCO_3 (calcium carbonate). This is merely a convention to allow the comparison of different results and does not necessarily indicate that the hardness is present in the water in this form.
 - 2 This test measures total hardness. For the specific measurement of calcium hardness or magnesium hardness refer to the Palintest Calcol (PHOT.12) and Magnecol (PHOT.21) tests respectively.
-

HYDROGEN PEROXIDE LR

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

**TEST FOR LOW LEVELS OF
HYDROGEN PEROXIDE IN WATER**

0 – 2.0 mg/l

Hydrogen peroxide is used in various water treatment processes. In such applications it is important to ensure that the hydrogen peroxide level is maintained within the correct range to ensure optimum operation of the water treatment process.

The Palintest Hydrogen Peroxide LR test provides a simple means of measuring Hydrogen Peroxide levels over the range 0 - 2.0 mg/l.

Method

Hydrogen peroxide reacts with potassium iodide under slightly acid conditions, and in the presence of a catalyst, to release iodine into solution. The iodine then reacts with diethyl-p-phenylene diamine (DPD) to produce a pink coloration. In the Palintest method the reagents are combined in the form of a single tablet and the test is simply carried out by adding a tablet to a sample of the water.

The intensity of the colour produced is proportional to the hydrogen peroxide concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Hydrogen Peroxide LR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Rinse test tube with sample leaving 2 to 3 drops of sample in the tube.
- 2 Add one Hydrogen Peroxide LR tablet, crush and then fill tube with sample to the 10 ml mark. Mix to dissolve tablet.
- 3 Stand for two minutes to allow full colour development.
- 4 Select Phot 16 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l H_2O_2 .

Notes

- 1 The sample should be free of other oxidising agents such as chlorine, bromine, etc, as these react in a similar manner and will interfere with the test. It is unlikely that these oxidising agents will be used in conjunction with hydrogen peroxide and, under normal circumstances, will not usually coexist in solution.
 - 2 For measuring high levels of hydrogen peroxide used in industrial processes, use the Palintest Hydrogen Peroxide HR test (see PHOT.17.).
-

HYDROGEN PEROXIDE HR

TEST FOR HIGH LEVELS OF HYDROGEN PEROXIDE IN WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 100 mg/l

Hydrogen peroxide is used as a bleach and oxidising agent in a number of industrial processes. Applications include textile bleaching, commercial laundering and paper manufacturing. It is important in such processes to control the hydrogen peroxide level within the correct range so as to achieve the desired bleaching or oxidising effect without causing damage to the goods under treatment. Hydrogen Peroxide is also used in swimming pool water to control algae and improve clarity.

The Palintest Hydrogen Peroxide HR test provides a simple means of monitoring hydrogen peroxide levels in water over the range 0 - 100 mg/l.

Method

Hydrogen peroxide reacts with potassium iodide under acid conditions to release iodine which gives a yellow solution. A catalyst is used to speed up the rate of reaction. In the Palintest Hydrogen Peroxide HR test the reagents are provided in the form of two tablets. The test is simply carried out by adding one of each tablet to a sample of the water.

The intensity of the colour produced in the test is proportional to the hydrogen peroxide concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Hydrogen Peroxide HR Tablets

Palintest Acidifying PT Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Acidifying PT tablet and one Hydrogen Peroxide HR tablet. Crush tablets and mix to dissolve.
- 3 Select Phot 17 on Photometer.
- 4 Take Photometer reading in usual manner (see Photometer instructions).
- 5 The result is displayed as mg/l H_2O_2 .

Notes

- 1 The sample should be free of other oxidizing agents such as chlorine, bromine etc. as these react in a similar manner and will interfere with the test. It is unlikely that these oxidizing agents will be used in conjunction with hydrogen peroxide and, under normal circumstances, will not usually coexist in solution.
 - 2 For measuring low levels of hydrogen peroxide, use the Palintest Hydrogen Peroxide LR test (see PHOT.16.).
-

Photometer Method

IRON LR

TEST FOR LOW LEVELS OF IRON IN NATURAL AND TREATED WATER

**AUTOMATIC
WAVELENGTH
SELECTION****0 – 1.0 mg/l**

Iron occurs widely in nature and is found in many natural and treated waters. Iron is an objectionable constituent in both domestic and industrial water supplies. The presence of iron affects the taste of beverages and causes unsightly staining of laundered clothes, plumbing fittings, swimming pool surfaces and the like. The formation of insoluble iron deposits is troublesome in many industrial applications and in agricultural water uses such as drip feed irrigation. In industry iron salts occur through corrosion of plant and equipment, and from industrial processes.

Iron is therefore an important test for the monitoring of natural and drinking waters, for corrosion control in industry and for the checking of effluents and waste waters. The Palintest Iron LR test provides a simple test for the determination of low levels of iron in water over the range 0 - 1 mg/l Fe. The test responds to both ferrous and ferric iron and thus gives a measure of the total iron content of the water.

Method

The Palintest Iron LR test is based on a single tablet reagent containing 3-(2-Pyridyl)-5, 6-bis(4-phenyl-sulphonic acid)-1, 2, 4-triazine (PPST) formulated with a decomplexing/reducing agent in an acid buffer. The test is simply carried out by adding a tablet to a sample of the water under test. The decomplexing/ reducing agent breaks down weakly complexed forms of iron, and converts the iron from the ferric to the ferrous form. The ferrous iron reacts with PPST to form a pink coloration.

The intensity of the colour produced is proportional to the iron concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Iron LR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes 10 ml glass (PT 595)

Test Procedure

- 1 Fill the test tube with sample to the 10 ml mark.
- 2 Add one Iron LR tablet, crush and mix to dissolve.
- 3 Stand for one minute to allow full colour development.
- 4 Select Phot 18 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l Fe.

Iron Complexes

The test colour development will normally be complete within one minute. Continued colour development after this time is indicative of more strongly bound iron complexes in the water. In such cases the test solution should be stood for a longer period, say 10 - 15 minutes, until colour development is complete.

In certain industrial applications strong complexing agents are added to act as corrosion inhibitors. Moreover, some samples may contain precipitated iron complexes or particles of metallic iron. These samples will require pre-treatment by a standard laboratory procedure if it is required to determine the total iron content. The usual method of pre-treatment is acidification-with or without boiling, depending, on the nature of the sample.

To use the Palintest Iron LR test after such pre-treatment procedures, add the Iron LR tablet to the acidified sample, adjust to pH 3.5 - 4.0 using ammonia or sodium hydroxide, then take the photometer reading in the normal manner.

Photometer Method

IRON HR

TEST FOR HIGH LEVELS OF IRON IN NATURAL AND TREATED WATER

AUTOMATIC WAVELENGTH SELECTION

0 – 10 mg/l

Iron occurs widely in nature and is found in many natural and treated waters. Iron is an objectionable constituent in both domestic and industrial water supplies. The presence of iron affects the taste of beverages and causes unsightly staining of laundered clothes, plumbing fittings, swimming pool surfaces and the like. The formation of insoluble iron deposits is troublesome in many industrial applications and in agricultural water uses such as drip feed irrigation. In industry iron salts occur through corrosion of plant and equipment, and from industrial processes.

Iron is therefore an important test for the monitoring of natural and drinking waters, for corrosion control in industry and for the checking of effluents and waste waters. The Palintest Iron HR test provides a simple test for the determination of high levels of iron in water over the range 0 - 10 mg/l Fe. The test responds to both ferrous and ferric iron and thus gives a measure of the total iron content of the water.

Method

The Palintest Iron HR test is based on a single tablet reagent containing an alkaline thioglycollate. The test is carried out simply by adding a tablet to a sample of the water under test. The thioglycollate reduces ferric iron to ferrous iron and this, together with any ferrous iron already present in the sample, reacts to give a pink coloration.

The intensity of the colour produced is proportional to the iron concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Iron HR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Iron HR tablet, crush and mix to dissolve.
- 3 Stand for one minute to allow full colour development.
- 4 Select Phot 19 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l Fe.

Iron Complexes

The test colour development will normally be completed within one minute. Continued colour development after this time is indicative of more strongly bound iron complexes in the water. In such cases the test solution should be stood for a longer period, say 10 - 15 minutes, until colour development is complete.

In certain industrial applications strong complexing agents are added to act as corrosion inhibitors. Moreover some samples may contain precipitated iron complexes or particles of metallic iron. These samples will require pre-treatment by a standard laboratory procedure if it is required to determine the total iron content. The usual method of pre-treatment is acidification - with or without boiling, depending on the nature of the sample.

To use the Palintest Iron HR test after such pre-treatment procedures, add the Iron HR tablet to the acidified sample, adjust to pH 6.0 - 9.0 using ammonia or sodium hydroxide, then take the photometer reading in the normal manner.

Photometer Method

MANGANESE

TEST FOR SOLUBLE MANGANESE IN WATER

AUTOMATIC WAVELENGTH SELECTION

0 – 0.030 mg/l

Manganese-containing minerals occur widely and manganese salts are commonly found in many natural waters. Manganese is an objectionable constituent in water used for domestic purposes or industrial applications. In domestic situations, manganese will cause brown or black staining to laundry or plumbing fittings even at very low concentrations. In process applications such as paper manufacturing or textile finishing similar staining can occur. Manganese salts may impart an astringent taste to drinking water supplies, and in swimming pool applications can give an aesthetically displeasing brown coloration to the water.

In most cases where manganese salts occur naturally in the water, it will be necessary to apply special methods of removal before the water can be used for domestic or industrial purposes. The Palintest Manganese test provides an extremely sensitive method of measuring low concentrations of manganese for the assessment of natural waters and the control of manganese removal plant. The test measures total manganese over the range 0 - 0.030 mg/l.

Method

Manganese may occur in water in various different valency states. In the first stage of the Palintest method, manganese in lower valency states is oxidised to form permanganate by the action of an oxidising agent. In the second stage the permanganate formed is further reacted with leucomalachite green to form an intense turquoise coloured complex. Catalysts and inhibitors are incorporated into the tablet reagents to ensure that the colour reaction proceeds correctly and interferences are eliminated.

The intensity of colour produced in the test is proportional to the total manganese concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Manganese No 1 Tablets

Palintest Manganese No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Sample Collection

Manganese is readily absorbed onto the surfaces of sample containers. To avoid loss of manganese test sample as soon as possible after collection.

It is important, because of the extreme sensitivity of this test, to ensure that glassware used for the sample collection and test procedure is scrupulously clean. For most accurate results in laboratory use it is recommended that all glassware is acid-rinsed and then thoroughly washed out with deionised water before use.

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark (see Note 1).
- 2 Add one Manganese No 1 tablet, crush and mix to dissolve.
- 3 Add one Manganese No 2 tablet, crush and mix to dissolve then cap the tube.
- 4 Stand for 20 minutes to allow colour development (see Note 2).
- 5 Select Phot 20 on Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l Mn.

Notes

- 1 Colour formation is extremely sensitive to temperature. The sample temperature should be $20^{\circ} \pm 1^{\circ}\text{C}$ for optimum test results.
 - 2 It is important to observe the standing period of 20 minutes \pm 1 minute for optimum test results. Any continuing colour development or colour change after this period should be ignored.
-

MAGNESIUM (MAGNECOL)

TEST FOR MAGNESIUM IN WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 100 mg/l

Magnesium is a widely occurring natural element and is found in most water supplies. Magnesium salts contribute to the hardness of water and higher levels of magnesium will be found therefore in hard water areas. Scale formation in heating and steam raising equipment is promoted by the presence of magnesium salts in the water. Magnesium salts do however have a lower scale forming tendency than calcium salts.

The Palintest Magnecol test provides a simple means of measuring magnesium levels in water over the range 0 - 100 mg/l Mg.

Method

The Palintest Magnecol test is based on a simple colorimetric procedure. Magnesium reacts with an organic reagent to produce an orange coloured complex. The reagent itself is yellow and thus over the range of the test a series of colours from yellow through to orange are produced.

The colour produced in the test is indicative of the magnesium concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Magnecol Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Measuring Syringe, 1 ml (PT 361)

Test Procedure

- 1 Using the measuring syringe take a 1 ml sample of the water under test. Transfer to the round test tube and make up to the 10 ml mark with deionised water.
- 2 Add one Magnecol tablet, crush and mix to dissolve.
- 3 Stand for five minutes to allow full colour development and the slight turbidity to clear.
- 4 Select Phot 21 on Photometer for result as mg/l Mg. Select Phot 61 for result as magnesium hardness, mg/l CaCO₃.
- 5 Take photometer reading in usual manner (see Photometer instructions).

Note: To convert mg/l Mg to magnesium hardness as CaCO₃, multiply by 4.2.

Photometer Method

MOLYBDATE HR

AUTOMATIC WAVELENGTH SELECTION

TEST FOR HIGH LEVELS OF MOLYBDATE IN INDUSTRIAL WATERS AND EFFLUENTS

0 – 100 mg/l MoO₄

Formulations containing Molybdate are used as corrosion inhibitors in industrial water treatment. In particular, molybdate finds application in closed recirculating systems such as hot water heating systems and chilled water systems. Molybdate-based formulations have replaced older forms of corrosion inhibitor such as chromate.

When using molybdate treatment it is necessary to control the Molybdate concentration within specified levels depending on the application involved. Moreover since molybdates are widely used in water treatment and in industrial processes, molybdate is an increasingly important test for effluents and industrial discharges.

The Palintest Molybdate HR test provides a simple means of measuring high levels of molybdate in industrial waters and effluents and covers the range 0 - 100 mg/l MoO₄.

Method

Molybdates react with thioglycollate under acid conditions to give a yellow coloured complex. Slightly oxidising conditions are maintained during the acidification stage in order to keep the molybdate in a fully oxidised state. Under the conditions of the test, iron does not interfere and there is no significant interference from other metals at levels likely to be found in industrial water systems. The reagents are provided in the form of two tablets for maximum convenience. The test is simply carried out by adding one of each tablet to a sample of water.

The intensity of the colour produced in the test is proportional to the molybdate concentration, and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Molybdate No 1 HR Tablets

Palintest Molybdate No 2 HR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill round test tube with sample to the 10 ml mark.
- 2 Add one Molybdate No 1 HR tablet, crush and mix to dissolve.
- 3 Add one Molybdate No 2 HR tablet, crush and mix to dissolve.
- 4 Select Phot 22 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l MoO_4 .

Note

Molybdate concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-

To convert from MoO_4 to Na_2MoO_4 - multiply by 1.3

To convert from MoO_4 to Mo - multiply by 0.6

Interferences

The presence of Fe^{2+} (ferrous) or Fe^{3+} (ferric) iron at 5 mg/l causes the rapid development of a red colour in the test. This may be prevented by the treatment of the 10 ml sample with one Palintest EDTA tablet prior to carrying out the test.

NITRATE (NITRATEST)

TEST FOR NITRATE IN NATURAL, DRINKING AND WASTE WATERS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 1 mg/l N
0 – 20 mg/l N

Nitrates are normally present in natural, drinking and waste waters. Nitrates enter water supplies from the breakdown of natural vegetation, the use of chemical fertilisers in modern agriculture and from the oxidation of nitrogen compounds in sewage effluents and industrial wastes.

Nitrate is an important control test for water supplies. Drinking waters containing excessive amounts of nitrates can cause methaemoglobinaemia in bottle-fed infants (blue babies). The EEC has set a recommended maximum of 5.7 mg/l N (25 mg/l NO₃) and an absolute maximum of 11.3 mg/l N (50 mg/l NO₃) for nitrate in drinking water.

The Palintest Nitratest method provides a simple test for nitrate nitrogen over the range 0 - 1 mg/l N. The test can however be extended to cover the range 0 - 20 mg/l by a simple dilution technique.

Method

In the Palintest Nitratest method nitrate is first reduced to nitrite, the resulting nitrite is then determined by a diazonium reaction to form a reddish dye.

The reduction stage is carried out using the unique zinc-based Nitratest Powder, and Nitratest Tablet which aids rapid flocculation after the one minute contact period. The test is conducted in a special Nitratest Tube - a graduated sample container with hopper bottom to facilitate settlement and decanting of the sample.

The nitrite resulting from the reduction stage, is determined by reaction with sulphanilic acid in the presence of N-(1-naphthyl)-ethylene diamine to form a reddish dye. The reagents are provided in a single Nitricol tablet which is simply added to the test solution.

The intensity of the colour produced in the test is proportional to the nitrate concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Nitratest Powder (Spoon Pack)

Palintest Nitratest Tablets

Palintest Nitricol Tablets

Palintest Nitratest Tube, 20 ml (PT 526)

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml (PT 595)

Test Procedure

- 1 Fill the Nitratest Tube with sample to the 20 ml mark.
- 2 Add one level spoonful of Nitratest Powder and one Nitratest tablet. Do not crush the tablet. Replace screw cap and shake tube well for one minute.
- 3 Allow tube to stand for about one minute then gently invert three or four times to aid flocculation. Allow tube to stand for two minutes or longer to ensure complete settlement.
- 4 Remove screw cap and wipe around the top of the tube with a clean tissue. Carefully decant the clear solution into a round test tube, filling to the 10 ml mark.
- 5 Add one Nitricol tablet, crush and mix to dissolve.
- 6 Stand for 10 minutes to allow full colour development.
- 7 Select Phot 23 on Photometer for result as mg/l N, or Phot 63 for result as mg/l NO_3 .
- 8 Take Photometer reading in usual manner (see Photometer instructions).

Note

To convert mg/l N to mg/l NO_3 multiply result by 4.4.

Concentrations of nitrate greater than 1.0 mg/l may be determined by diluting the original sample with deionised water. The test can be conveniently carried out over a range 0 - 20 mg/l N as follows :-

Take a clean Nitratest Tube. Add 1 ml of sample using a pipette or graduated dropper. Fill the Nitratest Tube to the 20 ml mark with deionised water. Continue the test procedure as given in steps 2 to 9 above. Multiply the chart reading obtained by 20 to obtain the nitrate concentration in the original sample.

Nitrite Correction

The Nitratest method will also respond to any nitrite present in the sample. In most natural and drinking waters the amount of nitrite will be small in comparison to the nitrate concentration. If it is desired to correct for nitrite, determine nitrite concentration (as mg/l N) in the prescribed manner (see PHOT.24.) and deduct from the nitrate concentration (as mg/l N) obtained from the Nitratest procedure.

NITRITE (NITRICOL)

TEST FOR NITRITE IN NATURAL, DRINKING AND WASTE WATERS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 0.5 mg/l N
(0 – 1.6 mg/l NO₂)

Nitrites are found in natural waters as an intermediate product in the nitrogen cycle. Nitrite is harmful to fish and other forms of aquatic life and the nitrite level must be carefully controlled in water used for fish farms and aquariums. The nitrite test is also applied for pollution control in waste waters, and for the monitoring of drinking water.

The Palintest Nitricol test provides a simple method of measuring Nitrite Nitrogen levels over the range 0 - 0.5 mg/l N. Higher levels can be determined by diluting the sample.

Method

Nitrites in acid solution react with sulphanilic acid. The resulting diazo compound couples with N-(1-naphthyl)-ethylene diamine to form a reddish dye. The Palintest Nitricol method features a single tablet reagent containing both of these reagents in an acidic formulation. The test is simply carried out by adding a tablet to a sample of the water under test.

The intensity of the colour produced in the test is proportional to the nitrite concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Nitricol Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill round test tube with sample to the 10 ml mark.
- 2 Add one Nitricol tablet, crush and mix to dissolve.
- 3 Stand for 10 minutes to allow full colour development.
- 4 Select Phot 24 on Photometer for result as mg/l N, or Phot 64 for result as mg/l NO₂).
- 5 Take Photometer reading in usual manner (see Photometer instructions).
To convert from mg/l N to mg/l NO₂ multiply result by 3.3.

Photometer Method

OZONE

**AUTOMATIC
WAVELENGTH
SELECTION**

TEST FOR OZONE IN WATER

0 – 2.0 mg/l

Ozone is used for the disinfection of swimming pool water, and in many other water treatment systems. In swimming pool water treatment ozone is normally introduced into the circulation system and then removed prior to the re-entry of the water to the pool. In other water treatment systems an ozone residual may be maintained in the water. In all cases accurate measurement of ozone residual is essential for the control of the system or to ensure that the ozone has been removed.

The Palintest DPD Ozone method provides a simple means of measuring ozone residuals up to a level of 2.0 mg/l. Other disinfectants such as chlorine and bromine are frequently used in conjunction with ozone. Supplementary procedures are therefore provided for the separate determination of these residuals.

Method

The Palintest Ozone test uses the DPD method now internationally recognised as the standard method of testing for disinfectant residuals. In the DPD method the reagents are provided in tablet form for maximum convenience and simplicity of use.

Ozone reacts with diethyl-p-phenylene diamine (DPD) in buffered solution in the presence of potassium iodide to produce a pink coloration. The intensity of the colour is proportional to the ozone concentration and is measured using a Palintest Photometer.

For the determination of ozone in the presence of chlorine or bromine, a supplementary procedure using glycine is used. The glycine destroys the ozone in the sample and the colour produced in the DPD test thus corresponds to the chlorine or bromine only. The ozone content is thus obtained by the difference between the test readings with and without glycine.

Reagents and Equipment

Palintest DPD No 4 Clear Tablets

Palintest DPD Glycine Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Ozone Residuals

The photometer is programmed for both ozone and the correction procedure. Use program **Phot 25** Ozone (+ Chlorine), then select the 'Follow On' option on screen to continue test using program **Phot 26** Correction Procedure. The corrected ozone residual is calculated automatically and displayed.

Test Procedure

- 1 Rinse test tube with sample leaving two to three drops of sample in the tube.
- 2 Add one DPD No 4 tablet, crush tablet and then fill the test tube with sample to the 10 ml mark. Mix to dissolve tablet.
- 3 Gently invert the tube to remove any bubbles from the inner walls of the tube
- 4 Select Phot 25 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result represents the **ozone** residual as milligrams per litre.

The test may be terminated at this stage for systems treated with ozone alone. For waters containing both ozone and chlorine or bromine, a correction should be made as indicated in the following section. Select 'Follow On' option on screen to continue the test program.

Correction for Chlorine or Bromine

- 1 Fill a test tube with sample to the 10 ml mark. Add one DPD Glycine tablet, crush and mix to dissolve.
 - 2 Take a second clean test tube and add two to three drops of solution from the first tube. Add one DPD No 4 tablet, crush and then add the remainder of the solution to make up to the 10 ml mark. Mix to dissolve tablet.
 - 3 Gently invert the tube to remove any bubbles from the inner walls of the tube
 - 4 Take Photometer reading in the usual manner.
 - 5 The Photometer carries out the necessary calculation and displays the corrected ozone residual as mg/l O₃.
-

pH (PHENOL RED)

TEST FOR pH VALUE OF WATER AND AQUEOUS SOLUTIONS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

6.8 – 8.4

pH measurement is one of the tests most frequently carried out on water and aqueous solutions. The phenol red indicator method provides a simple colorimetric means of pH determination for neutral and slightly alkaline waters over the range 6.8 - 8.4 units. The Phenol Red pH test is particularly applicable to testing swimming pools and spas.

Method

The Palintest Phenol Red test uses a tablet reagent containing the precise amount of phenol red indicator required for the test. Phenol red reacts in water at different pH values over the range 6.8 - 8.4 to produce a distinctive range of colours from yellow to red. The colour of the test solution is indicative of the pH value and is measured using a Palintest Photometer.

Phenol red tablets contain a dechlorinating agent so that the test can be carried out in water containing normal levels of chlorine or other disinfectant residuals.

Reagents and Equipment

Palintest Phenol Red Clear Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Phenol Red tablet, crush and mix to dissolve.
- 3 Select Phot 27 on Photometer.
- 4 Take Photometer reading in usual manner (see Photometer instructions).

Notes

- 1 The colour range of the phenol red test is yellow, through orange, to red. The formation of an intense purple coloration shows that the indicator has been affected by high chlorine or other disinfectant residuals. In such cases the result should be disregarded.
 - 2 Phenol red does not show any further colour change at pH values below 6.8 or above 8.4. Note therefore that when such values are recorded this could indicate that the sample has a much lower or much higher pH value.
 - 3 Ionic strength, temperature and other water factors may have an effect on pH readings. This test has been calibrated for conditions most likely to be encountered in a typical swimming pool at 30°C.
-

Photometer Method

PHOSPHATE LR

TEST FOR LOW LEVELS OF PHOSPHATE IN NATURAL AND DRINKING WATERS

**AUTOMATIC
WAVELENGTH
SELECTION****0 – 4.0 mg/l PO₄****0 – 1.3 mg/l P**

Phosphates are extensively used in detergent formulations and washing powders. Phosphates also find widespread application in the food processing industry and in industrial water treatment processes. Agricultural fertilizers normally contain phosphate minerals and phosphates also arise from the breakdown of plant materials and in animal wastes.

Phosphates can therefore enter water courses through a variety of routes - particularly domestic and industrial effluents and run-off from agricultural land. Phosphate is an important control test for natural and drinking waters.

Whilst phosphates are not generally considered harmful for human consumption, they do exhibit a complex effect on the natural environment. In particular phosphates are associated with eutrophication of water and with rapid unwanted plant growth in rivers and lakes. Phosphates present in natural water pass through into drinking water supplies.

The Palintest Phosphate LR test provides a simple method of measuring phosphate levels over the range 0 - 4 mg/l PO₄. For drinking water the EEC has set a guide level of 0.5 mg/l PO₄ (0.4 mg/l P₂O₅) and a maximum admissible concentration of 6.7 mg/l PO₄ (5 mg/l P₂O₅).

Method

In the Palintest Phosphate LR method, the phosphate reacts under acid conditions with ammonium molybdate to form phospho-molybdic acid. This compound is reduced by ascorbic acid to form the intensely coloured 'molybdenum blue' complex. A catalyst is incorporated to ensure complete and rapid colour development, and an inhibitor is used to prevent interference from silica. The reagents are provided in the form of two tablets for maximum convenience. The test is simply carried out by adding one of each tablet to a sample of the water.

The intensity of the colour produced in the test is proportional to the phosphate concentration, and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Phosphate No 1 LR Tablets

Palintest Phosphate No 2 LR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Phosphate No 1 LR tablet, crush and mix to dissolve.
- 3 Add one Phosphate No 2 LR tablet, crush and mix to dissolve.
- 4 Stand for 10 minutes to allow full colour development.
- 5 Select Phot 28 on Photometer for result as mg/l PO_4 , or Phot 70 for result as mg/l P.
- 6 Take Photometer reading in usual manner (see Photometer instructions).

Note

Phosphate concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-

To convert from PO_4 to P_2O_5 - multiply by 0.75

To convert from PO_4 to P - multiply by 0.33

PHOSPHATE HR

**TEST FOR HIGH LEVELS
OF PHOSPHATE IN
BOILER WATER**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 100 mg/l

Phosphates are extensively used for treating water in boilers and steam raising plant. Phosphates are added to control the deposition of sediment and deposits within the boiler. It is an essential part of the treatment programme to monitor the phosphate level to ensure this is within the correct range of deposition control.

The Palintest Phosphate HR test provides a simple method of measuring phosphate levels in boiler waters over the range 0 - 100 mg/l PO₄.

Method

The Palintest Phosphate HR test is based on the vanadomolybdate method. The distinct advantage of the Palintest method is that all reagents required are provided in the form of a test tablet. The test is carried out simply by adding a single tablet to a sample of the boiler water. A supplementary tablet may be optionally used for the removal of Silica interference.

In the test phosphates react with ammonium molybdate, in the presence of ammonium vanadate, to form the yellow phosphovanadomolybdate. The intensity of the colour produced in the test is proportional to the phosphate concentration and is measured using a Palintest Photometer.

Sample Collection

Samples drawn from boiler sampling points may be hot and contain particulate matter. Prior to analysis samples should be cooled to below 25°C and filtered through a Whatman No 42 filter paper.

Reagents and Equipment

Palintest Phosphate HR Tablets

Palintest Phosphate SR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 IN THE CASE OF SAMPLES CONTAINING SILICA (>20 mg/l SiO₂) ONLY :-
Add one Phosphate SR tablet, crush and mix to dissolve.
- 3 Add one Phosphate HR tablet, crush and mix to dissolve.
- 4 Stand for 10 minutes to allow full colour development.
- 5 Select Phot 29 on Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l PO₄.

Note

Phosphate SR tablets are supplied in the Phosphate HR Starter Pack (PM 114). They are subsequently available as an optional extra (AT 116) for use with Phosphate HR Replacement Reagent Packs.

POTASSIUM

TEST FOR POTASSIUM IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 12.0 mg/l

Potassium is an abundant natural element. However in fresh water potassium levels are normally low. Higher levels can be observed in brackish waters. The guide level prescribed for drinking water supplies under the EEC Regulations is 10 mg/l.

The Palintest Potassium test provides a simple means of testing potassium levels in water over the range 0 - 12.0 mg/l.

Method

The Palintest Potassium test is based on a single tablet reagent containing sodium tetraphenylboron. Potassium salts react with sodium tetraphenylboron to form an insoluble white complex. At the potassium levels encountered in the test, this is observed as a turbidity in the test sample. The degree of turbidity is proportional to the potassium concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Potassium K Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Potassium K tablet, crush and mix to dissolve. A cloudy solution indicates the presence of potassium.
- 3 Select Phot 30 on Photometer.
- 4 Take Photometer reading in usual manner (see Photometer instructions).
- 5 The result is displayed as mg/l K.

Photometer Method

SILICA LR

TEST FOR SILICA IN NATURAL, TREATED AND INDUSTRIAL WATER

AUTOMATIC WAVELENGTH SELECTION

0 – 4.0 mg/l SiO₂

Silicon, in the form of silica, is one of the earth's most abundant elements. Silicon is found widely in natural waters as colloidal silica or soluble silicates.

Silica and silicates do not normally cause any problems in water intended for domestic consumption. However their presence is undesirable in water used in a variety of industrial applications. This is because of the tendency of such water to form a hard scale on equipment. Silica and silicate containing waters are particularly troublesome in steam generating plant such as high pressure boilers since silica scale can build up on turbine blades.

The Palintest Silica LR test provides a simple means of measuring silica and silicate levels in natural, treated and industrial waters over the range 0 - 4 mg/l SiO₂.

Method

Ammonium molybdate reacts with silica under acid conditions to produce molybdosilicic acid. In the presence of a reducing agent, this compound is reduced to form an intense blue complex. Phosphate reacts in a similar manner. Interference by phosphate is prevented by introducing a reagent which destroys any molybdophosphoric acid which may form.

The reagents for the method are provided in tablet form and the test is carried out simply by adding tablets to a sample of water. The intensity of the colour produced in the test is proportional to the silica concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Silica No 1 Tablets

Palintest Silica No 2 Tablets

Palintest Silica PR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Instructions

- 1 Fill test tube with sample to the 10 ml mark
- 2 Add one Silica No 1 tablet, crush and mix to dissolve. Stand for five minutes to allow the silica to react.
- 3 Add one Silica PR tablet, crush and mix to dissolve. (This stage may be omitted if the sample is known to be completely free of phosphate).
- 4 Add one Silica No 2 tablet, crush and mix to dissolve. Stand for one minute to allow full colour development.
- 5 Select Phot 31 on Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l SiO_2 .

Note

For testing high levels of Silica the Palintest Silica HR test should be used. The range of this test is 0 - 100 mg/l (see Test Instruction Phot 56).

SULPHATE

TEST FOR SULPHATE IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 200 mg/l

Sulphates occur naturally in many waters. Sulphates are introduced into treated waters by the use of such chemicals as aluminium sulphate, sodium bisulphate (dry acid) and sulphuric acid. The presence of high levels of sulphate can be undesirable for a number of reasons.

In industrial waters containing sulphate localised corrosion of iron, steel and aluminium in plant and pipe work can occur through the action of sulphate-reducing bacteria. These bacteria, which generate sulphides, cause a characteristic pitting of the metal surface.

High sulphate levels can also cause damage to concrete and cement based materials through the formation of calcium sulphotoaluminate. This causes expansion and crumbling of the cement. It can affect concrete structures and pipes in water distribution systems carrying sulphate-bearing ground waters; and can attack grouting in tiled swimming pools using sodium bisulphate for pH adjustment.

The Palintest Sulphate test provides a simple method of measuring sulphates over the range 0 - 200 mg/l SO_4 . Higher levels may be determined by diluting the sample.

Method

The Palintest Sulphate test is based on a single tablet reagent containing barium chloride in a slightly acidic formulation. Barium salts react with sulphates to form insoluble barium sulphate. At the sulphate levels encountered in the test, this is observed as turbidity in the test sample. The degree of turbidity is proportional to the sulphate concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Sulphate Turb Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Sulphate Turb tablet, crush and mix to dissolve. A cloudy solution indicates the presence of sulphate.
- 3 Stand for five minutes then mix again to ensure uniformity.
- 4 Select Phot 32 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l SO₄.

Caution

Palintest Sulphate (Turb) tablets each contain 20 mg Barium Chloride. These tablets are harmful if ingested. Avoid handling tablets whenever possible and wash hands after use.

SULPHIDE

TEST FOR SULPHIDE IN NATURAL AND WASTE WATERS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 0.5 mg/l

Natural waters containing dissolved hydrogen sulphide and other sulphides are found in certain parts of the world, particularly in areas having hot springs. Sulphides are constituents of many industrial wastes such as those from tanneries, gas plants and chemical works. Sulphides can be toxic to fish and aquatic life; and their presence in water supplies gives rise to undesirable tastes and odours.

The Palintest Sulphide Test provides a simple method of measuring total available sulphide over the range 0 - 0.5 mg/l and is particularly applicable to natural and drinking waters. Higher levels, such as those found in effluents and waste waters, can be determined by diluting the sample.

Method

This simplified method for the determination of sulphide is based on a reagent containing diethyl-p-phenylene diamine (DPD) and potassium dichromate. Sulphide reacts with this reagent in acid solution to produce a blue coloured complex. In the absence of sulphide the reagent produces a pink colour. Chlorine, and other oxidizing agents which normally react with DPD, do not interfere with the test. The reagents are provided in the form of two tablets and the test is simply carried out by adding one of each tablet to a sample of the water.

The colour produced is indicative of the sulphide concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Sulphide No 1 Tablets

Palintest Sulphide No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Sample Collection

To prevent loss of sulphide collect the sample carefully with a minimum of agitation or aeration. Test the sample as soon as possible after collection.

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Sulphide No 1 tablet and one Sulphide No 2 tablet. Crush and mix gently to dissolve the tablets. Gentle mixing is essential to avoid loss of sulphide.
- 3 Stand for 10 minutes to allow full colour development.
- 4 Select Phot 33 on Photometer.
- 5 Take Photometer reading in usual manner (see Photometer instructions).
- 6 The result is displayed as mg/l S.

To convert from mg/l S to mg/l H₂S - multiply result by 1.06

SULPHITE (SULPHITEST)

TEST FOR SULPHITE IN BOILER WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 500 mg/l Na_2SO_3

Oxygen is a major cause of corrosion in boilers and steam raising plant. Sodium sulphite and catalysed sulphite formulations are extensively used as oxygen scavengers in boiler water treatment.

The Palintest Sulphitest test provides a simple means of measuring sulphite levels for the control of such treatments in boiler plant. The test covers the range 0 - 500 mg/l Na_2SO_3 .

Method

The Palintest Sulphitest method is based on a colorimetric procedure involving the reduction of an indicator dye. Sulphites react with the indicator dye under buffered conditions to destroy the original purple coloration. With increasing sulphite concentrations a range of colours from purple to colourless is produced.

An advantage of the Sulphitest method is that it does not respond to other reducing species as do traditional iodometric methods.

The degree of colour loss observed in the test is proportional to the sulphite concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Sulphitest No 1 Tablets

Palintest Sulphitest No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Fill the test tube with sample to the 10 ml mark.
- 3 Add one Sulphitest No 1 tablet, crush and mix to dissolve.
- 4 Add one Sulphitest No 2 tablet, crush and mix to dissolve. Cap tube immediately.
- 5 Stand for two minutes to allow full colour reduction to take place.
- 6 Select Phot 34 on the Photometer.
- 7 Take Photometer reading in the usual manner (see Photometer instructions).
- 8 The result is displayed as mg/l Na_2SO_3 .

Note

Equipment should be washed immediately after use, with a detergent if necessary, to prevent staining.

Sulphite concentrations may be expressed as mg/l SO_3 . To convert from mg/l Na_2SO_3 to mg/l SO_3 multiply by 0.63.

Interferences

- 1 This test is not affected by the presence of other reducing species such as nitrite (up to 200 mg/l) ferrous iron (up to 20 mg/l) and sulphide (up to 10 mg/l); or by the presence of polyacrylates.
 - 2 Chlorine up to 250 mg/l does not cause interference. However, since sulphite and chlorine do not normally co-exist, the test will not usually be carried out in the presence of chlorine.
 - 3 The test gives low results if used in the presence of tannic acid or tannin treated waters.
-

ZINC

TEST FOR ZINC IN NATURAL AND TREATED WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 4.0 mg/l

Zinc compounds are used as corrosion inhibitors in industrial cooling water systems and similar applications. Control of the zinc level is an important aspect of corrosion control in such systems. Zinc and zinc containing alloys are widely used in industry and zinc salts are commonly found in industrial effluents.

The Palintest Zinc test provides a simple means of testing zinc levels over the range 0 - 4 mg/l and is suitable for testing cooling waters and industrial effluents, and for the monitoring of natural and drinking waters.

Method

Zinc reacts with 5-(o-carboxyphenyl)-1-(2-hydroxy-5-sulphophenyl)-3-phenyl-formazan (Zincon) in alkaline solution to give an intense blue colour. The reagent itself is orange in solution. At different zinc levels a distinctive colour range from orange through purple to blue is produced. In the Palintest Zinc test a tablet reagent containing both Zincon and an alkaline buffer is used for maximum convenience. The test is simply carried out by adding a tablet to a sample of the water. Samples containing high chlorine residuals are pre-treated with a special dechlorinating tablet to prevent bleaching of the test colours.

The colour produced in the test is indicative of the zinc concentration and is measured using a Palintest Photometer.

Copper reacts in a similar manner to zinc and a correction procedure using EDTA is applied to those samples which contain both zinc and copper. EDTA destroys the colour complex formed with zinc.

Reagents and Equipment

Palintest Zinc Tablets

Palintest Zinc-Dechlor Tablets

Palintest EDTA Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Residuals

The photometer is programmed for both zinc and the copper correction procedure. Use program **Phot 35** Zinc (+ Copper), then select the 'Follow On' option on screen to continue test for program **Phot 36** Corrected Zinc. The corrected zinc value is calculated automatically.

Test Procedure

- 1 Fill test tube to the 10 ml mark.
 - 2 IN THE CASE OF CHLORINE CONTAINING SAMPLE ONLY :-
Add one Zinc-Dechlor tablet, crush and mix to dissolve.
 - 3 Add one Zinc tablet, crush and mix to dissolve.
 - 4 Allow the sample to stand for five minutes then mix again to ensure complete dissolution of the indicator.
 - 5 Select Phot 35 on Photometer.
 - 6 Take Photometer reading in usual manner (see Photometer instructions). The result is displayed as mg/l Zn.
 - 7 FOR COPPER CONTAINING SAMPLES ONLY :-
Continue the test on the same test portion. Select the 'Follow On' option on screen to continue the test program.
 - 8 Add one EDTA tablet, crush and mix to dissolve.
 - 9 Take Photometer reading in usual manner.
 - 10 The photometer displays the corrected zinc concentration as mg/l Zn.
-

ALKALINITY M and P (ALKAPHOT M and P)

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

**TESTS FOR ALKALINITY M and P IN BOILER
WATER AND OTHER INDUSTRIAL WATERS**

0 – 500 mg/l CaCO₃

The Alkalinity of water is caused by the presence of alkaline substances such as hydroxides, carbonates, bicarbonates and, to a lesser extent, silicates and phosphates. Quantitatively alkalinity is the capacity of the water to react with acid to a specified pH end point. The value obtained will depend on the pH indicator used. Two measures of alkalinity are conventionally applied - Alkaphot M (Alkalinity to methyl orange) and Alkaphot P (Alkalinity to phenolphthalein).

Alkalinity is an important test parameter in a number of industrial water uses, notably in boiler water treatment. Boilers and steam raising plant are normally operated under conditions of high alkalinity in order to minimise corrosion and the monitoring of alkalinity is an important control test.

The Palintest Alkaphot M and Alkaphot P tests provide a simple means of checking Alkalinity M and Alkalinity P levels over the range 0 - 500 mg/l CaCO₃. The tests are particularly suited to boiler and industrial waters. The alkalinities specifically due to carbonates, bicarbonates and hydroxides can be calculated from the various data obtained.

Method

The Palintest Alkaphot M and Alkaphot P tests are both based on unique colorimetric methods. These methods offer considerable advantages over the titrimetric methods traditionally used for measuring these parameters.

The tests are each based on the use of a single tablet reagent containing a precisely standardised amount of acid combined with a colour indicator. The tests are simply carried out by adding the appropriate tablet to a sample of the water under test. Over the alkalinity range of each test a distinctive series of colours is produced - from yellow through green to blue in the case of the Alkaphot M test and from colourless to purple in the case of the Alkaphot P test.

The colour produced in each of the tests is indicative of the alkalinity and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Alkaphot M Tablets

Palintest Alkaphot P Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure - Alkaphot M

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Fill the test tube to the 10 ml mark with sample.
- 3 Add one Alkaphot M tablet, crush and mix. Ensure all particles are dissolved.
- 4 Select Phot 37 on the Photometer.
- 5 Take Photometer reading in the usual manner.
- 6 The result is displayed as mg/l CaCO_3 .

Test Procedure - Alkalinity P

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Fill the test tube to the 10 ml mark with sample.
- 3 Add one Alkaphot P tablet, crush and mix to dissolve.
- 4 Stand two minutes to allow complete colour development.
- 5 Select Phot 38 on the Photometer.
- 6 Take Photometer reading immediately in the usual manner.
- 7 The result is displayed as mg/l CaCO_3 .

Alkalinity Relationships

From the results obtained from the foregoing procedures it is possible to classify the sample into the three main chemical forms of alkalinity present in most waters, namely hydroxides, carbonates and bicarbonates. This calculated relationship assumes the absence of other weak forms of alkalinity and also assumes that hydroxides and bicarbonates are not compatible in the same sample. The chemical forms of alkalinity, expressed as mg/l CaCO_3 are calculated by the following equations :-

- | | |
|-----------------------------------|-----------------------------------|
| a) If Alkalinity P = 0 | b) If Alkalinity P > 0 and M > 2P |
| Then Bicarbonate = M | Then Bicarbonate = M - 2P |
| Carbonate = 0 | Carbonate = 2P |
| Hydroxide = 0 | Hydroxide = 0 |
| | |
| c) If Alkalinity P > 0 and M < 2P | |
| Then Bicarbonate = 0 | |
| Carbonate = 2M - 2P | |
| Hydroxide = 2P - M | |

Where P and M are the results of the Alkaphot P and Alkaphot M tests respectively.

Note

The expression of alkalinity results sometimes causes confusion. It is normal practice to express the result as mg/l CaCO_3 (calcium carbonate). This is merely a convention to allow the comparison of different results and does not necessarily indicate that the alkalinity is present in the water in this form. The different chemical forms of alkalinity have been referred to in the test instructions.

Photometer Method

IRON MR

AUTOMATIC WAVELENGTH SELECTION

TEST FOR IRON IN NATURAL, TREATED AND INDUSTRIAL WATERS

0 – 5.0 mg/l

Iron occurs widely in nature and is found in many natural and treated waters. Iron is an objectionable constituent in both domestic and industrial water supplies. The presence of iron affects the taste of beverages and causes unsightly staining of laundered clothes, plumbing fittings, swimming pool surfaces and the like. The formation of insoluble iron deposits is troublesome in many industrial applications and in the agricultural water uses such as drip feed irrigation. In industry iron salts occur through corrosion of plant and equipment, and from industrial processes.

Iron is therefore an important test for the monitoring of natural and drinking waters, for corrosion control in industry and for the checking of effluents and waste waters. The Palintest Iron MR test provides a simple test for the determination of both ferrous and ferric iron. It is capable of dissolving colloidal and particulate iron and thus gives a measure of the total iron content of the water.

Method

In the Palintest Iron MR method iron is reduced to the ferrous form and then reacted with 1,10-phenanthroline to form an orange coloured complex. A decomplexing agent is incorporated into the reagent system in order to break down complexed forms of iron. The test is simply carried out by adding tablet reagents to a sample of the water under test.

The intensity of the colour produced is proportional to the iron concentration and is measured using a Palintest Photometer.

Interference can occur in industrial waters treated with molybdate and nitrite based treatment products. A supplementary reagent can be used to prevent this interference.

Reagents and Equipment

Palintest Iron MR No 1 Tablets

Palintest Iron MR No 2 Tablets

Palintest Citrate IR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill the test tube with sample to the 10 ml mark.
- 2 Add one Iron MR No 1 tablet, crush and mix to dissolve.
- 3 Add one Iron MR No 2 tablet, crush and mix to dissolve.
- 4 Stand for 10 minutes to allow full colour development.
- 5 Select Phot 39 on Photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l Fe.

Interferences

Hardness 500 mg/l CaCO_3 , Silica 150 mg/l SiO_2 and Copper 3 mg/l Cu do not interfere with the test. Chromium 10 mg/l may cause slightly high results.

Nitrite greater than 50 mg/l NO_2 causes low results and molybdate at any concentration causes precipitation. The pretreatment procedures described below using Citrate IR tablets remove interference from nitrite up to 500 mg/l NO_2 and molybdate up to 20 mg/l MoO_4 . This pretreatment does however reduce the tolerance to chromium and is not recommended therefore for chromium containing samples.

Pretreatment Procedure Using Citrate IR Tablets

Samples Containing Nitrite :-

- 1 Fill the test tube with sample to the 10 ml mark.
- 2 Add one Citrate IR tablet, crush and mix to dissolve. Ensure all particles are dissolved.
- 3 Continue the test as described in the test procedure from Stage 2 above but allow the tube to stand for 15 minutes to allow full colour development before taking the photometer reading.

Samples Containing Molybdate :-

- 1 Fill the test tube with sample to the 10 ml mark.
- 2 Add one Iron MR No 1 tablet, crush and mix to dissolve.
- 3 Add one Citrate IR tablet, crush and mix to dissolve. Ensure all particles are dissolved.
- 4 Continue the test as described in the test procedure from Stage 3 but allow the tube to stand for 15 minutes to allow full colour development before taking the reading.

Photometer Method

BORON

AUTOMATIC WAVELENGTH SELECTION

TEST FOR BORON IN WATER

0 – 2.5 mg/l

Boron is an abundant natural element. It usually occurs in the form of calcium or sodium borate.

Boron is an essential element for plant growth. However, some crops can be highly sensitive to boron at other than very low levels. For this reason the boron level in irrigation water should be checked. Borates are widely used in the industrial processes and boron can occur in effluent discharges. For drinking water supplies the guide level prescribed under the EEC Regulations is 1.0 mg/l.

The Palintest Boron test provides a simple means of testing boron levels in drinking water, irrigation water and effluents over the range 0 - 2.5 mg/l.

Method

Boron in the form of borates, react with azomethine under slightly acidic conditions to form a yellow coloured complex. In the Palintest Boron method two test tablets are used to provide the necessary buffer and indicator reagents. A sequestering agent is incorporated to eliminate any interference from cations. The test is simply carried out by adding one of each tablet to a sample of the water. The intensity of colour produced in the test is proportional to the boron concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Boron No 1 Tablets

Palintest Boron No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill the test tube with sample to the 10 ml mark.
- 2 Add one Boron No 1 tablet, crush and mix to dissolve.
- 3 Add one Boron No 2 tablet, crush and mix to dissolve.
- 4 Stand for exactly 20 minutes to allow full colour development.
- 5 Select Phot 40 on photometer.
- 6 Take Photometer reading in usual manner (see Photometer instructions).
- 7 The result is displayed as mg/l B.

Note

For optimum results this test should be carried out at a temperature of 20°C ± 2°C. Temperatures outside this range can be 'normalised' using the following correction factors:

Temperature, °C	Correction Factor
10	1.09
15	1.05
20	1.0
25	0.94
30	0.88

PHOT 40.Cal Chart

BORON CALIBRATION CHART

For use with Photometer 7100/7500

	mg/l B								450nm	
%T	9	8	7	6	5	4	3	2	1	0
90	-	-	-	-	-	-	-	-	-	-
80	-	-	-	-	-	-	-	-	-	-
70	-	-	0.00	0.02	0.04	0.06	0.09	0.11	0.13	0.15
60	0.17	0.20	0.22	0.24	0.27	0.29	0.32	0.35	0.37	0.40
50	0.43	0.45	0.48	0.51	0.54	0.57	0.60	0.63	0.67	0.70
40	0.73	0.77	0.81	0.84	0.88	0.92	0.96	1.01	1.05	1.10
30	1.15	1.20	1.25	1.30	1.36	1.42	1.48	1.54	1.61	1.68
20	1.76	1.83	1.92	2.00	2.09	2.18	2.27	2.36	2.45	2.50
10	-	-	-	-	-	-	-	-	-	-
0	-	-	-	-	-	-	-	-	-	--

HYDRAZINE

TEST FOR HYDRAZINE IN INDUSTRIAL WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 0.5 mg/l N₂H₄

Hydrazine is used as an oxygen scavenger in high pressure boilers and steam raising plant. Hydrazine is particularly advantageous in that it does not contribute solids to the boiler water.

The Palintest Hydrazine test provides a simple means of measuring hydrazine levels in boiler feed water and boiler water over the range 0 - 0.5 mg/l.

Method

The Palintest hydrazine test uses a special reagent powder containing p-dimethylaminobenzaldehyde in an acidic formulation. Hydrazine reacts with this reagent to produce a yellow coloration. The intensity of the colour produced is proportional to the hydrazine concentration and is measured using the Palintest Photometer.

Reagents and Equipment

Palintest Hydrazine Test Powder

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Scoop, 1g approx (PT 697)

Test Instructions

- 1 Filter sample if necessary to obtain clear solution.
- 2 Take two test tubes A and B.
- 3 Fill test tube A with sample to the 10 ml mark.
- 4 Fill test tube B with deionised water to the 10 ml mark.
- 5 To each tube add one level scoop (1g) of Hydrazine Test Powder. Mix to dissolve and cap tubes.
- 6 Stand for two minutes to allow full colour development.
- 7 Select Phot 41 on photometer.
- 8 Take photometer reading of Tube A in usual manner (see photometer instructions).
- 9 Use Tube B as the Blank to set the instrument.
- 10 The result is displayed as mg/l N₂H₄.

Photometer Method

MOLYBDATE LR

AUTOMATIC WAVELENGTH SELECTION

TEST FOR LOW LEVELS OF MOLYBDATE IN INDUSTRIAL WATERS AND EFFLUENTS

0 – 20 mg/l MoO₄

Formulations containing molybdate are used as corrosion inhibitors in industrial water treatment. In particular, low level molybdate treatment finds application in cooling systems. Molybdate based formulations have replaced older forms of corrosion inhibitors.

When using molybdate treatment it is necessary to control the molybdate concentration within specified levels depending on the application involved. Moreover, since molybdates are widely used in water treatment and in industrial processes, molybdate is an increasingly important test for effluents and industrial discharges.

The Palintest Molybdate LR test provides a simple means of measuring low levels of molybdate in industrial waters and effluents and covers the range 0 - 20 mg/l MoO₄ (0 – 12 mg/l Mo).

Method

Molybdates react with a dihydroxybenzene disulphonic acid salt under slightly acid conditions to give a yellow coloured complex. Under the conditions of the test, iron does not interfere and there is no significant interference from other metals at levels likely to be found in industrial water systems (see Notes). The reagents are provided in the form of two tablets for maximum convenience. The test is simply carried out by adding one of each tablet to a sample of water.

The intensity of the colour produced in the test is proportional to the molybdate concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Molybdate No 1 LR Tablets

Palintest Molybdate No 2 LR Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Photometer Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Fill test tube with sample to the 10 ml mark.
- 3 Add one Molybdate No 1 LR tablet, crush and mix to dissolve.
- 4 Add one Molybdate No 2 LR tablet, crush and mix to dissolve.
- 5 Stand for two minutes to allow full colour development.
- 6 Select Phot 42 on the Photometer.
- 7 Take Photometer reading in the usual manner (see Photometer instructions).
- 8 The result is displayed as mg/l MoO_4 .

Note

Molybdate concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of results :-

To convert from MoO_4 to Na_2MoO_4 - multiply by 1.3

To convert from MoO_4 to Mo - multiply by 0.6.

Interferences

- 1 Copper 20 mg/l, zinc 20 mg/l, phosphate 100 mg/l and calcium 200 mg/l do not interfere in this test.
 - 2 Iron 10 mg/l and chlorine 10 mg/l cause slightly high blank readings equivalent to 0.6 mg/l Mo. However, they do not cause any interference in samples which contain molybdate.
-

NITRITE (NITRIPHOT)

TEST FOR NITRITE IN COOLING WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 1500 mg/l NaNO₂

Nitrites and nitrite-based formulations are widely used for corrosion control in cooling water systems. The Palintest Nitriphot test provides a simple means of measuring nitrite for the control of such treatment products in cooling water. The test covers the range 0 – 1500 mg/l NaNO₂.

Method

The Palintest Nitriphot method is based on a colorimetric procedure using an iodide containing reagent system. Nitrites catalyse the oxidation of the iodide to iodine under mildly acid conditions to produce a brown coloration. Over the range of the test a series of colours from colourless through yellow to brown are produced.

The intensity of the colour produced in the test is proportional to the nitrite concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Nitriphot No 1 Tablets

Palintest Nitriphot No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Measuring Syringe, 1 ml (PT 361)

Test Procedure

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Using the measuring syringe take 1 ml of the sample. Transfer to the test tube and make up to the 10 ml mark with deionised water.
- 3 Add one Nitriphot No 1 tablet, crush and mix to dissolve.
- 4 Add one Nitriphot No 2 tablet, crush and mix to dissolve. Cap immediately.
- 5 Stand for exactly two minutes to allow full colour development. Ignore any further colour development after this time.
- 6 Select Phot 43 on the Photometer.
- 7 Take Photometer reading in the usual manner (see Photometer instructions).
- 8 The result is displayed as mg/l NaNO₂.

Interferences

Chlorine in excess of 30 mg/l may give slight positive interference. However, nitrite and chlorine are incompatible and do not normally co-exist.

The solution should be cooled to below 30°C before testing for the most accurate analytical results.

ORGANO- PHOSPHONATE (OP)

TEST FOR ORGANOPHOSPHONATE IN COOLING WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 20 mg/l PO₄

The use of organophosphonate compounds as inhibitors in cooling systems has become widespread in recent years. It is essential to monitor the active organophosphonate content of the cooling water to ensure the treatment is fully effective.

The Palintest OP test provides a reliable means of monitoring organophosphonate levels over the range 0 - 20 mg/l PO₄. The test has been developed for use with commercially available organophosphonate products such as those based on amino trimethyl phosphonic acid and hydroxyethane diphosphonic acid.

Method

In the Palintest OP test, organophosphonates are first converted to orthophosphate by a catalysed cold oxidation process. Excess oxidising agent is removed from the sample by precipitation and filtration. The orthophosphate formed in the reaction is then determined using the 'molybdenum blue' method. The reagents for the procedure are provided in tablet form and the test is simply carried out by adding the appropriate tablets in sequence to a diluted sample of the water.

The intensity of the blue coloration formed in the test is proportional to the organophosphonate concentration and is determined using a Palintest Photometer.

A separate correction procedure is applied to those samples known or suspected to contain orthophosphate. This compensates for the orthophosphate originally present in the sample so that a true value for organophosphonate concentration can be obtained.

Reagents and Equipment

Palintest Oxidising OP Tablets
Palintest OP-A Tablets
Palintest OP-B Tablets
Palintest OP-AX Tablets
Palintest Automatic Wavelength Selection Photometer
Palintest Test Tube, 20 ml plastic (PT 526)
Round Test Tubes, 10 ml glass (PT 595)

A filtration is required during the course of this procedure. The use of Palintest Filtration Kit (PT 600) is recommended for this purpose. Alternatively, standard laboratory equipment with Whatman GF/B or equivalent papers may be used.

Correction Procedure

The photometer is programmed for both Organophosphonate and correction calibrations, and will automatically calculate the corrected organophosphonate concentration. Use program Phot 44 Organophosphonate, then select the 'Follow On' option on screen to continue test for program Phot 45 Correction Factor.

Sample Preparation and Dilution

- 1 Filter sample if necessary to obtain a clear solution.
- 2 Prepare x5 dilution of the sample using the Palintest dilution tube.

This diluted sample is used for both the Organophosphonate and correction procedures. The test calibrations take this dilution into account - it is not necessary to apply a dilution factor in the result calculation.

Test Procedure - Organophosphonate

- 1 Fill the plastic test tube with **diluted** sample to the 20 ml mark.
- 2 Add one Oxidising-OP tablet. Replace screw cap and shake tube until tablet dissolves.
- 3 Allow the tube to stand for five minutes.
- 4 Add one OP-A tablet. Replace screw cap and shake tube until tablet dissolves.
- 5 Allow the tube to stand for two minutes.
- 6 Filter a portion of the solution into a round glass test tube filling to the 10 ml mark.
- 7 Add one OP-B tablet, crush tablet and mix to dissolve.
- 8 Stand for five minutes to allow full colour development.
- 9 Select Phot 44 on Photometer.
- 10 Take Photometer reading in the usual manner (see Photometer instructions).

The test may be terminated at this stage if the original sample is known not to contain orthophosphate.

Test Procedure - Correction Factor

If it is suspected that the sample contains orthophosphate, carry out the following correction procedure. On the photometer, select the 'Follow On' option on screen to continue the test program.

- 1 Fill a round glass test tube with diluted sample to the 10 ml mark.
- 2 Add one OP-AX tablet. Crush and mix to dissolve.
- 3 Add one OP-B tablet. Crush and mix to dissolve.
- 4 Stand for five minutes to allow full colour development.
- 5 Take Photometer reading in the usual manner.
- 6 The instrument displays the corrected organophosphonate concentration as active PO_4 .

Interferences

Chloride in excess of 350 mg/l will cause low results for organophosphonate. Samples containing chloride levels in excess of this value should be further diluted prior to the start of the test.

Note

The results of this test are expressed in terms of mg/l (ppm) active phosphate content. Commercially available products are normally sold as aqueous formulations with a given active content. To utilise the test results, regard must be paid to the active content of the product in use.

Photometer Method

CHLORIDE (CHLORIDOL)

AUTOMATIC WAVELENGTH SELECTION

TEST FOR CHLORIDE SALT IN WATER

0 – 50 mg/l Cl to

0 – 50,000 mg/l NaCl

The Palintest Chloridol test provides a simple method for measuring chloride salt levels. There are many applications in water technology that require determination of chlorides. These include the measurement of low levels of chloride to determine the extent of carry-over in boiler condensates; chloride determination to assess salt build-up in swimming pools or boiler waters; and measurement of high chloride levels for testing sea water or determining the saltness of brackish waters. A further application is for checking swimming pools where salt has been artificially added to simulate sea water bathing, or where this is necessary for the operation of certain types of electrolytic hypochlorite generator.

The test can be used for measuring these widely different chloride concentrations by varying the sample size selected. The test ranges covered are 0 - 50 mg/l Cl, 0 - 500 mg/l Cl, 0 - 10,000 mg/l NaCl and 0 - 50,000 mg/l NaCl.

Method

The Palintest Chloridol test is based on a tablet reagent system containing silver nitrate. Chlorides react with the silver nitrate to produce insoluble silver chloride. At the chloride levels encountered in the test, the insoluble silver chloride is observed as turbidity in the test sample. The degree of turbidity is proportional to the chloride concentration and is measured using a Palintest Photometer.

The test is carried out under acidic and oxidising conditions so as to prevent interference from complexing agents such as EDTA and polyphosphates, and from any reducing substances which may be present in the water. Polyacrylates do however interfere and the test should not be used on industrial waters using polyacrylate-based treatments.

The formation of the precipitate in the Chloridol test may be subject to matrix effects in the presence of high total dissolved solids (TDS). The 0 – 50 mg/l Cl range is calibrated only for use on softened waters and condensates, and should not be used for other samples. The dilution step in the other ranges reduces the TDS to acceptable levels and prevents this effect.

Reagents and Equipment

Palintest Acidifying CD Tablets

Palintest Chloridol Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Measuring Syringe, 1 ml (PT 361)

Sample Container, 100/50/10 ml plastic (PT 510)

Test Calibration

Select Program **Phot 46** Range 0 – 50 mg/l Cl
or **Phot 51** Range 0 – 500 mg/l Cl
or **Phot 101** Range 0 – 10,000 mg/l NaCl
or **Phot 102** Range 0 – 50,000 mg/l NaCl

Test Instructions

1 For Testing Boiler Condensate and Softened Waters ONLY

Range 0 - 50 mg/l Cl

Fill test tube with sample to the 10 ml mark.

For Testing Natural Waters, Drinking Water, Swimming Pools and Boiler Waters

Range 0 - 500 mg/l Cl

Using the measuring syringe, take 1 ml of sample. Transfer to the test tube and make-up to the 10 ml mark with deionised water.

For Testing Salt Chlorinator Treated Swimming Pools

Range 0 - 10,000 mg/l NaCl

Using the measuring syringe, take 0.5 ml of sample. Transfer to the sample container (PT 510) then make-up to the 100 ml mark with deionised water. Cap tube and mix. Fill test tube to the 10 ml mark with solution from the sample container.

For Testing Sea Water and Brackish Waters

Range 0 - 50,000 mg/l NaCl

Using the measuring syringe, take 0.1 ml of sample. Transfer to the sample container (PT 510) then make-up to the 100 ml mark with deionised water. Cap tube and mix. Fill test tube to the 10 ml mark with solution from the sample container.

- 2 Add one Acidifying CD tablet, crush and mix to dissolve.
- 3 Add one Chloridol tablet, allow the tablet to disintegrate for two minutes then crush any remaining particles and mix. A cloudy solution indicates the presence of chloride.
- 4 Select the appropriate program number on the photometer for the test range required.
- 5 Take the photometer reading in usual manner (see Photometer instructions). Use the light cap whilst taking readings.

Conversion Factors

In different applications it may be conventional to express the results of chloride tests in different ways. The following conversion factors are provided for the convenience of users :-

TO CONVERT RESULT		MULTIPLY BY
From	To	
mg/l Cl	mg/l CaCO ₃	1.41
mg/l NaCl	mg/l CaCO ₃	0.85

COLOUR

TEST FOR COLOUR IN NATURAL AND TREATED WATERS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

10 – 500 mg/l Pt
(10 – 500 mg/l Hazen Units)

Pure water exhibits a light blue colour when viewed in depth. This colour may be modified by the presence of organic material, typically to a yellow or brown colour. An estimate of this colour intensity is used as a simple means of monitoring natural and treated water.

Method

The colour of the water is determined photoelectrically using the Palintest Photometer. The sample should be filtered to remove suspended solids before analysis to determine the 'true colour' due to dissolved matter.

The colour of water is expressed using the platinum/cobalt colour scale (Pt/Co scale). Each unit is equivalent to the colour produced by 1 mg/l platinum in the form of chloroplatinic acid in the presence of 2 mg/l cobaltous chloride hexahydrate. These units are identical with 'Hazen' units, which have been traditionally used to express results from the visual estimation of water colour.

Reagents and Equipment

Palintest Colour/Turbidity Set (PM 269)

Palintest Automatic Wavelength Selection Photometer

Test Procedure

- 1 Filter sample through a GF/B filter paper.
- 2 Fill a test tube with filtered sample to the 10 ml mark.
- 3 Fill a test tube with deionised water to the 10 ml mark and retain for use as the BLANK tube.
- 4 Select Phot 47 on photometer.
- 5 Take photometer reading in usual manner (see photometer instructions) using the deionised water as the blank.
- 6 The result is displayed as mg/l Pt.

Note

Samples, which contain metallic impurities, dyestuffs or other industrial pollutants, may exhibit a different colour to the natural yellow-brown coloration. This test may not be suitable for samples of this type.

TURBIDITY

TEST FOR TURBIDITY IN NATURAL AND TREATED WATERS

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

5 – 400 Turbidity Units

Turbidity is an important parameter for characterising water quality. Turbidity is caused by the scattering of light by suspended matter such as clay, silt, finely divided organic and inorganic matter. A knowledge of turbidity facilitates estimation of the concentration of undissolved substances.

Method

The turbidity of the water is determined photoelectrically using the Palintest Photometer. In many samples both colour and turbidity will be present. In order to separate the effect of turbidity and colour, the sample is compared against a filtered portion of the same water.

The Palintest method has been calibrated against the widely recognised formazin turbidity solutions. Turbidity is expressed in terms of Formazin Turbidity Units (FTU). These units are broadly equivalent to Jackson Turbidity Units (JTU) and Nephelometric Turbidity Units (NTU).

Reagents and Equipment

Palintest Colour/Turbidity Set (PM 269)

Palintest Automatic Wavelength Selection Photometer

Test Procedure

- 1 Filter a portion of the sample through a GF/B filter paper.
- 2 Fill a test tube with filtered sample and retain for use as the BLANK tube.
- 3 Fill a test tube with unfiltered sample to the 10 ml mark.
- 4 Select Phot 48 on photometer.
- 5 Take photometer reading in usual manner (see photometer instructions) using the filtered sample as the blank.

Note

An optional light shield is available for use with the photometer. This shield fits over the test chamber and reduces stray light reaching the photocell. It is not necessary to use the light shield when carrying out this test indoors or under shaded outdoor light. The use of the light shield is however recommended when testing for turbidity under bright or variable lighting conditions.

DISSOLVED OXYGEN/0.8/1.4

TEST FOR DISSOLVED OXYGEN IN BOILER WATER AND BOILER FEEDWATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 0.8 mg/l/0 – 1.4 mg/l

The presence of dissolved oxygen in water used in boilers and steam raising plant is wholly undesirable. At high temperatures even small amounts of dissolved oxygen render the water highly corrosive to boiler plant, the oxygen must be removed from boiler feedwater by chemical treatment or by mechanical de-aeration.

The DO/0.8/1.4 test provides a simple means of testing for dissolved oxygen in boiler water or boiler feedwater. The tests cover the range 0 - 0.8 mg/l on most photometers and 0 – 1.4 mg/l on the Photometer 8000.

Method

Special techniques must be employed when testing for dissolved oxygen as the water sample can be readily contaminated by the oxygen in the atmosphere. CHEMetrics Vacu-Vials self-filling reagent ampoules provide the ideal means of carrying out this test. The tip of the ampoule is dipped into the water sample and is then broken to allow the vial to fill with water. In this way there is no possibility of contamination from the air.

The Vacu-Vial DO/0.8 test uses a methodology based on Rhodazine D reagent. The Rhodazine D compound in reduced form reacts with dissolved oxygen to form a bright pink complex.

The intensity of the colours formed in the tests is proportional to the dissolved oxygen content of the water and is measured using a Palintest Photometer.

Reagents and Equipment

CHEMetrics Vacu-Vial Reagent Set DO/0.8

Palintest Automatic Wavelength Selection Photometer

DO test reagents are light sensitive. Store tubes in original containers and keep the box closed when not in use.

Test Procedure

Read the Oxygen test instructions leaflet contained in the CHEMetrics Vacu-Vials pack. Observe these various recommendations regarding sample handling and use of Vacu-Vials.

- 1 Carry out the test in accordance with the test procedure given in the CHEMetrics instruction leaflet. Observe the time periods given in the test instructions.
- 2 Select Phot 49 on the photometer. The wavelength is set automatically.
- 3 Use the colourless blank ampoule provided in the Starter Pack as the blank for setting the instrument.
- 4 The photometer provides direct readings in mg/l O₂.

Note

Note for measuring dissolved oxygen in natural waters, use the DO 15/20 test (see instructions Phot.75).

Vacu-Vials is a registered trade mark of CHEMetrics Inc

Photometer Method

DISSOLVED OXYGEN/2

TEST FOR DISSOLVED OXYGEN IN BOILER WATER AND BOILER FEEDWATER

AUTOMATIC WAVELENGTH SELECTION

0 – 2.0 mg/l

The presence of dissolved oxygen in water used in boilers and steam raising plant is wholly undesirable. At high temperatures even small amounts of dissolved oxygen render the water highly corrosive to boiler plant, the oxygen must be removed from boiler feedwater by chemical treatment or by mechanical de-aeration.

The DO/2.0 tests provide a simple means of testing for dissolved oxygen in boiler water or boiler feedwater. The test covers the range and 0 - 2.0 mg/l.

Method

Special techniques must be employed when testing for dissolved oxygen as the water sample can be readily contaminated by the oxygen in the atmosphere. CHEMetrics Vacu-Vials self-filling reagent ampoules provide the ideal means of carrying out this test. The tip of the ampoule is dipped into the water sample and is then broken to allow the vial to fill with water. In this way there is no possibility of contamination from the air.

The Vacu-Vial DO/2.0 test uses a reagent based on the indigo carmine method. Indigo carmine, in its reduced form, reacts with dissolved oxygen to form a blue complex.

The intensity of the colours formed in the tests is proportional to the dissolved oxygen content of the water and is measured using a Palintest Photometer.

Reagents and Equipment

CHEMetrics Vacu-Vial Reagent Set DO/2.0

Palintest Automatic Wavelength Selection Photometer

DO test reagents are light sensitive. Store tubes in original containers and keep the box closed when not in use.

Test Procedure

Read the Oxygen test instructions leaflet contained in the CHEMetrics Vacu-Vials pack. Observe these various recommendations regarding sample handling and use of Vacu-Vials.

- 1 Carry out the test in accordance with the test procedure given in the CHEMetrics instruction leaflet. Observe the time periods given in the test instructions.
- 2 Select Phot 50 on the photometer. The wavelength is set automatically.
- 3 Use the colourless blank ampoule provided in the Starter Pack as the blank for setting the instrument.
- 4 The photometer provides direct readings in mg/l O₂.

Note

Note for measuring dissolved oxygen in natural waters use the DO 15/20 test (see instructions Phot.75).

Vacu-Vials is a registered trade mark of CHEMetrics Inc

PHMB

TEST FOR PHMB-BASED SANITISERS IN SWIMMING POOL WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 100 mg/l

Polyhexamethylbiguanide (PHMB) is an organic biocide used for water disinfection. PHMB-based sanitisers are widely used for the treatment of swimming pool water. These sanitisers are typically sold under branded names, for example Baquacil* (Zeneca), Softswim* (Biolab), Revosil* (Mareva) and Nicosil* (Nico Norge).

The Palintest PHMB test provides a simple means of measuring PHMB-based sanitiser levels in swimming pool waters over the range 0 - 100 mg/l. The test is calibrated in terms of commercially available sanitiser products which normally contain 20% active biocide.

Method

The Palintest PHMB test is based on a colorimetric method developed by Palintest and now established as the standard method of testing for polyhexamethylbiguanide. In the test the PHMB reacts with a sulphone-phthalein indicator under mildly acid conditions to form an intense blue complex. The indicator itself is yellow in colour. Thus at different PHMB levels a distinctive range of colours from yellow, through green, to blue are produced.

In the Palintest method, the reagents are combined in the form of a single tablet and the test is simply carried out by adding a tablet to a sample of water. The intensity of the colour produced is proportional to the PHMB concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest PHMB-Phot Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill the test tube with sample to the 10 ml mark.
- 2 Add one PHMB-Phot tablet crush and mix to dissolve.
- 3 Select Phot 52 on Photometer.
- 4 Take Photometer reading immediately in usual manner (see Photometer instructions).
- 5 The result is displayed as mg/l of PHMB.

* All trade marks acknowledged

NICKEL (NICKELTEST)

TEST FOR NICKEL IN NATURAL AND TREATED WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 10 mg/l

Nickel does not occur naturally in water but is found in many industrial waste waters, such as those from the steel and plating industries. It is considered an undesirable constituent of water, and hence requires close and careful monitoring. The EC maximum admissible concentration for drinking water (MAC) is 0.05 mg/l.

The Palintest Nickeltest method provides a simple test for the determination of nickel in water over the range 0 - 10 mg/l Ni. The test responds to both Ni^{2+} and Ni^{4+} and thus gives a measure of total soluble inorganic nickel content of the water.

Method

In the Palintest Nickeltest method, nickel salts are reduced to the nickelous form and then reacted with nioxime indicator to give a pink coloured complex. Reagents are included to prevent copper interference, and a complexing powder is provided to prevent iron interference.

The reagents are provided in tablet form and the test is simply carried out by adding tablets to a sample of the water. The intensity of colour produced in the test is proportional to the nickel concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Nickeltest PR Powder (Spoon Pack)

Palintest Nickeltest No 1 Tablets

Palintest Nickeltest No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one Nickeltest No 1 tablet, crush and mix to dissolve. Ensure tablet is completely dissolved before proceeding.
- 3 If iron is thought to be present in the sample, add one level spoonful of Nickeltest PR powder and mix.
- 4 Add one Nickeltest No 2 tablet, crush and mix to dissolve.
- 5 Stand for two minutes to allow full colour development.
- 6 Select Phot 53 on Photometer.
- 7 Take photometer reading in usual manner (see Photometer instructions).
- 8 The result is displayed as mg/l Ni.

Interferences

- 1 The presence of cobalt at 0.5 mg/l gives a positive response in the test.
 - 2 The presence of significant levels of EDTA (at least 25 mg/l) complexes nickel and reduces response in the test. Complexing agents used in water treatment, such as polyphosphates, do not affect the results.
-

Photometer Method

PHENOL (PHENOLTEST)

AUTOMATIC WAVELENGTH SELECTION

**TEST FOR PHENOL AND ORTHO/META
SUBSTITUTED PHENOLS IN NATURAL,
DRINKING AND INDUSTRIAL WASTE WATERS**

**0 – 5.0 mg/l
as Phenol**

Phenols and substituted phenols may occur in natural, drinking and industrial waste waters. Phenols are not readily removed from water by conventional water treatment processes. These compounds arise typically from oil and chemical refining, livestock dips, the breakdown of pesticides, human and animal wastes and from naturally occurring sources. Chlorination of such waters may produce odorous and objectionable-tasting chlorophenols.

The Palintest Phenoltest method provides a simple means of measuring the concentration of phenol and phenolic compounds present in water over the range 0 - 5.0 mg/l. The concentration of phenol determined in the test is due to unsubstituted and to ortho and meta substituted phenols. A proportion of para substituted phenols will give a positive response.

Method

In the Phenoltest method, phenol and phenolic compounds react with 4-amino-antipyrine in the presence of ferricyanide ions to form a red colour. The reagents are provided in tablet form and the test is carried out simply by adding the appropriate tablets to a sample of the water. A further tablet reagent is used to prevent interference due to metal ions.

The intensity of the red colour produced in the test is proportional to the concentration of phenolic compounds present in the sample and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Phenoltest No 1 Tablets

Palintest Phenoltest No 2 Tablets

Palintest Phenoltest PR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill round test tube to the 10 ml mark with sample.
- 2 In the case of samples known to contain copper, zinc, iron or manganese ions, add one Phenoltest PR tablet. Crush and mix to dissolve.
- 3 Add one Phenoltest No 1 tablet, crush and mix to dissolve.
- 4 Add one Phenoltest No 2 tablet, crush and mix to dissolve.
- 5 Stand for 10 minutes to allow full colour development.
- 6 Select Phot 54 on the Photometer.
- 7 Take photometer reading in the usual manner (see Photometer instructions).
- 8 The result is displayed as mg/l C_6H_5OH .

Interferences

- 1 Use of the Phenoltest PR tablet will prevent interference from metal ions up to a concentration of 350 mg/l. The test is unaffected by free chlorine in the sample up to 10 mg/l.
 - 2 Low results may be obtained in samples containing more than 150 mg/l alkalinity (as $CaCO_3$), 10 mg/l sulphite or 2 mg/l sulphide. Certain organic keto-enol compounds may cause high results. In the case of known or suspected interferences, then the sample should be pre-treated in accordance with standard analytical procedures.
-

CHROMIUM (CHROMICOL)

TEST FOR SOLUBLE CHROMIUM IN NATURAL AND INDUSTRIAL WASTE WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 1.0 mg/l

Chromium may be present in certain industrial waste waters, such as those from the tanning, plating and coating industries. Chromium may occur in hexavalent form as chromates and dichromates, or in trivalent form as chromium salts. In water supplies hexavalent chromium is a particularly objectionable constituent. Trivalent chromium, although relatively inert, is also regarded as undesirable.

The Palintest Chromicol test provides a means of measuring chromium over the range 0 - 1.0 mg/l. The test is particularly useful since it can be used to differentiate between the concentrations of trivalent (Cr^{III}) and hexavalent (Cr^{VI}) chromium present.

Method

In the Palintest Chromicol method, hexavalent chromium salts in acidic conditions react with diphenylcarbazide to give a purple coloured complex. This provides a measure of the hexavalent chromium (Cr^{VI}) present in the sample. The reagents are provided in tablet form and the test is simply carried out by adding tablets to a sample of the water.

To determine total chromium (Cr^{III}) plus (Cr^{VI}) a fresh sample of the solution is oxidised using a powder reagent to convert the trivalent chromium to the hexavalent form. The test is then repeated to give a measure of the total soluble chromium content of the water. The difference between the two readings gives a measure of trivalent chromium

The intensity of colour produced in the tests is proportional to the chromium concentrations and is measured using a Palintest Photometer.

Reagents and Equipment

For Hexavalent Chromium :-

Palintest Chromicol No 1 Tablets

Palintest Chromicol No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Test Tubes, 10 ml glass (PT 595)

For Trivalent and Total Chromium :-

Palintest Chromicol CR Reagent (Spoon Pack)

Palintest Pretreatment Tube, 20 ml plastic (PT 526)

Filtration Kit, 0.45µm (PT 601)

A filtration is required during the course of the procedure for total and trivalent chromium. The use of Palintest Filtration Kit (PT 601) is recommended for this purpose. Alternatively, standard laboratory equipment with Whatman 0.45µm or equivalent membrane filters may be used.

Test Procedure - Hexavalent Chromium

- 1 Fill round test tube to the 10 ml mark.
- 2 Add one Chromicol No 1 tablet, crush and mix to dissolve.
- 3 Add one Chromicol No 2 tablet, crush and mix to dissolve.
- 4 Stand for 10 minutes without disturbing the solution to allow full colour development and to enable any undissolved particles to settle.
- 5 Select Phot 55 on the Photometer.
- 6 Take photometer reading in the usual manner (see Photometer instructions). (**Result A**).
- 7 The result represents the hexavalent chromium concentration (chromates and dichromates) as mg/l Cr. Stop the test at this stage if only hexavalent chromium determination is required.

Test Procedure - Total Chromium

- 1 Fill the pretreatment tube with sample to the 20 ml mark.
- 2 Add one level spoonful of Chromicol CR powder. Replace screw cap and shake tube well for two minutes.
- 3 Allow tube to stand for two minutes.
- 4 Filter a portion of the solution **dropwise** into a round glass test tube. Discard the first few drops and then fill to the 10 ml mark.
- 5 Add one Chromicol No 1 tablet, crush and mix to dissolve.
- 6 Add one Chromicol No 2 tablet, crush and mix to dissolve.
- 7 Stand for 10 minutes without disturbing the solution to allow full colour development and to enable any undissolved particles to settle.
- 8 Select Phot 100 on Photometer.
- 9 Take photometer reading in usual manner (see Photometer instructions).
- 10 The result represents the total soluble chromium concentration (trivalent and hexavalent) as mg/l Cr (**Result B**).
- 11 The trivalent Chromium (Cr^{III}) concentration is obtained by subtracting Result A from Result B :-

$$\text{Trivalent Chromium} = \text{Result B} - \text{Result A}$$

Interferences

Levels of dissolved iron above 1 mg/l cause low results for chromium. To increase the tolerance, repeat the test using two Chromicol No 1 tablets and one Chromicol No 2 tablet. Tannin causes complexation which prevents a response in the test.

SILICA HR

TEST FOR SILICA IN NATURAL AND INDUSTRIAL WATERS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 150 mg/l SiO₂

Silicon, in the form of silica, is one of the earth's most abundant elements. Silicon is found widely in natural waters as colloidal silica or soluble silicates.

Silica and silicates do not normally cause any problems in water intended for domestic consumption. However, their presence is undesirable in water used in a variety of industrial applications. This is because of the tendency of such water to form a hard scale on equipment. Silica and silicate containing waters are particularly troublesome in steam generating plant such as high pressure boilers since silica scale can build up on turbine blades.

Formulations containing silicate are used in industrial water treatment, as it is necessary to control the silicate within specified levels.

The Palintest Silica test provides a simple means of measuring silica and silicate levels in natural, treated, industrial and cooling waters over the range 0 - 150 mg/l SiO₂.

Method

Sodium molybdate reacts with silica under acid conditions to produce molybdosilicic acid. Phosphate reacts in a similar manner. Interference by phosphate is prevented by introducing a reagent that destroys any molybdophosphoric acid which may form.

The reagents for the method are provided in tablet form and the test is carried out simply by adding tablets to a sample of water. The intensity of the colour produced in the test is proportional to the silica concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Silica HR No 1 Tablets

Palintest Silica HR No 2 Tablets

Palintest Silica PR Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Fill the test tube with sample to the 10 ml mark.
 - 2 Add one Silica No 1 tablet, crush and mix to dissolve.
 - 3 Add one Silica No 2 tablet, crush and mix to dissolve. Stand for 10 minutes to allow full colour development.
 - 4 Add one Silica PR tablet, crush and mix to dissolve. Stand for two minutes. (This stage may be omitted if the sample is known to be completely free of phosphate and chlorine).
 - 5 Select Phot 56 on Photometer.
 - 6 Take Photometer reading in usual manner (see Photometer instructions).
 - 7 The result is displayed as mg/l SiO_2 .
-

CHLORINE DIOXIDE LR

TEST FOR CHLORINE DIOXIDE RESIDUALS IN WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 2.5 mg/l

Chlorine dioxide is increasingly being chosen ahead of chlorine for use in many applications where it is believed to have several advantages.

Chlorine dioxide is used in water disinfection, where its ability to disrupt biofilm in pipe work makes it useful against certain waterborne micro organisms such as Legionella. Chlorine dioxide is also used in the food industry for control of micro organisms in fruit washing and is popular in the pulp and paper industry as a bleaching agent. The USEPA has rated chlorine dioxide as the best available technology for paper pulp bleaching due to its low environmental impact.

Chlorine dioxide may be generated on-site either electrolytically or by reacting chlorine with sodium chlorite solution. Water treated with chlorine dioxide may therefore also contain amounts of chlorine and chlorite. For the control of water treatment systems it is necessary to determine chlorine dioxide in the presence of these other residuals.

The Palintest Chlorine Dioxide method provides a specific method of determining chlorine dioxide in treated water, in the presence of free and combined chlorine and chlorite.

Method

In the Palintest Chlorine Dioxide test, Lissamine Green B is bleached under alkaline conditions by chlorine dioxide. An ammonium salt is used to prevent any interference by chlorine. This method can determine chlorine dioxide accurately in the presence of free and complexed chlorine, chlorite, chlorate, ozone, bromine and permanganate.

Reagents and Equipment

Palintest Chlordiox Buffer Tablets

Palintest Chlordiox LR Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Round Test Tubes, 10 ml glass (PT 595)

Important Note

Chlorine dioxide is *extremely volatile* and can be lost from solution very easily. Extreme care must be taken when extracting and dispensing samples to minimise any loss from solution to ensure accurate measurement. When pouring the sample into a test tube, it is recommended that this is done by holding the tube at a slight angle and pouring slowly and gently down the side of the tube to minimise any splashing and turbulence which may cause loss of chlorine dioxide vapour.

Test Procedure - Chlorine Dioxide

- 1 Rinse a clean test tube with sample leaving **a few drops** in the tube.
- 2 Add one Chlordiox Buffer tablet and one Chlordiox LR tablet, crush and mix both together in the small volume of sample left in the tube to produce an even mixture of well crushed material.
- 3 Rinse and fill another clean test tube with sample to the 10 ml mark and slowly pour this into the tube containing the crushed tablets.
- 4 Mix the tube contents with a crushing rod to ensure complete dissolution of the tablet material.
- 5 Stand for **one minute** to allow the chlorine dioxide to react with the indicator.
- 6 Select Phot 74 on photometer.
- 7 Take photometer reading in usual manner (see photometer instructions). This result represents the chlorine dioxide residual in terms of mg/l ClO_2 .

Note - to obtain the chlorine dioxide residual as mg/l Cl_2 divide the result by 1.9.

Interferences

Studies of the effect of expected levels of common species which may be present in chlorine dioxide containing waters were undertaken to determine if these would detrimentally affect the results of the test. These included other chlorine compounds and oxidising agents, metal ions, hardness, alkalinity, nitrate, phosphate and sulphate. No interference effect was observed.

Temperature Effect

The method is calibrated for use at 15 – 25°C - lower temperatures will cause a slightly high bias to results. For accurate results, equilibrate a full glass bottle of sample, with no headspace, to room temperature for analysis.

DISSOLVED OXYGEN 15

TEST FOR DISSOLVED OXYGEN IN NATURAL WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 15 mg/l

Natural water contains oxygen dissolved from the atmosphere. The presence of oxygen is essential to sustain aquatic life and to prevent the water from stagnation. The extent to which oxygen from the air dissolves in water is determined largely by the water temperature. The solubility is approximately 14.6 mg/l at 0°C, 11.3 mg/l at 10°C, 9.1 mg/l at 20°C and 7.6 mg/l at 30°C. In saline waters, the solubility of oxygen from the air is lower.

Dissolved oxygen is easily lost from the water through pollution or through high temperature conditions during summer months. Water in reservoirs and lakes may therefore be aerated by vigorous agitation in order to prevent stagnation. In extreme cases, oxygen may be directly injected into rivers or lakes in order to preserve aquatic life.

The DO/20 tests provide a simple means of testing for dissolved oxygen in natural or other waters over the range 0 - 20.0 mg/l or 0 – 15 .0 mg/l depending on the photometer used.

Method

Special techniques must be employed when testing for dissolved oxygen as the water sample can be readily contaminated by the oxygen in the air. CHEMetrics Vacu-Vials self-filling reagent ampoules provide the ideal means of carrying out this test. The tip of the ampoule is dipped into the water sample and is then broken to allow the vial to fill with water. In this way there is no possibility of contamination from the air.

The Vacu-Vial DO/20 test uses a reagent based on the indigo carmine method. Indigo carmine, reacts with dissolved oxygen under the conditions of the test to form a blue complex. The intensity of the colours formed in the test is proportional to the dissolved oxygen content of the water and is measured using a Palintest Photometer.

Reagents and Equipment

CHEMetrics Vacu-Vial Reagent Set DO/20

Palintest Automatic Wavelength Selection Photometer

DO test reagents are light sensitive. Store tubes in original containers and keep the box closed when not in use.

Test Procedure

Read the Oxygen test instructions leaflet contained in the CHEMetrics Vacu-Vials pack. Observe these various recommendations regarding sample handling and use of Vacu-Vials.

- 1 Carry out the test in accordance with the test procedure given in the CHEMetrics instruction leaflet. Observe the time periods given in the test instructions.
- 2 Select Phot 75 on the photometer.
- 3 Use the colourless blank ampoule provided in the Starter Pack as the blank for setting the instrument.
- 4 Take the photometer reading (see Photometer instructions).
- 5 The result is displayed as mg/l O₂.

Note

For testing dissolved oxygen in water used in boilers and steam raising plant, use the DO/0.8 or DO/2 tests (see instructions Phot.49 and Phot.50).

Vacu-Vials is a registered trade mark of CHEMetrics Inc

Photometer Method**CHLORINE DIOXIDE HR****AUTOMATIC
WAVELENGTH
SELECTION****TEST FOR CHLORINE DIOXIDE
RESIDUALS IN WATER****0 – 20 mg/l**

Chlorine dioxide is increasingly being chosen ahead of chlorine for use in many applications where it is believed to have several advantages.

Chlorine dioxide is used in water disinfection, where its ability to disrupt biofilm in pipe work makes it useful against certain waterborne micro organisms such as Legionella. Chlorine dioxide is also used in the food industry for control of micro organisms in fruit washing and is popular in the pulp and paper industry as a bleaching agent. The USEPA has rated chlorine dioxide as the best available technology for paper pulp bleaching due to its low environmental impact.

Chlorine dioxide may be generated on-site either electrolytically or by reacting chlorine with sodium chlorite solution. Water treated with chlorine dioxide may therefore also contain amounts of chlorine and chlorite. For the control of water treatment systems it is necessary to determine chlorine dioxide in the presence of these other residuals.

The Palintest Chlorine Dioxide method provides a specific method of determining chlorine dioxide in treated water, in the presence of free and combined chlorine and chlorite.

Method

In the Palintest Chlorine Dioxide test, Lissamine Green B is bleached under alkaline conditions by chlorine dioxide. An ammonium salt is used to prevent any interference by chlorine. This method can determine chlorine dioxide accurately in the presence of free and complexed chlorine, chlorite, chlorate, ozone, bromine and permanganate.

Reagents and Equipment

Palintest Chlordiox Buffer Tablets

Palintest Chlordiox HR Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Round Test Tubes, 10 ml glass (PT 595)

Important Note

Chlorine dioxide is extremely volatile and can be lost from solution very easily. Extreme care must be taken when extracting and dispensing samples to minimise any loss from solution to ensure accurate measurement. When pouring the sample into a test tube, it is recommended that this is done by holding the tube at a slight angle and pouring slowly and gently down the side of the tube to minimise any splashing and turbulence which may cause loss of chlorine dioxide vapour.

Test Procedure - Chlorine Dioxide

- 1 Rinse a clean test tube with sample leaving **a few drops** in the tube.
- 2 Add one Chlordiox Buffer tablet and one Chlordiox HR tablet, crush and mix both together in the small volume of sample left in the tube to produce an even mixture of well crushed material.
- 3 Rinse and fill another clean test tube with sample to the 10 ml mark and slowly pour this into the tube containing the crushed tablets.
- 4 Mix the tube contents with a crushing rod to ensure complete dissolution of the tablet material.
- 5 Stand for **two minutes** to allow the chlorine dioxide to react with the indicator.
- 6 Select Phot 76 on photometer.
- 7 Take photometer reading (%T) in usual manner (see photometer instructions). This result represents the chlorine dioxide residual in terms of mg/l ClO_2 .

Notes

- 1 To obtain the chlorine dioxide residual as mg/l Cl_2 , divide the result by 1.9.
- 2 For the greatest accuracy below 2.5 mg/l ClO_2 , use the Palintest Chlorine Dioxide LR test.

Interferences

Studies of the effect of expected levels of common species which may be present in chlorine dioxide containing waters were undertaken to determine if these would detrimentally affect the results of the test. These included other chlorine compounds and oxidising agents, metal ions, hardness, alkalinity, nitrate, phosphate and sulphate. No interference effect was observed.

Temperature Effect

The method is calibrated for use at 15 – 25°C - lower temperatures will cause a slightly high bias to results. For accurate results, equilibrate a full glass bottle of sample, with no headspace, to room temperature for analysis.

TUBETESTS® COPPER/20

TEST FOR COPPER IN EFFLUENTS, WASTE
WATERS AND INDUSTRIAL WATER SAMPLES

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 20 mg/l

Copper is widely found in natural and waste waters. Copper-bearing ores occur commonly and copper is extensively used in industrial products. Copper finds particular application in electrical and electronic products, in the production of alloys, in electroplating processes and as an additive in paints and wood preservatives.

At low levels, copper is not known to have an adverse effect on humans. Nevertheless, control of copper concentrations in effluents and waste waters is necessary in order to prevent pollution of the aquatic environment and to meet industrial consent limits.

The Palintest Tubetests Copper/20 test is designed to measure total recoverable copper concentration over the range 0 - 20 mg/l.

Method

The Palintest Tubetests Copper/20 test is particularly applicable to the analysis of effluents, waste waters and industrial water samples. In such samples, the metal ions are often present in complexed, colloidal or particulate form. Moreover, effluents and waste waters typically contain colour and suspended solids. Special techniques are necessary therefore for the analysis of metals in these types of waters. The Palintest Tubetests Copper/20 test is designed to measure the total recoverable copper concentration in such samples.

In the Palintest Tubetests Copper/20 test the sample is first digested in a sulphuric acid/nitric acid mixture in order to solubilise particulate matter, break down complexes and remove colour. The acid digest mixture is provided pre-dispensed into special digestion tubes for ease of use and maximum safety.

Following the digestion stage, the sample is neutralised and buffered to provide the correct pH conditions for the test. A reducing agent is then added to convert all of the copper to cuprous form and this is then reacted with a 2,2-biquinoline-4,4-dicarboxylic salt to form a purple-coloured complex. A decomplexing agent is incorporated into the test reagent system in order to breakdown chelated copper which is present in the sample.

The intensity of the colour produced in the test is proportional to the copper concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Copper/20 Pack (PL 427) containing :-

- Metaltube Digest Tubes
- Metaltube Neut Reagent
- Coppertube Buffer
- Coppertube No 1 Tablet
- Coppertube No 2 Tablet

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 2 ml (PT 572)

Palintest Pipettor, 5 ml (PT 576)

Working Procedure

The Palintest Tubetests Copper/20 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

Palintest Metaltube Digest tubes contain approximately 40% mixed nitric/sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the digest tubes to add the sample, or to add reagents, as gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples the test must always be carried out in a fume cupboard. It is generally recommended that the test be conducted in a fume cupboard where available.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage.

Sample Preparation

Effluents and waste waters often contain undissolved or particulate material. Such samples should be homogenised thoroughly prior to taking the test sample in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests Heater, set the control to 105°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest Reagent tube and add 5 ml of sample using a Palintest pipettor with disposable tip or a standard laboratory pipette.
- 3 Replace the cap tightly and invert tube to mix contents. Place the tube in the Tubetests heater. Digest the tube for 60 minutes then remove and transfer to a test tube rack. Allow the tube to cool for approximately 10 minutes.

- 4 Add 2 ml of Metaltube Neut Reagent to the tube using a Palintest pipettor with disposable tip or a standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents. The tube will become hot on mixing. Allow the tube to cool for approximately 10 minutes.
- 5 Add 2 ml of Coppertube Buffer to the tube using a Palintest pipettor with disposable tip or a standard laboratory pipette.
- 6 Remove the cap from the tube and then add one Coppertube No 1 tablet, crush and mix to dissolve.
- 7 Add one Coppertube No 2 tablet, crush and mix to dissolve and then replace the cap on the tube.
- 8 Stand for 5 minutes without disturbing the solution to allow full colour development and to allow any undissolved particles to settle.
- 9 Prepare a Blank tube by filling a Metaltube Digest Reagent tube to the graduation line (10 ml) with deionised water. Cap the tube and invert to mix. This tube can be kept and used again for any subsequent Coppertube/20 testing.
- 10 Select Phot 77 on the photometer.
- 11 Wipe the tubes with a clean tissue to remove any finger marks and smears and then take photometer reading in the usual manner (see Photometer instructions).
- 12 The result is displayed as mg/l Cu.

Interferences

In interference studies the presence of metals such as cadmium, chromium, iron, nickel and zinc have all been found not to cause any effect on the test result.

Tests with samples containing common anions and non-metallic species showed that there was no significant interference.

Tests using this procedure with a variety of industrial waste waters showed that in most cases the colour and turbidity found in such samples were reduced to a level where they did not interfere with the test result.

However, in some extreme cases there may be noticeable colour or turbidity remaining. This may be the case for example with samples taken from pretreatment streams or effluent treatment tanks. Unless compensation is made for this colour or turbidity, it will lead to an inaccurate result. In such cases it is recommended to use a compensating blank by using the following procedure :-

Prepare two tubes of the same sample by following the test procedure up to and including Step 5. However at this point only continue the procedure using one of the tubes. Use the other tube, the 'compensating blank', in place of the normal blank tube described in Step 9 when taking the photometer reading. This will help compensate for any colour/turbidity present in the sample.

Tubetests Heater

The Palintest Digital Tubetests Heater (PT 589) is a 12-tube block heater featuring a digital display. The heater is dedicated for use with the Palintest Tubetests system. It comprises an electrically controlled dry bath which heats an aluminium test block. The heater is designed to provide the correct digesting and refluxing conditions for Tubetests tubes.

The heater features a digital display for the operating temperature and set temperature. The heater should be set to the temperature stated in the test procedure. On no account must the heater be set to a higher temperature than that specified as this may cause a hazard through pressure build-up in the tubes. It is not necessary to use a thermometer with the heater - the operating temperature is shown on the display. The temperature setting takes into account the thermal lag between the block and the heating tubes. The heater also features a timer - which is located on the base of the heater. The heater can be pre-set to operate for a predetermined time to suit particular test procedures.

TUBETESTS® TOTAL CHROMIUM/10

TEST FOR TOTAL CHROMIUM IN
EFFLUENTS, WASTE **WATERS AND**
INDUSTRIAL WATER SAMPLES

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 10 mg/l

Chromium occurs commonly in nature but is only found in natural waters at very low concentrations. However, chromium and chromium compounds are widely used in industrial processes such as tanning, plating, coating, metal finishing and water treatment. Chromium is therefore found in many effluents and industrial waste waters.

Chromium may be present in hexavalent form as chromates or dichromates, or in trivalent form as chromium salts. Hexavalent chromium is regarded as a particularly objectionable constituent in water supplies. Trivalent chromium, whilst relatively inert, is also regarded as undesirable. Careful monitoring of chromium in industrial effluents and waste waters is therefore necessary in order to conform to discharge consent limits and to prevent this element entering the aqueous environment.

The Palintest Tubetests Total Chromium/10 test is designed to measure total recoverable chromium concentrations over the range 0 - 10 mg/l.

Method

The Palintest Tubetests Total Chromium/10 test is particularly applicable to the analysis of effluents, waste waters and industrial water samples. In such samples, the metal ions are often present in complexed, colloidal or particulate form. Moreover, effluents and waste waters typically contain colour and suspended solids. Special techniques are necessary therefore for the analysis of metals in these types of waters. The Palintest Tubetests Total Chromium/10 test is designed to measure the total recoverable chromium concentration in such samples.

In the Palintest Tubetests Total Chromium/10 test the sample is first digested in a sulphuric acid/nitric acid mixture in order to solubilise particulate matter, break down complexes and remove colour. The acid digest mixture is provided pre-dispensed into special digestion tubes for ease of use and maximum safety.

Following the digestion stage, the acid is neutralised and an oxidising agent is added to convert any trivalent chromium to hexavalent form. This is then reacted under acid conditions with diphenylcarbazide to form a purple coloured complex. Decomplexing agents and inhibitors are incorporated into the test reagent system in order to break down any complexes which may be present and to prevent interference from other species commonly found in effluents and waste water samples.

The intensity of the colour produced in the test is proportional to the total chromium concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Total Chromium/10 Pack (PL 436) containing

Metaltube Digest Tubes

Chromitube Alkali Reagent

Chromitube Acid Reagent

Chromitube No 1 Tablet

Chromitube No 2 Tablet

Chromitube Indicator Tablet

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 1 ml (PT 574)

Palintest Pipettor, 2 ml (PT 572)

Working Procedure

The Palintest Tubetests Total Chromium/10 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

Palintest Metaltube Digest tubes contain approximately 40% mixed nitric/sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the reagent tubes to add the sample as gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples the test must always be carried out in a fume cupboard. It is generally recommended that the test be conducted in a fume cupboard where available.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage.

Sample Preparation

Effluents and waste waters often contain undissolved or particulate material. Such samples should be homogenised thoroughly prior to taking the test sample in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests Heater, set the control to 105°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest tube and add 1 ml of sample using a Palintest pipettor with a disposable tip or a standard laboratory pipette.
- 3 Replace the cap tightly and invert tube to mix contents. Place the tube in the Tubetests heater. Digest the tube for 60 minutes then remove and transfer to a test tube rack. Allow the tube to cool for approximately 10 minutes.
- 4 Add 2 ml of Chromitube Alkali Reagent to the tube using a Palintest pipettor with a disposable tip or a standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents. The tube will become hot on mixing. Allow the tube to cool for approximately 10 minutes.
- 5 Remove the cap from the tube and add one Chromitube No 1 tablet, crush and mix to dissolve.
- 6 Replace the cap tightly. Place the tube back in the heater for five minutes then transfer to a test tube rack. Allow the tube to cool for five minutes.
- 7 Add one Chromitube No 2 tablet, crush and mix to dissolve. Allow to stand for one minute.
- 8 Add 2 ml of Chromitube Acid Reagent into the tube using a Palintest pipettor with a disposable tip or a standard laboratory pipette, then fill the tube to the graduation line (10 ml) with deionised water. Replace the cap tightly and invert the tube gently to mix the contents.
- 9 Remove the cap and add one Chromitube Indicator tablet, crush and mix to dissolve, then replace cap.
- 10 Stand for 10 minutes without disturbing the solution to allow full colour development and to allow any undissolved particles to settle.
- 11 Prepare a Blank tube by filling a Metaltube Digest Reagent tube to the graduation line (10 ml) with deionised water, cap the tube and invert to mix. This tube can be kept and used again for any subsequent Tubetests Total Chromium/10 testing.
- 12 Select Phot 78 on the photometer.
- 13 Wipe the tubes with a soft tissue to remove any finger marks and smears and then take photometer reading in the usual manner (see Photometer instructions).
- 14 The result is displayed as mg/l Cr.

Interferences

In interference studies the presence of copper, iron, nickel and zinc have been found not to cause any effect on the test result. Tests with samples containing common anions and non-metallic species showed that there was no significant interference.

Tests using this procedure with a variety of industrial waste waters showed that in most cases the colour and turbidity found in such samples were reduced to a level where they did not interfere with the test result.

However in some extreme cases there may be noticeable colour or turbidity remaining. This may be the case for example with samples taken from pre-treatment streams or effluent treatment tanks. Unless compensation is made for this colour or turbidity it will lead to an inaccurate result. In such cases it is recommended to use a compensating blank by using the following procedure :-

Prepare two tubes of the same sample by following the test procedure up to and including Step 8. However at this point only continue the procedure using one of the tubes. Use the other tube, the 'Compensating Blank', in place of the normal blank tube described in Step 11 when taking the photometer reading. This will help compensate for any colour/turbidity present in the sample.

Tubetests Heater

The Palintest Digital Tubetests Heater (PT 589) is a 12-tube block heater featuring a digital display. The heater is dedicated for use with the Palintest Tubetests system. It comprises an electrically controlled dry bath which heats an aluminium test block. The heater is designed to provide the correct digesting and refluxing conditions for Tubetests tubes.

The heater features a digital display for the operating temperature and set temperatures. The heater should be set to the temperature stated in the test procedure. On no account must the heater be set to a higher temperature than that specified as this may cause a hazard through pressure build-up in the tubes. It is not necessary to use a thermometer with the heater - the operating temperature is shown on the display. The temperature setting takes into account the thermal lag between the block and the heating tubes. The heater also features a timer which is located on the base of the heater. The heater can be pre-set to operate for a predetermined time to suit particular test procedures.

Hexavalent Chromium

This test is for the determination of total chromium. For the specific determination of hexavalent chromium (Chromium VI) see test instructions PHOT.79. Use Palintest Tubetests Hexavalent Chromium Pack (PT 440).

TUBETESTS® HEXAVALENT CHROMIUM/10

TEST FOR HEXAVALENT CHROMIUM IN
EFFLUENTS, WASTE WATERS AND
INDUSTRIAL WATER SAMPLES

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 10 mg/l

Hexavalent Chromium (Chromium - VI) is not normally found in natural waters. However chromates and dichromates are widely used in industrial processes such as tanning, coating and water treatment. Hexavalent chromium is therefore commonly found in many effluents and industrial waste waters.

Hexavalent chromium is regarded as a particularly objectionable constituent in water supplies. Careful monitoring of industrial effluents and waste waters is therefore necessary in order to ensure conformity to consent discharge limits and to prevent hexavalent chromium entering the aqueous environment.

The Palintest Tubetests Hexavalent Chromium/10 test is designed to measure recoverable hexavalent chromium concentrations over the range 0 - 10 mg/l.

Method

The Palintest Tubetests Hexavalent Chromium/10 test is particularly applicable to the analysis of effluents, waste waters and industrial water samples. In such samples the metal ions are often present in complexed, colloidal or particulate form. Moreover, effluents and waste waters typically contain colour and suspended solids. Special techniques are necessary therefore for the analysis of metals in these types of waters. The Palintest Tubetests Hexavalent Chromium/10 test is designed to measure the recoverable hexavalent chromium concentration in such samples.

In the Palintest Tubetests Hexavalent Chromium/10 test the sample is first digested in a sulphuric acid/nitric acid mixture in order to solubilise particulate matter, break down complexes and remove colour. The acid digest mixture is provided pre-dispensed into special digestion tubes for ease of use and maximum safety.

Following the digestion stage, the acid is partially neutralised and then reacted with diphenylcarbazide to form a purple coloured complex. Decomplexing agents and inhibitors are incorporated into the test reagent system in order to break down any complexes which may be present and to prevent interference from other species commonly found in effluents and waste water samples.

The intensity of the colour produced in the test is proportional to the hexavalent chromium concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Hexavalent Chromium/10 Pack (PL 440) containing :-

Metaltube Digest Tubes

Chromitube Neut Reagent

Chromitube Indicator Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 1 ml (PT 574)

Palintest Pipettor, 2 ml (PT 572)

Working Procedure

The Palintest Tubetests Hexavalent Chromium/10 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

Palintest Metaltube Digest tubes contain approximately 40% mixed nitric/sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the reagent tubes to add the sample as gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples the test must always be carried out in a fume cupboard. It is generally recommended that the test be conducted in a fume cupboard where available.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage.

Sample Preparation

Effluents and waste waters often contain undissolved or particulate material. Such samples should be homogenised thoroughly prior to taking the test sample in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests Heater, set the control to 105°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest tube and add 1 ml of sample using a Palintest pipettor with a disposable tip or a standard laboratory pipette.
- 3 Replace the cap tightly and invert tube to mix contents. Place the tube in the Tubetests heater. Digest the tube for 60 minutes then remove and transfer to a test tube rack. Allow the tube to cool for approximately 10 minutes.
- 4 Add 2 ml of Chromitube Neut Reagent to the tube using a Palintest pipettor with a disposable tip or a standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents. The tube will become hot on mixing.
- 5 Allow the tube to cool for approximately 10 minutes then remove the cap and fill to the graduation line (10 ml) with deionised water.
- 6 Add one Chromitube Indicator tablet, crush and mix to dissolve then replace cap.
- 7 Stand for 10 minutes without disturbing the solution to allow full colour development and to allow any undissolved particles to settle.
- 8 Prepare a Blank tube by filling a Metaltube Digest Reagent tube to the graduation line (10 ml) with deionised water, cap the tube and invert to mix. This tube can be kept and used again for any subsequent Tubetests Hexavalent Chromium/10 testing.
- 9 Select Phot 79 on the photometer.
- 10 Wipe the tubes with a soft tissue to remove any finger marks and smears and then take photometer reading in the usual manner (see Photometer instructions).
- 11 The result is displayed as mg/l Cr.

Interferences

In interference studies the presence of copper, nickel and zinc have been found not to cause any effect on the test result. Iron levels greater than 1 mg/l have been found to cause slightly low results. Tests with samples containing common anions and non-metallic species showed that there was no significant interference.

Tests using this procedure with a variety of industrial waste waters showed that in most cases the colour and turbidity found in such samples were reduced to a level where they did not interfere with the test result.

However, in some extreme cases there may be noticeable colour or turbidity remaining. This may be the case for example with samples taken from pre-treatment streams or effluent treatment tanks. Unless compensation is made for this colour or turbidity it will lead to an inaccurate result. In such cases it is recommended to use a compensating blank by using the following procedure :-

Prepare two tubes of the same sample by following the test procedure up to and including Step 5. However at this point only continue the procedure using one of the tubes. Use the other tube, the 'Compensating Blank', in place of the normal blank tube described in Step 8 when taking the photometer reading. This will help compensate for any colour/turbidity present in the sample.

Tubetests Heater

The Palintest Digital Tubetests Heater (PT 589) is a 12-tube block heater featuring a digital display. The heater is dedicated for use with the Palintest Tubetests system. It comprises an electrically controlled dry bath which heats an aluminium test block. The heater is designed to provide the correct digesting and refluxing conditions for Tubetests tubes.

The heater features a digital display for the operating temperature and set temperatures. The heater should be set to the temperature stated in the test procedure. On no account must the heater be set to a higher temperature than that specified as this may cause a hazard through pressure build-up in the tubes. It is not necessary to use a thermometer with the heater - the operating temperature is shown on the display. The temperature setting takes into account the thermal lag between the block and the heating tubes. The heater also features a timer which is located on the base of the heater. The heater can be pre-set to operate for a predetermined time to suit particular test procedures.

Total Chromium

This test is for the determination of hexavalent chromium. For the specific determination of total chromium see test instructions PHOT.78. Use Palintest Tubetests Total Chromium Pack (PT 436).

TUBETESTS®
CHEMICAL OXYGEN
DEMAND – COD/150

**TEST FOR ASSESSING EFFLUENT AND WASTE
WATER QUALITY PRIOR TO DISCHARGE**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

5 – 150 mg/l O₂

Chemical oxygen demand is a vital test for assessing the quality of effluents and waste waters prior to discharge. The Chemical Oxygen Demand (COD) test predicts the oxygen requirement of the effluent and is used for monitoring and control of discharges, and for assessing treatment plant performance.

The impact of an effluent or waste water discharge on the receiving water is predicted by its oxygen demand. This is because the removal of oxygen from the natural water reduces its ability to sustain aquatic life. The COD test is therefore performed as routine in laboratories of water utilities and industrial companies.

Method

The Palintest COD method conforms to the sealed tube reflux version of methods detailed in the following reference texts :-

- 1 Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, American Public Health Association, American Water Works Association and Water Environment Federation.
Section 5220 D, Pages 5-14 to 5-19.
- 2 Methods for the Examination of Waters and Associated Materials 2006, Standing Committee of Analysts.
The Determination of Chemical Oxygen Demand in Waters and Effluents (2006).
- 3 British Standard BS ISO 15705:2002 BS 6068-2.80:2002
Water Quality - Determination of the Chemical Oxygen Demand Index (ST-COD) – small-scale, sealed-tube method.

Over the range of the test a series of colours from yellow through green to blue are produced. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests Tubes are available in different formats (see Interferences) :

Palintest COD/150, COD/150/M or COD/150/2M Tubetests Tubes

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 2 ml (PT 572)

COD test reagents are light-sensitive. Store tubes in the original container and keep the box closed when not in use. Store in cool, dry conditions.

Working Practice

The Palintest COD test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice. The reagent tubes contain 84% sulphuric acid and must be handled with care.

The Material Safety Data Sheet (MSDS) is the document that contains information on the potential hazards (health, fire, reactivity and environmental) and how to work safely with the chemical product. It is an essential starting point for the development of a complete health and safety program. It also contains information on the use, storage, handling and emergency procedures all related to the hazards of the material.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage. **Do not open tubes during or after sample digestion.**

Reagent Blank

In this test a reagent blank is used instead of the usual water blank referred to in the general photometer operating instructions. The reagent blank is prepared by adding deionised or distilled water to the reagent tube (see Test Procedure, Step 4) and then digesting the tube in the same manner as for the water sample.

It is not necessary to prepare a reagent blank each time the test is carried out. The reagent blank tube may be prepared weekly and used repeatedly with all samples prepared from the same batch of reagent tubes. The reagent blank should be stored in the dark between uses.

Sample Preparation

Effluents and waste water samples may contain undissolved or particulate material. Such samples may be homogenised in a blender prior to the test in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests heater, set the control to 150°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the SAMPLE TUBE as follows. Shake tube vigorously to suspend all sediment. Remove the cap of the COD Tubetests tube and add 2 ml of sample using a Palintest pipettor.
- 3 Replace the cap tightly and invert tube gently to mix contents. The tube will become hot on mixing. Ensure all of the precipitate is suspended before proceeding. Label the tube using the labels provided in the reagent pack and place the tube in the Tubetests heater. Ensure the safety screen is in position.
- 4 Prepare a REAGENT BLANK by repeating Steps 2 and 3 using 2 ml of deionised or distilled water in place of the sample. This stage may be omitted if a suitable reagent blank tube is already available (see Reagent Blank).
- 5 Digest the tubes for two hours then turn off the heater unless it is required for further tests.
- 6 Carefully remove each tube, invert gently to mix and then transfer to a test tube rack.
- 7 Allow the tubes to cool to room temperature.
- 8 Select Phot 80 on Photometer.
- 9 Take the photometer reading (see photometer instructions).
- 10 The result is displayed as mg/l O₂.

Interferences

Chloride is the main potential interference in the COD test. High chloride levels may result in an apparent high COD result. The Palintest COD/150 test will not be significantly affected by chloride levels up to 100 mg/l. Samples containing above this level should be diluted so as to reduce the concentration to 100 mg/l or below and the test carried out on the diluted sample.

If sample dilution is not possible then it may be necessary to suppress chloride interference. The method most commonly prescribed in standard analytical methods is the addition of mercuric sulphate to the reagent system.

In the Palintest COD/150/M test 0.04g of mercuric sulphate is provided in each tube of reagent and will suppress interference up to 2,000 mg/l chloride in sample containing 50 to 2,000 mg/l COD. In the Palintest COD/150/2M test 0.08g of mercuric sulphate is provided and will suppress interference up to 4,000 mg/l chloride in samples containing from 50 to 2,000 mg/l COD.

Disposal

The used COD Tubetests tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents should be disposed of in accordance with Local Authority requirements. A COD tube disposal service is available through Palintest Ltd (UK only). The tubes must not be re-used as they are designed for single use only.

Tubetests Heater

The Palintest Tubetests heater is a dedicated heater for use with the COD Tubetests system. It comprises an electronically controlled dry bath which heats aluminium test tube blocks. The heater is designed to provide the correct digesting and refluxing conditions necessary for the COD test. It provides the correct digestion temperature of $150^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in the reagent tubes.

The Palintest Digital Tubetests Heater (PT 589) is a 12 tube heater featuring a digital display.

To use the digital heater for the COD test, set the temperature on the digital display to 150°C .

On no account must the heater be set at a higher temperature than that specified as this may cause a hazard through pressure build-up in the COD tubes.

TUBETESTS®

CHEMICAL OXYGEN DEMAND – COD/400

Photometer Method

AUTOMATIC
WAVELENGTH
SELECTION

**TEST FOR ASSESSING EFFLUENT AND WASTE
WATER QUALITY PRIOR TO DISCHARGE**

20 - 400 mg/l O₂

Chemical oxygen demand is a vital test for assessing the quality of effluents and waste waters prior to discharge. The Chemical Oxygen Demand (COD) test predicts the oxygen requirement of the effluent and is used for monitoring and control of discharges, and for assessing treatment plant performance.

The impact of an effluent or waste water discharge on the receiving water is predicted by its oxygen demand. This is because the removal of oxygen from the natural water reduces its ability to sustain aquatic life. The COD test is therefore performed as routine in laboratories of water utilities and industrial companies.

Method

The Palintest COD method conforms to the sealed tube reflux version of methods detailed in the following reference texts :

- 1 Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, American Public Health Association, American Water Works Association and Water Environment Federation.
Section 5220 D, Pages 5-14 to 5-19.
- 2 Methods for the Examination of Waters and Associated Materials 2006, Standing Committee of Analysts.
The Determination of Chemical Oxygen Demand in Waters and Effluents (2006).
- 3 British Standard BS ISO 15705:2002 BS 6068-2.80:2002
Water Quality - Determination of the Chemical Oxygen Demand Index (ST-COD) - small-scale, sealed-tube method.

Over the range of the test, a series of colours from yellow through green to blue are produced. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests Tubes are available in different formats (see Interferences) :

Palintest COD/400 or COD/400/M or COD/400/2M Tubetests Tubes

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 2 ml (PT 572)

COD test reagents are light-sensitive. Store tubes in the original container and keep the box closed when not in use. Store in cool, dry conditions.

Working Practice

The Palintest COD test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice. The reagent tubes contain 84% sulphuric acid and must be handled with care.

The Material Safety Data Sheet (MSDS) is the document that contains information on the potential hazards (health, fire, reactivity and environmental) and how to work safely with the chemical product. It is an essential starting point for the development of a complete health and safety program. It also contains information on the use, storage, handling and emergency procedures all related to the hazards of the material.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage. **Do not open tubes during or after sample digestion.**

Reagent Blank

In this test a reagent blank is used instead of the usual water blank referred to in the general photometer operating instructions. The reagent blank is prepared by adding deionised or distilled water to the reagent tube (see Test Procedure, Step 4) and then digesting the tube in the same manner as for the water sample.

It is not necessary to prepare a reagent blank each time the test is carried out. The reagent blank tube may be prepared weekly and used repeatedly with all samples prepared from the same batch of reagent tubes. The reagent blank should be stored in the dark between uses.

Sample Preparation

Effluents and waste water samples may contain undissolved or particulate material. Such samples may be homogenised in a blender prior to the test in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests heater, set the control to 150°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the SAMPLE TUBE as follows. Shake tube vigorously to suspend all sediment. Remove the cap of the COD Tubetests tube and add 2 ml of sample using a Palintest pipettor.
- 3 Replace the cap tightly and invert tube gently to mix contents. The tube will become hot on mixing. Ensure all of the precipitate is suspended before proceeding. Label the tube using the labels provided in the reagent pack and place the tube in the Tubetests heater. Ensure the safety screen is in position.
- 4 Prepare a REAGENT BLANK by repeating Steps 2 and 3 using 2 ml of deionised or distilled water in place of the sample. This stage may be omitted if a suitable reagent blank tube is already available (see Reagent Blank).
- 5 Digest the tubes for two hours then turn off the heater unless it is required for further tests.
- 6 Carefully remove each tube, invert gently to mix and then transfer to a test tube rack.
- 7 Allow the tubes to cool to room temperature.
- 8 Select Phot 81 on Photometer.
- 9 Take the photometer reading (see photometer instructions).
- 10 The result is displayed as mg/l O₂.

Interferences

Chloride is the main potential interference in the COD test. High chloride levels may result in an apparent high COD result. The Palintest COD/400 test will not be significantly affected by chloride levels up to 100 mg/l. Samples containing above this level should be diluted so as to reduce the concentration to 100 mg/l or below and the test carried out on the diluted sample.

If sample dilution is not possible then it may be necessary to suppress chloride interference. The method most commonly prescribed in standard analytical methods is the addition of mercuric sulphate to the reagent system.

In the Palintest COD/400/M test 0.04g of mercuric sulphate is provided in each tube of reagent and will suppress interference up to 2,000 mg/l chloride in sample containing 50 to 2,000 mg/l COD. In the Palintest COD/400/2M test 0.08g of mercuric sulphate is provided and will suppress interference up to 4,000 mg/l chloride in samples containing from 50 to 2,000 mg/l COD.

Disposal

The used COD Tubetests tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents should be disposed of in accordance with Local Authority requirements. A COD tube disposal service is available through Palintest Ltd (UK only). The tubes must not be re-used as they are designed for single use only.

Tubetests Heater

The Palintest Tubetests heater is a dedicated heater for use with the COD Tubetests system. It comprises an electronically controlled dry bath which heats aluminium test tube blocks. The heater is designed to provide the correct digesting and refluxing conditions necessary for the COD test. It provides the correct digestion temperature of $150^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in the reagent tubes.

The Palintest Digital Tubetests Heater (PT 589) is a 12 tube heater featuring a digital display.

To use the digital heater for the COD test, set the temperature on the digital display to 150°C .

On no account must the heater be set at a higher temperature than that specified as this may cause a hazard through pressure build-up in the COD tubes.

TUBETESTS®

CHEMICAL OXYGEN DEMAND – COD/2000

Photometer Method

AUTOMATIC
WAVELENGTH
SELECTION

**TEST FOR ASSESSING EFFLUENT AND WASTE
WATER QUALITY PRIOR TO DISCHARGE**

50 – 2,000 mg/l O₂

Chemical oxygen demand is a vital test for assessing the quality of effluents and waste waters prior to discharge. The Chemical Oxygen Demand (COD) test predicts the oxygen requirement of the effluent and is used for monitoring and control of discharges, and for assessing treatment plant performance.

The impact of an effluent or waste water discharge on the receiving water is predicted by its oxygen demand. This is because the removal of oxygen from the natural water reduces its ability to sustain aquatic life. The COD test is therefore performed as routine in laboratories of water utilities and industrial companies.

Method

The Palintest COD method conforms to the sealed tube reflux version of methods detailed in the following reference texts :-

- 1 Standard Methods for the Examination of Water and Wastewater. 21st Edition. 2005. American Public Health Association, American Water Works Association and Water Environment Federation.
Section 5220 D, Pages 5-14 to 5-19.
- 2 Methods for the Examination of Waters and Associated Materials 2006, Standing Committee of Analysts.
The Determination of Chemical Oxygen Demand in Waters and Effluents (2006).
- 3 British Standard BS ISO 15705:2002 BS 6068 - 2.80:2002
Water Quality - Determination of the Chemical Oxygen Demand Index (ST-COD) - small-scale, sealed-tube method.

Over the range of the test, a series of colours from yellow through green to blue are produced. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests Tubes are available in different formats (see Interferences) :

Palintest COD/2000, COD/2000/M or COD/2000/2M Tubetests Tubes

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 2 ml (PT 572)

COD test reagents are light-sensitive. Store tubes in the original container and keep the box closed when not in use. Store in cool, dry conditions.

Working Practice

The Palintest COD test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice. The reagent tubes contain 84% sulphuric acid and must be handled with care. The Material Safety Data Sheet (MSDS) is the document that contains information on the potential hazards (health, fire, reactivity and environmental) and how to work safely with the chemical product. It is an essential starting point for the development of a complete health and safety program. It also contains information on the use, storage, handling and emergency procedures all related to the hazards of the material.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage. **Do not open tubes during or after sample digestion.**

Reagent Blank

In this test a reagent blank is used instead of the usual water blank referred to in the general photometer operating instructions. The reagent blank is prepared by adding deionised or distilled water to the reagent tube (see Test Procedure, Step 4) and then digesting the tube in the same manner as for the water sample.

It is not necessary to prepare a reagent blank each time the test is carried out. The reagent blank tube may be prepared weekly and used repeatedly with all samples prepared from the same batch of reagent tubes. The reagent blank should be stored in the dark, for example in the original packaging between use.

Sample Preparation

Effluents and waste water samples may contain undissolved or particulate material. Such samples may be homogenised in a blender prior to the test in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests heater, set the control to 150°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the SAMPLE TUBE as follows. Shake tube vigorously to suspend all sediment. Remove the cap of the COD Tubetests tube and add 2 ml of sample using a Palintest pipettor disposable tip dispenser or a standard laboratory pipette.
- 3 Replace the cap tightly and invert tube gently to mix contents. The tube will become hot on mixing. Ensure all of the precipitate is suspended before proceeding. Label the tube using the labels provided in the reagent pack and place the tube in the Tubetests heater. Ensure the safety screen is in position.
- 4 Prepare a REAGENT BLANK by repeating Steps 2 and 3 using 2 ml of deionised or distilled water in place of the sample. This stage may be omitted if a suitable reagent blank tube is already available (see Reagent Blank).
- 5 Digest the tubes for two hours then turn off the heater unless it is required for further tests.
- 6 Carefully remove each tube, invert gently to mix and then transfer to a test tube rack.
- 7 Allow the tubes to cool to room temperature.
- 8 Select Phot 82 on Photometer.
- 9 Take the photometer reading (see photometer instructions).
- 10 The result is displayed as mg/l O₂.

Interferences

Chloride is the main potential interference in the COD test. High chloride levels may result in an apparent high COD result. The Palintest COD/2000 test will not be significantly affected by chloride levels up to 100 mg/l. Samples containing above this level should be diluted so as to reduce the concentration to 100 mg/l or below and the test carried out on the diluted sample.

If sample dilution is not possible then it may be necessary to suppress chloride interference. The method most commonly prescribed in standard analytical methods is the addition of mercuric sulphate to the reagent system.

In the Palintest COD/2000/M test 0.04g of mercuric sulphate is provided in each tube of reagent and will suppress interference up to 2,000 mg/l chloride in sample containing 50 to 2,000 mg/l COD. In the Palintest COD/2000/2M test 0.08g of mercuric sulphate is provided and will suppress interference up to 4,000 mg/l chloride in samples containing from 50 to 2,000 mg/l COD.

Disposal

The used COD Tubetests tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents should be disposed of in accordance with Local Authority requirements. A COD tube disposal service is available through Palintest Ltd (UK only). The tubes must not be re-used as they are designed for single use only.

Tubetests Heater

The Palintest Tubetests heater is a dedicated heater for use with the COD Tubetests system. It comprises an electronically controlled dry bath which heats aluminium test tube blocks. The heater is designed to provide the correct digesting and refluxing conditions necessary for the COD test. It provides the correct digestion temperature of $150^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in the reagent tubes.

The Palintest Digital Tubetests Heater (PT 589) is a 12 tube heater featuring a digital display.

To use the digital heater for the COD test, set the temperature on the digital display to 150°C .

On no account must the heater be set at a higher temperature than that specified as this may cause a hazard through pressure build-up in the COD tubes.

TUBETESTS®

CHEMICAL OXYGEN DEMAND – COD/20,000

Photometer Method

AUTOMATIC
WAVELENGTH
SELECTION

TEST FOR ASSESSING EFFLUENT AND WASTE
WATER QUALITY PRIOR TO DISCHARGE

500 – 20,000 mg/l
(0.50 – 20.00 g/l O₂)

Chemical oxygen demand is a vital test for assessing the quality of effluents and waste waters prior to discharge. The Chemical Oxygen Demand (COD) test predicts the oxygen requirement of the effluent and is used for monitoring and control of discharges, and for assessing treatment plant performance.

The impact of an effluent or waste water discharge on the receiving water is predicted by its oxygen demand. This is because the removal of oxygen from the natural water reduces its ability to sustain aquatic life. The COD test is therefore performed as routine in laboratories of water utilities and industrial companies.

Method

The Palintest COD method conforms to the sealed tube reflux version of methods detailed in the following reference texts :-

- 1 Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, American Public Health Association, American Water Works Association and Water Environment Federation.
Section 5220 D, Pages 5-14 to 5-19.
- 2 Methods for the Examination of Waters and Associated Materials 2006, Standing Committee of Analysts.
The Determination of Chemical Oxygen Demand in Waters and Effluents (2006).
- 3 British Standard BS ISO 15705:2002 BS 6068-2.80:2002
Water Quality - Determination of the Chemical Oxygen Demand Index (ST-COD) – small-scale, sealed-tube method.

Over the range of the test a series of colours from yellow through green to blue are produced. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests Tubes are available in different formats (see Interferences) :

Palintest COD/20,000, COD/20,000/M or COD/20,000/2M Tubetests Tubes

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 0.2 ml (PT 570)

COD test reagents are light-sensitive. Store tubes in the original container and keep the box closed when not in use. Store in cool, dry conditions.

Working Practice

The Palintest COD test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice. The reagent tubes contain 84% sulphuric acid and must be handled with care. The Material Safety Data Sheet (MSDS) is the document that contains information on the potential hazards (health, fire, reactivity and environmental) and how to work safely with the chemical product. It is an essential starting point for the development of a complete health and safety program. It also contains information on the use, storage, handling and emergency procedures all related to the hazards of the material.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage. **Do not open tubes during or after sample digestion.**

Reagent Blank

In this test a reagent blank is used instead of the usual water blank referred to in the general photometer operating instructions. The reagent blank is prepared by adding deionised or distilled water to the reagent tube (see Test Procedure, Step 4) and then digesting the tube in the same manner as for the water sample.

It is not necessary to prepare a reagent blank each time the test is carried out. The reagent blank tube may be prepared weekly and used repeatedly with all samples prepared from the same batch of reagent tubes. The reagent blank should be stored in the dark, for example in the original packaging between use.

Sample Preparation

Effluents and waste water samples may contain undissolved or particulate material. Such samples may be homogenised in a blender prior to the test in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests heater, set the control to 150°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the SAMPLE TUBE as follows. Shake tube vigorously to suspend all sediment. Remove the cap of the COD Tubetests tube and add 0.2 ml of sample using a Palintest pipettor.
- 3 Replace the cap tightly and invert tube gently to mix contents. The tube will become hot on mixing. Ensure all of the precipitate is suspended before proceeding. Label the tube using the labels provided in the reagent pack and place the tube in the Tubetests heater. Ensure the safety screen is in position.
- 4 Prepare a REAGENT BLANK by repeating Steps 2 and 3 using 0.2 ml of deionised or distilled water in place of the sample. This stage may be omitted if a suitable reagent blank tube is already available (see Reagent Blank).
- 5 Digest the tubes for two hours then turn off the heater unless it is required for further tests.
- 6 Carefully remove each tube, invert gently to mix and then transfer to a test tube rack.
- 7 Allow the tubes to cool to room temperature.
- 8 Select Phot 83 on Photometer.
- 9 Take the photometer reading (see photometer instructions).
- 10 The result is displayed as mg/l O₂.

Interferences

Chloride is the main potential interference in the COD test. High chloride levels may result in an apparent high COD result. The Palintest COD/20,000 test will not be significantly affected by chloride levels up to 100 mg/l. Samples containing above this level should be diluted so as to reduce the concentration to 100 mg/l or below and the test carried out on the diluted sample.

If sample dilution is not possible then it may be necessary to suppress chloride interference. The method most commonly prescribed in standard analytical methods is the addition of mercuric sulphate to the reagent system.

In the Palintest COD/20,000/M test 0.04g of mercuric sulphate is provided in each tube of reagent and will suppress interference up to 2,000 mg/l chloride in sample containing 50 to 2,000 mg/l COD. In the Palintest COD/20,000/2M test 0.08g of mercuric sulphate is provided and will suppress interference up to 4,000 mg/l chloride in samples containing from 50 to 2,000 mg/l COD.

Disposal

The used COD Tubetests tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents should be disposed of in accordance with Local Authority requirements. A COD tube disposal service is available through Palintest Ltd (UK only). The tubes must not be re-used as they are designed for single use only.

Tubetests Heater

The Palintest Tubetests heater is a dedicated heater for use with the COD Tubetests system. It comprises an electronically controlled dry bath which heats aluminium test tube blocks. The heater is designed to provide the correct digesting and refluxing conditions necessary for the COD test. It provides the correct digestion temperature of $150^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in the reagent tubes.

The Palintest Digital Tubetests Heater (PT 589) is a 12 tube heater featuring a digital display.

To use the digital heater for the COD test, set the temperature on the digital display to 150°C .

On no account must the heater be set at a higher temperature than that specified as this may cause a hazard through pressure build-up in the COD tubes.

TUBETESTS®
CHEMICAL OXYGEN
DEMAND – COD/1000

Photometer Method

AUTOMATIC
WAVELENGTH
SELECTION

TEST FOR ASSESSING EFFLUENT AND WASTE
WATER QUALITY PRIOR TO DISCHARGE

10 – 1,000 mg/l O₂

Chemical oxygen demand is a vital test for assessing the quality of effluents and waste waters prior to discharge. The Chemical Oxygen Demand (COD) test predicts the oxygen requirement of the effluent and is used for monitoring and control of discharges, and for assessing treatment plant performance.

The impact of an effluent or waste water discharge on the receiving water is predicted by its oxygen demand. This is because the removal of oxygen from the natural water reduces its ability to sustain aquatic life. The COD test is therefore performed as routine in laboratories of water utilities and industrial companies.

Method

The Palintest COD method conforms to the sealed tube reflux version of methods detailed in the following reference texts :

- 1 Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, American Public Health Association, American Water Works Association and Water Environment Federation.
Section 5220 D, Pages 5-14 to 5-19.
- 2 Methods for the Examination of Waters and Associated Materials 2006, Standing Committee of Analysts.
The Determination of Chemical Oxygen Demand in Waters and Effluents (2006).
- 3 British Standard BS ISO 15705:2002 BS 6068 - 2.80:2002
Water Quality - Determination of the Chemical Oxygen Demand Index (ST-COD) – small-scale, sealed-tube method.

Over the range of the test, a series of colours from yellow through green to blue are produced. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests tubes are available in different formats (see Interferences) :

Palintest COD/1000, COD/1000/M or COD/1000/2M Tubetests Tubes

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 2 ml (PT 572)

COD test reagents are light-sensitive. Store tubes in the original container and keep the box closed when not in use. Store in cool, dry conditions.

Working Practice

The Palintest COD test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice. The reagent tubes contain 84% sulphuric acid and must be handled with care. The Material Safety Data Sheet (MSDS) is the document that contains information on the potential hazards (health, fire, reactivity and environmental) and how to work safely with the chemical product. It is an essential starting point for the development of a complete health and safety program. It also contains information on the use, storage, handling and emergency procedures all related to the hazards of the material.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage. **Do not open tubes during or after sample digestion.**

Sample Preparation

Effluents and waste water samples may contain undissolved or particulate material. Such samples may be homogenised in a blender prior to the test in order to improve accuracy and reproducibility.

Reagent Blank

In this test a reagent blank is used instead of the usual water blank referred to in the general photometer operating instructions. The reagent blank is prepared by adding deionised or distilled water to the reagent tube (see Test Procedure, Step 4) and then digesting the tube in the same manner as for the water sample.

It is not necessary to prepare a reagent blank each time the test is carried out. The reagent blank tube may be prepared weekly and used repeatedly with all samples prepared from the same batch of reagent tubes. The reagent blank should be stored in the dark between uses.

Test Procedure

- 1 Turn on Tubetests heater, set the control to 150°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the SAMPLE TUBE as follows. Shake tube vigorously to suspend all sediment. Remove the cap of the COD Tubetests tube and add 2 ml of sample using a Palintest pipettor.
- 3 Replace the cap tightly and invert tube gently to mix contents. The tube will become hot on mixing. Ensure all of the precipitate is suspended before proceeding. Label the tube using the labels provided in the reagent pack and place the tube in the Tubetests heater. Ensure the safety screen is in position.
- 4 Prepare a REAGENT BLANK by repeating Steps 2 and 3 using 2 ml of deionised or distilled water in place of the sample. This stage may be omitted if a suitable reagent blank tube is already available (see Reagent Blank).
- 5 Digest the tubes for two hours then turn off the heater unless it is required for further tests.
- 6 Carefully remove each tube, invert gently to mix and then transfer to a test tube rack.
- 7 Allow the tubes to cool to room temperature.
- 8 Select Phot 84 on Photometer.
- 9 Take the photometer reading (see photometer instructions).
- 10 The result is displayed as mg/l O₂.

Interferences

Chloride is the main potential interference in the COD test. High chloride levels may result in an apparent high COD result. The Palintest COD/1000 test will not be significantly affected by chloride levels up to 100 mg/l. Samples containing above this level should be diluted so as to reduce the concentration to 100 mg/l or below and the test carried out on the diluted sample.

If sample dilution is not possible then it may be necessary to suppress chloride interference. The method most commonly prescribed in standard analytical methods is the addition of mercuric sulphate to the reagent system.

In the Palintest COD/1000/M test 0.04g of mercuric sulphate is provided in each tube of reagent and will suppress interference up to 2,000 mg/l chloride in sample containing 50 to 2,000 mg/l COD. In the Palintest COD/1000/2M test 0.08g of mercuric sulphate is provided and will suppress interference up to 4,000 mg/l chloride in samples containing from 50 to 2,000 mg/l COD.

Disposal

The used COD Tubetests tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents should be disposed of in accordance with Local Authority requirements. A COD tube disposal service is available through Palintest Ltd (UK only). The tubes must not be re-used as they are designed for single use only.

Tubetests Heater

The Palintest Tubetests heater is a dedicated heater for use with the COD Tubetests system. It comprises an electronically controlled dry bath which heats aluminium test tube blocks. The heater is designed to provide the correct digesting and refluxing conditions necessary for the COD test. It provides the correct digestion temperature of $150^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in the reagent tubes.

The Palintest Digital Tubetests Heater (PT 589) is a 12 tube heater featuring a digital display.

To use the digital heater for the COD test, set the temperature on the digital display to 150°C .

On no account must the heater be set at a higher temperature than that specified as this may cause a hazard through pressure build-up in the COD tubes.

TUBETESTS® AMMONIA/12N/50N (INDOPHENOL)

TEST FOR AMMONIA IN NATURAL,
SEA AND WASTE WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 12 mg/l N

0 – 50 mg/l N

Ammonia occurs as a breakdown product of nitrogenous material in natural waters. It is also found in domestic effluents and certain industrial waste waters. Ammonia is harmful to fish and other forms of aquatic life, and the ammonia level must be carefully controlled in water used for fish farms and aquariums. Ammonia tests are routinely applied for the monitoring of natural water, sea water; and for pollution control on effluents and waste waters.

The Palintest Tubetests Ammonia/12N/50N (Indophenol) test provides a simple method of measuring ammonia (ammoniacal nitrogen) over the ranges 0 - 12 mg/l and 0 - 50 mg/l N.

Method

The Palintest Tubetests Ammonia/12N/50N (Indophenol) test is based on the Indophenol Blue method. Ammonia reacts with alkaline salicylate in the presence of chlorine to form a green-blue indophenol complex. Catalysts are incorporated to ensure complete and rapid colour development. The reagents are provided in the form of a predispensed tube and a tablet for maximum convenience. The test is simply carried out by adding a sample of the water and a tablet to a tube.

The intensity of the colour produced in the test is proportional to the ammonia concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Ammonia/12N/50N (Indophenol) Tubes

Palintest Tubetests Ammonia (Indophenol) Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 0.2 ml (PT 570) or 1 ml (PT 574)

Test Instructions

- 1 Remove the cap of the Tubetests Ammonia/12N/50N (Indophenol) tube and add 0.2 ml (0 - 50 mg/l range) or 1.0 ml (0 - 12 mg/l range) of sample using a pipettor. Swirl tube to mix.
- 2 Add one Tubetests Ammonia (Indophenol) tablet, crush and mix to dissolve. Replace cap.
- 3 Stand for exactly 10 minutes to allow colour development. (See Note 2).
- 4 Select Phot 85 on Photometer for 0 – 50 mg/l range. Select Phot 86 on Photometer for 0 – 12 mg/l range.
- 5 Take photometer reading in usual manner (see Photometer instructions). Use an unused Tubetests Ammonia/12N/50N (Indophenol) Tube to set the blank on the photometer. Alternatively, a Tubetests tube containing deionised water only may be used.
- 6 The Ammonia Nitrogen result is displayed as mg/l N.

Notes

- 1 At low temperatures the rate of colour development in the test is substantially slower. Colour development should be carried out between 18 and 22°C. To ensure correct conditions for the test, the Tubetests tubes should be brought within this temperature range prior to use.
 - 2 It is important to observe the standing period of 10 minutes \pm 1 minute for optimum test results. Any continuing colour development or colour change after this period should be ignored.
 - 3 Ammonia concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-

To convert from N to NH_4 multiply by 1.3
To convert from N to NH_3 multiply by 1.2
 - 4 Tubetests tubes are light sensitive. Store in the original packs and keep the lid closed.
 - 5 Interferences. Any substances that consume chlorine may lead to low results.
 - 6 The test can be used on sea or salt water without the need for pretreatment of the sample.
-

TUBETESTS® NITRATE/30N

TEST FOR NITRATE IN NATURAL,
DRINKING AND WASTE WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

**0 – 30 mg/l N
0 – 150 mg/l NO₃**

Nitrates are normally present in natural, drinking and waste waters. Nitrates enter water supplies from the breakdown of natural vegetation, the use of chemical fertilisers in modern agriculture and from the oxidation of nitrogen compounds in sewage effluents and industrial wastes.

Nitrate is an important control test for water supplies. Drinking waters containing excessive amounts of nitrates can cause methaemoglobinaemia in bottle-fed infants (blue babies). The EEC has set a recommended maximum level of 25 mg/l NO₃ (5.7 mg/l N) and an absolute maximum of 50 mg/l NO₃ (11.3 mg/l N) for nitrate in drinking water.

The Palintest Tubetests Nitrate/30N method provides a simple test for nitrate over the range 0 - 30 mg/l N (0 - 150 mg/l NO₃).

Note that the Tubetests Nitrate/30N reagent system is also used in the colour development stage of the Palintest Tubetests Total Nitrogen/30 test. See Instruction Sheet Phot.89 for the test instructions for this test.

Method

In the Palintest Tubetests Nitrate/30N method, nitrate reacts with chromotropic acid, under strongly acidic conditions to produce a yellow colour. Chemicals are incorporated to prevent interference from nitrite, chloride, iron (Fe^{III}), chlorine and other oxidising agents. The reagents are provided in the form of a predispensed tube and a powder. The test is simply carried out by adding a sample of the water and a scoop of powder to a tube.

The intensity of the colour produced in the test is proportional to the nitrate concentration and is measured using a Palintest Photometer.

Working Practice

The Palintest Tubetests Nitrate test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice. The reagent tubes contain 90% sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the reagent tubes to add the water sample as heat will be produced and gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples, the test must always be carried out in a fume cupboard. It is generally recommended that the test be conducted in a fume cupboard where available.

Reagents and Equipment

Palintest Tubetests Nitrate/30N Tubes

Palintest Tubetests Nitrate Powder

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 1 ml (PT 574)

Palintest Dosing Scoop - Size 1 (PT 691)

Palintest Dosing Funnel (PT 690)

Palintest Dosing Scoop Scraper (PT 695)

Use of Dosing Scoop and Funnel

This Tubetests method uses a Palintest Dosing Scoop and Funnel. The scoop and funnel are specially designed to ensure accurate dosing of reagent powders into the Tubetests tubes :-

- 1 Dip the scoop into the powder and ensure that it is completely filled. Draw the scraper across the top of the scoop to ensure a level fill.
- 2 Place the funnel on top of the Tubetests tube. Locate the scoop in the groove on the side of the funnel. Rotate the scoop to invert then tap gently to ensure that all the reagent goes into the tube.

Test Instructions

- 1 Remove the cap of the Tubetests Nitrate/30N Tube and add 1.0 ml of sample using a pipettor. For optimum results, the sample should be added slowly without disturbing the contents of the tube. DO NOT SHAKE THE TUBE.
- 2 Add one level scoop of Tubetests Nitrate Powder using a Size 1 dosing scoop. Cap tube and gently invert five or six times to dissolve and mix the reagents and sample.
- 3 Stand for five minutes to allow colour development.
- 4 Select Phot 87 on Photometer for results as mg/l N or Phot 88 for results as m/l NO_3 .
- 5 Take photometer reading in usual manner (see Photometer instructions). Use an unused Tubetests Nitrate tube to set the blank on the photometer.

Interferences

The test system incorporates reagents to prevent potential interferences from nitrite, chloride, iron (Fe^{III}), and chlorine and other oxidising agents. Interference studies have shown that levels up to nitrite 10 mg/l, chloride 1,000 mg/l, iron 40 mg/l and chlorine 5 mg/l do not effect the result of the test.

Notes

- 1 Tubetests Nitrate Powder is light sensitive. Store in original pack and keep lid closed when not in use.
- 2 Disposal. The used Tubetests Nitrate/30N Tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents must be disposed of in accordance with waste regulations and the laid-down disposal procedures of the laboratory of use.

TUBETESTS® TOTAL NITROGEN/30

TEST FOR TOTAL PERSULPHATE NITROGEN
IN NATURAL AND WASTE WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 30 mg/l N

Total nitrogen is a vital test for assessing the quality of effluents and waste water prior to discharge. In the UK the Urban Waste Water Treatment Regulations (1994) make provision for the control of discharge of total nitrogen to sensitive bodies of natural water. The monitoring of the rate of nitrogen removal is therefore of great importance in waste water treatment. Total nitrogen is composed of nitrate, nitrite, ammonium and organic nitrogen compounds. The Palintest Tubetests Total Nitrogen/30 test provides a simple method of measuring total persulphate nitrogen over the range 0 – 30 mg/l N.

Method

The Palintest Tubetests Total Nitrogen/30 test is a simple two stage procedure. The sample is initially digested with alkaline persulphate to break down nitrogenous compounds which are then converted to nitrate. The digested sample is then transferred to a Palintest Tubetests Nitrate/30N Tube for determination of the total nitrogen present. The reagents are provided in the form of predispensed tubes and powders. The powders are added using a specially designed scoop and funnel.

The intensity of the colour produced in the test is proportional to the total nitrogen concentration and is measured using a Palintest Photometer.

In total nitrogen determinations, the recovery of different compounds depends to an extent on the method of oxidation used to make the conversion to nitrate. It is normal practice to refer to the method of oxidation when stating test results for any formal purpose. Results from the Palintest Total Nitrogen test should therefore be expressed as 'Total Persulphate Nitrogen'.

Working Practice

The Palintest Tubetests Total Nitrogen/30 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

The Total Nitrogen Tubes contain sodium hydroxide solution, to which potassium persulphate is added. The Tubetests Nitrate/30N Tubes contain strong sulphuric acid. These reagents must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when adding Tubetests Total Nitrogen Reagent No 2 to the digestion tubes. Sulphur dioxide will be evolved. Care should be taken when opening the Tubetests Nitrate/30N Tube which contains concentrated acid. On adding the digestate heat will be produced, the tube will become hot and gases may be evolved. It is generally recommended that the test be conducted in a fume cupboard where available, particularly in the case of samples originally known to contain toxic materials such as cyanide or sulphide.

Reagents and Equipment - Digestion Stage

Palintest Tubetests Total Nitrogen Tubes
Palintest Tubetests Total Nitrogen Reagent No 1
Palintest Tubetests Total Nitrogen Reagent No 2
Palintest Digital Tubetests Heater (PT 589)
Palintest Tubetests Heater Safety Screen (PT 590)
Palintest Pipettor 5 ml (PT 576)
Palintest Dosing Scoop - Size 1 (PT 691)
Palintest Dosing Scoop - Size 4 (PT 694)
Palintest Dosing Funnel (PT 690)
Palintest Dosing Scoop Scraper (PT 695)

Reagents and Equipment - Colour Development Stage

Palintest Tubetests Nitrate/30N Tubes
Palintest Tubetests Nitrate Powder
Palintest Automatic Wavelength Selection Photometer
Palintest Pipettor, 1 ml (PT 574)
Palintest Dosing Scoop - Size 1 (PT 691)
Palintest Dosing Funnel (PT 690)
Palintest Dosing Scoop Scraper (PT 695)

Use of Dosing Scoop and Funnel

This Tubetests method uses Palintest Dosing Scoops and Funnels. The scoops and funnels are specially designed to ensure accurate dosing of reagent powders into the Tubetests tubes :-

- 1 Select the correct size scoop. Dip the scoop into the powder and ensure that it is completely filled. Draw the scraper across the top of the scoop to ensure a level fill.
- 2 Place the funnel on top of the Tubetests tube. Locate the scoop in the groove on the side of the funnel. Rotate the scoop to invert then tap gently to ensure that all of the reagent goes into the tube.

Test Instructions - Digestion Stage

- 1 Turn on the Tubetests heater, set the control to the 105°C mark and allow to heat up to temperature.
- 2 Remove the cap of the Tubetests Total Nitrogen Tube and add three level scoops of Tubetests Total Nitrogen Reagent No 1 using the Size 1 dosing scoop and funnel.
- 3 Add 5.0 ml of sample using a pipettor. Replace the cap tightly and shake the tube vigorously for 30 seconds.
- 4 Label the tube and place in the Tubetests heater. Ensure the safety screen is in position and digest the tube for 30 minutes, then turn off the heater.
- 5 Carefully remove each tube and transfer to a test tube rack. Handle hot tubes by the cap only.
- 6 Allow tubes to cool to room temperature.
- 7 Remove the cap of the Tubetests Total Nitrogen Tube and add one level scoop of Tubetests Total Nitrogen Reagent No 2 using the Size 4 dosing scoop and funnel. *Take care - sulphur dioxide will be evolved.*
- 8 Cap the tube and shake for 15 seconds, then stand for 3 minutes.

Test Instructions - Colour Development Stage

- 1 Using a pipettor, transfer 1 ml of digested sample from the Tubetests Total Nitrogen Tube to a Tubetests Nitrate/30N Tube. Take care to add the digestate slowly. DO NOT SHAKE THE TUBE.
- 2 Add one level scoop of Tubetests Nitrate Powder using the Size 1 dosing scoop and funnel. Cap tube and invert slowly ten times to dissolve and mix the reagents and sample. Take care! The tube will become hot.
- 3 Stand for five minutes to allow colour development.
- 4 Select Phot 89 on Photometer.
- 5 Take photometer reading in usual manner (see Photometer instructions). Use an unused Tubetests Nitrate/30N Tube to set the blank on the photometer.
- 6 The result is displayed as mg/l N.

Notes

- 1 This method is based on the Persulphate Method from 'Standard Methods for the Examination of Water and Waste Water' 19th Edition 1995, Pages 4 - 95. The method, in general, does not yield 100% recovery. Recoveries of various nitrogen compounds have been tested in the Palintest laboratories. Inorganic compounds such as potassium nitrate, sodium nitrite and ammonium chloride yield in excess of 95% recovery. The typical recoveries of some organic nitrogen compounds are quoted below :-

Compound	Typical Recovery
Glycine	95% all levels
Urea	90% all levels
Nicotinic Acid	95% at 10 mg/l, 45% at 30 mg/l
Creatinine	100% at 10 mg/l, 70% at 30 mg/l

- 2 Tubetests Nitrate Powder is light sensitive. Store in original pack and keep lid closed when not in use.
 - 3 Disposal. The used Tubetests Nitrate/30N Tubes contain strong sulphuric acid and other chemical reagents and care must therefore be exercised in their disposal. The tube contents should be disposed of in accordance with the laid-down disposal procedures of the laboratory of use.
-

TUBETESTS® PHOSPHATE/12P

TEST FOR PHOSPHATE IN NATURAL,
DRINKING WATER AND WASTE WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 12 mg/l P

0 – 36 mg/l PO₄

Phosphates are extensively used in detergent formulations, in food processing and in industrial water treatment processes. These phosphates may be in the form of orthophosphates, or are broken down to orthophosphates in the process concerned. Agricultural fertilisers normally contain phosphate minerals. Phosphates also arise from the breakdown of plant materials and are found in animal wastes.

Phosphates can therefore enter water courses through a variety of routes particularly domestic and industrial effluents and run-off from agricultural land. Phosphate is an important control test for natural and drinking waters.

Whilst phosphates are not generally considered harmful for human consumption, they do exhibit a complex effect on the natural environment. In particular, phosphates are associated with eutrophication of water and with rapid unwanted plant growth in rivers and lakes. Phosphates present in natural water pass through into drinking water supplies.

The Palintest Tubetests Phosphate/12P test provides a simple method of measuring orthophosphate levels over the range 0 - 12 mg/l P (0 - 36 mg/l PO₄). For drinking water, the EC has set a maximum admissible concentration of 2.2 mg/l P (6.7 mg/l PO₄).

Method

In the Palintest Tubetests Phosphate/12P method, the phosphate reacts under acid conditions with ammonium molybdate to form phospho-molybdic acid. The compound is reduced by ascorbic acid to form the intensely coloured 'molybdenum blue' complex. A catalyst is incorporated to ensure complete and rapid colour development, and an inhibitor is used to prevent interference from silica. The reagents are provided in the form of a predispensed tube and two tablets for maximum convenience. The test is simply carried out by adding a sample of the water and one of each tablet.

Reagents and Equipment

Palintest Tubetests Phosphate/12P Tubes

Palintest Tubetests Phos No 1 Tablets

Palintest Tubetests Phos No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 2 ml (PT 572)

Test Instructions

- 1 Remove the cap of the Tubetests Phosphate/12P Tube and add 2.0 ml of sample using a pipettor.
- 2 Add one Tubetests Phos No 1 tablet, crush and mix to dissolve. Ensure the tablet is completely dissolved.
- 3 Add one Tubetests Phos No 2 tablet, crush and mix to dissolve. Cap tube and gently invert several times to mix.
- 4 Stand for 10 minutes to allow colour development.
- 5 Select Phot 90 on Photometer for result as mg/l P or Phot 91 for result as mg/l PO_4 .
- 6 Take photometer reading in the usual manner (see Photometer instructions). Use an unused Tubetests Phosphate Tube to set the blank on the photometer. Alternatively, a Tubetests tube containing deionised water only may be used.

Notes

- 1 Phosphate concentrations can be expressed in a number of different ways :-

To convert from PO_4 to P_2O_5 - multiply by 0.75

TUBETESTS®

TOTAL PHOSPHORUS/12

**TEST FOR PHOSPHATE COMPOUNDS
IN NATURAL AND WASTE WATER**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 12 mg/l P

Total Phosphorus is composed of orthophosphates, polyphosphates and organic phosphorus compounds. Ortho and polyphosphates are extensively used in detergent formulations and washing powders. Phosphates also find widespread application in the food processing industry and in industrial water treatment processes. Agricultural fertilisers normally contain phosphate minerals. Phosphates also arise from the breakdown of plant materials and are found in animal wastes. Organic phosphate compounds are used in industrial and water treatment applications; and arise from certain manufacturing processes.

Phosphorus compounds can therefore enter water courses through a variety of routes - particularly domestic and industrial effluents and run-off from agricultural land. Phosphates are associated with eutrophication of water and with rapid unwanted plant growth in rivers and lakes.

The Total Phosphorus test is a vital test for assessing the quality of effluents and waste water prior to discharge. In the UK the Urban Waste Water Treatment Regulations make provision for the control of discharge of total phosphorus to sensitive bodies of natural waste water. The monitoring of the rate of phosphorus removal is therefore of great importance in waste water treatment. The Palintest Tubetests Total Phosphorus Test provides a simple method of measuring total phosphorus compounds over the range 0 – 12 mg/l P.

Method

The Palintest Tubetests Total Phosphorus/12 test is a simple two-stage procedure. The sample is first digested with acid persulphate to break down polyphosphates and organic phosphorus compounds and convert them to orthophosphate. The resulting orthophosphate, together with that originally present in the sample, is then determined by reaction with ammonium molybdate and ascorbic acid to form the intensely coloured 'molybdenum blue' complex. In this way, the total phosphorus content of the sample can be determined. A catalyst is incorporated to ensure complete and rapid colour development, and an inhibitor is used to prevent interference from silica.

The intensity of the colour produced in the test is proportional to the total phosphorus concentration, and is measured using a Palintest Photometer.

Reagents and Equipment - Digestion Stage

Palintest Tubetests Total Phosphorus/12 Tubes

Palintest Digest Ox Tablets

Palintest Tubetests Heater Palintest Pipettor, 2 ml (PT 572)

Reagents and Equipment - Colour Development Stage

Palintest Tubetests PhosNeut Solution

Palintest Tubetests Phos No 1 Tablets

Palintest Tubetests Phos No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Test Instructions - Digestion Stage

- 1 Turn on Tubetests Heater, set the control to 100 - 105°C (212 - 221°F) and allow to heat up to temperature.
- 2 Remove the cap of the Tubetests Total Phosphorus/12 Tube and add 2.0 ml of sample using a pipettor.
- 3 Add two Digest Ox tablets, crush and mix to dissolve.
- 4 Replace the cap tightly and invert tube gently to mix. Label the tube and place in the Tubetests heater. Ensure the safety screen is in position.
- 5 Digest the tube for one hour (minimum 45 minutes) then turn off the heater unless it is required for further tests.
- 6 Carefully remove the tube, transfer to a test tube rack and allow to cool to room temperature.

Test Instructions - Colour Development Stage

- 1 Carefully remove the cap from the cooled tube and add 2.0 ml of PhosNeut Solution using a pipettor.
 - 2 Add one Tubetests Phos No 1 tablet, and crush and mix to dissolve. Ensure all particles of the tablet have dissolved.
 - 3 Add one Tubetests Phos No 2 tablet, crush and mix to dissolve. Cap tube and gently invert several times to mix.
 - 4 Stand tube for 10 minutes to allow colour development.
 - 5 Select Phot 92 on Photometer.
 - 6 Take the photometer reading in usual manner (see Photometer instructions). Use an unused Tubetests Total Phosphorus/12 Tube to set the blank on the photometer. Alternatively, a Tubetests tube containing deionised water only may be used.
 - 7 The result is displayed as mg/l P.
-

TUBETESTS® AMMONIA/15N (NESSLER)

**TEST FOR AMMONIA IN NATURAL,
SEA AND WASTE WATER**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 15 mg/l N

Ammonia occurs as a breakdown product of nitrogenous material in natural waters. It is also found in domestic effluents and certain industrial waste waters. Ammonia is harmful to fish and other forms of aquatic life and the ammonia level must be carefully controlled in water used for fish farms and aquariums. Ammonia tests are routinely applied for the monitoring of natural water, sea water; and for pollution control on effluents and waste waters.

The Palintest Tubetests Ammonia/15N (Nessler) test provides a simple method of measuring ammonia (ammoniacal nitrogen) over the range 0 - 15 mg/l N.

Method

The Palintest Tubetests Ammonia/15N (Nessler) test is based on the Nessler method. Nessler's reagent (potassium tetraiodomercurate (II)) reacts rapidly with ammonia under alkaline conditions to form an orange-brown product. Turbidity due to hardness salts is prevented by adding the sample to a solution of Rochelle salt prior to adding the Nessler reagent.

The intensity of the colour produced in the test is proportional to the ammonia concentration and is measured using a Palintest Photometer or Ammonia Meter.

Reagents and Equipment

Palintest Tubetests Ammonia 15N (Nessler) Tubes

Palintest Tubetests Ammonia (Nessler) Reagent

Palintest Automatic Wavelength Selection Photometer or Ammonia Meter

Palintest Syringe, 5 ml (PT 365)

Test Instructions

- 1 Remove the cap of the Tubetests Ammonia/15N (Nessler) Tube and add 5.0 ml of sample to fill the tube to the 10 ml line. Cap tube and invert three times to mix.
- 2 Add **12 drops** of Tubetests Ammonia (Nessler) Reagent. Replace cap and invert several times to mix.
- 3 Stand for one minute to allow colour development.
- 4 Select Phot 93 on Photometer, or select range 15N on Ammonia Meter.
- 5 Take photometer reading in usual manner (see Photometer instructions).
Use an unused Tubetests Ammonia/15N (Nessler) Tube to set the blank on the photometer.

Alternatively, a Tubetests tube containing deionised water only may be used. If the test sample has an inherent colour, then the sample must be used to create the blank.
- 6 The result is displayed as mg/l N.

Notes

- 1 Nessler's reagent is toxic. Handle with care. This reagent is for use in professional water testing applications only.
- 2 Nessler's reagent is sensitive to air. Replace cap when not in use.
- 3 Ammonia concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-

To convert from N to NH_4 - multiply by 1.3
To convert from N to NH_3 - multiply by 1.2
- 4 Interferences. Sufficient Rochelle salt is present to prevent turbidity due to at least 1,000 mg/l hardness. The test can be used on sea or salt water without the need for pre-treatment of the sample.

Disposal

Used Ammonia (Nessler) tubes contain alkaline mercury salts - which are toxic. Care must therefore be exercised in their disposal. The tubes must be disposed of in accordance with current waste legislation and consent limits. Used tubes must always be treated using a proper waste disposal system. A tube disposal service is available through Palintest Ltd (UK only). The tubes must not be reused as they are designed for single use only.

TUBETESTS® AMMONIA/50N (NESSLER)

**TEST FOR AMMONIA IN NATURAL,
SEA AND WASTE WATER**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 50 mg/l N

Ammonia occurs as a breakdown product of nitrogenous material in natural waters. It is also found in domestic effluents and certain industrial waste waters. Ammonia is harmful to fish and other forms of aquatic life and the ammonia level must be carefully controlled in water used for fish farms and aquariums. Ammonia tests are routinely applied for the monitoring of natural water, sea water; and for pollution control on effluents and waste waters.

The Palintest Tubetests Ammonia/50N (Nessler) test provides a simple method of measuring ammonia (ammoniacal nitrogen) over the range 0 - 50 mg/l N.

Method

The Palintest Tubetests Ammonia/50N (Nessler) test is based on the Nessler method. Nessler's reagent (potassium tetraiodomercurate (II)) reacts rapidly with ammonia under alkaline conditions to form an orange-brown product. Turbidity due to hardness salts is prevented by adding the sample to a solution of Rochelle salt prior to adding the Nessler reagent.

The intensity of the colour produced in the test is proportional to the ammonia concentration and is measured using a Palintest Photometer or Ammonia Meter.

Reagents and Equipment

Palintest Tubetests Ammonia 50N (Nessler) Tubes

Palintest Tubetests Ammonia (Nessler) Reagent

Palintest Automatic Wavelength Selection Photometer or Ammonia Meter

Palintest Syringe, 1 ml (PT 361)

Test Instructions

- 1 Remove the cap of the Tubetests Ammonia/50N (Nessler) Tube and add 1.0 ml of sample to fill to the 10ml line. Cap tube and invert three times to mix.
- 2 Add **12 drops** of Tubetests Ammonia (Nessler) Reagent. Replace cap and invert several times to mix.
- 3 Stand for one minute to allow colour development.
- 4 Select Phot 94 on Photometer, or select range 50N on Ammonia Meter.
- 5 Take photometer reading in usual manner (see Photometer instructions).

Use an unused Tubetests Ammonia/50N (Nessler) Tube to set the blank on the photometer.

Alternatively, a Tubetests tube containing deionised water only may be used. If the test sample has an inherent colour, then the sample must be used to create the blank.

- 6 The result is displayed as mg/l N.

Notes

- 1 Nessler's reagent is toxic. Handle with care. This reagent is for use in professional water testing applications only.
- 2 Nessler's reagent is sensitive to air. Replace cap when not in use.
- 3 Ammonia concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-

To convert from N to NH_4 - multiply by 1.3

To convert from N to NH_3 - multiply by 1.2

- 4 Interferences. Sufficient Rochelle salt is present to prevent turbidity due to at least 1,000 mg/l hardness. The test can be used on sea or salt water without the need for pre-treatment of the sample.

Disposal

Used Ammonia (Nessler) tubes contain alkaline mercury salts - which are toxic. Care must therefore be exercised in their disposal. The tubes must be disposed of in accordance with current waste legislation and consent limits. Used tubes must always be treated using a proper waste disposal system. A tube disposal service is available through Palintest Ltd (UK only). The tubes must not be reused as they are designed for single use only.

TUBETESTS® IRON/25

TEST FOR IRON IN EFFLUENTS, WASTE
WATERS AND INDUSTRIAL WATER SAMPLES

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 25 mg/l

Iron occurs widely in nature and is found in many natural and treated waters. Iron is an objectionable constituent in both domestic and industrial water supplies. The presence of iron affects the taste of beverages and causes unsightly staining of laundered clothes, plumbing fittings, swimming pool surfaces and the like. The formation of insoluble iron deposits is troublesome in many industrial applications and in agricultural uses such as drip feed irrigation.

Iron is an important test for effluents, waste waters and industrial water samples. The sources of iron in such samples are many and varied and include the corrosion of plant and equipment and waste from industrial processes. The Palintest Tubetests Iron/25 test is designed to measure the total recoverable iron concentration over the range 0 - 25 mg/l.

Method

The Palintest Tubetests Iron/25 test is particularly applicable to the analysis of effluents, waste waters and industrial water samples. In such samples, the metal ions are often present in complexed, colloidal or particulate form. Moreover, effluents and waste waters typically contain colour and suspended solids. Special techniques are necessary therefore for the analysis of metals in these types of waters. The Palintest Tubetests Iron/25 test is designed to measure the total recoverable iron concentration in such samples.

In the Palintest Tubetests Iron/25 test the sample is first digested in a sulphuric acid/nitric acid mixture in order to solubilise particulate matter, break down complexes and remove colour. The acid digest mixture is provided pre-dispensed into special digestion tubes for ease of use and maximum safety.

Following the digestion stage, the sample is neutralised and buffered to provide the correct pH conditions for the test. A reducing agent is then added to convert all of the iron to ferrous form and this is then reacted with 1,10 phenanthroline to form an orange coloured complex. Decomplexing agents and inhibitors are incorporated into the test reagent system in order to break down any chelated iron which is present and to prevent interference from other metal ions commonly found in effluents and waste water samples.

The intensity of the colour produced in the test is proportional to the iron concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Iron/25 Pack (PL 434) containing :-

Metaltube Digest Tubes

Metaltube Neut Reagent

Metaltube Buffer

Irontube No 1 Tablet

Irontube No 2 Tablet

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 2 ml (PT 572)

Working Procedure

The Palintest Tubetests Iron/25 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

Palintest Metaltube Digest tubes contain approximately 40% mixed nitric/sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the digest tubes to add the sample, or to add reagents, as gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples, the test must always be carried out in a fume cupboard. It is generally recommended that the test be conducted in a fume cupboard where available.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage.

Sample Preparation

Effluents and waste waters often contain undissolved or particulate material. Such samples should be homogenised thoroughly prior to taking the test sample in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests Heater, set the control to 105°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest tube and add 2 ml of sample using a Palintest pipettor with disposable tip or a standard laboratory pipette.
- 3 Replace the cap tightly and invert tube to mix contents. Place the tube in the Tubetests heater. Digest the tube for 60 minutes then remove and transfer to a test tube rack. Allow the tube to cool for approximately 10 minutes.
- 4 Add 2 ml of Metaltube Neut Reagent to the tube using a Palintest pipettor with disposable tip or a standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents. The tube will become hot on mixing. Allow the tube to cool for approximately 10 minutes.
- 5 Add 2 ml of Metaltube Buffer to the tube using a Palintest pipettor with disposable tip or a standard laboratory pipette, then fill the tube to the graduation line (10 ml) with deionised water. Replace the cap tightly and invert the tube gently to mix the contents.
- 6 Remove the cap from the tube and then add one Irontube No 1 tablet, crush and mix to dissolve.
- 7 Add one Irontube No 2 tablet, crush and mix to dissolve and then replace the cap on the tube.
- 8 Stand for 10 minutes without disturbing the solution to allow full colour development and to allow any undissolved particles to settle.
- 9 Prepare a Blank tube by filling a Metaltube Digest Reagent tube to the graduation line (10 ml) with deionised water. Cap the tube and invert to mix. This tube can be kept and used again for any subsequent Tubetests Iron/25 testing.
- 10 Select Phot 95 on the Photometer.
- 11 Wipe the tubes with a soft tissue to remove any finger marks and smears and then take photometer reading in the usual manner (see Photometer instructions).
- 12 The result is displayed as mg/l Fe.

Interferences

In interference studies the presence of chromium, nickel and zinc have been found not to cause any effect on the test result. The presence of molybdate will cause proportionately high readings and will give a false positive response in the absence of iron. Copper levels greater than 2 mg/l and lead levels greater than 4 mg/l will cause slightly high readings and will give a false positive response in the absence of iron.

Tests with samples containing common anions and non-metallic species showed that there was no significant interference.

Tests using this procedure with a variety of industrial waste waters showed that in most cases the colour and turbidity found in such samples were reduced to a level where they did not interfere with the test result :-

However, in some extreme cases there may be noticeable colour or turbidity remaining. This may be the case for example with samples taken from pre-treatment streams or effluent treatment tanks. Unless compensation is made for this colour or turbidity, it will lead to an inaccurate result. In such cases, it is recommended to use a compensating blank by using the following procedure :-

Prepare two tubes of the same sample by following the test procedure up to and including Step 6. However at this point only continue the procedure using one of the tubes. Use the other tube, the 'Compensating Blank', in place of the normal blank tube described in Step 9 when taking the photometer reading. This will help compensate for any colour/turbidity present in the sample.

Tubetests Heater

The Palintest Digital Tubetests Heater (PT 589) is a 12-tube block heater featuring a digital display. The heater is dedicated for use with the Palintest Tubetests system. It comprises an electrically controlled dry bath which heats an aluminium test block. The heater is designed to provide the correct digesting and refluxing conditions for Tubetests tubes.

The heater features a digital display for the operating temperature and set temperature. The heater should be set to the temperature stated in the test procedure. On no account must the heater be set to a higher temperature than that specified as this may cause a hazard through pressure build-up in the tubes. It is not necessary to use a thermometer with the heater - the operating temperature is shown on the display. The temperature setting takes into account the thermal lag between the block and the heating tubes. The heater also features a timer, which is located on the base of the heater. The heater can be pre-set to operate for a predetermined time to suit particular test procedures.

TUBETESTS® NICKEL/20

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

TEST FOR NICKEL IN EFFLUENTS, WASTE
WATERS AND INDUSTRIAL WATER SAMPLES

0 – 20 mg/l

Nickel does not occur naturally in water apart from a few areas around the world where nickel-bearing ores are present. Nickel is however commonly found in industrial waste waters such as those from the steel industry and from plating processes. Nickel is considered an undesirable element in water supplies and careful monitoring of effluent and waste waters is necessary to prevent this element entering the aqueous environment.

The Palintest Tubetests Nickel/20 test is designed to measure total recoverable nickel concentration over the range 0 - 20 mg/l.

Method

The Palintest Tubetests Nickel/20 test is particularly applicable to the analysis of effluents, waste waters and industrial water samples. In such samples the metal ions are often present in complexed, colloidal or particulate form. Moreover effluents and waste waters typically contain colour and suspended solids. Special techniques are necessary therefore for the analysis of metals in these types of waters. The Palintest Tubetests Nickel/20 test is designed to measure the total recoverable nickel concentration in such samples.

In the Palintest Tubetests Nickel/20 test the sample is first digested in a sulphuric acid/nitric acid mixture in order to solubilise particulate matter, break down complexes and remove colour. The acid digest mixture is provided pre-dispensed into special digestion tubes for ease of use and maximum safety.

Following the digestion stage, the sample is neutralised and buffered to provide the correct pH conditions for the test. A reducing agent is then added to convert all of the nickel to nickelous form and this is then reacted with nioxime indicator to form a pink-coloured complex. Inhibitors are incorporated into the test reagent system in order to prevent interference from iron and other metal ions commonly found in effluents and waste water samples.

The intensity of the colour produced in the test is proportional to the nickel concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Nickel/20 Pack (PL 430) containing :-

- Metaltube Digest Tubes
- Metaltube Neut Reagent
- Metaltube Buffer
- Nickeltube No 1 Tablet
- Nickeltube No 2 Tablet
- Nickeltube Powder
- Dosing Scoop - Size 1
- Dosing Funnel
- Dosing Scoop Scraper

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 2 ml (PT 572)

Palintest Pipettor, 5 ml (PT 576)

Working Procedure

The Palintest Tubetests Nickel/20 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

Palintest Metaltube Digest tubes contain approximately 40% mixed nitric/sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the digest tubes to add the sample, or to add reagents, as gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples the test must always be carried out in a fume cupboard, it is generally recommended that the test be conducted in a fume cupboard where available.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage.

Use of Dosing Scoop and Funnel

This Tubetests method uses a Palintest Dosing Scoop and Funnel. The scoop and funnel are specially designed to ensure accurate dosing of reagent powders into the Tubetests tubes :-

- 1 Dip the scoop into the powder and ensure that it is completely filled. Draw the scraper across the top of the scoop to ensure a level fill.
- 2 Place the funnel on top of the Tubetests tube. Locate the scoop in the groove on the side of the funnel. Rotate the scoop to invert then tap gently to ensure that all the reagent goes into the tube.

Sample Preparation

Effluents and waste waters often contain undissolved or particulate material. Such samples should be homogenised thoroughly prior to taking the test sample in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests Heater, set the control to 105°C and place the safety shield in position. Allow the heater to heat up to temperature (see Tubetests Heater).
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest Reagent tube and add 5 ml of sample using a Palintest pipettor with disposable tip or a standard laboratory pipette.
- 3 Replace the cap tightly and invert tube to mix contents. Place the tube in the Tubetests heater. Digest the tube for 60 minutes then remove and transfer to a test tube rack. Allow the tube to cool for approximately 10 minutes.
- 4 Add 2 ml of Metaltube Neut Reagent to the tube using a Palintest pipettor with disposable tip or a standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents. The tube will become hot on mixing. Allow the tube to cool for approximately 10 minutes.
- 5 Add 2 ml of Metaltube Buffer to the tube using a Palintest pipettor with disposable tip or a standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents.
- 6 Remove the cap from the tube and then add one Nickeltube No 1 tablet, crush and mix to dissolve.
- 7 Add one level scoop of Nickeltube Powder using a Size 1 dosing scoop. Cap tube and shake to dissolve powder.
- 8 Remove the cap and then add one Nickeltube No 2 tablet. Crush tablet and mix to dissolve and then replace the cap on the tube.
- 9 Stand for 5 minutes without disturbing the solution to allow full colour development. Invert the tube to ensure even distribution of indicator and then stand for 2 minutes to allow any undissolved particles to settle.
- 10 Prepare a Blank tube by filling a Metaltube Digest Reagent tube to the graduation line (10 ml) with deionised water. Cap the tube and invert to mix. This tube can be kept and used again for any subsequent Nickel/20 testing.
- 11 Select Phot 96 on the Photometer.
- 12 Wipe the tubes with a soft tissue to remove any finger marks and smears and then take photometer reading in the usual manner (see Photometer instructions).
- 13 The result is displayed as mg/l Ni.

Interferences

In interference studies the presence of chromium, copper, iron and zinc have been found not to cause any effect on the test result. Cobalt levels greater than 1 mg/l have been found to give a false positive response in the absence of nickel.

Tests with samples containing common anions and non-metallic species showed that there was no significant interference.

Tests using this procedure with a variety of industrial waste waters showed that in most cases the colour and turbidity found in such samples were reduced to a level where they did not interfere with the test result.

However, in some extreme cases there may be noticeable colour or turbidity remaining. This may be the case for example with samples taken from pre-treatment streams or effluent treatment tanks. Unless compensation is made for this colour or turbidity, it will lead to an inaccurate result. In such cases it is recommended to use a compensating blank by using the following procedure :-

Prepare two tubes of the same sample by following the test procedure up to and including Step 7. However at this point only continue the procedure using one of the tubes. Use the other tube, the 'Compensating Blank', in place of the normal blank tube described in Step 10 when taking the photometer reading. This will help compensate for any colour/turbidity present in the sample.

Tubetests Heater

The Palintest Digital Tubetests Heater (PT 589) is a 12-tube block heater featuring a digital display. The heater is dedicated for use with the Palintest Tubetests system. It comprises an electrically controlled dry bath, which heats an aluminium test block. The heater is designed to provide the correct digesting and refluxing conditions for Tubetests tubes.

The heater features a digital display for the operating temperature and set temperature. The heater should be set to the temperature stated in the test procedure. On no account must the heater be set to a higher temperature than that specified as this may cause a hazard through pressure build-up in the tubes. It is not necessary to use a thermometer with the heater - the operating temperature is shown on the display. The temperature setting takes into account the thermal lag between the block and the heating tubes. The heater also features a timer, which is located on the base of the heater. The heater can be pre-set to operate for a predetermined time to suit particular test procedures.

TUBETESTS® ZINC/7/35

TEST FOR ZINC IN NATURAL AND
EFFLUENT WASTE WATERS AND
INDUSTRIAL WATER SAMPLES

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

**0 – 7 mg/l and
0 – 35 mg/l**

Zinc is found in nature usually as metallic sulphide ores. Zinc and zinc compounds are extensively used in galvanising, in the manufacture of alloys and as corrosion inhibitors in industrial cooling water systems. Zinc is therefore a common constituent of industrial effluents and careful monitoring is necessary to prevent this element entering the aqueous environment.

The Palintest Tubetests Zinc/7/35 test is designed to measure total recoverable zinc concentrations over the ranges 0 - 7 mg/l and 0 - 35 mg/l.

Method

The Palintest Tubetests Zinc/7/35 test is particularly applicable to the analysis of effluents, waste waters and industrial water samples. In such samples, the metal ions are often present in complexed, colloidal or particulate form. Moreover effluents and waste waters typically contain colour and suspended solids. Special techniques are necessary therefore for the analysis of metals in these types of waters. The Palintest Tubetests Zinc/7/35 test is designed to measure the total recoverable zinc concentration in such samples.

In the Palintest Tubetests Zinc/7/35 test the sample is first digested in a sulphuric acid/nitric acid mixture in order to solubilise particulate matter, break down complexes and remove colour. The acid digest mixture is provided pre-dispensed into special digestion tubes for ease of use and maximum safety.

Following the digestion stage, the sample is neutralised and then complexed with thiocyanate ions to form zinc thiocyanate. This is then reacted with brilliant green indicator under acidic conditions to give a green coloration. The indicator itself is yellow so over the range of zinc levels under test a distinctive range of colours from pale yellow to dark green is produced. Inhibitors are incorporated into the test reagent system in order to prevent interference from other species commonly found in effluent and waste water samples.

The colour produced in the test is indicative of the zinc concentration and is measured using a Palintest Photometer. Since the colour fades rapidly for a short time after formation, the measurement is not taken until after 10 minutes standing period in order to allow the colour to stabilise.

Reagents and Equipment

Palintest Tubetests Zinc/7/35 Pack (PL 442) containing :-

Metal tube Digest Tubes

Zinc tube Powder

Dosing Scoop - Size 4

Zinc tube Neut Reagent

Zinc tube Indicator Tablet *

Dosing Funnel

Zinc tube IR Tablet

Dosing Scoop Scraper

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 0.2 ml (PT 570) - for 0 - 35 mg/l Range

Palintest Pipettor, 1 ml (PT 574) - for 0 - 7 mg/l Range

Palintest Pipettor, 5 ml (PT 576)

*The indicator required in this test is provided in tablet form but must be made-up in deionised water prior to use in the test. The indicator solution is only stable for 10 minutes so the time of preparation is critical. A recommended approach is to measure 10 ml of deionised water into a 10 ml photometer tube or empty Tubetests tube before beginning the test and then dissolve the Zinc Indicator tablet during the appropriate two minute standing period. It is important to ensure that the tablet is thoroughly crushed and that all of the solid particles are dissolved.

Working Procedure

The Palintest Tubetests Zinc/7/35 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

Palintest Metal tube Digest tubes contain approximately 40% mixed nitric/sulphuric acid and must be handled with care. The use of appropriate protective clothing, gloves and safety spectacles is recommended. In the event of skin or eye contact, or spillage, wash immediately with large amounts of water.

Particular care should be taken when opening the reagent tubes to add the sample as gases may be evolved. Samples containing cyanide or sulphide will release toxic fumes and for such samples the test must always be carried out in a fume cupboard. It is generally recommended that the test be conducted in a fume cupboard where available.

Reagent tubes should not be opened whilst hot as pressure build-up may cause acid spillage.

Use of Dosing Scoop and Funnel

This Tubetests method uses a Palintest Dosing Scoop and Funnel. The scoop and funnel are specially designed to ensure accurate dosing of reagent powders into the Tubetests tubes :-

- 1 Dip the scoop into the powder and ensure that it is completely filled. Draw the scraper across the top of the scoop to ensure a level fill.
- 2 Place the funnel on top of the Tubetests tube. Locate the scoop in the groove on the side of the funnel. Rotate the scoop to invert then tap gently to ensure that all the reagent goes into the tube.

Sample Preparation

Effluents and waste waters often contain undissolved or particulate material. Such samples should be homogenised thoroughly prior to taking the test sample in order to improve accuracy and reproducibility.

Test Procedure

- 1 Turn on Tubetests Heater, set the control to 105°C and place the safety shield in position. Allow the heater to heat up to temperature. On no account must the heater be set to a higher temperature than that specified as this may cause a hazard through pressure build-up in the tubes.
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest Reagent tube and add either 1 ml of sample (for 0 - 7 mg/l range) or 0.2 ml of sample (for 0 - 35 mg/l range) using a Palintest pipettor with disposable tip or standard laboratory pipette.
- 3 Replace the cap tightly and invert tube to mix contents. Place the tube in the Tubetests heater. Digest the tube for 60 minutes then remove and transfer to a test tube rack. Allow the tube to cool for approximately 10 minutes.
- 4 Add 5 ml of Zinctube Neut Reagent to the tube using a Palintest pipettor with disposable tip or standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents. The tube will become hot on mixing. Allow the tube to cool for approximately 10 minutes. NB: The temperature of the solution affects the rate of colour development. For the most accurate result, ensure that the temperature remains between 15 and 25°C from this step onwards.
- 5 Fill the sample tube to the graduation mark (10 ml) with deionised water. Replace the cap tightly and invert the tube gently to mix the contents.
- 6 Remove the cap from the tube and add one Zinctube IR tablet, crush and mix to dissolve.
- 7 Add two level scoops of Zinctube Powder using a Size 4 dosing scoop. Cap tube and shake to dissolve powder. Stand the tube for two minutes to allow complete reaction.
- 8 During the two minute standing time make-up 10 ml of indicator solution by adding one Zinctube Indicator tablet to 10 ml of deionised water. Crush the tablet and mix thoroughly to dissolve. It is very important to ensure the tablet is completely dissolved. This indicator solution is only stable for 10 minutes and must be discarded and a fresh solution made for any further testing after this time.

- 9 When the two minute standing time has expired remove the cap from the sample tube and add 1 ml of the indicator solution using a Palintest pipettor with disposable tip or standard laboratory pipette. Replace the cap tightly and invert the tube gently to mix the contents.
- 10 Stand for 10 minutes without disturbing the solution to allow any undissolved particles to settle and to allow the colour to stabilise.
- 11 Prepare a Blank tube by filling a Metaltube Digest Reagent tube to the graduation line (10 ml) with deionised water, cap the tube and invert to mix. This tube can be kept and used again for any subsequent Zinc/7/35 testing.
- 12 Select Phot 97 on the Photometer for range 0 – 7 mg/l or Phot 98 for 0 – 35 mg/l.
- 13 Wipe the tube with a soft tissue to remove any finger marks and smears and then take photometer reading in the usual manner (see Photometer Instructions).

Interferences

In interference studies the presence of metals such as cadmium, copper, chromium, iron and nickel have all been found not to cause any effect on the test result.

Tests with samples containing common anions and non-metallic species showed that there was no significant interference.

Tests using this procedure with a variety of industrial waste waters showed that in most cases the colour and turbidity found in such samples were reduced to a level where they did not interfere with the test result.

However, in some extreme cases, there may be noticeable colour or turbidity remaining. This may be the case for example with samples taken from pretreatment streams or effluent treatment tanks. Unless compensation is made for this colour or turbidity, it will lead to an inaccurate result. In such cases it is recommended to use a compensating blank by using the following procedure :-

Prepare two tubes of the same sample by following the test procedure up to and including Step 7. However at this point only continue the procedure using one of the tubes. Use the other tube, the 'compensating blank', in place of the normal blank tube described in Step 11 when taking the photometer reading This will help compensate for any colour/turbidity present in the sample.

TUBETESTS® AMMONIA/100N (NESSLER)

**TEST FOR AMMONIA IN NATURAL,
SEA AND WASTE WATER**

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 100 mg/l N

Ammonia occurs as a breakdown product of nitrogenous material in natural waters. It is also found in domestic effluents and certain industrial waste waters. Ammonia is harmful to fish and other forms of aquatic life and the ammonia level must be carefully controlled in water used for fish farms and aquariums. Ammonia tests are routinely applied for the monitoring of natural water, sea water; and for pollution control on effluents and waste waters.

The Palintest Tubetests Ammonia/100N (Nessler) test provides a simple method of measuring ammonia (ammoniacal nitrogen) over the range 0 - 100 mg/l N.

Method

The Palintest Tubetests Ammonia/100N (Nessler) test is based on the Nessler method. Nessler's reagent (potassium tetraiodomercurate (II)) reacts rapidly with ammonia under alkaline conditions to form an orange-brown product. Turbidity due to hardness salts is prevented by adding the sample to a solution of Rochelle salt prior to adding the Nessler reagent.

The intensity of the colour produced in the test is proportional to the ammonia concentration and is measured using a Palintest Photometer or Ammonia Meter.

Reagents and Equipment

Palintest Tubetests Ammonia 100N (Nessler) Tubes
Palintest Tubetests Ammonia (Nessler) Reagent
Palintest Automatic Wavelength Selection Photometer
Palintest Syringe, 1 ml (PT 361)

Test Instructions

- 1 Remove the cap of the Tubetests Ammonia/100N (Nessler) Tube and add 0.5 ml of sample to fill to the 10ml line. Cap tube and invert three times to mix.
- 2 Add **12 drops** of Tubetests Ammonia (Nessler) Reagent. Replace cap and invert several times to mix.
- 3 Stand for one minute to allow colour development.
- 4 Select Phot 107 on Photometer *
- 5 Take photometer reading in usual manner (see Photometer instructions).

Use an unused Tubetests Ammonia/100N (Nessler) Tube to set the blank on the photometer.

Alternatively, a Tubetests tube containing deionised water only may be used. If the test sample has an inherent colour, then the sample must be used to create the blank.

- 6 The result is displayed as mg/l N.

* = not necessary when using the Compact Ammonia Meter

Notes

- 1 Nessler's reagent is toxic. Handle with care. This reagent is for use in professional water testing applications only.
- 2 Nessler's reagent is sensitive to air. Replace cap when not in use.
- 3 Ammonia concentrations can be expressed in a number of different ways. The following factors may be used for the conversion of readings :-

To convert from N to NH_4 - multiply by 1.3

To convert from N to NH_3 - multiply by 1.2

- 4 Interferences. Sufficient Rochelle salt is present to prevent turbidity due to at least 2,000 mg/l hardness. The test can be used on sea or salt water without the need for pre-treatment of the sample.

Disposal

Used Ammonia (Nessler) tubes contain alkaline mercury salts - which are toxic. Care must therefore be exercised in their disposal. The tubes must be disposed of in accordance with current waste legislation and consent limits. Used tubes must always be treated using a proper waste disposal system. A tube disposal service is available through Palintest Ltd (UK only). The tubes must not be reused as they are designed for single use only.

CHLORINE/10

TEST FOR FREE, COMBINED AND TOTAL CHLORINE IN WATER

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 10.0 mg/l

Chlorine and chlorine-release compounds are widely used for the disinfection of drinking water and swimming pools, for the control of micro-biological growth in cooling water, and in many other water treatment systems. Accurate measurement of the chlorine residual is an essential aspect of the control of these chlorination processes.

The chlorine level can be expressed in terms of the free chlorine, combined chlorine or total chlorine residuals. For the majority of applications measurement of the free chlorine residual is the most important. The Palintest Chlorine/10 method provides a simple means of measuring free, combined and total chlorine residuals over the range 0 – 10 mg/l.

It is recommended that if any shock treatment compounds are known to have been used in the treatment of the water to be tested, that a DPD Oxystop tablet be included in the test procedure as outlined below.

Method

This Palintest Chlorine/10 test uses the DPD method developed by Dr A T Palin and now internationally recognised as the standard method of testing for chlorine and other disinfectant residuals. In the Palintest DPD method the reagents are provided in tablet form for maximum convenience and simplicity of use.

Free chlorine reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. The intensity of the colour is proportional to the free chlorine concentration. Subsequent addition of excess potassium iodide induces a further reaction with any combined chlorine present. The colour intensity is now proportional to the total chlorine concentration; the increase in intensity represents the combined chlorine concentration. In this way it is possible to differentiate between free and combined chlorine present in the sample. The colour intensities are measured using a Palintest Photometer.

The DPD Oxystop tablet is added after measurement for free chlorine but before the DPD-XT tablet. It prevents the reaction between shock treatment chemicals and potassium iodide which would give a positive response.

Reagents and Equipment

Palintest DPD-XF Tablets

Palintest DPD Oxystop Tablets (Optional)

Palintest DPD-XT Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595)

Separation of Chlorine Residuals

The photometer is programmed for both free and total chlorine. Use program Phot 108 Free Chlorine, then select the 'Follow On' option on screen to continue test for program Phot 109 Total Chlorine.

Test Instructions

- 1 Rinse test tube with sample leaving two or three drops of sample in the tube.
- 2 Add one DPD XF tablet, crush tablet and then fill the test tube with sample to the 10 ml mark. Mix to dissolve tablet.
- 3 Select Phot 108 on Photometer.
- 4 Take Photometer reading immediately in usual manner - see Photometer instructions.
- 5 The result represents the free chlorine residual as milligrams per litre. Stop the test at this stage if only **free chlorine** determination is required.
- 6 If it is desired to measure combined or total chlorine residual continue the test on the same test portion. Select the 'Follow On' from screen options to continue the test program.
- 7 If shock treatment chemicals are present in the pool, add one DPD Oxystop tablet, crush and mix to dissolve. Stand for one minute before proceeding.
- 8 Add one DPD XT tablet, crush and mix to dissolve.
- 9 Stand for two minutes to allow full colour development.
- 10 Take photometer reading.
- 11 The result represents the **total chlorine** residual as milligrams per litre.
- 12 The **combined chlorine** residual is obtained by subtracting the free chlorine residual result from the total chlorine residual result :-

ie Combined Chlorine = Total Chlorine - Free Chlorine

Notes

A too high chlorine level (above 20 mg/l) can cause bleaching of the pink coloration formed in the DPD test and give a false negative or lower than expected result. If a colourless or pale pink test solution is obtained then a high level chlorine may be present, check for the possibility of bleaching by repeating the test on a sample diluted with chlorine-free water.

pH VALUE

TEST FOR pH VALUE OF WATER AND AQUEOUS SOLUTIONS

Photometer Method

AUTOMATIC
WAVELENGTH
SELECTION

4.4 – 9.6

pH value is a parameter frequently determined on water and aqueous solutions. The Palintest pH method provides a simple test for the determination of pH for a variety of applications. The test is available in four narrow ranges covering pH values between 4.4 and 9.6.

Method

Palintest pH methods use standard pH indicators in tablet form. Different indicators are used to cover different ranges. Each tablet contains the precise amount of indicator needed for the test. All Palintest pH tablets contain a dechlorinating agent so that the test can be carried out in water containing chlorine or other disinfectant residuals.

Reagents and Equipment

Palintest pH Indicator Tablets (see table below)
Palintest Automatic Wavelength Selection Photometer
Round Test Tubes, 10 ml glass (PT 595)

pH Range	Phot Number	Indicator Tablet	Code
4.4 – 6.0	110	Bromocresol Green	PM 136*
6.0 – 7.6	111	Bromothymol Blue	PM 138*
6.8 – 8.4	27	Phenol Red	PM 130*
8.0 – 9.6	112	Thymol Blue	PM 139*

Test Procedure

- 1 Select the appropriate indicator tablets for the pH range under test.
- 2 Fill test tube with sample to the 10 ml mark.
- 3 Add one pH indicator tablet, crush and mix to dissolve.
- 4 Select the appropriate Phot number on the Photometer.
- 5 Take Photometer reading in the usual manner (see Photometer instructions).
- 6 If the reading is outside the range of the indicator tablet selected, the test should be repeated using a higher or lower range.

* Tablets are available as 'Starter Packs' coded 'PM'
or 'Replacement Reagent Packs' coded 'AP'.

MANGANESE

TEST FOR SOLUBLE MANGANESE IN WATER

Photometer Method

**Auto Wavelength
Selection**

0 – 5 mg/l

Manganese-containing minerals occur widely and manganese salts are commonly found in many natural waters. Manganese is an objectionable constituent in water used for domestic purposes or industrial applications. In domestic situations, manganese will cause brown or black staining to laundry or plumbing fittings even at very low concentrations. In process applications such as paper manufacturing or textile finishing similar staining can occur. Manganese salts may impart an astringent taste to drinking water supplies, and in swimming pool applications can give an aesthetically displeasing brown coloration to the water.

In most cases where manganese salts occur naturally in the water, it will be necessary to apply special methods of removal before the water can be used for domestic or industrial purposes. The Palintest Manganese test provides an extremely sensitive method of measuring low concentrations of manganese for the assessment of natural waters and the control of manganese removal plant. The test measures total manganese over the range 0 - 5 mg/l.

Method

Manganese may occur in water in various different valency states. This method offered is the Formaldoxime Method, with a range of 0 - 5.0 mg/l Mn. In alkaline solution manganese reacts with formaldoxime to form an orange-red complex. The color developed is proportional to the manganese concentration. Iron II ions also form a coloured complex, which will interfere.

The intensity of colour produced in the test is proportional to the total manganese concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Manganese HR No 1 Tablets

Palintest Manganese HR No 2 Tablets

Palintest Automatic Wavelength Selection Photometer

Round Test Tubes, 10 ml glass (PT 595/5)

Sample Collection

Manganese is readily absorbed onto the surfaces of sample containers. To avoid loss of manganese test sample as soon as possible after collection.

It is important, because of the extreme sensitivity of this test, to ensure that glassware used for the sample collection and test procedure is scrupulously clean. For most accurate results in laboratory use it is recommended that all glassware is acid-rinsed and then thoroughly washed out with deionised water before use.

Interferences

- 1 Iron (II) and Iron (III) will interfere with the test and cause false positive results. Even low levels of Iron (0.1mg/l) will interfere.
- 2 Copper will interfere and cause false positive results, this is characterised by the formation of a blue solution during the test.

Test Procedure

- 1 Fill test tube with sample to the 10 ml mark
 - 2 Add one Manganese HR No 1 tablet, crush and mix to dissolve.
 - 3 Add one Manganese HR No 2 tablet, crush and mix to dissolve then cap the tube.
 - 4 Stand for 5 minutes to allow colour development
 - 5 Select Phot 113 on the Photometer.
 - 6 Take Photometer reading in usual manner (see Photometer instructions).
 - 7 The photometer will display the reading as mg/l Mn reading.
-

Photometer Method

CHLORINE DIOXIDE

TEST FOR CHLORINE DIOXIDE AND OTHER RESIDUALS IN WATER

AUTOMATIC WAVELENGTH SELECTION

0 – 10 mg/l as ClO₂

0 – 25 mg/l as Cl

Chlorine dioxide is used for the disinfection of water in a variety of different applications. Chlorine dioxide is normally generated by reacting chlorine with sodium chlorite solution in specially designed plant and equipment. Water treated with chlorine dioxide may therefore also contain amounts of chlorine and chlorite. For the control of such water treatment systems it is necessary to determine and differentiate between these different residual species.

The Palintest Chlorine Dioxide method provides a precise method of determining chlorine dioxide in treated water. Supplementary procedures provide for the determination of free and combined chlorine and chlorite.

Method

Chlorine dioxide reacts with diethyl-p-phenylene diamine (DPD) in buffered solution to produce a pink coloration. Chlorine reacts in a similar manner. Glycine is used to prevent the reaction with chlorine so as to give specific determination of chlorine dioxide.

In the supplementary part of the test the glycine is omitted and it is then possible, by differences, to measure the free chlorine content. Subsequent addition of potassium iodide induces a further reaction with any combined chlorine present. Continuation of the test using an acidification and neutralisation procedure produces a further reaction and in this way the chlorite concentration can be determined.

The colour intensities at each stage of the test are measured using a Palintest Photometer and the concentration of each individual component are obtained by a simple calculation. It is normal practice to express the concentration of each component in terms of the equivalent chlorine concentration.

Reagents and Equipment

Palintest DPD No 1 Tablets

Palintest DPD Glycine Tablets

Palintest Automatic Wavelength Selection Photometer

Palintest Round Test Tubes, 10 ml glass (PT 595)

Test Procedure

- 1 Rinse a clean test tube with sample, then fill with sample to the 10 ml mark. Add one Glycine tablet, crush and mix to dissolve.
 - 2 Decant two or three drops of Glycine treated sample into a second clean test tube. Add one DPD No 1 tablet then crush to disintegrate.
 - 3 Add the remaining contents of the first test tube to the second test tube and mix.
 - 4 Gently invert the tube to remove any bubbles from the inner walls of the tube
 - 4 Select Phot 115 on the photometer.
 - 5 Take photometer reading.
 - 6 The result displayed on screen represents the chlorine dioxide concentration as mg/l of ClO_2 .
 - 7 Divide your result by 1.9 and then multiply by 5 to obtain the chlorine dioxide residual in terms of mg/l Chlorine.
-

DEHA

(N,N-Diethylhydroxylamine)

TEST FOR BOILER AND COOLER WATER SYSTEMS

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

**0.01 – 0.500 mg/l
DEHA**

Method

The Palintest test is based on N,N-Diethylhydroxylamine (DEHA) or other oxygen scavengers present in the sample reacting with Ferric Iron to produce Ferrous Iron. The Ferrous Iron concentration then forms a purple colour proportional to the concentration of the oxygen scavenger.

Ferrous Iron already present in the sample will interfere, hence the method is defined as DEHA + Fe(II). To correct for any Ferrous Iron not introduced during the method use the Follow-On method Phot 117. This will automatically remove any offset and present the 'true' DEHA result as mg/l DEHA.

Reagents and Equipment

Palintest DEHA Tablets

Palintest DEHA Solution

Palintest DEHA Iron Correction Solution

Two 1 ml Syringes

Palintest Photometer 8000, 7500 or 7100

Round Test Tubes, 10 ml glass (PT 595)

Test Procedure – DEHA + Fe(II)

- 1 Fill test tube with sample to the 10 ml mark.
- 2 Add one DEHA tablet, crush and mix until completely dissolved.
- 3 Add 0.5 ml of DEHA test solution with the first syringe, mix the solution and cap the photometer tube.
- 4 The solution is photosensitive. In order to prevent inaccurate results, place the tube in the photometer with the light cap in place.
- 5 Stand for 10 minutes to allow full colour development.
- 6 Select Phot 116 on the photometer and take the reading in the usual manner. The result is displayed in mg/l DEHA.

The test may be terminated at this stage if the sample is known to contain no Ferrous Iron.

Test Procedure – Fe(II) Correction

- 1 Select 'Follow-On' on the photometer
- 2 Fill test tube with the same sample to the 10 ml mark.
- 3 Add one DEHA tablet, crush and mix until completely dissolved.
- 4 Add 0.5 ml of DEHA Iron Correction solution with the second syringe, mix the solution and cap the photometer tube.
- 5 The solution is photosensitive. In order to prevent inaccurate results, place the tube in the photometer with the light cap in place.
- 6 Stand for 10 minutes to allow full colour development.
- 7 Select 'Read' and the result will be displayed as mg/l DEHA.
- 8 Press 'Return' to complete the test sequence.

Interferences

This method reacts with similar oxygen scavengers (carbohydrazide, DEHA, hydroquinone, iso-ascorbic acid [ISA], methylethyl ketoxime [MEKO]) and does not differentiate samples containing more than one type of oxygen scavenger.

During colour development the samples must be protected from light.

The test has been calibrated at 18°C, sample temperature during the test should be maintained as close to this temperature as possible. Samples of a temperature lower than 18°C will give a low response; samples of a higher temperature will give a high response.

For a standard in the region of 0.2 mg/l the result will drift by approximately 0.017 mg/l for every 5°C away from 18°C (ie at 23°C the instrument would give a result of 0.217 mg/l).

For a result in the region of 0.4 mg/l the result will drift by approximately 0.03 mg/l for every 5°C away 18°C (ie at 23°C the instrument would give a result of 0.43 mg/l).

Any chemical that will reduce ferric iron, or that will complex with iron strongly, will interfere. The species below **will interfere** at the levels indicated:

Borate >500 mg/l, Cobalt >0.025 mg/l, Copper >8 mg/l, Hardness >1000 mg/l, Manganese >0.8 mg/l, Molybdenum >80 mg/l, Nickel >0.8 mg/l, Phosphate >10 mg/l, Phosphonates >10 mg/l, Sulphate >1000 mg/l, Zinc >50 mg/l.

Notes

- 1 If carrying out the correction procedure for ferrous iron and on addition of the DEHA tablet no colour is produced, this indicates there is no ferrous iron in the sample and the correction procedure isn't necessary.
- 2 To convert a **mg/l** reading into **ppb**, multiply the result by 1000.

PHOT 116.Cal Chart

DEHA CALIBRATION CHART

For use with Photometer 7100/7500

	mg/l DEHA								550nm	
%T	9	8	7	6	5	4	3	2	1	0
90	-	-	-	-	-	0.000	0.003	0.005	0.008	0.010
80	0.013	0.015	0.018	0.021	0.023	0.026	0.029	0.032	0.034	0.037
70	0.040	0.043	0.046	0.049	0.052	0.055	0.058	0.062	0.065	0.068
60	0.072	0.075	0.078	0.082	0.086	0.089	0.093	0.097	0.101	0.104
50	0.108	0.112	0.117	0.121	0.125	0.130	0.134	0.139	0.143	0.148
40	0.153	0.158	0.163	0.169	0.174	0.180	0.185	0.191	0.197	0.203
30	0.209	0.216	0.223	0.230	0.237	0.244	0.252	0.260	0.268	0.276
20	0.285	0.294	0.303	0.313	0.323	0.333	0.344	0.355	0.367	0.379
10	0.392	0.405	0.420	0.435	0.451	0.469	0.488	0.500	-	-
0	-	-	-	-	-	-	-	-	-	--