DISSOLVED OXYGEN/0.8/1.4

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

0 – 0.8 mg/l/0 – 1.4 mg/l

TEST FOR DISSOLVED OXYGEN IN BOILER WATER AND BOILER FEEDWATER

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 O_2.

Note

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

Photometer Method

DISSOLVED OXYGEN/2

AUTOMATIC WAVELENGTH SELECTION

0 – 2.0 mg/l

TEST FOR DISSOLVED OXYGEN IN BOILER WATER AND BOILER FEEDWATER

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 O_2.

Note

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

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

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

5 – 150 mg/l O_2

TEST FOR ASSESSING EFFLUENT AND WASTE WATER QUALITY PRIOR TO DISCHARGE

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).

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 O_2.

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°C ± 3°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.

Palintest®

TEST INSTRUCTIONS

PHOT.81.AUTO

TUBETESTS®
CHEMICAL OXYGEN DEMAND – COD/400

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

20 - 400 mg/l O_2

TEST FOR ASSESSING EFFLUENT AND WASTE WATER QUALITY PRIOR TO DISCHARGE

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).

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 O_2.

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°C ± 3°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.

Palintest®

TEST INSTRUCTIONS

PHOT.82.AUTO

TUBETESTS®
CHEMICAL OXYGEN DEMAND–COD/2000

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

50 – 2,000 mg/l O_2

TEST FOR ASSESSING EFFLUENT AND WASTE WATER QUALITY PRIOR TO DISCHARGE

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).

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 O_2.

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°C ± 3°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.

Palintest®

TEST INSTRUCTIONS

PHOT.83.AUTO

TUBETESTS®
CHEMICAL OXYGEN DEMAND–COD/20,000

Photometer Method

AUTOMATIC WAVELENGTH SELECTION

500 – 20,000 mg/l (0.50 – 20.00 g/l O_2)

TEST FOR ASSESSING EFFLUENT AND WASTE WATER QUALITY PRIOR TO DISCHARGE

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).

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 O_2.

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 20,000 mg/l chloride in sample containing 500 to 20,000 mg/l COD. In the Palintest COD/20,000/2M test 0.08g of mercuric sulphate is provided and will suppress interference up to 40,000 mg/l chloride in samples containing from 500 to 20,000 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°C ± 3°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

10 – 1,000 mg/l O_2

TEST FOR ASSESSING EFFLUENT AND WASTE WATER QUALITY PRIOR TO DISCHARGE

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).

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 O_2.

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°C ± 3°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 ± 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 NO_3

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 NO_3 (5.7 mg/l N) and an absolute maximum of 50 mg/l NO_3 (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 NO_3).

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.

PL 404, PL 999 AUTO

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 oxidisation used to make the conversion to nitrate. It is normal practice to refer to the method of oxidisation 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.

PL 408, PL 999 AUTO

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 PO_4

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 PO_4). For drinking water, the EC has set a maximum admissible concentration of 2.2 mg/l P (6.7 mg/l $P0_4$).

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

Palintest®

TEST INSTRUCTIONS

PHOT.92.AUTO

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.

PL 416 AUTO

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.

Palintest®

TEST INSTRUCTIONS

PHOT.96.AUTO

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 :-

- Metaltube Digest Tubes
- Zinctube Powder
- Dosing Scoop - Size 4
- Zinctube Neut Reagent
- Zinctube Indicator Tablet *
- Dosing Funnel
- Zinctube 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 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.

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.

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