

TEST INSTRUCTIONS

**TUBETESTS®
CHEMICAL OXYGEN
DEMAND – COD/150****Photometer Method**

490 nm

**TEST FOR ASSESSING EFFLUENT AND WASTE
WATER QUALITY PRIOR TO DISCHARGE****5 – 150 mg/l O₂**

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

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

Method

In the Palintest COD method, the water sample is oxidised by digesting in a sealed reaction tube with sulphuric acid and potassium dichromate in the presence of a silver sulphate catalyst. The amount of dichromate reduced is proportional to the COD. A reagent blank is prepared for each batch of tubes so as to compensate for the oxygen demand of the reagent itself.

Over the range of the test a series of colours from yellow through green to blue are produced. The colour is indicative of the chemical oxygen demand and is measured using a Palintest Photometer. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests Tubes are available in COD (mercury free), COD/M and COD/2M (with mercuric sulphate) 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 Photometer (with Tubetests Adaptor)

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 a refrigerator for maximum storage life. Inspect tubes before use - do not use any which show green discoloration.

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 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, particularly when using the COD/M format.

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

Test Calibration

Transmittance-display photometer - use calibration chart

Direct-reading photometer - select program **Phot 80** (Photometer 7000)
or **TTest 1** (Tubetests Photometer)

The photometer is used in conjunction with a special Tubetests Adaptor (PT 586) which fits into the test chamber. The adaptor must be located with the position mark  facing towards the user and must be pushed fully home to the bottom of the test chamber.

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 wavelength 490 nm on Photometer.
- 9 Take the photometer reading (see photometer instructions).

Photometer 5000 and Photometer 7000 (to version 7000.9) :-

The usual photometer operating sequence is reversed. Firstly insert the SAMPLE TUBE to set the instrument, then insert the REAGENT BLANK and take the photometer reading.

Photometer 7000 (Version 7000.10 Onwards) and Tubetests Photometer :-

The usual photometer operating sequence is used. Firstly insert the REAGENT BLANK to set the instrument, then insert the SAMPLE TUBE and take the photometer reading.

- 10 Consult COD/150 calibration chart (Transmittance-display photometer only).

| COD/150 | | mg/l O ₂ | | | | | | | | |
|---------|-----|---------------------|-----|-----|-----|-----|-----|-----|-----|-----|
| %T | 9 | 8 | 7 | 6 | 5 | 4 | 3 | 2 | 1 | 0 |
| 90 | 3 | 6 | 9 | 12 | 15 | 19 | 23 | 27 | 31 | 35 |
| 80 | 39 | 43 | 47 | 52 | 56 | 60 | 65 | 70 | 75 | 80 |
| 70 | 85 | 90 | 95 | 100 | 105 | 110 | 115 | 120 | 125 | 130 |
| 60 | 136 | 142 | 147 | 153 | - | - | - | - | - | - |

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 50 mg/l. Samples containing above this level should be diluted so as to reduce the concentration to 50 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 from up to 2,000 mg/l chloride in the sample. In the Palintest COD/150/2M test 0.08g of mercuric sulphate is provided and will suppress interference from up to 4,000 mg/l chloride in the sample.

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 the laid down disposal procedures of the laboratory of use. Used COD/150 tubes may, subject to consent limits, be decanted into running water, or into a sink of water, and then run to waste. Used COD/150/M tubes must always be treated using a proper waste disposal system. 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 Heaters

The Palintest Tubetests heaters are dedicated heaters for use with the COD Tubetests system. They comprise an electronically controlled dry bath which heats aluminium test tube blocks. The heaters are designed to provide the correct digesting and refluxing conditions necessary for the COD test. They provide the correct digestion temperature of $150^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in the reagent tubes. The Palintest Digital Tubetests Heater (PT 589) is a 12 tube heater with an integral safety screen. The heater features a digital display for the operating and set temperatures.

To use the digital heater for the COD test, set the temperature on the digital display to 150°C . It is not necessary to use a thermometer with this heater - the operating temperature is shown on the digital display. The display takes into account thermal lag between the block and the heater tubes.

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