

TUBETESTS[®]

CHEMICAL OXYGEN DEMAND – COD/150

Photometer Method

AUTOMATIC
WAVELENGTH
SELECTION

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

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, the colour changes from orange to green. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

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

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

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Automatic Wavelength Selection Photometer

Palintest Pipettor, 2 mL (PT 572)

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

Working Practice

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

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

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

Reagent Blank

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

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

Sample Preparation

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

Test Procedure

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

Interferences

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

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

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

Disposal

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

Tubetests Heater

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

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

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

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