TUBETESTS®
CHEMICAL OXYGEN DEMAND – COD/M-C

TEST FOR ASSESSING EFFLUENT AND WASTE WATER QUALITY PRIOR TO DISCHARGE

Photometer Method
420 and 620 nm

0 – 150 mg/l O_2
20 – 1,500 mg/l O_2
200 – 15,000 mg/l O_2

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 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 in order 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 Photometer. The results are expressed as milligrams of oxygen consumed per litre of sample.

Reagents and Equipment

COD Tubetests Tubes are available in different strengths depending on the range of test required :-

COD/150/M-C (PL 481)
COD/1500/M-C (PL 484)
COD/15000/M-C (PL 486)

Palintest Digital Tubetests Heater (PT 589)
Palintest Tubetests Heater Safety Screen (PT 590)
Suitable Photometer
Pipettors, 2 ml and 0.2 ml

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 eye contact, rinse with large amounts of water and seek medical attention immediately. For skin contact, or spillage, wash immediately with large quantities 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.

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

These COD reagents are designed for use with any compatible photometer. For example, they have been tested and found to be compliant with the direct-reading Hach DR/2000 and DR/2500 Odyssey instruments.

They are not compatible with Palintest direct-reading instruments, for which dedicated COD reagents are available.

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. Add 2 ml of sample (150 and 1,500 ranges), or 0.2 ml of sample (15,000 range), 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 (150 and 1,500 ranges), or 0.2 ml (15,000 range) 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 appropriate wavelength on photometer.

9 Take the photometer reading (see photometer instructions).

Interferences

Chloride is the main potential interference in the COD test. High chloride levels may result in an apparent high COD result.

The method most commonly prescribed in standard analytical methods is the addition of mercuric sulphate to the reagent system. In the Palintest COD/M-C tests, 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.

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 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

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 and integral safety screen. 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°C ± 3°C in the reagent tubes.

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