

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).