

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 :-

Metal tube Digest Tubes

Zinc tube Powder

Dosing Scoop - Size 4

Zinc tube Neut Reagent

Zinc tube Indicator Tablet *

Dosing Funnel

Zinc tube 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 Metal tube 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.
