

TUBETESTS® IRON/25

TEST FOR IRON IN EFFLUENTS, WASTE
WATERS AND INDUSTRIAL WATER SAMPLES

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 25 mg/l

Iron occurs widely in nature and is found in many natural and treated waters. Iron is an objectionable constituent in both domestic and industrial water supplies. The presence of iron affects the taste of beverages and causes unsightly staining of laundered clothes, plumbing fittings, swimming pool surfaces and the like. The formation of insoluble iron deposits is troublesome in many industrial applications and in agricultural uses such as drip feed irrigation.

Iron is an important test for effluents, waste waters and industrial water samples. The sources of iron in such samples are many and varied and include the corrosion of plant and equipment and waste from industrial processes. The Palintest Tubetests Iron/25 test is designed to measure the total recoverable iron concentration over the range 0 - 25 mg/l.

Method

The Palintest Tubetests Iron/25 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 Iron/25 test is designed to measure the total recoverable iron concentration in such samples.

In the Palintest Tubetests Iron/25 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 buffered to provide the correct pH conditions for the test. A reducing agent is then added to convert all of the iron to ferrous form and this is then reacted with 1,10 phenanthroline to form an orange coloured complex. Decomplexing agents and inhibitors are incorporated into the test reagent system in order to break down any chelated iron which is present and to prevent interference from other metal ions commonly found in effluents and waste water samples.

The intensity of the colour produced in the test is proportional to the iron concentration and is measured using a Palintest Photometer.

Reagents and Equipment

Palintest Tubetests Iron/25 Pack (PL 434) containing :-

Metaltube Digest Tubes

Metaltube Neut Reagent

Metaltube Buffer

Irontube No 1 Tablet

Irontube No 2 Tablet

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 2 ml (PT 572)

Working Procedure

The Palintest Tubetests Iron/25 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 digest tubes to add the sample, or to add reagents, 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 2 ml of sample using a Palintest pipettor with 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 Metaltube Neut Reagent to the tube using a Palintest pipettor with 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 Add 2 ml of Metaltube Buffer to the tube using a Palintest pipettor with 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.
- 6 Remove the cap from the tube and then add one Irontube No 1 tablet, crush and mix to dissolve.
- 7 Add one Irontube No 2 tablet, crush and mix to dissolve and then replace the cap on the tube.
- 8 Stand for 10 minutes without disturbing the solution to allow full colour development and to allow any undissolved particles to settle.
- 9 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 Iron/25 testing.
- 10 Select Phot 95 on the Photometer.
- 11 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).
- 12 The result is displayed as mg/l Fe.

Interferences

In interference studies the presence of chromium, nickel and zinc have been found not to cause any effect on the test result. The presence of molybdate will cause proportionately high readings and will give a false positive response in the absence of iron. Copper levels greater than 2 mg/l and lead levels greater than 4 mg/l will cause slightly high readings and will give a false positive response in the absence of iron.

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 6. 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 9 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 temperature. 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.
