

TUBETESTS® NICKEL/20

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

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

0 – 20 mg/l

Nickel does not occur naturally in water apart from a few areas around the world where nickel-bearing ores are present. Nickel is however commonly found in industrial waste waters such as those from the steel industry and from plating processes. Nickel is considered an undesirable element in water supplies and careful monitoring of effluent and waste waters is necessary to prevent this element entering the aqueous environment.

The Palintest Tubetests Nickel/20 test is designed to measure total recoverable nickel concentration over the range 0 - 20 mg/l.

Method

The Palintest Tubetests Nickel/20 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 Nickel/20 test is designed to measure the total recoverable nickel concentration in such samples.

In the Palintest Tubetests Nickel/20 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 nickel to nickelous form and this is then reacted with nioxime indicator to form a pink-coloured complex. Inhibitors are incorporated into the test reagent system in order to prevent interference from iron and other metal ions commonly found in effluents and waste water samples.

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

Reagents and Equipment

Palintest Tubetests Nickel/20 Pack (PL 430) containing :-

Metaltube Digest Tubes

Metaltube Neut Reagent

Metaltube Buffer

Nickeltube No 1 Tablet

Nickeltube No 2 Tablet

Nickeltube Powder

Dosing Scoop - Size 1

Dosing Funnel

Dosing Scoop Scraper

Palintest Automatic Wavelength Selection Photometer

Palintest Digital Tubetests Heater (PT 589)

Palintest Tubetests Heater Safety Screen (PT 590)

Palintest Pipettor, 2 ml (PT 572)

Palintest Pipettor, 5 ml (PT 576)

Working Procedure

The Palintest Tubetests Nickel/20 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.

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 (see Tubetests Heater).
- 2 Prepare the Sample tube as follows. Remove the cap of the Metaltube Digest Reagent tube and add 5 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. Replace the cap tightly and invert the tube gently to mix the contents.
- 6 Remove the cap from the tube and then add one Nickeltube No 1 tablet, crush and mix to dissolve.
- 7 Add one level scoop of Nickeltube Powder using a Size 1 dosing scoop. Cap tube and shake to dissolve powder.
- 8 Remove the cap and then add one Nickeltube No 2 tablet. Crush tablet and mix to dissolve and then replace the cap on the tube.
- 9 Stand for 5 minutes without disturbing the solution to allow full colour development. Invert the tube to ensure even distribution of indicator and then stand for 2 minutes to allow any undissolved particles to settle.
- 10 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 Nickel/20 testing.
- 11 Select Phot 96 on the Photometer.
- 12 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).
- 13 The result is displayed as mg/l Ni.

Interferences

In interference studies the presence of chromium, copper, iron and zinc have been found not to cause any effect on the test result. Cobalt levels greater than 1 mg/l have been found to give a false positive response in the absence of nickel.

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 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 10 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.
