

TUBETESTS® TOTAL NITROGEN/30

TEST FOR TOTAL PERSULPHATE NITROGEN
IN NATURAL AND WASTE WATER

Photometer Method

**AUTOMATIC
WAVELENGTH
SELECTION**

0 – 30 mg/l N

Total nitrogen is a vital test for assessing the quality of effluents and waste water prior to discharge. In the UK the Urban Waste Water Treatment Regulations (1994) make provision for the control of discharge of total nitrogen to sensitive bodies of natural water. The monitoring of the rate of nitrogen removal is therefore of great importance in waste water treatment. Total nitrogen is composed of nitrate, nitrite, ammonium and organic nitrogen compounds. The Palintest Tubetests Total Nitrogen/30 test provides a simple method of measuring total persulphate nitrogen over the range 0 – 30 mg/l N.

Method

The Palintest Tubetests Total Nitrogen/30 test is a simple two stage procedure. The sample is initially digested with alkaline persulphate to break down nitrogenous compounds which are then converted to nitrate. The digested sample is then transferred to a Palintest Tubetests Nitrate/30N Tube for determination of the total nitrogen present. The reagents are provided in the form of predispensed tubes and powders. The powders are added using a specially designed scoop and funnel.

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

In total nitrogen determinations, the recovery of different compounds depends to an extent on the method of oxidation used to make the conversion to nitrate. It is normal practice to refer to the method of oxidation when stating test results for any formal purpose. Results from the Palintest Total Nitrogen test should therefore be expressed as 'Total Persulphate Nitrogen'.

Working Practice

The Palintest Tubetests Total Nitrogen/30 test is a simplified laboratory procedure and should be carried out in accordance with good laboratory working practice.

The Total Nitrogen Tubes contain sodium hydroxide solution, to which potassium persulphate is added. The Tubetests Nitrate/30N Tubes contain strong sulphuric acid. These reagents 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 adding Tubetests Total Nitrogen Reagent No 2 to the digestion tubes. Sulphur dioxide will be evolved. Care should be taken when opening the Tubetests Nitrate/30N Tube which contains concentrated acid. On adding the digestate heat will be produced, the tube will become hot and gases may be evolved. It is generally recommended that the test be conducted in a fume cupboard where available, particularly in the case of samples originally known to contain toxic materials such as cyanide or sulphide.

Reagents and Equipment - Digestion Stage

Palintest Tubetests Total Nitrogen Tubes
Palintest Tubetests Total Nitrogen Reagent No 1
Palintest Tubetests Total Nitrogen Reagent No 2
Palintest Digital Tubetests Heater (PT 589)
Palintest Tubetests Heater Safety Screen (PT 590)
Palintest Pipettor 5 ml (PT 576)
Palintest Dosing Scoop - Size 1 (PT 691)
Palintest Dosing Scoop - Size 4 (PT 694)
Palintest Dosing Funnel (PT 690)
Palintest Dosing Scoop Scraper (PT 695)

Reagents and Equipment - Colour Development Stage

Palintest Tubetests Nitrate/30N Tubes
Palintest Tubetests Nitrate Powder
Palintest Automatic Wavelength Selection Photometer
Palintest Pipettor, 1 ml (PT 574)
Palintest Dosing Scoop - Size 1 (PT 691)
Palintest Dosing Funnel (PT 690)
Palintest Dosing Scoop Scraper (PT 695)

Use of Dosing Scoop and Funnel

This Tubetests method uses Palintest Dosing Scoops and Funnels. The scoops and funnels are specially designed to ensure accurate dosing of reagent powders into the Tubetests tubes :-

- 1 Select the correct size scoop. 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 of the reagent goes into the tube.

Test Instructions - Digestion Stage

- 1 Turn on the Tubetests heater, set the control to the 105°C mark and allow to heat up to temperature.
- 2 Remove the cap of the Tubetests Total Nitrogen Tube and add three level scoops of Tubetests Total Nitrogen Reagent No 1 using the Size 1 dosing scoop and funnel.
- 3 Add 5.0 ml of sample using a pipettor. Replace the cap tightly and shake the tube vigorously for 30 seconds.
- 4 Label the tube and place in the Tubetests heater. Ensure the safety screen is in position and digest the tube for 30 minutes, then turn off the heater.
- 5 Carefully remove each tube and transfer to a test tube rack. Handle hot tubes by the cap only.
- 6 Allow tubes to cool to room temperature.
- 7 Remove the cap of the Tubetests Total Nitrogen Tube and add one level scoop of Tubetests Total Nitrogen Reagent No 2 using the Size 4 dosing scoop and funnel. *Take care - sulphur dioxide will be evolved.*
- 8 Cap the tube and shake for 15 seconds, then stand for 3 minutes.

Test Instructions - Colour Development Stage

- 1 Using a pipettor, transfer 1 ml of digested sample from the Tubetests Total Nitrogen Tube to a Tubetests Nitrate/30N Tube. Take care to add the digestate slowly. DO NOT SHAKE THE TUBE.
- 2 Add one level scoop of Tubetests Nitrate Powder using the Size 1 dosing scoop and funnel. Cap tube and invert slowly ten times to dissolve and mix the reagents and sample. Take care! The tube will become hot.
- 3 Stand for five minutes to allow colour development.
- 4 Select Phot 89 on Photometer.
- 5 Take photometer reading in usual manner (see Photometer instructions). Use an unused Tubetests Nitrate/30N Tube to set the blank on the photometer.
- 6 The result is displayed as mg/l N.

Notes

- 1 This method is based on the Persulphate Method from 'Standard Methods for the Examination of Water and Waste Water' 19th Edition 1995, Pages 4 - 95. The method, in general, does not yield 100% recovery. Recoveries of various nitrogen compounds have been tested in the Palintest laboratories. Inorganic compounds such as potassium nitrate, sodium nitrite and ammonium chloride yield in excess of 95% recovery. The typical recoveries of some organic nitrogen compounds are quoted below :-

Compound	Typical Recovery
Glycine	95% all levels
Urea	90% all levels
Nicotinic Acid	95% at 10 mg/l, 45% at 30 mg/l
Creatinine	100% at 10 mg/l, 70% at 30 mg/l

- 2 Tubetests Nitrate Powder is light sensitive. Store in original pack and keep lid closed when not in use.
 - 3 Disposal. The used Tubetests Nitrate/30N 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.
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