



TN HR TT

281

5 - 150 mg/l N<sup>b)</sup>

Persulphate Digestion

### Instrument specific information

The test can be performed on the following devices. In addition, the required cuvette and the absorption range of the photometer are indicated.

Instrument Type	Cuvette	$\lambda$	Measuring Range
MD 600, MD 610, MD 640, MultiDirect	ø 16 mm	430 nm	5 - 150 mg/l N <sup>b)</sup>
SpectroDirect, XD 7000, XD 7500	ø 16 mm	410 nm	5 - 150 mg/l N <sup>b)</sup>

### Material

Required material (partly optional):

Reagents	Packaging Unit	Part Number
VARIO Total Nitrogen HR, Set	1 Set	535560

The following accessories are required.

Accessory	Packaging Unit	Part Number
Thermoreactor RD 125	1 pc.	2418940

### Application List

- Waste Water Treatment
- Drinking Water Treatment
- Raw Water Treatment

### Preparation

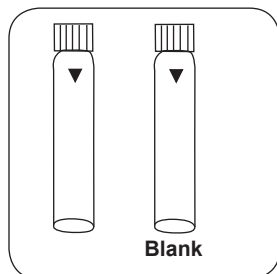
1. Large quantities of nitrogen free, organic compounds that are included in some water samples may reduce the effectiveness of the digestion by reacting with the Persulphate reagent. Samples which are well known to contain large quantities of organic compounds must be diluted and digestion and measurement must be repeated for checking the effectiveness of the digestion.

## Notes

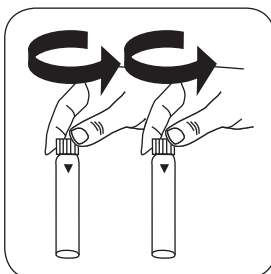
1. Persulphate reagent may not get on the vial threads. To remove spattered or spilt Persulphate reagent, thoroughly wipe the vial threads with a clean cloth.
2. Volumes for samples and blank should always be metered by using suitable pipettes (class A).
3. One blank is sufficient for each set of samples.
4. The reagents TN hydroxide LR, TN persulphates RGT. and TN reagent B may not completely dissolve.
5. The blank (stored in the dark) can be used for 7 days, if the measured samples were prepared with the same batch of reagent.

## Implementation of the provision Nitrogen, total HR with Vial Test

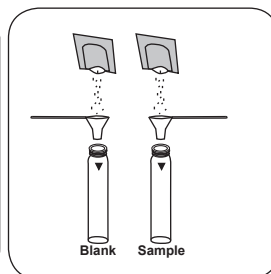
Select the method on the device



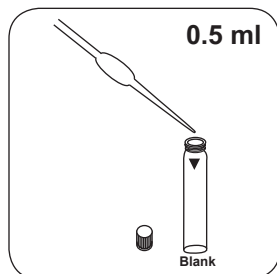
Prepare two **digestion vials** TN Hydroxide HR . Mark one as a blank.



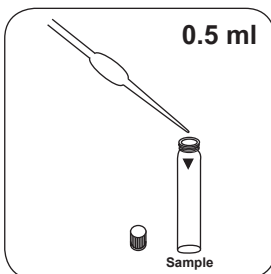
Open the vial.



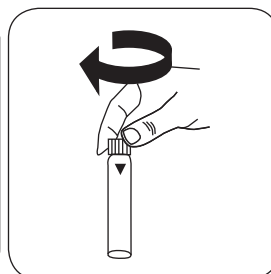
Add a **Vario TN Persulfate Rgt. powder pack** in each vial.



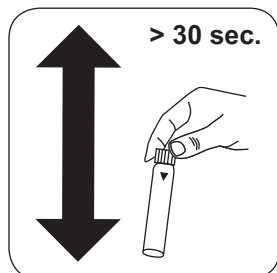
Put **0.5 ml deionised water** in the blank.



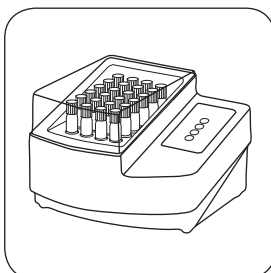
Put **0.5 ml sample** in the sample vial.



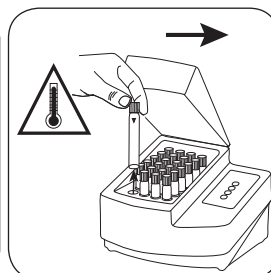
Close vial(s).



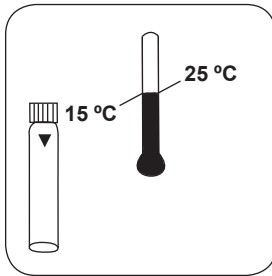
Mix the contents by shaking vigorously. (> 30 sec.).



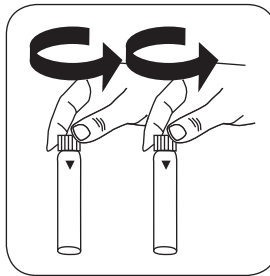
Seal the vials in the pre-heated thermoreactor for **30 minutes at 100 °C** .



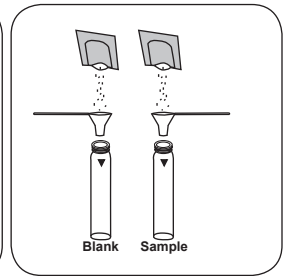
Remove the vial from the thermoreactor. **Note: vial will be hot!**



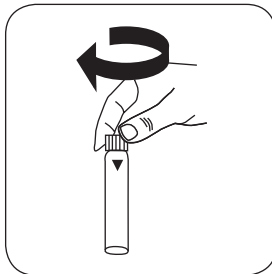
Allow the sample to cool to room temperature.



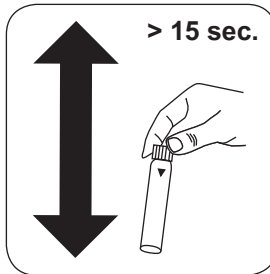
Open the vial.



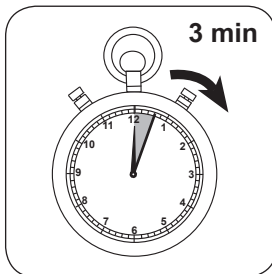
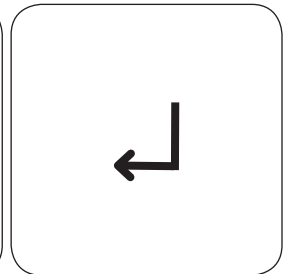
Add a **Vario TN Reagent A powder pack** in each vial.



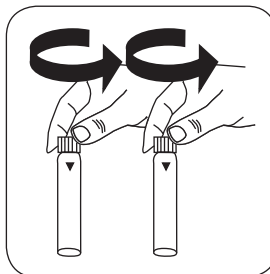
Close vial(s).



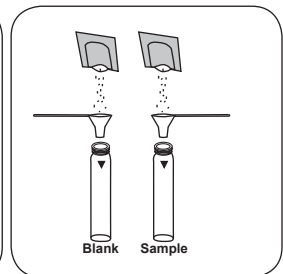
Mix the contents by shaking. Press the **ENTER** button. (> 15 sec.).



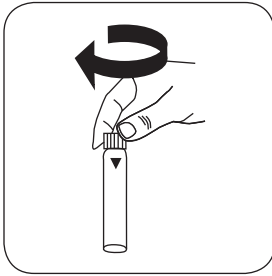
Wait for **3 minute(s) reaction time**.



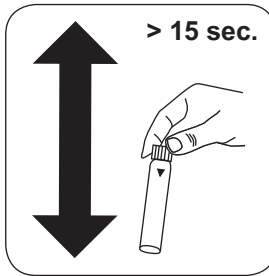
Open the vial.



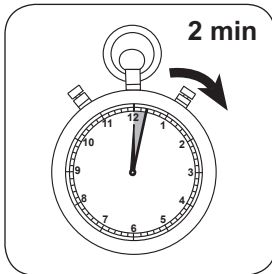
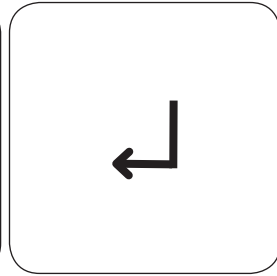
Add a **Vario TN Reagent B powder pack** in each vial.



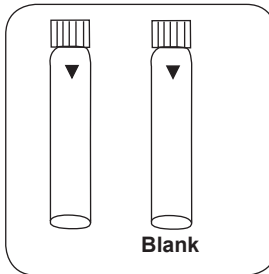
Close vial(s).



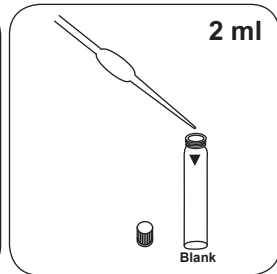
Mix the contents by shaking. Press the **ENTER** button. (> 15 sec.).



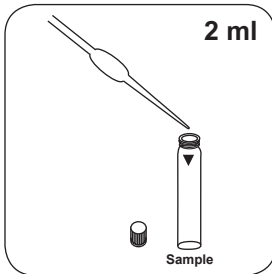
Wait for **2 minute(s)** reaction time.



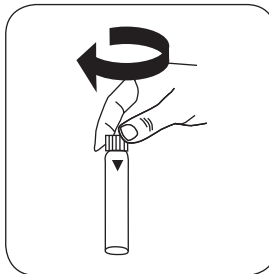
Prepare two **TN Acid LR/HR (Reagent C)** vials. Mark one as a blank.



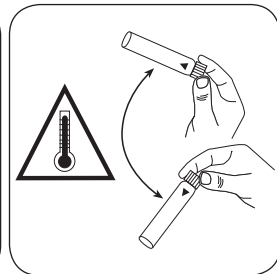
Place **2 ml** of digested, pre-prepared zero sample in the blank.



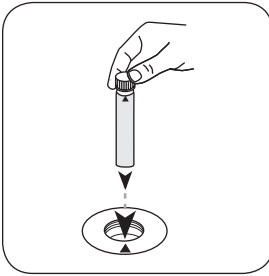
Fill sample vial with **2 ml** prepared, digested sample.



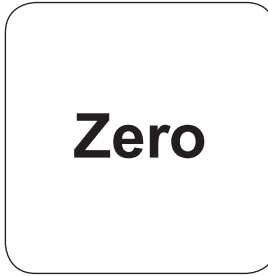
Close vial(s).



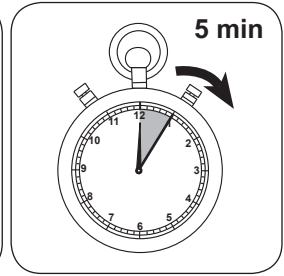
Invert several times to mix the contents (10 x). **Note: Will get hot!**



Place **blank** in the sample chamber. • Pay attention to the positioning.

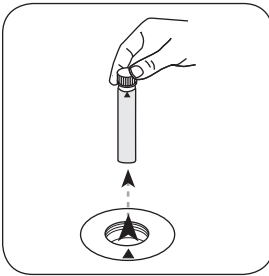


Press the **ZERO** button.

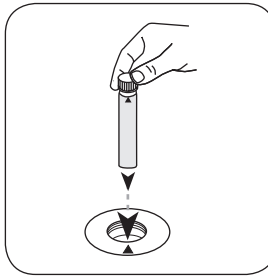


Wait for **5 minute(s) reaction time**.

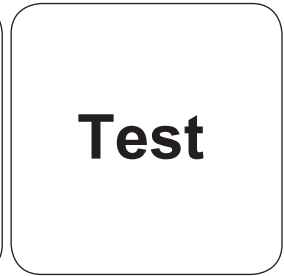
Once the reaction period is finished, the measurement takes place automatically.



Remove **vial** from the sample chamber.



Place **sample vial** in the sample chamber. • Pay attention to the positioning.



Press the **TEST (XD: START)** button.

The result in mg/l Nitrogen appears on the display.

## Chemical Method

Persulphate Digestion

## Appendix

### Interferences

Interference	from / [mg/l]
Cr <sup>6+</sup>	5
Fe <sup>2+</sup>	50
Sn <sup>2+</sup>	50
Ca <sup>2+</sup>	100
Co <sup>2+</sup>	100
Cu <sup>2+</sup>	100
Fe <sup>3+</sup>	100
Ni <sup>2+</sup>	100
Pb <sup>2+</sup>	100
Zn <sup>2+</sup>	100
Cd <sup>2+</sup>	200
K <sup>+</sup>	500
NO <sub>2</sub> <sup>-</sup>	2
Cl <sup>-</sup>	500

### Method Validation

Limit of Detection	1.87 mg/l
Limit of Quantification	5.6 mg/l
End of Measuring Range	150 mg/l
Sensitivity	0.00498 mg/l / Abs
Confidence Range	2.27 mg/l
Standard Deviation	0.94 µg
Variation Coefficient	1.20 %

### Bibliography

M. Hosomi, R. Sudo, Simultaneous determination of total nitrogen and total phosphorus in freshwater samples using persulphate digestion, Int. J. of Env. Stud. (1986), 27 (3-4), p. 267-275

<sup>a)</sup> determination of free, combined and total | <sup>b)</sup> Reactor is necessary for COD (150 °C), TOC (120 °C) and total -chromium, - phosphate, -nitrogen, (100 °C) | <sup>c)</sup> MultiDirect: Adapter is necessary for Vacu-vials<sup>®</sup> (Order code 19 20 75) | <sup>d)</sup> Spectroquant<sup>®</sup> is a Merck KGaA Trademark | <sup>e)</sup> alternative reagent, used instead of DPD No.1/No.3 in case of turbidity in the water sample caused by high concentration of calcium and/or high conductivity | <sup>f)</sup> additionally required for determination of bromine, chlorine dioxide and ozone in the presence of chlorine | <sup>g)</sup> Reagent recovers most insoluble iron oxides without digestion | <sup>h)</sup> additionally required for samples with hardness values above 300 mg/l CaCO<sub>3</sub> | <sup>i)</sup> high range by dilution | <sup>j)</sup> including stirring rod, 10 cm