

DETERMINATION OF HIGH-RANGE SIMPLIFIED TKN WITH TNTPLUS

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Introduction

Hach® currently offers TNT880 for simplified Total Kjeldahl Nitrogen (s-TKN), using the TNTplus reagent vials. The current s-TKN test has a concentration range of 0-16 mg/L N, and for many customers this range is too low. The analyst can dilute the sample into this concentration range. This technique works with known sample concentrations that are frequently analyzed, but what about unknown samples? With unknown nitrogen sample concentrations you have to analyze the sample first to figure out what dilution series is necessary to accurately determine the TKN value; this step wastes time and reagent vials. This application note will describe how to analyze s-TKN up to 40 mg/L of nitrogen using the HR Nitrate TNT836 and the HR Total Nitrogen TNT827 TNTplus reagent sets.

Material

- TNT827 – High Range Total Nitrogen, 5-40 mg/L N
- TNT836 – High Range Nitrate, 5-35 mg/L NO₃-N
- Method 10206 – HR Nitrate procedure (DOC316.53.01071)
- Method 10208 – HR Total Nitrogen procedure (DOC316.53.01088)
- BBP065 – Pipet, adjustable volume, 1.0 - 5.0 mL
- BBP068 – Pipet tips, for 1.0 - 5.0 pipet
- BBP078 – Pipet, adjustable volume, 0.2 - 1.0 mL
- BBP079 – Pipet tips, for 0.2 - 1.0 mL pipet
- DR photometer to analyze vials (DR1900, DR2800, DR3800, DR5000 or DR6000)
- DBR200 Reactor Block (with at least 4, 20-mm wells)

Procedure

Follow the HR TN procedure and digest the sample for 1 hour in the DRB200 reactor. After the digestion period, allow the reaction tube to cool to room temperature and follow procedure to neutralize the sample and react with test vial and solution D. While the TN test vial is reacting for the 15 minute reaction period, follow the HR Nitrate procedure by pipetting the unreacted sample to the test vial and adding solution A to the test vial, mixing and allowing to react for the 15 minute reaction period.

After both the 15 minute reaction periods are completed, clean and place the TN vial into the photometer and record the TN value. After measuring the TN vial, clean and place the NO₃ vial into the photometer and record the NO₃ value.

Calculation

Once you have both the TN and NO₃ values, subtract the NO₃ value from the TN value to get the s-TKN concentration.

$$\text{TN value} - \text{NO}_3 \text{ value} = \text{s-TKN} \quad (21 \text{ mg/L N} - 14 \text{ mg/L NO}_3\text{-N} = 7 \text{ mg/L s-TKN})$$

This calculation reports the same values that the TNT880 test does:

TN = 21 mg/L N

NO₃ & NO₂ = 14 mg/L NO₃-N

s-TKN = 7 mg/L s-TKN

COD Interference

One of the benefits with using the LR s-TKN with higher concentration range samples is being able to dilute out the possible COD interference. The method states that a concentration of 500 mg/L of COD will interfere with the s-TKN results. The HR TN method can handle >1000 mg/L interference of COD.



Be Right™

30 mg/L NO₃-N standard spiked with KHP, Glutamic Acid and Glucose

Spike Concentration (as COD)	KHP		Glutamic Acid	Glucose	
	TN	NO ₃ -N	NO ₃ -N	TN	NO ₃ -N
30 mg/L NO ₃ -N Stnd	29.2	29.0	28.7	27.8	28.3
500 mg/L Spike	229.9	29.5	29.1	28.8	28.4
1000 mg/L Spike	29.2	29.0	28.8	28.9	28.8
2000 mg/L Spike	29.9	29.1	28.8	28.6	28.8
3000 mg/L Spike	28.9	29.0	29.3	28.9	29.2
4000 mg/L Spike	29.5	29.2	29.1	28.7	29.0

This table shows that the HR TN and HR NO₃ reagents can handle up to ~4000 mg/L of COD in the 30 mg/L NO₃-N standard DI water matrix. Further lab studies showed that effluent samples spiked with ~2000 mg/L of a combined COD spike with KHP and glucose. This COD spike study did not exhibit any synergistic or antagonistic effects due to the presence of the presumed interference.

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