

Nessler Method¹

1 to 150 mg/L TKN

Method 8075

Reagent Solution

Scope and application: For water, wastewater and sludge; digestion is required.

¹ Adapted from Hach, et. al., Journal of Association of Official Analytical Chemists, 70(5) 783-787 (1987); Hach, et. al., Journal of Agricultural and Food Chemistry, 33(6) 1117-1123 (1985); Standard Methods for the Examination of Water and Wastewater.



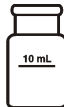
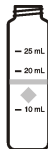
Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Before starting

Hach no longer offers the Digesdahl Digestion Apparatus. Hach does offer an EPA approved method for TKN following the sTKN method using TNT880.

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

If the Pour-Thru Cell is used (for applicable instruments), clean the cell periodically. Pour a few sodium thiosulfate pentahydrate crystals into the cell funnel or rinse the cell with a solution of sodium thiosulfate. Flush the crystals through the funnel and cell with enough deionized water to dissolve. Rinse the cell with deionized water.

The Nessler reagent contains mercuric iodide. Both the reacted sample and blank will contain mercury. Do not pour these solutions down the drain. Collect the reacted samples and the blank for proper disposal.

Hold the reagent droppers and dropper bottles vertically, not at an angle, when the reagent is added.

Use the Standard Adjust option with each new lot of reagent for the best results. Refer to the Standard solution method in [Accuracy check](#) on page 6.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

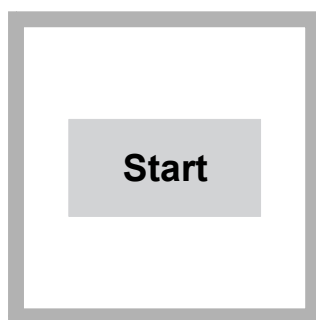
Description	Quantity
Boiling chips, silicon carbide	2-3
Cylinder, graduated mixing, 25-mL	2
Finger cots	2
Digesdahl Digestion Apparatus (Obsolete, no replacement available)	1
Safety shield	1
Hydrogen Peroxide, 50%	20 mL
Mineral Stabilizer	6 drops
Nessler Reagent	2 mL
Polyvinyl Alcohol Dispersing Agent	6 drops
Potassium Hydroxide (KOH) Standard Solution, 1.0 N	varies
Potassium Hydroxide (KOH) Standard Solution, 8.0 N	varies
Sulfuric Acid, ACS, concentrated	6 mL
TKN Indicator Solution	2 drops
Pipet, TenSette, 0.1–1.0 mL, plus tips	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 7 for order information.

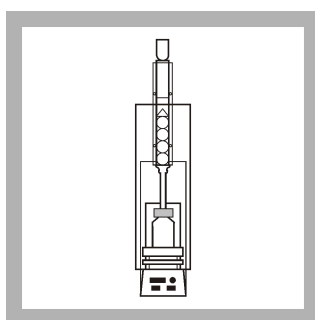
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 3–5 with 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution caused by the volume additions.

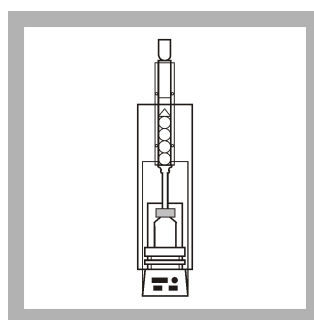
Nessler method



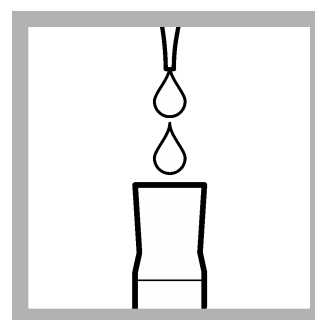
1. Start program **399 Nitrogen, TKN**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



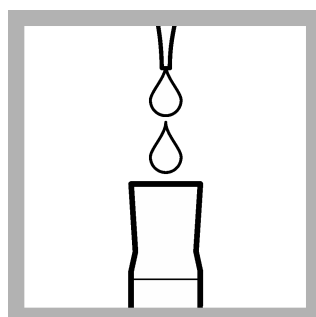
2. **Prepare the sample:** Use the Digesdahl Digestion Apparatus¹ Instruction Manual to digest the sample amount. Refer to [Digested sample volumes](#) on page 5.



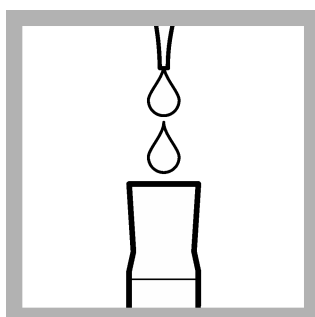
3. **Prepare the blank:** Digest an equal amount of deionized water for use as the blank .



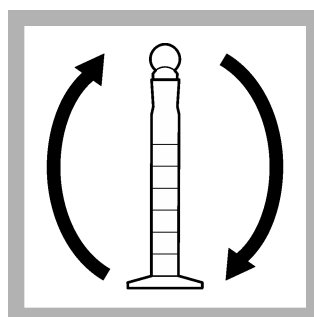
4. Use a pipet to move an analysis volume of the digested sample to a graduated mixing cylinder. Refer to [Digested sample volumes](#) on page 5.



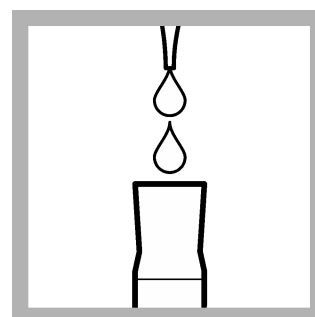
5. Use a pipet to transfer an equal amount of digested deionized water to a second graduated mixing cylinder.



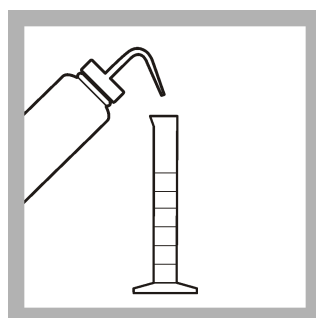
6. Add one drop of TKN Indicator to each cylinder.



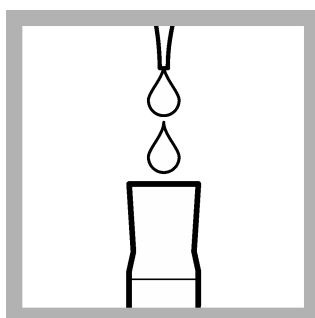
7. If the aliquot is less than 1 mL, go to step 8. If the aliquot is greater than 1 mL, add drops of 8.0 KOH to each cylinder until the first flash of blue color shows. Put the stopper in the cylinder and invert after each addition.



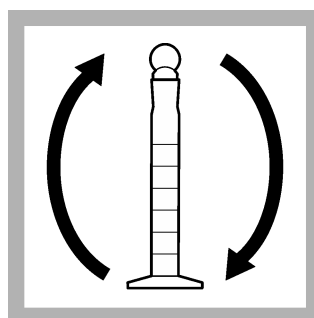
8. Add 1.0 N KOH to each cylinder, one drop at a time. Mix after each addition. Continue until the first permanent blue color shows.



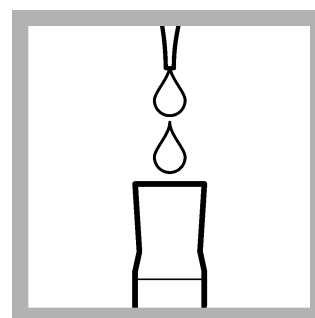
9. Fill both cylinders to the 20-mL mark with deionized water.



10. Add three drops of Mineral Stabilizer to each cylinder.

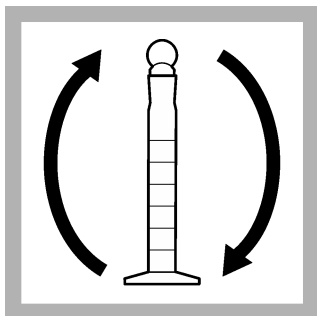


11. Put the stoppers in the cylinders and invert to mix.

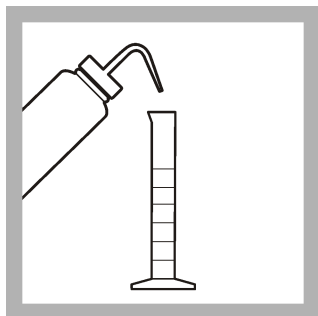


12. Add three drops of Polyvinyl Alcohol Dispersing Agent to each cylinder.

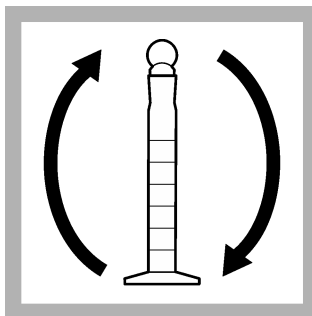
¹ Hach no longer offers the Digesdahl Digestion Apparatus.



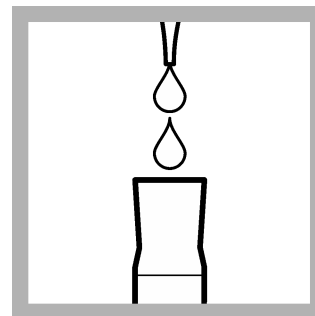
13. Put the stoppers in the cylinders and invert to mix.



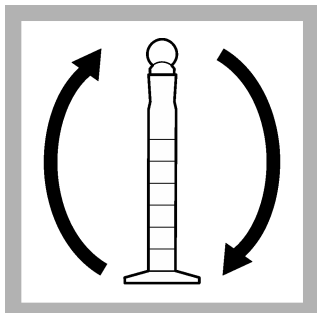
14. Fill both cylinders to the 25-mL mark with deionized water.



15. Put the stoppers in the cylinders and invert several times to mix.



16. Use a pipet to add 1.00 mL of Nessler Reagent to each cylinder.



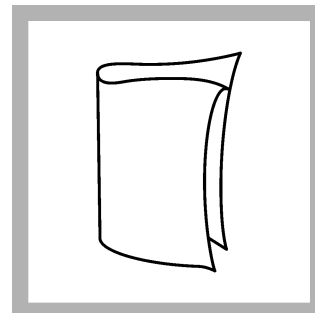
17. Put the stoppers in the cylinders and invert to mix. The solution should not be hazy. Any turbidity (haze) will cause incorrect results.



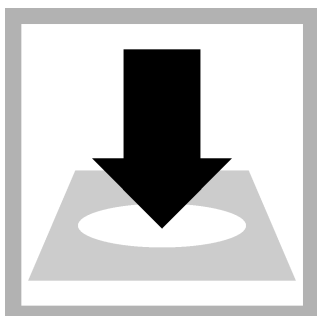
18. Start the instrument timer. A 2-minute reaction time starts.



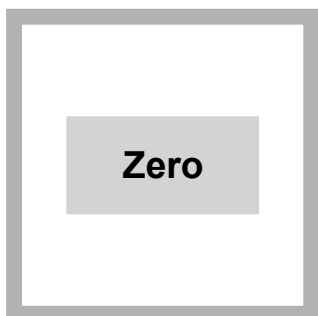
19. When the timer expires, pour the contents of each cylinder into separate sample cells.



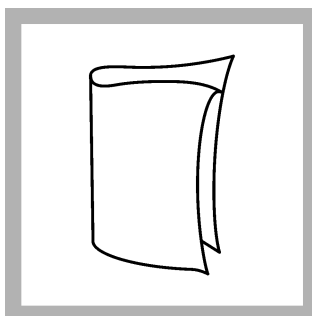
20. Clean the blank sample cell.



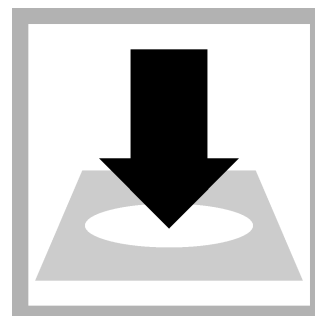
21. Insert the blank into the cell holder.



22. Push **ZERO**. The display shows 0 mg/L TKN.



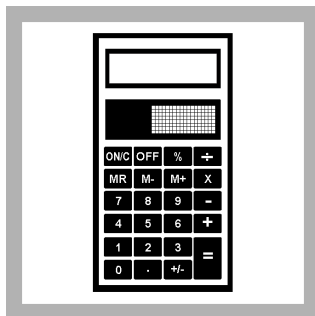
23. Clean the prepared sample cell.



24. Insert the prepared sample into the cell holder.



25. Push **READ**. Results show in mg/L TKN.



26. Calculate the sample TKN in ppm:

$$\text{TKN} = (75 \times A) \div (B \times C)$$

Where:

- A = mg/L read from the display
- B = g (or mL of water) sample taken for the digestion
- C = mL analysis volume of the digested sample

Digested sample volumes

Table 2 Aqueous samples (solutions or suspensions in water—less than 1% solids)

Expected nitrogen concentration (mg/L)	Analysis volume (mL)
0.5–28	10
2–112	5
11–560	2
45–2250	1
425–22500	0.5

Table 3 Dry samples

Expected nitrogen concentration (mg/L)	Analysis volume (mL)
42–2200	10
106–5600	5
350–18,000	2
1000–56,000	1
4200–220,000	0.5

Table 4 Oils and fats

Expected nitrogen concentration (mg/L)	Analysis volume (mL)
85–4500	10
210–11,000	5
2100–110,000	1

Accuracy check

Digestion method

To validate the digestion method, use the Primary Standards for Kjeldahl Nitrogen that are given in the Accuracy Check section of the *Digesdahl® Digestion Apparatus Instruction Manual*². Use the accuracy check procedure to find the digestion efficiency and the amount of bound nitrogen that is released during digestion.

Use the digested Kjeldahl standard in the Nessler test procedure to measure the TKN of the primary standard. The TKN value should be within $\pm 3\%$ of the value of the prepared Kjeldahl standard.

Standard solution method

Items to collect:

- 1.0-mg/L NH₃-N standard solution
- TKN indicator
- Dropper
- 25-mL graduated mixing cylinders (2)
- Deionized water
- Mineral Stabilizer
- Polyvinyl Alcohol Dispersing agent

1. Add one drop of TKN Indicator to each 25-mL graduated mixing cylinder.
2. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0-mg/L NH₃-N standard solution.
3. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix.
4. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder. Invert several times to mix.
5. Continue with the TKN procedure to measure the concentration of the standard solution. Accurate calibrations will show 26–27 mg/L TKN.

Note: The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
399	76 mg/L NH ₃ -N	70–82 mg/L NH ₃ -N	1 mg/L NH ₃ -N

Summary of method

The term Total Kjeldahl Nitrogen refers to the combination of ammonia and organic nitrogen. However, only the organic nitrogen compounds that are present as organically bound nitrogen in the trinegative state are determined in this test. Nitrogen in this form is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified Nessler method test. The measurement wavelength is 460 nm for spectrophotometers or 420 nm for colorimeters.

Pollution prevention and waste management

The Nessler reagent contains mercuric iodide. The reacted samples and blanks will contain mercury and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

² Hach no longer offers the Digesdahl Digestion Aparatus

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Nitrogen Reagent Set, 0-150 mg/L, Nessler Method	—	250 tests	2495300
Includes:			
Hydrogen Peroxide, 50%	20 mL	490 mL	2119649
Mineral Stabilizer	6 drops	50 mL SCDB	2376626
Nessler Reagent	2 mL	500 mL	2119449
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	2376526
Potassium Hydroxide Standard Solution, 1.0 N	varies	50 mL SCDB	2314426
Potassium Hydroxide Standard Solution, 8.0 N	varies	100 mL MDB	28232H
Sulfuric Acid, concentrated, ACS	varies	500 mL	97949
TKN Indicator Solution	2 drops	50 mL SCDB	2251926

Required apparatus

Description	Quantity/test	Unit	Item no.
Boiling chips, silicon carbide	2–3	500 g	2055734
Cylinder, graduated, 25 mL, Certified	2	each	2636240
Finger cots	2	2/pkg	1464702
Pipet, TenSette, 0.1–1.0 mL	1	each	1970001
Pipet tips, for TenSette Pipet, 0.1–1.0 mL	2	50/pkg	2185696
Safety shield	1	each	5003000

Recommended standards

Description	Unit	Item no.
Kjeldahl Nitrogen Primary Standard Set	set of 3	2277800
Nitrogen Ammonia Standard Solution, 1.0-mg/L NH ₃ -N	500 mL	189149
Nitrogen, Ammonia Standard Solution, 10-mL Voluette Ampules, 150 mg/L	16/pkg	2128410
Wastewater Influent Standard Solution, Mixed Parameter, for NH ₃ -N, NO ₃ -N, PO ₄ , COD, SO ₄ , TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Item no.
Sodium Thiosulfate, Pentahydrate	454 g	46001
Pour-Thru Cell Kit (DR 2700, DR 2800)	each	5940400
Pour-Thru Cell Kit (DR 5000)	each	LZV479
PourRite Ampule Breaker, 2-mL	each	2484600
Ampule Breaker, 10-mL Voluette Ampules	each	2196800
Paper, for weighing, 100 x 100 mm	500/pkg	1473885
Pipet, TenSette, 1.0–10.0 mL	each	1970010
Pipet tips for TenSette Pipet, 1.0–10.0 mL	50/pkg	2199796

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Pipet tips for TenSette Pipet, 1.0–10.0 mL	250/pkg	2199725
Pipet tips for TenSette Pipet, 0.1–1.0 mL	1000/pkg	2185628
Nitrogen Ammonia Standard Solution, 10-mg/L NH ₃ -N	500 mL	15349
Nitrogen Ammonia Standard Solution, 100-mg/L as NH ₃ -N	500 mL	2406549
Nitrogen, Ammonia Standard Solution, 1000-mg/L NH ₃ -N	1 L	2354153
Nitrogen Ammonia Standard Solution, 10-mL Voluette Ampule, 50-mg/L NH ₃ -N	16/pkg	1479110
Balance, analytical, 80 g x 0.1 mg 100–240 VAC	each	2936701
Nitrogen Ammonia Standard Solution, 2-mL PourRite Ampules, 50 mg/L	20/pkg	1479120



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