



Scope

This method is used for determining Total Kjeldahl Nitrogen (TKN) in drinking water, surface water, and domestic and industrial wastes according to USEPA Method 351.2 and Standard Methods $4500-N_{org}$ D. This method can also be used for the determination of TKN in potassium chloride (KCl) extracts of soils and plants.

Summary

Digest the sample prior to analysis in the presence of sulfuric acid, potassium sulfate, and a mercury catalyst at a final temperature of 380 °C. Free ammonia and organic nitrogen compounds convert to ammonium sulfate under these conditions. Copper catalysts can be used, however, the green color of the catalyst interferes with the method. The ammonium reacts with salicylate and hypochlorite in a buffered alkaline solution in the presence of sodium nitroferricyanide (pH 12.8-13) to form the salicylic acid analog of indophenol blue. Measure the blue-green color produced at 660 nm.

Interferences

Eliminate precipitation of calcium and magnesium hydroxides by adding potassium sodium tartrate in the working buffer. Filter or centrifuge turbid digestates prior to the analysis. Digestates with background absorbances at the analytical wavelength may interfere with the analysis. Mercury interference is minimized by complexation with chlorine ion.

Performance Specifications

0.05-20.0 mg/L Range: Throughput: 55 samples/hour Precision (at 1.0 mg/L): <1% RSD Precision (at 5.0 mg/L): <1% RSD Method Detection Limit (MDL): 0.01 mg/L 99% Accuracy*:

Chemicals

Ammonium Sulfate, (NH₄)₂SO₄ Sodium Hydroxide, NaOH

Chloroform, CHCl₃ Sodium Hypochlorite, 5.25% available chlorine (OI

Analytical does not recommend household Deionized Water (ASTM Type I or II)

bleach), NaOCl

Hydrochloric Acid, concentrated, HCl Sodium Nitroferricyanide Dihydrate,

Potassium Sodium Tartrate

Tetrahydrate, KNaC₄H₄O₆•4H₂O

Na₂Fe(CN)₅NO•2H₂O (FW 297.95)

Sodium Phosphate Dibasic Anhydrous, Na₂HPO₄

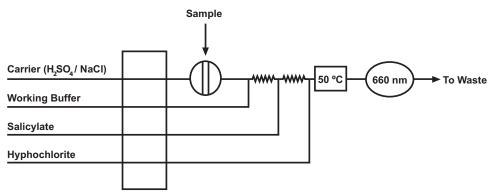
Potassium Sulfate, K₂SO₄ Sodium Salicylate, NaC₇H₅O₃ Red Mercuric Oxide, HgO Sulfuric Acid, concentrated, H₂SO₄

Sodium Chloride, NaCl

^{*}Undigested; ERA (Environmental Resources Associates) WasteWatR Simple Nutrients Quality Control Sample.



Basic Flow Diagram



Note

This method complies with USEPA Method 351.2.

Selected References

Methods for Chemical Analysis of Water and Wastewater; EPA-600/4-79-020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; Method 351.2.

Standard Methods for the Examination of Water and Wastewater, 21st ed.; American Public Health Association: Washington, D.C., 2005.

Figures

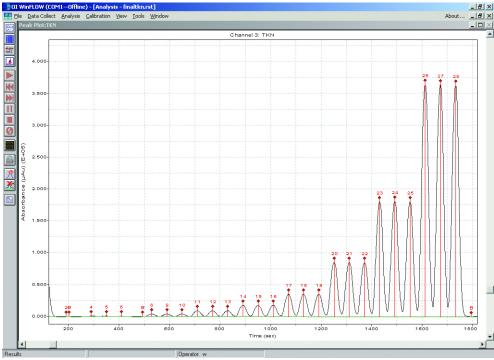


Figure 1. TKN Calibration (0.05–20 ppm)



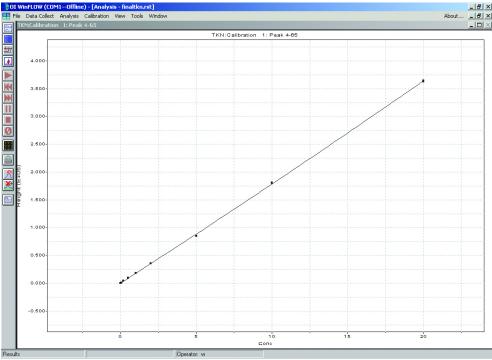


Figure 2. TKN Calibration Curve (0.05–20 ppm)

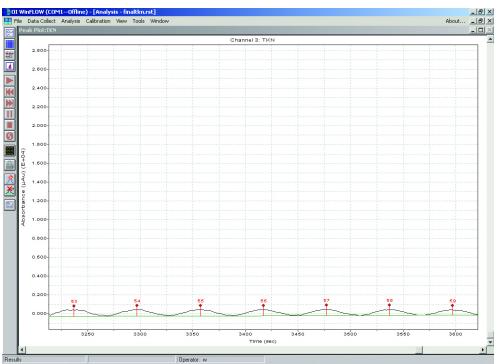


Figure 3. TKN MDL (at 0.01 ppm)



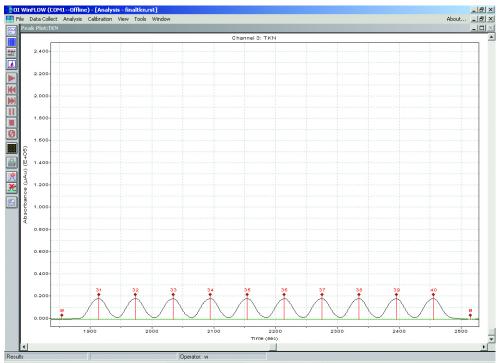


Figure 4. TKN Precision (at 1 ppm)

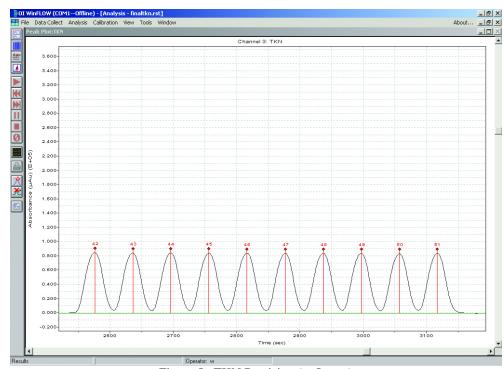


Figure 5. TKN Precision (at 5 ppm)



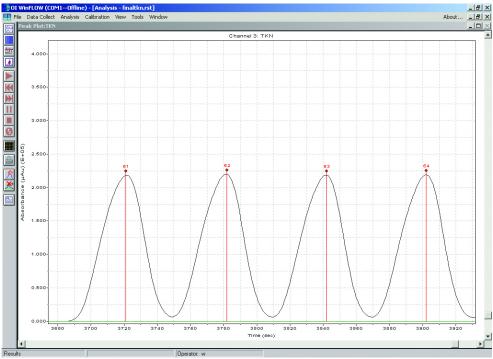


Figure 6. TKN IDAC QC Samples

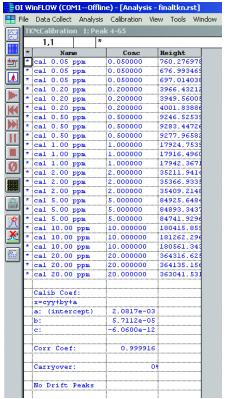


Figure 7. TKN Calibration Results (0.05–20 ppm)





Table 1. Total Kjeldahl Nitrogen Precision Calculations

	1.00 mg N/L	5.00 mg N/L	0.10 mg N/L	ERA
				P127-739B
Rep 1	0.985	4.76	0.118	12.9
Rep 2	0.978	4.74	0.132	12.8
Rep 3	0.974	4.73	0.116	12.8
Rep 4	0.969	4.74	0.116	12.8
Rep 5	0.974	4.73	0.116	_
Rep 6	0.972	4.74	0.116	_
Rep 7	0.968	4.73	0.114	_
Rep 8	0.978	4.73	_	_
Rep 9	0.975	4.74	_	_
Rep 10	0.971	4.70	_	_
Mean	0.974	4.73	0.118	12.8
Standard Deviation	0.0005016	0.014789	0.006157	0.035151
% RSD	0.51	0.31	5.20	0.27
% Recovery	97	95	118	98
MDL	_	_	0.019	_

ERA sample recovery on non-digested, simple nutrient Ready-to-Use Quality Control Sample.