Method Abstract

Scope

This method is used for the determination of 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

The sample is digested 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 are converted to ammonium sulfate under these conditions. 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. The blue-green color produced is measured at 660 nm.

Interferences

High nitrate concentrations (10 times or more than the TKN level) result in low TKN values. If interference is suspected, dilute samples and analyze again. Precipitation of calcium and magnesium hydroxides is eliminated by potassium sodium tartrate in the working buffer. Filter turbid samples prior to analysis. Digestates with background absorbance at the analytical wavelength may interfere.

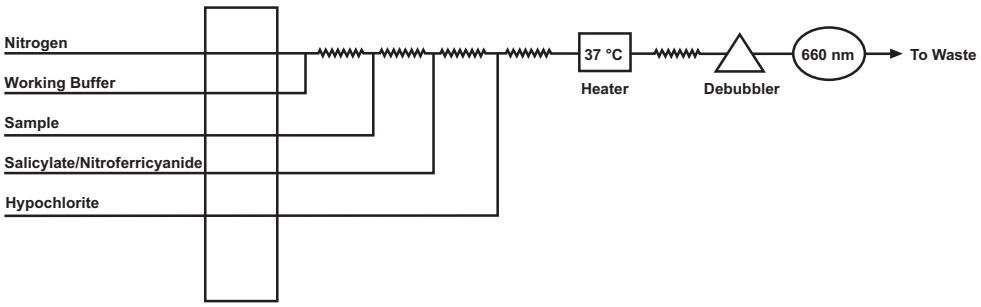
Performance Specifications

Range:	0.05–20 mg/L
Throughput:	60 samples/hour
Precision (at 0.2 mg/L):	<1% RSD
Precision (at 2.0 mg/L):	<1% RSD
Precision (at 20 mg/L):	<2% RSD
Method Detection Limit (MDL):	0.008 mg/L
Accuracy:	94.4%

Chemicals

- Ammonium Sulfate, $(NH_4)_2SO_4$
- Brij®-35, 21% solution (part number A21-0110-33)
- Chloroform, $CHCl_3$
- Deionized (DI) Water, ASTM Type I or II
- Potassium Sodium Tartrate Tetrahydrate, $KNaC_4H_4O_6 \bullet 4H_2O$
- Potassium Sulfate, K_2SO_4
- Red Mercuric Oxide, HgO
- Sodium Hydroxide, NaOH
- Sodium Hypochlorite, 5.25% available chlorine, NaOCl
- Sodium Nitroferricyanide Dihydrate, $Na_2Fe(CN)_5NO \bullet 2H_2O$
- Sodium Phosphate Dibasic Anhydrous, Na_2HPO_4
- Sodium Salicylate, $NaC_7H_5O_3$
- Sulfuric Acid, concentrated, H_2SO_4

Basic Flow Diagram



Selected Reference

Methods for the Determination of Inorganic Substances in Environmental Samples; EPA/600/R-93/100; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1993; Method 351.2.

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

Figures

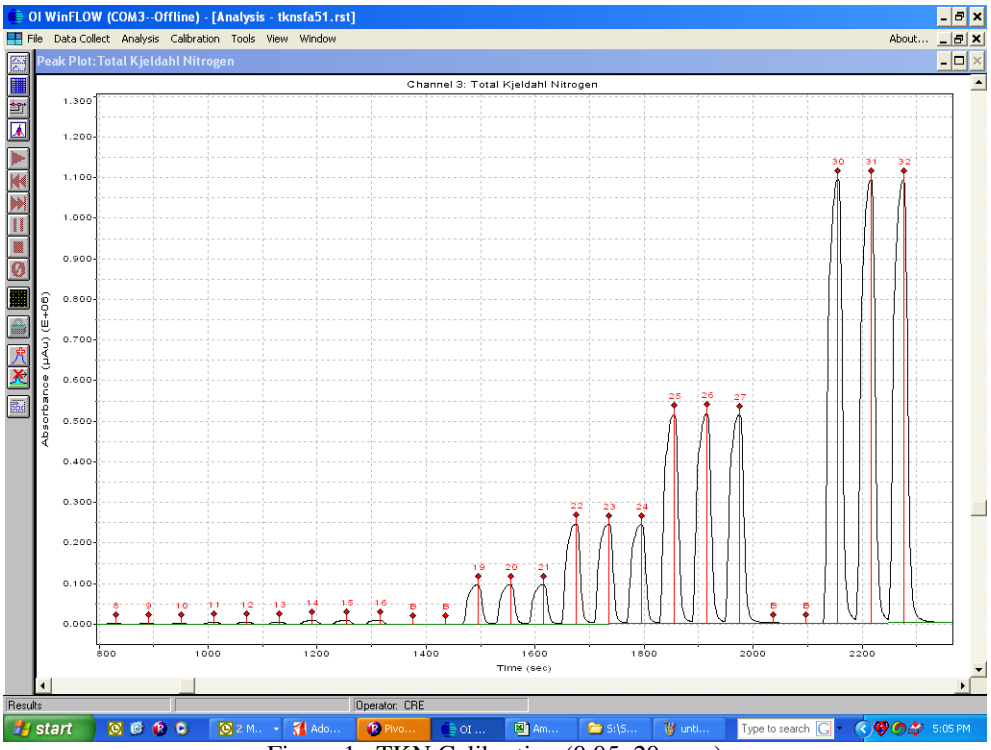


Figure 1. TKN Calibration (0.05–20 ppm)

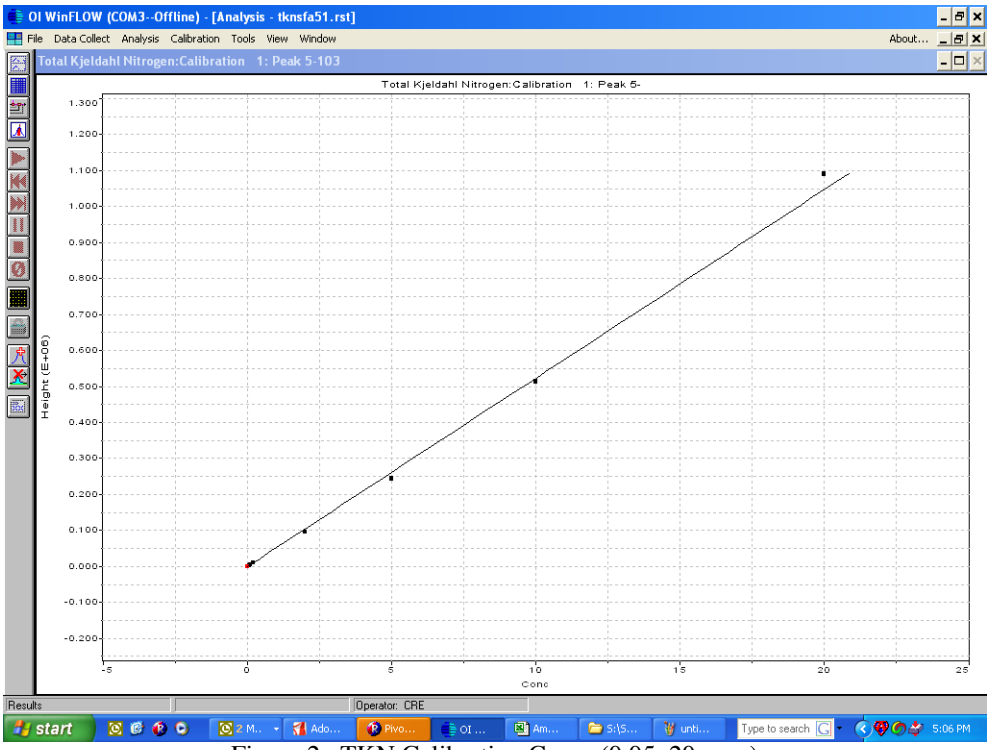


Figure 2. TKN Calibration Curve (0.05–20 ppm)

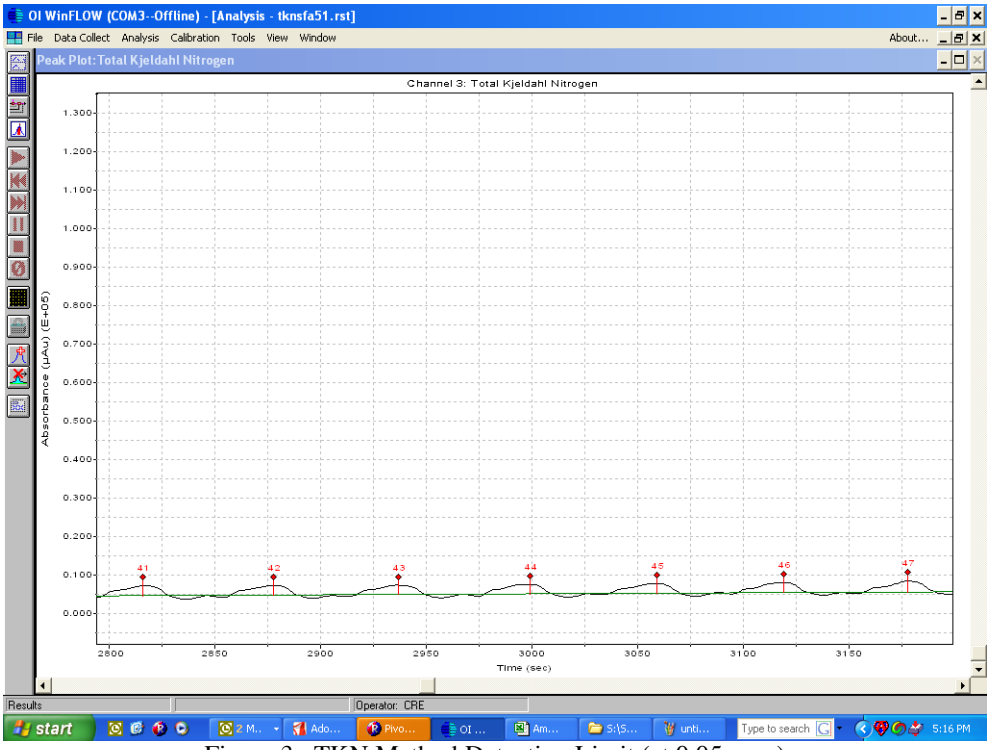


Figure 3. TKN Method Detection Limit (at 0.05 ppm)

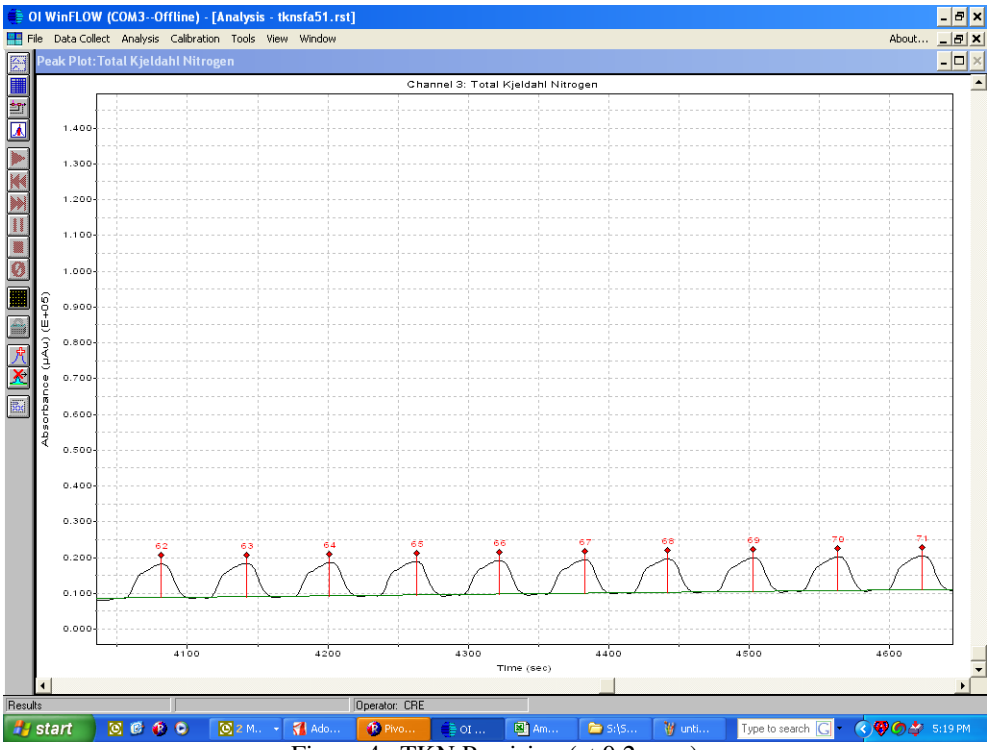


Figure 4. TKN Precision (at 0.2 ppm)

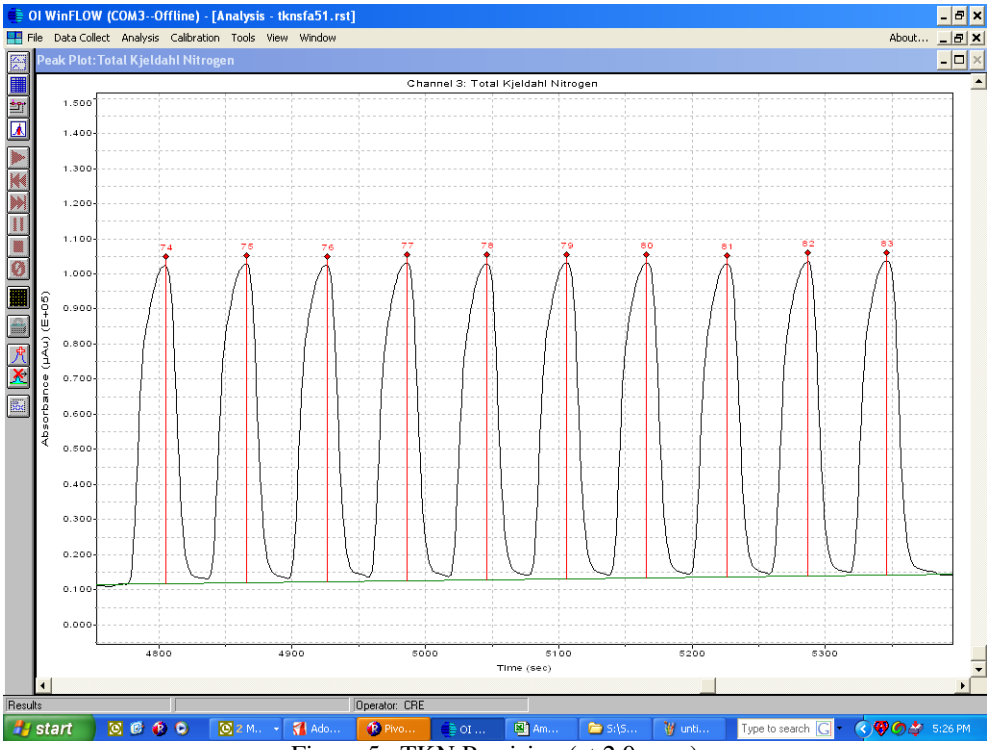


Figure 5. TKN Precision (at 2.0 ppm)

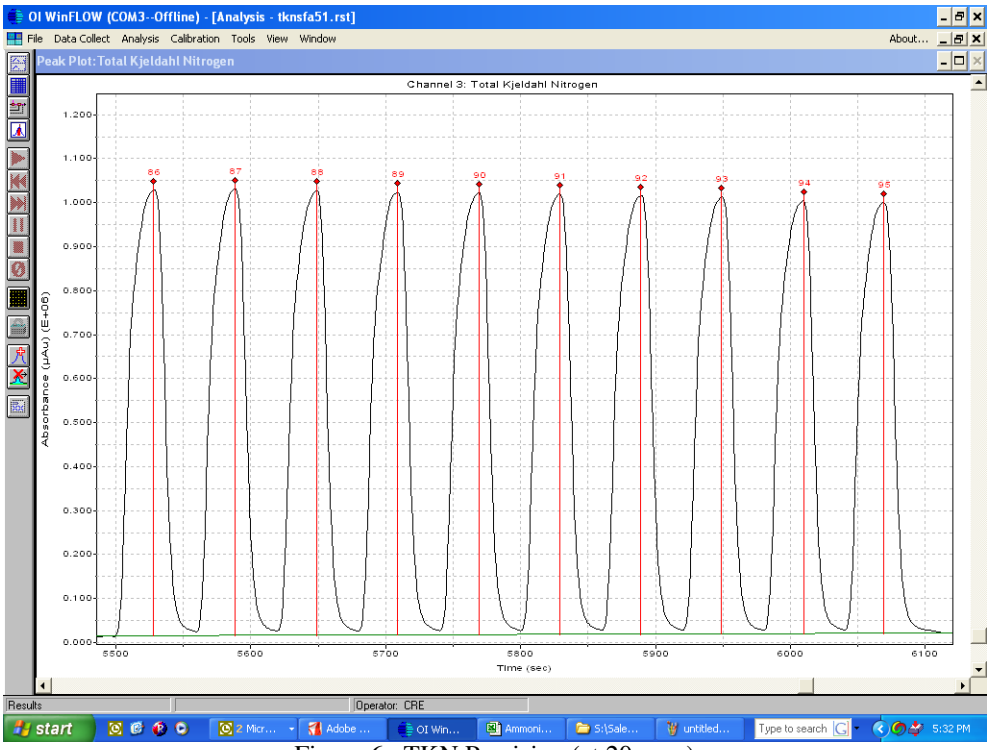


Figure 6. TKN Precision (at 20 ppm)

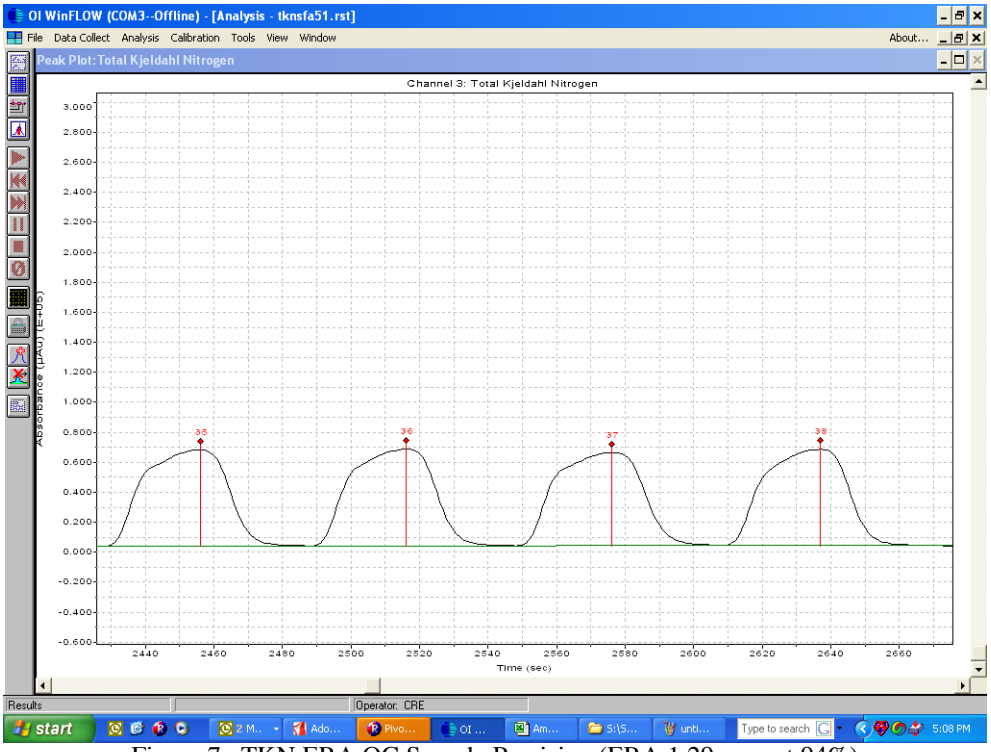


Figure 7. TKN ERA QC Sample Precision (ERA 1.29 ppm at 94%)

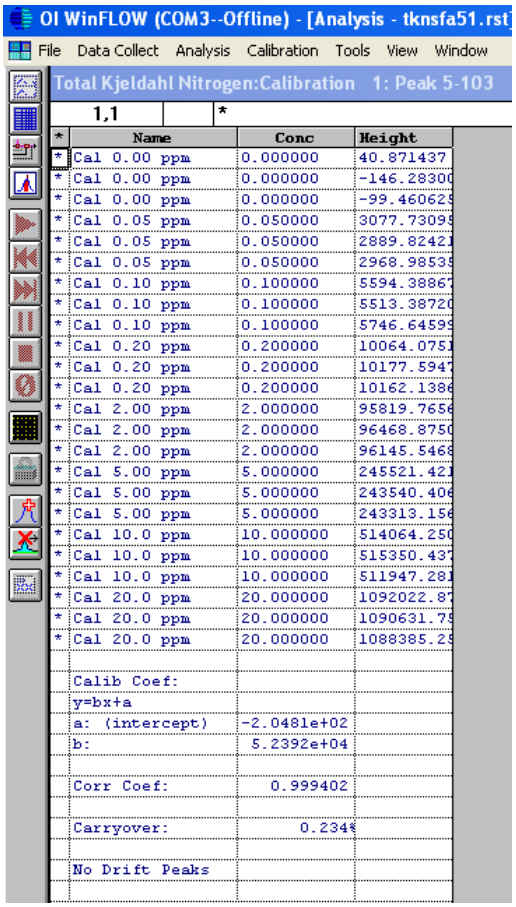


Figure 8. TKN Calibration Results (0.05–20 ppm)

Table 1. TKN Method Data

Parameter	Calibrant 0.05 mg/L	Calibrant 0.2 mg/L	Calibrant 2.0 mg/L	Calibrant 20 mg/L	ERA QC Standard 1.29 mg/L
Rep 1	0.05381	0.1835	1.732	19.38	1.232
Rep 2	0.05206	0.1817	1.730	19.36	1.239
Rep 3	0.05070	0.1819	1.722	19.28	1.188
Rep 4	0.05189	0.1817	1.727	19.19	1.229
Rep 5	0.05141	0.1814	1.718	19.18	—
Rep 6	0.05472	0.1811	1.716	19.10	—
Rep 7	0.05845	0.1808	1.710	19.04	—
Rep 8	—	0.1804	1.701	18.96	—
Rep 9	—	0.1825	1.708	18.79	—
Rep 10	—	0.1811	1.706	18.72	—
Average	0.05329	0.1816	1.717	19.10	1.222
Standard Deviation	0.0002668	0.0008964	0.01074	0.02249	0.02299
% RSD	5.007	0.4936	0.6253	1.177	1.882
MDL	0.0084	—	—	—	—
% Accuracy	—	—	—	—	94.40%

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