

NITROGEN, KJELDAHL, TOTAL
 EPA Method 351.1 (Colorimetric, Automated Phenate),
 EPA Method 351.2 (Colorimetric, Semi-Automated Block Digester, AAI), or
 EPA Method 351.4 (Potentiometric, Ion Selective Electrode)

Table 1. Summary of Contract Required Detection Limits, Holding Times, and Preservation for Total Kjeldahl Nitrogen

Analytical Parameter	Contract Required Detection Limit (CRDL)	Technical and Contract Holding Times	Preservation
Total Kjeldahl-Nitrogen as (TKN-N)	0.10 mg/L	Technical: 28 days from collection; Contract: 26 days from receipt at laboratory	H ₂ SO ₄ to pH <2, Cool to 4°C ±2°C

Data Calculations and Reporting Units:

Calculate the sample results according to Section 9 of EPA Method 351.1 or Section 8 of EPA Methods 351.2 and 351.4.

Report sample results for Total Kjeldahl-Nitrogen in concentration units of milligram per liter (mg/L) as nitrogen (TKN-N). Report Total Kjeldahl-N concentrations that are less than 10 mg/L to 2 significant figures, and Total Kjeldahl-N concentrations that are greater than or equal to 10 mg/L to 3 significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 2. Summary of Calibration Procedures for Total Kjeldahl-Nitrogen by EPA 351.1, 351.2, and 351.4

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points) (ICAL) ^a	With each set of samples analyzed;	r \geq 0.995	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Initial Calibration Verification (ICV) (Separate source from ICAL standards)	Daily, prior to sample analysis, immediately following ICAL	\pm 10% from expected concentration	1. Reprep ICV and, reanalyze all associated samples 2. Identify and document problem 3. Recalibrate and reanalyze reprepared ICV and all associated samples
CRDL Verification Standard (< 2X CRDL)	Daily, after ICV and before samples	\pm 20% from expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify
Continuing Calibration Verification (CCV)	Before sample analysis; after every 10 samples and end of run	\pm 10% from expected concentration	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV
Calibration Blank Verification (ICB, CCB)	After ICV and CCVs	< CRDL	1. Terminate analysis 2. Identify and document the problem 3. Recalibrate, verify and reanalyze all associated samples

Prepare a standard curve with each set of samples analyzed.

Dilute and reanalyze samples with Total Kjeldahl-N concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

^a Method 351.1: Analyze calibration standards in order of increasing concentration for the colorimetric procedure, lowest to highest. Reanalyze any sample with a Total Kjeldahl-N concentration that is less than 10% of the sample run immediately prior to it.

Table 3. Summary of Internal Quality Control Procedures for Total Kjeldahl-Nitrogen by EPA 351.1, 351.2, and 351.4

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB) ^a	One per Batch or SDG (1 per 20 samples minimum) ^b	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all affected samples must be prepared again with another method blank and reanalyzed
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples \geq 5X CRDL; \pm CRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike (MS) ^c	One per batch or SDG (1 per 20 samples minimum)	\pm 25% from expected value ^d	1. Flag associated data with an "N"
Laboratory Control Sample (LCS) ^a	One per batch or SDG (1 per 20 samples minimum)	\pm 20% from expected concentration	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples

^a The MB and LCS must be treated identically to the samples. If the environmental samples are preserved with sulfuric acid, the LCS and MB should be acidified with sulfuric acid. Similarly, if the environmental samples are filtered due to presence of suspended matter, the LCS and MB should be filtered also.

^b SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^c Spike the Matrix Spike sample prior to sample digestion.

^d An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of 4. In such an event, the data shall be reported unflagged.