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Method 1686

Nitrate/Nitrite-N in Water and Biosolids by Manual Colorimetry

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Disclaimer

This draft method has been reviewed and approved for publication by the Analytical Methods Staff within the Engineering and Analysis Division of EPA. Mention of trade names or commercial products does not constitute endorsement or recommendation for use. EPA plans further validation of this draft method. The method may be revised following validation to reflect results of the study. This method version contains minor editorial changes to the November 1999 version.

EPA welcomes suggestions for improvement of this method. Suggestions and questions concerning this method or its application should be addressed to:

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Note: This method is performance based. The laboratory is permitted to omit any step or modify any procedure provided that all performance requirements in this method are met. The laboratory may not omit any quality control analyses. The terms "shall," "must," and “may not” define procedures required for producing reliable results. The terms "should" and "may" indicate optional steps that may be modified or omitted if the laboratory can demonstrate that the modified method

Method 1686
Nitrate/Nitrite - N in Water and Biosolids by Manual Colorimetry

1.0 Scope and Application

1.1 This method describes procedures for the determination of nitrate/nitrite-nitrogen, or oxidized nitrogen, in drinking, ground, and surface water; domestic and industrial waste; and biosolids (municipal sewage sludge). Manual colorimetry is used to determine the nitrate/nitrite-N concentration. This method is based on U.S. Environmental Protection Agency (EPA) Method 353.3: *Nitrogen, Nitrate-Nitrite (Spectrophotometric Cadmium Reduction)* (Reference 16.1). This method is associated with Method 1691: *Municipal Biosolids Sampling Guidance* (Reference 16.2).

1.2 This method is for use in EPA's data gathering and monitoring programs under the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, the Solid Waste Disposal Act, and the Safe Drinking Water Act.

1.3 Method detection limits and minimum levels for nitrate/nitrite-nitrogen have not been formally established for this draft method. These values will be determined during the validation of the method.

1.4 This method is performance based. The laboratory is permitted to omit any step or modify any procedure, provided that all performance requirements in this method are met. Requirements for establishing method equivalency are given in Section 9.1.2.

1.5 Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedures in Section 9.2.

2.0 Summary of Method

2.1 Aqueous samples are filtered then passed through a cadmium-copper reduction column prior to analysis.

2.2 The oxidized nitrogen (the sum of nitrite and nitrate) in solid samples is extracted in reagent water, filtered, and passed through a cadmium-copper reduction column prior to analysis. The cadmium-copper reduction column converts any nitrate present in the samples to nitrite.

2.3 The nitrite concentration of samples (nitrite originally present plus reduced nitrate) is determined by diazotizing with sulfanilimide and coupling with N-(1-naphthyl)ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically.

2.4 Quality is assured through calibration and testing of the sample preparation and analytical instruments.

3.0 Definitions

Definitions for terms used in this method are given in Section 18.

4.0 Interferences

4.1 Large concentrations of fat, oil and grease (FOG) will coat the cadmium particles used for reduction of nitrate to nitrite. FOG should be removed by sample extraction with hexane or a 80%/20% hexane/methyl *t*-butyl ether mixture prior to passing the sample through the reduction column (Section 11.2.3).

4.2 Residual chlorine, if present, must be removed by pretreatment of the sample with sodium thiosulfate before digestion/distillation. Typically, this will be necessary if the sample contains free water or is aqueous.

4.3 Color present in the sample that absorbs at about 540 nm interferes with the photometry.

4.4 Certain metal ions can interfere with the colorimetry. An ethylenediamine tetraacetate (EDTA) solution is added to the sample extract to remove these potential interferences.

5.0 Safety

5.1 The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should be available to all personnel involved in the chemical analysis. Additional information on laboratory safety can be found in Reference 16.3.

5.2 If samples originate from a highly contaminated area, appropriate sample handling procedures must be followed to minimize worker exposure.

5.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.

6.0 Equipment and Supplies

NOTE: *Brand names, suppliers, and part numbers are for illustration only, and no endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here. Meeting the performance requirements of this method is the responsibility of the sampling team and laboratory.*

6.1 Equipment for Extraction

6.1.1 Erlenmeyer flasks, 500 mL.

6.1.2 Wrist shaker or orbital shaker capable of holding 500 mL flasks.

6.1.3 Syringe filters—0.45µm pore diameter. These filters should be rinsed with a dilute sulfuric acid solution before use.

6.2 Equipment for Analysis

6.2.1 Chromatographic column—3.5 mm inside diameter, with a packing tube length of 20 cm and a liquid reservoir capacity of 85 mL.

6.2.2 Photometer—Capable of analysis at 540 nm wavelength, and recorder or data system.

6.3 General equipment

6.3.1 Drying oven-Capable of maintaining a constant temperature in the range of 103-105°C.

6.3.2 Analytical balance-Capable of weighing to 0.001 g (1 mg).

6.3.3 Glass wool.

6.3.4 PTFE thread sealing tape.

7.0 Reagents and Standards

7.1 Use deionized or distilled water, shown to be free of nitrate and nitrite, for all solutions and reagents used in this method.

7.2 6N Hydrochloric acid (HCl)—50 mL of concentrated HCl, diluted to 100 mL with distilled water.

7.3 2% Copper sulfate ($CuSO_4$) solution—20 g $CuSO_4 \cdot 5H_2O$ in 1 L reagent water.

7.4 Cadmium granules

7.4.1 Rinse 25 g cadmium granules, 20-100 mesh, with 6N HCl followed by a rinse with reagent water. The cadmium should have a silver color.

7.4.2 Add 100 mL of 2% $CuSO_4$ solution and swirl the mixture for 5 minutes or until the blue color fades noticeably. Decant the liquid, and repeat with fresh $CuSO_4$ until a brown colloidal precipitate begins to form.

7.4.3 Rinse the granules with reagent water until the brown copper precipitate is removed. The cadmium should now have a black color.

7.5 85% Phosphoric acid.

7.6 Sulfanilimide (CAS # 63-74-1), suitable for diazotization titration.

7.7 N-(1-naphthyl)-ethylenediamine dihydrochloride (CAS # 1465-25-4).

7.8 Color Reagent

7.8.1 Add 100 mL 85% phosphoric acid (H_3PO_4) and 10 g sulfanilimide to about 800 mL reagent water and mix to completely dissolve the sulfanilimide.

7.8.2 Add 1 g N-(1-naphthyl)-ethylenediamine dihydrochloride and mix to dissolve. Dilute to 1 L with reagent water. Store the solution refrigerated in an amber glass container.

7.8.3 This reagent is stable for about one month. Remake if the solution is older than a month.

7.9 Ammonium chloride (NH_4Cl)

7.10 Concentrated ammonium hydroxide (NH_4OH)

7.11 Potassium nitrate (KNO_3)

7.12 Sodium nitrite ($NaNO_2$)

7.13 Dechlorinating reagent—Dissolve 0.35 g sodium thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) in reagent water and dilute to 100 mL. One mL of this reagent will neutralize 1 mg/L of residual chlorine in a 500 mL sample aliquot.

7.14 Ammonium chloride—EDTA solutions

7.14.1 Stock solution

7.14.1.1 Dissolve 13 g NH_4Cl in about 900 mL reagent water.

7.14.1.2 Add 0.1 g disodium EDTA and dilute to 1 L.

7.14.1.3 Adjust the pH to 8.5 with conc. NH_4OH.

7.14.2 Dilute solution—Dilute 300 mL of stock NH_4Cl-EDTA solution to 500 mL in reagent water.

7.15 Blank sand—Bake 500 g of clean sand or diatomaceous earth at 400°C for eight hours. Cool and store in a glass container with a sealing lid.

7.16 Chloroform ($CHCl_3$)

7.17 Oxidized nitrogen standards

7.17.1 Nitrate stock solution—1000 mg/L NO_3-N.

7.17.1.1 Dry several grams of potassium nitrate (KNO_3) in an oven at 105°C for 24 hours. Cool in a dessicator.

7.17.1.2 Dissolve 0.7218 g KNO_3 in reagent water and dilute to 100 mL in a volumetric flask. Preserve with 0.2 mL chloroform ($CHCl_3$).

7.17.2 Nitrate working standard—10 mg/L NO_3-N. Dilute 10 mL of nitrate stock solution (Section 7.16.1) to 1 L in reagent water in a volumetric flask.

7.17.3 Nitrite (NO_2) stock solution—1000 mg/L NO_2-N.

7.17.3.1 Because nitrite is oxidized rapidly in the presence of moisture, use a fresh unopened bottle of sodium nitrite ($NaNO_2$) for preparing the stock solution.

7.17.3.2 Dissolve 0.4928 g $NaNO_2$ in reagent water and dilute to 100 mL in a volumetric flask. Preserve with 0.2 mL $CHCl_3$.

7.17.4 Nitrite working standard—10 mg/L NO_2-N. Dilute 10 mL of nitrite stock solution (Section 7.17.3) to 1 L in reagent water in a volumetric flask.

7.18 Quality control sample (QCS)—A prepared quality control sample from a standards vendor (ERA catalog # 545, or equivalent).

8.0 Sample Collection, Preservation, and Storage

8.1 A sufficient volume of sample for analysis must be collected using the procedures found in Reference 16.2 for biosolids samples and Reference 16.4 for water and wastewater samples. Samples should be collected in wide mouth jars with a minimum of air space above the biosolids sample. Minimize exposure of samples to air and intense light as much as possible.

8.2 Nitrate can be formed or lost during storage due to biological activity or lost by oxidation. The following preservation procedures will help prevent significant changes in the analyte concentration.

8.2.1 If the sample contains free water or is aqueous:

8.2.1.1 Samples should be checked for residual chlorine and treated with sodium thiosulfate, if necessary, during collection.

8.2.1.2 Samples should be preserved with 2 mL concentrated H_2SO_4 and cooled to 4°C as soon as possible after collection. The holding time for samples should not exceed 28 days from sampling.

8.2.2 If the sample contains no free water or is solid, the sample should be cooled to 4°C as soon as possible after collection. The holding time for samples should not exceed 28 days from sampling.

8.3 Collect a separate sample for total solids determination (Appendix A). The holding time for total solids determination is seven days.

9.0 Quality Control

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, the ongoing analysis of laboratory reagent blanks, precision and recovery standards, and matrix-spiked samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

9.1.2 In recognition of advances that are occurring in analytical technology, the analyst is permitted certain options to improve separations or lower the costs of measurements, provided that all performance specifications are met. Changes that degrade method performance are not allowed. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for nitrate/nitrite-N in the sample of interest. Specificity is defined as producing results equivalent to the results produced by this method for analytical standards (Section 9.4) and, where applicable, environmental samples (Section 9.5), and as meeting all of the QC criteria stated in this method.

9.1.2.1 Each time a modification is made to this method, the analyst is required to repeat the IPR test in Section 9.2.2 to demonstrate that the modification produces results equivalent to or better than results produced by this method. If the detection limit of the method will be affected by the modification, the analyst must demonstrate that the MDL (40 CFR part 136, appendix B) is less than or equal to the MDL in this method or one-third the regulatory compliance level, whichever is higher. The tests required for this equivalency demonstration are given in Section 9.1.2.2.4.

9.1.2.2 The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:

9.1.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.

9.1.2.2.2 A listing of pollutant(s) measured (nitrate/nitrite-N).

9.1.2.2.3 A narrative stating reason(s) for the modification.

9.1.2.2.4 Results from all quality control (QC) tests comparing the modified method to this method, including:

(a) Calibration (Section 10).
(b) Calibration verification (Section 9.6).
(c) Initial precision and recovery (Section 9.2.2).
(d) Analysis of blanks (Section 9.3).
(e) Accuracy assessment (Sections 9.5 and 9.7).
(f) Ongoing precision and recovery (Section 9.4).
(g) Method detection limit (Section 9.2.1)
(h) Nitrate Reduction Efficiency (Section 9.8)

9.1.2.2.5 Data that will allow an independent reviewer to validate each determination by tracing the instrument output (weight, absorbance, or other signal) to the final result. These data are to include:

(a) Sample numbers and other identifiers.
(b) Sample preparation dates.
(c) Analysis dates and times.
(d) Analysis sequence/run chronology.

(e) Sample weight or volume.
(f) Dry weight ratio (solid and semi-solid samples only; Appendix A).
(g) Distillate solution volume.
(h) Make and model of analytical balance and weights traceable to NIST.
(i) Copies of logbooks, printer tapes, and other recordings of raw data.
(j) Data system outputs, and other data to link the raw data to the results reported.

9.1.3 Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedures and criteria for blank analyses are described in Section 9.3

9.1.4 Analyses of ongoing precision and recovery samples are required to demonstrate that the sample preparation and analysis are within the specified limitations. The procedure and criteria for OPR sample analysis are described in Section 9.4.

9.1.5 Analyses of matrix spike and matrix spike duplicate samples are required to demonstrate method accuracy and precision, and to monitor interferences caused by the sample matrix. The procedure and criteria for spiking are described in Section 9.5.

9.1.6 Analyses of calibration verification standards are required to demonstrate accuracy and stability of the initial calibration. The procedure and criteria for calibration verification analyses are described in Section 9.6.

9.1.7 Analyses of quality control samples (QCS) are required to demonstrate the accuracy of the calibration standards and the analytical system. The procedure and criteria for the QCS sample analyses are described in Section 9.7.

9.2 Initial demonstration of laboratory capability—The initial demonstration of laboratory capability is used to characterize laboratory performance and method detection limits. The MDL and IPR for solid samples should be determined using blank sand as a reference matrix. The MDL and IPR for aqueous samples should be determined using reagent water as a reference matrix.

9.2.1 Method detection limit (MDL)—The MDL should be established for nitrate/nitrite-N according to the procedures at 40 CFR Part 136, Appendix B (Reference 16.5). First, spike a reference matrix with nitrate working standard (Section 7.17.2) to produce a concentration one to five times the estimated detection limit. To determine the MDL, take seven replicate aliquots of the spiked reference matrix and process each aliquot through each step of the analytical method. Perform all calculations and report the concentration values in the appropriate units. Aqueous and/or solid method detection limits should be determined every year or whenever a modification to the method or analytical system is made that will affect the MDL.

9.2.2 Initial precision and recovery (IPR)—To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.2.1 Prepare four spiked samples as detailed in Section 9.4. Using the procedures in Section 11, prepare and analyze these spiked samples for nitrate/nitrite-N.

9.2.2.2 Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation (s) of the percent recovery for nitrate/nitrite-N.

9.2.2.3 Compare s and X with the corresponding limits for initial precision and recovery in Table 1. If s and X meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or X falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.

9.3 Laboratory blanks—Laboratory blanks are analyzed to demonstrate freedom from contamination. Aqueous samples should be run with an aqueous blank, and solid samples should be run with a solid blank.

9.3.1 Prepare and analyze a laboratory blank initially (i.e., with the tests in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample, and will consist of 250 mL of nitrate/nitrite-free reagent water (aqueous blank) or 5 g aliquot of blank sand in 250 mL of nitrate/nitrite-free reagent water (solid blank).

9.3.2 If material is detected in the blank at a concentration greater than the MDL (Section 1.3), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination. All samples must be associated with an uncontaminated laboratory blank before the results may be reported for regulatory compliance purposes.

9.4 Ongoing precision and recovery (OPR)—The laboratory must analyze at least one ongoing precision and recovery sample with each analytical batch. A solid OPR should be run with solid samples, and an aqueous OPR should be run with aqueous samples. An aqueous OPR is prepared by spiking reagent water with nitrate working standard (Section 7.17.2) so that the concentration of nitrate/nitrite-N in the OPR is one to five times the ML. A solid OPR is prepared by mixing 5 g of blank sand with 250 mL reagent water and spiking with nitrate working standard (Section 7.17.2) so that the concentration of nitrate/nitrite-N in the OPR is one to five times the ML. The spiked aliquot is carried through the entire analytical process (Section 11). Calculate accuracy as percent recovery. If the recovery of the analyte falls outside the control limits in Table 1, the system performance is unacceptable, and the source of the problem should be identified and resolved before continuing analyses.

9.5 Matrix spike and matrix spike duplicates (MS/MSD)—To assess the performance of the method on a given sample matrix, the laboratory must spike, in duplicate, a minimum of 10% (one sample in 10) of the samples from a given sampling site or, if for compliance monitoring, from a given discharge. Blanks may not be used for MS/MSD analysis.

9.5.1 The concentration of the MS and MSD shall be determined as follows:

9.5.1.1 If, as in compliance monitoring, the concentration of analytes in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit or at 1-5 times the background concentration of the sample, whichever is greater.

9.5.1.2 If the concentration of nitrate/nitrite-N in a sample is not being checked against a regulatory concentration limit, the spike shall be at 1-5 times the background concentration.

9.5.1.3 For solid and biosolids samples, the concentration added should be expressed as mg/kg and is calculated for a one gram aliquot by multiplying the added analyte concentration (mg/L) in solution by the conversion factor 100 (mg/L x 0.1L/0.001kg = 100).

9.5.2 Assessing spike recovery

9.5.2.1 To determine the background concentration, analyze one sample aliquot from each set of 10 samples from each site or discharge according to the procedure in Section 11. If the expected background concentration is known from previous experience or other knowledge, the spiking level may be established *a priori*.

9.5.2.2 Prepare the MS/MSD samples by spiking two sample aliquots with nitrate working standard (Section 7.17.2). Analyze the MS/MSD aliquots as described in Section 11 to determine the concentration of the samples after spiking.

9.5.3 Calculate the percent recovery (P) and relative percent difference (RPD) of the two matrix spike samples for the analyte, corrected for the background concentration measured in the sample, and compare these values to the control limits given in Table 1. Percent recovery is calculated in units appropriate to the matrix, using Equation 1. RPD is calculated using Equation 2.

Equation 1

$$percent\ recovery = \frac{(C_s - C_b)}{S} * 100$$

where: C_s = *Measured sample concentration after spiking*
C_b = *Measured sample background concentration*
S = *known concentration of the spike*

Equation 2

$$RPD = \frac{(|D_1 - D_2|)}{(D_1 + D_2)} * 200$$

where: D_1 = *concentration nitrate/nitrite-N of MS sample*
D_2 = *concentration nitrate/nitrite-N of MSD sample*

9.5.4 If the percent recovery or the RPD of the analyte in the MS/MSD samples falls outside the designated range, and the laboratory performance on the OPR for the analyte is within the specified limits (Section 9.4), the recovery problem encountered with the MS/MSD sample is judged to be matrix-related instead of method-related.

9.5.5 Recovery for samples should be assessed and records maintained.

9.5.5.1 After the analysis of five samples of a given matrix type (wastewater, heat-dried biosolids, etc.) for which the results pass the tests in Section 9.5.3, compute the average percent recovery (R) and the standard deviation of the percent recovery (SR) for the analyte(s). Express the accuracy assessment as a percent recovery interval from R - 2SR to R + 2SR for each matrix. For example, if R=90% and SR = 10% for five analyses of wastewater, the accuracy interval is expressed as 70-110%.

9.5.5.2 Update the accuracy assessment for each matrix regularly (e.g., after each five to ten new measurements).

9.6 Calibration verification (CV)—The laboratory must analyze a calibration verification standard before running any samples and once per ten analyses thereafter. The CV should be prepared at a concentration that is at or near the midpoint of the calibration curve. The source of the CV standard should be different from the source used to prepare the calibration standards. If a different nitrate compound is used for the CV stock, the amount weighed will have to be adjusted according to the ratio of nitrate atomic weight to the molecular weight. Results of the CV analysis should be evaluated according to the specifications in Table 1. If the CV does not meet acceptance criteria, the problem must be identified and corrected, including possible recalibration of the instrument.

9.7 Quality control sample (QCS)—It is suggested that the laboratory analyze a QCS with each day's distillations, or every twelve hours, whichever is more frequent. The results of the QCS analysis should be evaluated according to the manufacturer's specifications.

9.8 Nitrate Reduction Efficiency Standard—Prepare a nitrite standard at the same concentration as the CV nitrate standard (Section 9.6). Analyze these standards together and compare the recoveries of each. If the recovery of the nitrate standard falls below 80% of the nitrite standard's recovery, replace the cadmium reduction column with a fresh column. The cadmium granules can be reactivated by following the procedure in Section 11.1.4.

10.0 Calibration and Standardization

10.1 Calibrate the photometer with a minimum of five standards and a blank that cover the expected range of the samples. Develop a weighted linear regression formula from the calibration data using concentration versus response. If the correlation coefficient falls below 0.995, check the system for faults, correct them if found, and reanalyze the calibration standards.

10.2 Preparation of calibration curve—Table 2 gives the volume of nitrate working standard (Section 7.17.2) to make the calibration standards in 100-mL volumetric flasks. Analyze the standards according to the procedure in Section 11, beginning with the lowest concentration standard.

10.3 Balance calibration—Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights. Calibration shall be within ± 10 % at 2 mg and ± 0.5% at 1000 mg. If values are not within these limits, recalibrate the balance.

11.0 Procedure

11.1 Preparation of reduction column

11.1.1 Insert a glass wool plug in the end of the chromatographic column (Section 6.2.1). Then, close the stopcock and fill the tube with reagent water to prevent entrapment of air bubbles during filling.

11.1.2 Pour the copper-cadmium granules into the column until a column height of 18.5 cm is created. Tap the sides of the column gently to release any trapped air bubbles.

11.1.3 Wash the column with about 200 mL of dilute ammonium chloride-EDTA solution. (Section 7.14.2) Do not allow any air to enter the column.

11.1.4 Before using the column for analysis, activate by passing through the column 100 mL of a solution consisting of 25 mL of 1.0 mg/L nitrate ($NO_{3)}$ standard and 75 mL dilute ammonium chloride-EDTA solution (Section 7.14.2) at a flow rate of 7-10 mL/min.

11.2 Sample Preparation

11.2.1 Aqueous samples—Mix the sample thoroughly. Using a 0.45 µm membrane filter, filter up to 25 mL of the sample into a volumetric flask. Record the amount collected.

11.2.2 Solid samples—Thoroughly homogenize the sample. Weigh 5 g into a 500 mL Erlenmeyer flask. Add 100 mL reagent water.

11.2.2.1 Seal the flask with a stopper, secured with a small portion of PTFE thread sealing tape, and place in the shaker.

11.2.2.2 Shake the samples for 4 hours. Once the shaking is complete, remove the flasks from the assembly and allow any suspended sediment to settle out.

11.2.2.3 Using a 0.45 µm membrane filter, filter up to 25 mL of the extracted sample into a volumetric flask. Record the amount of filtered extract collected.

11.2.3 If FOG removal is necessary, adjust the pH of the extract to 2 by addition of concentrated HCl. Remove the FOG by serial extraction with hexane or a 80%/20% hexane/methyl *t*-butyl ether mixture in a separatory funnel. Proceed with filtration.

11.3 Reduction of nitrate to nitrite

11.3.1 Check the pH of the filtrates. If any are above 9 or below 5, adjust to between 5 and 9 with concentrated HCl or concentrated NH_4OH.

11.3.2 Add 75 mL of dilute ammonium chloride-EDTA solution (Section 7.14.2) to 25 mL of sample extract or sample extract diluted to 25 mL.

11.3.3 Pass the sample through the reduction column at a rate of 7-10 mL/min. Discard the first 25 mL of eluent, then collect the rest in the sample flask. Do not allow the reduced samples to sit for more than 15 minutes before analysis.

11.4 Analysis

11.4.1 Add 2 mL of color reagent (Section 7.8) to 50.0 mL of sample.

11.4.2 Allow ten minutes for the color to develop, then measure the absorbance of the sample at 540 nm. Developed color is accurate for two hours afer the reagent addition.

11.4.3 Generate a calibration curve as detailed in Section 10. Once a curve meeting the requirements in Section 10 is created, set up the samples so that every tenth sample measured is a calibration verification standard.

11.4.4 Compare the sample absorbance to the calibration curve. If the absorbance exceeds that of the highest concentration standard, use the remaining reduced filtrate to prepare a dilution of the sample extract, and re-run the diluted sample.

11.5 The solid sample dry weight/wet weight ratio must be determined separately (Appendix A).

12.0 Data Analysis and Calculations

12.1 Aqueous samples—Compare the absorbance reading for each sample to the calibration curve and determine the sample concentration using Equation 3. Report all values in mg/L to three significant figures.

12.2 Solid samples—Compare the absorbance reading for each sample to the calibration curve and determine the sample concentration using Equations 3 and 4. Report all values in mg/kg to three significant figures.

Equation 3

$$C_s = \frac{(C_{extract})(V_{sample})(R_{vol})(F_{dil})}{1000\ mL/L}$$

where: C_s *= Oxidized nitrogen in the sample (mg/L)*
$C_{extract}$ *= Concentration of oxidized nitrogen in extract, mg/L*
Vol_{sample} *= Volume of sample, mL (100 mL)*
F_{dil} *= Dilution factor of extract, if any*
R_{vol} *= ratio of initial sample volume to volume of filtrate collected (100/25, or 4 for a typical sample)*

For solid samples, oxidized nitrogen, mg/kg = the above equation multiplied by: 1000 (g/kg) / weight of biosolids extracted, g.

Equation 4

$$N\ (mg / kg) = \frac{C_s}{W}$$

where: N = *Oxidized nitrogen in solid sample (mg/kg)*
C_s = *Oxidized nitrogen in sample (Equation 3, mg/L)*
W = *Dry weight ratio (Appendix A*)

12.3 Report all results below the ML as "less than the ML."

12.4 The QC data obtained during the analysis provides an indication of the quality of the sample data and should be provided with the sample results.

13.0 Method Performance

This is a draft method, and is currently undergoing validation. Method performance criteria will be set following the validation of the method.

14.0 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, EPA recommends recycling as the next best option. The acids used in this method should be reused as practicable by purifying by electrochemical techniques. Most other chemicals used in this method are the neat materials used in preparing standards. These standards are used in extremely small amounts and pose little threat to the environment when managed properly. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Office of Society Services, 1155 16th Street NW, Washington DC 20036, 202-872-4600.

15.0 Waste Management

15.1 The laboratory is responsible for complying with all Federal, State, and local regulations governing waste management, particularly hazardous waste identification rules and land disposal restrictions, and for protecting the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. An overview of requirements can be found in *Environmental Management Guide for Small Laboratories* (EPA 233-B-98-001).

15.2 Samples containing strong acids or bases are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste. For further information on waste management,

consult, "The Waste Management Manual for Laboratory Personnel" and "Less is Better: Laboratory Chemical Management for Waste Reduction", both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington, DC 20036.

16.0 References

16.1 U.S. Environmental Protection Agency, 1979. Methods for Chemical Analysis of Water and Wastes. Publ. 600/4-79-020, rev. March 1983. Environmental Monitoring and Support Lab., U.S. Environmental Protection Agency, Cincinnati, Ohio.

16.2 U.S. Environmental Protection Agency, 1998. Method 1691: Municipal Biosolids Sampling Guidance. Draft, September 1998. Office of Water, Washington, DC.

16.3 Sax, N.I. and R.I. Lewis, Sr. Dangerous Properties of Industrial Materials, 5th. ed., Van Nostrand Reinhold, New York, 1989.

16.4 U.S. Environmental Protection Agency, 1982. Handbook for Sampling and Sample Preservation of Water and Wastewater. Publ. 600/4-82-029, Environmental Monitoring and Support Lab., U.S. Environmental Protection Agency, Cincinnati, Ohio.

16.5 Code of Federal Regulations 40, Ch. 1, Part 136, Appendix B.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Table 1. QC Sample Acceptance Criteria (to be established from validation study)

Analyte	Blank limit	IPR recovery (x) limit	IPR precision (s) limit	OPR recovery limit	CV recovery limit	QCS recovery limit
Oxidized nitrogen						

Table 2. The volume of working standard necessary to make the calibration standards in 100-mL volumetric flasks

Volume of standard (7.17.2) added, (mL)	Concentration of calibration standard (mg/L)
0.1	0.01
0.2	0.02
0.5	0.05
1.0	0.10
2.0	0.20

18.0 Glossary, Acronyms, and Abbreviations

The definitions and purposes below are specific to this method, but conform to common usage as much as possible.

18.1 **Analyte**—A compound or element tested for by the methods referenced in this method.

18.2 **Apparatus**—The sample container and other containers, filters, filter holders, labware, tubing, pipets, and other materials and devices used for sample collection or sample preparation, and that will contact samples, blanks, or analytical standards.

18.3 **Biosolids**—The treated residuals from wastewater treatment that can be used beneficially.

18.4 **Calibration Standard**—A solution prepared from a dilute mixed standard and/or stock solution and used to calibrate the response of the instrument with respect to analyte concentration.

18.5 **Calibration Verification Standard (CV)**—A solution prepared from a different source than the calibration standards that is used to confirm the accuracy of the instrument's calibration.

18.6 **Initial Precision and Recovery (IPR)**—Four aliquots of the OPR standard analyzed to establish the ability to generate acceptable precision and accuracy. IPRs are performed before a method is used for the first time and any time the method or instrumentation is modified.

18.7 **Laboratory Blank**— An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The laboratory blank is used to determine if method analytes or interferences are present in the laboratory environment, the reagents, or the apparatus (Section 9.3).

18.8 **Matrix Spike (MS) and Matrix Spike Duplicate (MSD)**—Aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample. Their purpose is to quantify the bias and precision caused by the sample matrix. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for background concentrations (Section 9.5).

18.9 **May**—This action, activity, or procedural step is optional.

18.10 **May Not**—This action, activity, or procedural step is prohibited.

18.11 **Method Detection Limit (MDL)**—The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero (Section 9.2.1).

18.12 **Minimum Level (ML)**—The lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point.

18.13 **Must**—This action, activity, or procedural step is required.

18.14 **Ongoing Precision and Recovery (OPR) Standard**—A laboratory blank spiked with known quantities of the method analytes. The OPR is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to assure that the results produced by the laboratory remain within the method-specified limits for precision and accuracy (Section 9.4).

18.15 **Oxidized Nitrogen**—The sum of nitrate- and nitrite-nitrogen.

18.16 **Reagent Water**—Water demonstrated to be free from the method analytes and potentially interfering substances at the MDL for the method.

18.17 **Reduction Efficiency Standard**—A nitrate standard analyzed to verify the completeness of reduction of the copper-cadmium column.

18.18 **Sewage Sludge**—Sewage sludge is solid, semi-solid, or liquid residue generated during the treatment process of domestic sewage in a treatment works. Sewage sludge includes but is not limited to, domestic septage; scum or solids removed in primary, secondary, or advanced wastewater treatment processes; and a material derived from sewage sludge. Sewage sludge does not include ash generated during the firing of sewage sludge in a sewage sludge incinerator or grit and screenings generated during preliminary treatment of domestic sewage in a treatment works.

18.19 **Shall**—This action, activity, or procedural step is required

18.20 **Should**—This action, activity, or procedural step is suggested but not required.

18.21 **Stock Standard Solution—A** solution containing one or more method analytes that is prepared using a reference material traceable to EPA, NIST, or a source that will attest to the purity and authenticity of the reference material.

Appendix A: Total Solids in Solids and Biosolids

1.0 Scope and Application

1.1 This procedure is applicable to the determination of total solids in such solid and semisolid samples as soils, sediments, biosolids (municipal sewage sludge) separated from water and wastewater treatment processes, and biosolids cakes from vacuum filtration, centrifugation, or other biosolids dewatering processes.

1.2 This procedure is taken from EPA Method 1684: *Total, Fixed, and Volatile Solids in Solids and Semisolid Matrices.*

1.3 Method detection limits (MDLs) and minimum levels (MLs) have not been formally established for this draft procedure. These values will be determined during the validation of Method 1684.

1.4 This procedure is performance based. The laboratory is permitted to omit any step or modify any procedure (e.g. to overcome interferences, to lower the cost of measurement), provided that all performance requirements in this procedure are met. Requirements for establishing equivalency are given in Section 9.1.2 of Method 1686.

1.5 Each laboratory that uses this procedure must demonstrate the ability to generate acceptable results using the procedure in Section 9.2 of this appendix.

2.0 Summary of Method

2.1 Sample aliquots of 25-50 g are dried at 103°C to 105°C to drive off water in the sample.

2.2 The mass of total solids in the sample is determined by comparing the mass of the sample before and after each drying step.

3.0 Definitions

3.1 Analytical batch–The set of samples analyzed at the same time, to a maximum of 10 samples. Each analytical batch of 10 or fewer samples must be accompanied by a laboratory blank, an ongoing precision and recovery sample, and a set of MS/MSD, resulting in a minimum of five analyses (1 sample, 1 blank, 1 OPR, and 2 matrix spike samples) and a maximum of 14 samples.

3.2 Total Solids–The residue left in the vessel after evaporation of liquid from a sample and subsequent drying in an oven at 103°C to 105°C.

3.3 Additional definitions are given in Sections 3.0 and 18.0 of Method 1686.

4.0 Interferences

4.1 Sampling, subsampling, and pipeting multi-phase samples may introduce serious errors (Reference 16.1). Make and keep such samples homogeneous during transfer. Use special handling to ensure sample integrity when subsampling. Mix small samples with a magnetic stirrer. If visible suspended solids are present, pipet with wide-bore pipets. If part of a sample adheres to the sample container,

intensive homogenization is required to ensure accurate results. When dried, some samples form a crust that prevents evaporation; special handling such as extended drying times are required to deal with this. Avoid using a magnetic stirrer with samples containing magnetic particles.

4.2 The temperature and time of residue drying has an important bearing on results (Reference 16.1). Problems such as weight losses due to volatilization of organic matter, and evolution of gases from heat-induced chemical decomposition, weight gains due to oxidation, and confounding factors like mechanical occlusion of water and water of crystallization depend on temperature and time of heating. It is therefore essential that samples be dried at a uniform temperature, and for no longer than specified. Each sample requires close attention to desiccation after drying. Minimize the time the desiccator is open because moist air may enter and be absorbed by the samples. Some samples may be stronger desiccants than those used in the desiccator and may take on water. If uptake of water by a sample is suspected, the operator should weigh the sample to see if it gains weight while in the desiccator. If the sample is indeed taking on water, then a vacuum desiccator should be used.

4.3 Residues dried at 103°C to 105°C may retain some bound water as water of crystallization or as water occluded in the interstices of crystals. They lose CO_2 in the conversion of bicarbonate to carbonate. The residues usually lose only slight amounts of organic matter by volatilization at this temperature. Because removal of occluded water is marginal at this temperature, attainment of constant weight may be very slow.

4.4 Results for residues high in oil or grease may be questionable because of the difficulty of drying to constant weight in a reasonable time.

4.5 The determination of total solids is subject to negative error due to loss of ammonium carbonate and volatile organic matter during the drying step at 103°C to 105°C. Carefully observe specified ignition time and temperature to control losses of volatile inorganic salts if these are a problem.

5.0 Safety

5.1 Refer to Section 5.0 of Method 1686 for safety precautions

6.0 Equipment and Supplies

Note: Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.

6.1 Evaporating Dishes–Dishes of 100-mL capacity. The dishes may be made of porcelain (90-mm diameter), platinum, or high-silica glass.

6.2 Watch glass–Capable of covering the evaporating dishes (Section 6.1).

6.3 Steam bath for evaporation of liquid samples.

6.4 Desiccator–Moisture concentration in the desiccator should be monitored by an instrumental indicator or with a color-indicator desiccant.

6.5 Drying oven–Thermostatically-controlled, capable of maintaining a uniform temperature of 103°C to 105°C throughout the drying chamber.

6.6 Analytical balance–Capable of weighing to 0.1 mg for samples having a mass up to 200 g.

6.7 Reference weights–2 mg, 1000 mg, and 50 g class "S" weights.

6.8 Container handling apparatus–Gloves, tongs, or a suitable holder for moving and handling hot containers after drying.

6.9 Sample handling apparatus–Spatulas, spoonulas, funnels, or other equipment for transfer and manipulation of samples.

6.10 Bottles–Glass or plastic bottles of a suitable size for sample collection.

6.11 Rubber gloves (Optional).

6.12 No. 7 Cork borer (Optional).

6.13 Desiccant (Optional).

7.0 Reagents and Standards

7.1 Reagent water–Deionized, distilled, or otherwise purified water.

7.2 Quality control spiking solution–If a commercially available standard can be purchased that contains standard total solids, the laboratory may use that standard. The laboratory may also prepare a spiking solution. One possible recipe is given below for a NaCl-KHP solution.

7.2.1 Dissolve 0.10 g sodium chloride (NaCl) in 500 mL reagent water. Mix to dissolve.

7.2.2 Add 0.10 g potassium hydrogen phthalate (KHP) to the NaCl solution (Section 7.2.1) and mix. If the KHP does not dissolve readily, warm the solution while mixing. Dilute to 1 L with reagent water. Store at 4°C. Assuming 100% volatility of the acid phthalate ion, this solution contains 200 mg/L total solids, 81.0 mg/L volatile solids, and 119 mg/L fixed solids.

8.0 Sample Collection, Preservation, and Storage

8.1 Use resistant-glass or plastic bottles to collect sample for solids analysis, provided that the material in suspension does not adhere to container walls. Sampling should be done in accordance with Reference 16.2. Begin analysis as soon as possible after collection because of the impracticality of preserving the sample. Refrigerate the sample at 4°C up to the time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours. Under no circumstances should the sample be held more than seven days. Bring samples to room temperature before analysis.

9.0 Quality Control

9.1 Quality control requirements and requirements for performance-based methods are given in Section 9.1 of Method 1686.

9.2 Initial demonstration of laboratory capability—The initial demonstration of laboratory capability is used to characterize laboratory performance and method detection limits.

9.2.1 Method detection limit (MDL)—The method detection limit should be established for total solids using the QC spiking solution (Section 7.2). To determine MDL values, take seven replicate aliquots of the diluted QC spiking solution and process each aliquot through each step of the analytical method. Perform all calculations and report the concentration values in the appropriate units. MDLs should be determined every year or whenever a modification to the method or analytical system is made that will affect the method detection limit.

9.2.2 Initial precision and recovery (IPR)—To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.2.1 Prepare four samples by diluting the QC spiking solution (Section 7.2) to 1 to 5 times the MDL. Using the procedures in Section 11, analyze these samples for total solids.

9.2.2.2 Using the results of the four analyses, compute the average percent recovery (x) and the standard deviation (s, Equation 1) of the percent recovery for total solids.

Equation 1

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x^2)}{n}}{n-1}}$$

Where:

n = number of samples
x = % recovery in each sample
s = standard deviation

9.2.2.3 Compare s and x with the corresponding limits for initial precision and recovery in Table 2 (to be determined in validation study). If s and x meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or x falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.

9.3 Laboratory blanks

9.3.1 Prepare and analyze a laboratory blank initially (i.e. with the tests in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample, and will consist of approximately 25 g of reagent water.

9.3.2 If material is detected in the blank at a concentration greater than the MDL (Section 1.3), analysis of samples must be halted until the source of contamination is eliminated and a new

blank shows no evidence of contamination. All samples must be associated with an uncontaminated laboratory blank before the results may be reported for regulatory compliance purposes.

9.4 Ongoing precision and recovery

9.4.1 Prepare an ongoing precision and recovery (OPR) solution identical to the IPR solution described in Section 9.2.2.1.

9.4.2 An aliquot of the OPR solution must be analyzed with each sample batch (samples started through the sample preparation process (Section 11) on the same 12-hour shift, to a maximum of 20 samples).

9.4.3 Compute the percent recovery of total solids in the OPR sample.

9.4.4 Compare the results to the limits for ongoing recovery in Table 2 (to be determined in validation study). If the results meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, the recovery of total solids falls outside of the range given, the analytical processes are not being performed properly. Correct the problem, reprepare the sample batch, and repeat the OPR test. All samples must be associated with an OPR analysis that passes acceptance criteria before the sample results can be reported for regulatory compliance purposes.

9.4.5 Add results that pass the specifications in Section 9.4.4 to IPR and previous OPR data. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory accuracy for each analyte by calculating the average percent recovery (R) and the standard deviation of percent recovery (SR). Express the accuracy as a recovery interval from R-2SR to R+2SR. For example, if R=05% and SR=5%, the accuracy is 85-115%.

9.5 Duplicate analyses

9.5.1 Ten percent of samples must be analyzed in duplicate. The duplicate analyses must be performed within the same sample batch (samples whose analysis is started within the same 12-hour period).

9.5.2 The total solids of the duplicate samples must be within 10% of the solids determination.

10.0 Calibration and Standardization

10.1 Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights.

10.2 Calibration shall be within ± 10% (i.e. ±0.2 mg) at 2 mg and ± 0.5% (i.e. ±5 mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

10.3 Place a 50 g weight and a 2 mg weight on the balance. Verify that the balance reads 50.002 ±10% (i.e. ±0.2 mg)

11.0 Procedure

11.1 Preparation of evaporating dishes–Heat dishes and watch glasses at 103°C to 105°C for 1 hour in an oven. Cool and store the dried equipment in a desiccator. Weigh each dish and watch glass prior to use (record combined weight as "W_{dish}").

11.2 Preparation of samples

11.2.1 Fluid samples–If the sample contains enough moisture to flow readily, stir to homogenize, place a 25 to 50 g sample aliquot on the prepared evaporating dish. If the sample is to be analyzed in duplicate, the mass of the two aliquots may not differ by more than 10%. Spread each sample so that it is evenly distributed over the evaporating dish. Cover each sample with a watch glass, and weigh (record weight as "W_{sample}"). Evaporate the samples to dryness on a steam bath.

Note: Weigh wet samples quickly because wet samples tend to lose weight by evaporation. Samples should be weighed immediately after aliquots are prepared.

11.2.2 Solid samples–If the sample consists of discrete pieces of solid material (dewatered biosolids, for example), take cores from each piece with a No. 7 cork borer or pulverize the entire sample coarsely on a clean surface by hand, using rubber gloves. Place a 25 to 50 g sample aliquot of the pulverized sample on the prepared evaporating dish. If the sample is to be analyzed in duplicate, the mass of the two aliquots may not differ by more than 10%. Spread each sample so that it is evenly distributed over the evaporating dish. Cover each sample with a watch glass, and weigh (record weight as "W_{sample}").

11.3 Dry the samples at 103°C to 105°C for a minimum of 12 hours, cool to balance temperature in an individual desiccator containing fresh desiccant, and weigh. Heat the residue again for 1 hour, cool it to balance temperature in a desiccator, and weigh. Repeat this heating, cooling, desiccating, and weighing procedure until the weight change is less than 5% or 50 mg, whichever is less. Record the final weight as "W_{total}."

Note: It is imperative that dried samples be weighed quickly since residues often are very hygroscopic and rapidly absorb moisture from the air. Samples must remain in the dessicator until the analyst is ready to weigh them.

12.0 Data Analysis and Calculations

12.1 Calculate the % solids or the mg solids/kg sample for total solids (Equation 2).

Equation 2

$$\%\ total\ solids = \frac{W_{total} - W_{dish}}{W_{sample} - W_{dish}} * 100$$

or

$$\frac{mg\ total\ solids}{kg\ sludge} = \frac{W_{total} - W_{dish}}{W_{sample} - W_{dish}} * 1{,}000{,}000$$

Where:

W_{dish} = *Weight of dish (mg)*
W_{sample} = *Weight of wet sample and dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*

12.2 Sample results should be reported as % solids or mg/kg to three significant figures. Report results below the ML as less than the ML, or as required by the permitting authority or in the permit.

13.0 Method Performance

13.1 Method performance (MDL and quality control acceptance criteria) will be determined during the multi-lab validation of this method.

13.2 Total solids duplicate determinations must agree within 10% to be reported for permitting purposes. If duplicate samples do not meet this criteria, the problem must be discovered and the sample must be run over.

14.0 Pollution Prevention

14.1 Pollution prevention details are given in Section 14 of Method 1686.

15.0 Waste Management

15.1 Waste management details are given in Section 15 of Method 1686.

16.0 References

16.1 "Standard Methods for the Examination of Water and Wastewater," 18th ed. and later revisions, American Public Health Association, 1015 15th Street NW, Washington, DC 20005. 2-59: Section 2540 G (Total, Fixed, and Volatile Solids in Solid and Semisolid Matrices), 1992.

16.2 U.S. Environmental Protection Agency, 1992. Control of Pathogens and Vector Attraction in Sewage Sludges. Publ 625/R-92/013. Office of Research and Development, Washington, DC.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Tables containing method requirements for QA/QC will be added after the validation study has been performed.