Appendix B

Guidelines and Format for Water, Wastewater and Process Liquid Methods Included in the NCASI Methods Manual

West Coast Regional Center
Organic Analytical Program

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Introduction

As part of the NCASI Technical Studies Program, it is often necessary to develop methods for analytical parameters for which no rigorously tested or validated methods are available. Once developed, these methods are frequently used for other research applications and to respond to regulatory and member company requests. Previously these methods have been made available to member companies in the form of technical bulletins or special reports. More recently, NCASI member companies requested that these methods be compiled into a single manual to facilitate their distribution and use.

In selecting a standardized format for water, wastewater and process liquids, consideration was given to various formats commonly used for trace environmental analytical procedures. Utilizing a standardized format familiar to analysts should facilitate the implementation of the procedures in the laboratory, workplace or in the field.

Consideration was also given to activities taking place at the Environmental Protection Agency (EPA). As part of the Agency's efforts to harmonize analytical methods, the Environmental Methods Monitoring Council (EMMC) has developed a guideline and format for preparing analytical procedures. In the Fall of 1995, EPA's Office of Water announced it's plans to "Streamline" the 304(h) approval process and indicated that it would adopt the EMMC format for this purpose. In July of 1996, the EPA published "Guidelines and Format for Methods to be Proposed at 40 CFR Part 136 or Part 141."

Given the efforts of the EPA to standardize on a single format, it was logical for NCASI to adopt those guidelines and format to the maximum extent possible consistent with NCASI standards of quality. Furthermore, adopting this format will facilitate submitting wastewater methods developed by NCASI for approval under the Streamlined 304(h) approval process in the event the industry finds that to be of benefit.

The guidelines and format presented in this document for the analysis of water, wastewater and process liquid matrices closely follow the EPA EMMC format to the extent possible. Some sections have been modified to reflect the different intent of the NCASI Methods Manual and to reflect standards of quality and scientific principals which NCASI advocates. Otherwise, efforts have been made to stay as close to the EPA format as possible.

The NCASI Methods incorporated into the Methods Manual will be revised as modifications or additional technical information become available. If changes in the EPA recommended method format occur, those changes will only be incorporated in new methods or at the time existing methods are updated.

Guidelines and Format for Water, Wastewater and Process Liquid Methods Included in the NCASI Methods Manual

1.0 Elements

1.1 Cover page

The cover page will include the number NCASI assigned to the method, the title of the method, the origin of the method (NCASI Center), the current revision number(if applicable), and the date the method was issued or the date the revision went into effect.

1.2 Title page

1.2.1 When titling the method, use a concise title that cites (in sequence) the particular analyte(s) or property being determined, the type of sample or sample matrix(ces) to which the method is applicable, as appropriate, and the determinative technique or instrumentation. Apply the following guidelines in titling methods.

1.2.1.1 If the method applies to numerous matrices (such as water, soil, sediment, sludge, tissue, and others), it may not be practical to include matrices in the title; however, if the method applies to a single matrix or a limited number of matrices, matrix(ces) should be specified in the title.

1.2.1.2 If the method is used to determine a number of analytes or properties, analytes or properties can be named as a group (e.g., trace elements), and the names of specific analytes or properties omitted.

1.2.1.3 Avoid the use of the terms "analysis of..." or "determination of..." in method titles, since these terms are understood within the context of the term "method."

1.2.1.4 Method titles may use familiar abbreviations or acronyms for determinative techniques, e.g., HRGC/HRMS.

1.2.2 Examples of suitable method titles are:

1.2.2.1 Resin and Fatty Acids in Pulp Industry Wastewaters by GC/MS

1.2.2.2 Chlorinated Phenolics in Pulp Industry Wastewaters by *In-Situ* Acetylation and GC/MS

1.3 Acknowledgments

Acknowledgments should identify the author and editor, and provide credit to researchers, peer reviewers, and organizations or individuals that contributed substantively in preparing the method. In addition, the name, address, phone and FAX numbers of the NCASI contact(s) to which questions should be directed should be included.

1.4 Disclaimer

The disclaimer may appear on the same page with the acknowledgments or may be on the page following the acknowledgments. It may contain one or more disclaimer statements. All disclaimers should include the following statement:

> The mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Statements of interest such as the following may be included:

> This method has been submitted for proposal at 40 CFR Part 136 but has not been approved for use by the U. S. Environmental Protection Agency.

For draft or proposed methods, include the following statement:

> This method is included in the NCASI Methods Manual as a DRAFT or PROPOSED method. The purpose of including DRAFT or PROPOSED methods in the NCASI Methods Manual is to make it known that a method is under development and to solicit comment regarding the technical merit and applicability of the method. This method should not be construed as having been rigorously validated on any matrix and therefore, those electing to apply the method are strongly encouraged to conduct rigorous QA/QC or validation such that the quality of the data generated can be evaluated. Please note that DRAFT or PROPOSED methods are not suitable for use as regulatory monitoring or compliance methods.

1.5 Table of contents

A table of contents is required. The table of contents should cite the titles and page numbers of all first- and second-order headings (see section 2.9, "Section headings") and tables and figures.

1.6 Introduction

In the introduction, provide background on the method, describe the purpose of the method, and include a summary-level description of the method.

1.7 Notice of performance-based method

Notice of performance-based methods is not applicable to NCASI Methods contained in the Manual.

1.8 Body of method (water and wastewater matrices)

NCASI water and wastewater methods are to contain the following sections. All sections are considered mandatory for all methods. If the description of the analytical procedure for a particular method is extensive, starting with section 11.0 Procedure, additional numbered sections may be inserted as required to allow logical division of the procedure (e.g., extraction, cleanup, concentration); however, the sections listed below must appear in each method in the order listed.

1.0 Scope and Application

2.0 Summary of Method

3.0 Definitions

4.0 Interferences

5.0 Safety

6.0 Equipment and Supplies

7.0 Reagents and Standards

8.0 Sample Collection Preservation and Storage

9.0 Quality Control

10.0 Calibration and Standardization

11.0 Procedure

12.0 Data Analysis and Calculations

13.0 Method Performance

14.0 Pollution Prevention

15.0 Waste Management

16.0 References

17.0 Tables, Diagrams, Flowcharts, and Validation Data

The required content for each of these sections is described in section 4.0 of this document.

2.0 Format

2.1 Page numbering

Page numbers should appear in the bottom center of the page. For methods that are prepared double-sided, page numbers may appear on the outside bottom corner of the page (i.e., on the bottom right for right-hand pages and on the bottom left for left-hand pages).

2.1.1 Numbering front matter–Number the front matter (i.e., everything preceding the body of the method) consecutively using lower-case Roman numerals. The numerals should appear on the bottom of each page of the front matter, except for the cover and title pages. The cover page is unnumbered. The title page holds the place of page i but the numeral is not displayed.

2.1.2 Numbering body of method–Number the body of the method consecutively with Arabic numerals on the bottom of each page, starting with the number 1.

2.3 Method identification

2.2.1 The method introduction page(s) should contain a header that identifies the method number and revision number or letter. The first page of the body of the method should start with the method number and title in the top center of the page (preceding 1.0 Scope and Application), with no header, as shown in Appendix A. Each pursuant page of the method should contain a header that identifies the method number and revision number or letter. The header must also be separated from the main body of the method by a horizontal line running the width of the page.

2.2.2 In the event that NCASI methods are submitted to the EPA for proposal at 40 CFR Part 136, and are assigned an EPA number, the method manual will reflect both the former NCASI number or designation and the newly assigned EPA number. When preparing the method for submission to EPA for proposal at 40 CFR Part 136, edit the header to reflect the method number assigned by EPA (i.e., Method 1664). Append the EPA number to the NCASI number in the method manual.

2.3 Method date

The method status and date of the method (month and year) should appear on the bottom of each page of the method.

2.4 Font

For text, use a 12-point Times New Roman font (typeface). For first-order headings, use a bold, 14-point font. For section numbering, use a bold, 12-point font. Times Roman fonts may be used in tables as appropriate.

2.5 Margins

Left and right margins should be one inch. The header should be 0.5 inch from the top of the page, with the text starting one inch from the top of the page. The page number should appear 0.5 inch from the bottom of the page, with the text starting one inch from the bottom of the page.

2.6 Justification

Use left justification for text. This results in a ragged-right margin.

2.7 Line spacing

The method should be single-spaced. (If preferred, 1.1 line spacing can be used to enhance readability.) One blank line should appear between each paragraph and section.

2.8 Method sections

Each method must contain sections 1.0 through 17.0, as identified in section 4.0, "Body of Method." Section 4.0 contains a detailed description of this format. If a section does not apply to a particular method, include the section with a statement that it is not applicable in that method.

2.9 Section headings and numbering

Use the Modified Decimal Numbering (MDN) system to organize material presented in methods and methods manuals. In this system, each method section and subsection is assigned a unique number that shows the relationship of a specific section/subsection to all previous sections/subsections and allows for easy reference. This numbering system is used in this document.

The first-order headings are the 17 sections identified in section 1.7, "Body of Method," starting with 1.0 Scope and Application. First-order headings must appear on a separate line, with a blank line appearing between the heading and the section text. Subsections are numbered and may or may not have a heading preceding the text. Second-order headings or sections are numbered 1.1, 1.2, 1.3, 1.4, etc. Third-order headings or sections are numbered 1.1.1, 1.1.2, 1.1.3, etc. Fourth-order headings or sections are numbered 1.1.1.1, 1.1.1.2, 1.1.1.3, etc.

Do not number beyond the fourth-order heading or section. If additional subdivisions are necessary, use (a), (b), (c), etc. to identify further divisions. Use of subdivisions below

the fourth-order heading or section should be avoided when possible by organizing the material differently.

2.10 Indentation

First-order headings should appear flush left. Each subsequent order heading should be block-indented to align with the text of the previous order heading. This indentation method is illustrated in this document.

2.11 Electronic submission

Methods to be incorporated into the methods manuals should be prepared and submitted in both hard copy and electronic formats. Electronic methods must be submitted in machine-readable format, Microsoft Word.

2.12 References

Use the following format for order, content, and punctuation when listing references.

2.12.1 Books–author's name or names (initials last), title of book (underline, period, no quotation marks), name of publisher, address of publisher (city and state), year of publication, and page number, if applicable

2.12.2 Magazines and Journals–author's name or names (initials last), "title of paper" (quotation marks, comma), volume number, issue number (this may be omitted if the journal page numbers are continuous throughout the volume), date of publications, and page numbers. Example: Jones, J.J., and Smith, R.R., "Correlation of Brinell Hardness and Tensile Strength, *Materials in Design Engineering*," Vol. 10, No. 2, February 1958, pp. 52-67

2.12.3 Proceedings, Transactions, Reports, Bulletins, etc.–author's name or names (initials last), "title of paper" (in quotation marks), name of publication (underline, no quotation marks, comma), name of publisher, volume number, if any date of publication, and page numbers.

2.12.4 Symposium Volumes or Other Books Comprising Collections of Papers–Follow style for books, above and add title of paper, in quotes, after author's name. Patents–patent number and data.

2.12.5 EPA methods–Method number and name, EPA report number, U.S. Environmental Protection Agency, laboratory and/or office, location, date.

3.0 Conventions

3.1 Capitalization, italics, underlining, and boldface

3.1.1 Capitalization

3.1.1.1 For first-order headings (numbered 1.0, 2.0, 3.0, etc.), use initial capitalization of major words.

3.1.1.2 For second-, third-, or fourth-order headings, capitalize the first word of the heading only.

3.1.2 Italics–Italicize words or blocks of text for emphasis. Equations and notes interspersed in the text also should be italicized.

3.1.3 Underlining–Underline words that are defined in the definitions section (or glossary). Use underlining in tables as appropriate for clear presentation of material. Do not use underlining for emphasis; use italics instead.

3.1.4 Boldface–Boldface the following items:

The method number and title on the cover page, title page, and page 1 of the method

Acknowledgments, Disclaimer, and Introduction headings

First-order headings

Section numbering

Equation numbers

The word "Note:" preceding text notes.

3.2 Punctuation

3.2.1 Always use a comma after the second to last entry in a series.

3.2.2 A dash may be used between a subheading and text that directly follows the subheading. There should be no blank space before or after the dash, e.g., "Matrix Spikes–The laboratory must spike..."

3.2.3 As a general rule, use a hyphen in compound modifiers to avoid ambiguity, e.g., 1-L flask. (In some cases, the hyphen can be left out without ambiguity, e.g., toxic chemical waste.) Do not use a hyphen after an adverb ending in "ly," e.g., commonly accepted practice.

3.2.4 Bullets are not to be used in the body of the method. If used in introductory material, the text following the bullet should start with a capital letter. Short

bullets do not require periods at the end; long (multiple-line) bullets do. Semicolons or commas should not be used after bulleted text.

3.3 Footnotes

Use footnotes only in tables. Footnotes should be designated with lower case letters in superscript, and should appear below the body of the table.

3.4 Text notes

Notes may be used within the text to highlight important information regarding use of the method. Use a margin-to-margin line across the page both preceding and following the note to set it off from the text.

3.5 Equations

Equations should be numbered Equation 1, Equation 2, etc., consecutively as they appear in the text. Use a margin-to-margin line across the page both preceding and following the equation to set it off from the text. Equations should be presented in italics. The equation is followed by "where:" and a list of terms used in the equation (e.g., n = number of samples, x = concentration in each sample).

3.6 Tables and Figures

Tables and figures appear in Section 17.0.

3.6.1 Number tables and figures consecutively with Arabic numerals, and give each a title that is complete and descriptive.

3.6.2 In table column headings, specify the quantity being tabulated, followed by the units of measurement shown in parentheses. For example, "Amount spiked (µg/L)."

3.6.3 Place table and figure titles above the information presented.

3.6.4 Figures may be enclosed in a box if desired.

3.7 Trademarks

3.7.1 Avoid the use of trademarks or brand names whenever possible. For example, use the term "borosilicate glass" rather than the trademarks Pyrex™ or Kimax™. (See Section 4.6.3.)

3.7.2 When a trademark or brand name is used, capitalize it.

3.8 Text references

Text references are references to other locations within the method at hand, not references to any outside source. References to other sources appear in Section 16.0. Do not incorporate essential information into the method by referring to another method.

In the method text, refer to other sections of the method capitalizing the word "Section." Section references should appear in parentheses at the end of the phrase or sentence to which the reference applies, for example, (Section 9.6).

3.9 Units, symbols, abbreviations, and acronyms

3.9.1 Units and symbols from the international metric system (SI, from the French name, Le Systeme International d Unites) are to be used. SI is based on seven basic units that are dimensionally independent. The SI unit of time is the second (symbol = s) which should be used if practical. The SI unit of volume is the cubic meter (symbol = m) but the spectral name liter (symbol = L) can be used for liquids and gases. Although the SI unit for mass is kilogram (symbol = kg), the use of gram (g) with or without prefixes is appropriate.

3.9.2 Symbols, not abbreviations, should be used for units. Symbols are not followed by a period except when used at the end of a sentence. Except for the symbol for liter (L), unit symbols are written in lower case, unless the unit name was derived from a proper name, such as Pa, from Pascal. When a quantity is expressed as a numerical value and a unit symbol, a space should be left between them, except between the number and symbol for degree Celsius (20°C) and for degree, minute, and second of plane angle.

3.9.3 Use commonly accepted abbreviations in tables as appropriate to conserve space. Abbreviations should not be used in text.

3.9.4 Use commonly accepted acronyms in text and tables. (In many cases, an acronym is more readily identifiable than its narrative counterpart.) Always spell out the term the first time it is used and follow it with the acronym shown in parentheses, e.g., material safety data sheet (MSDS), relative percent difference (RPD), or United States Environmental Protection Agency (USEPA). Acronyms have no periods or spaces between letters. As depicted in these examples, although the acronym is capitalized, the narrative version of it is not capitalized unless it is a proper name such as a government agency, society, or association. Once an acronym is introduced in this manner, use only the acronym subsequently.

3.9.5 When a long word or phrase for which there is no standard acronym is used frequently, it may be replaced by an acronym that is explained when it first occurs. For example, relative centrifugal force (rcf).

3.9.6 A list of common symbols, abbreviations, and acronyms is included in Appendix A to this document.

3.10 Numerals

3.10.1 Spell out single-digit numbers (one through nine), with the following exceptions:

3.10.1.1 Use numerals when the quantity is partly fractional, e.g., 1.15, 1- 1/2.

3.10.1.2 Use numerals when the number is followed by a unit symbol, e.g., 1 m, 9%, 3 ppm. (In the method text, units should be spelled out, so the numbers one through nine associated with the units would be spelled out also, e.g., one meter, nine percent, three parts per million.)

3.10.1.3 Use numerals to identify equations and tables, e.g., Equation 2, Table 5.

3.10.1.4 In statements containing multiple numbers, if some numbers must be numerals, use numerals for all (e.g., 2 tests and 16 weighings).

3.10.2 Use numerals for multiple-digit numbers (10 and above), with the following exceptions:

3.10.2.1 Do not begin a sentence with a numeral. When the numeral is spelled out, also spell out the unit following (e.g., One gram is usually sufficient.)

3.10.2.2 Spell out round numbers that are used in an indefinite sense (e.g., a hundred feet or so).

3.10.3 When a number is used as an adjective, insert a hyphen between the number and the unit symbol (e.g., 100-mL volumetric flask, 1-L sample).

3.10.4 When writing decimal numbers of value less than one, place a zero before the decimal point (e.g., 0.45 g).

3.10.5 Do not point-off numbers of four figures (1234) except in tables when they occur in a column containing numbers of more than four figures. Point-off numbers of more than four figures, using commas with no spaces (e.g., 1,325,000).

3.10.6 In expressing ranges and ratios in text, use 1 to 10 or 1:10, not 1-10. A hyphen may be used for ranges in tables.

3.11 Significant Digits

Handle numbers with careful regard for correspondence between the data accuracy and the given number of digits. The number of significant digits should neither sacrifice nor exaggerate accuracy.

3.11.1 Any digit that is necessary to define the specific value or quantity is significant. For example, when measured to the nearest 1 m, a distance may be 157 m, which has three significant figures; when measured to the nearest 0.1 m, the distance may be 157.4 m, which has four significant figures.

3.11.2 When adding or subtracting numbers with different degrees of precision, the answer should contain no significant digits farther to the right than the least precise number. Numbers should first be rounded to one significant digit farther to the right than that of the least precise number. The answer is then rounded to the same number of significant figures as the least precise number.

3.11.3 For multiplication and division, the product or quotient should contain no more significant digits than are contained in the number with the fewest significant digits.

3.11.4 Examples to distinguish the addition/subtraction and multiplication/division rules are:

Addition:
113.2 + 1.43 = 114.63, rounded to 114.6

Subtraction:
113.1 - 1.43 = 111.77, rounded to 111.8

Multiplication:
113.2 x 1.43 = 161.876, (rounded to 162)

Division:
113.1 / 1.43 = 79.16, rounded to 79.2

Note: *The product and quotient above should contain only three significant digits because the number 1.43 contains only three significant digits. The above sum and difference, however, contain four significant figures, because digits that occur to the right of the last significant in the least precise number are rounded.*

3.12 Order of magnitude

Zeros may be used to indicate a specific value or to indicate the order of magnitude of a number. For example, in the number 203,185,000, representing population rounded to

thousands, the first six digits are significant. The last three digits are zeros that indicate the order of magnitude.

3.13 Rounding

3.13.1 When the first digit discarded is less than five, the last digit retained is not changed.

3.13.2 When the first digit discarded is five or greater, or when five is followed by a digit other than zero, the last digit retained is increased by one.

3.13.3 When the first digit discarded is exactly five followed only by zeros, the last digit retained is rounded upward if it is an odd number and is not adjusted if it is an even number.

4.0 Content (Water and Wastewater Methods)

In accordance with NCASI format, each analytical method for water and wastewater must contain all of the following sections in a designated order, as shown.

1.0 Scope and Application
2.0 Summary of Method
3.0 Definitions
4.0 Interferences
5.0 Safety
6.0 Equipment and Supplies
7.0 Reagents and Standards
8.0 Sample Collection Preservation and Storage
9.0 Quality Control
10.0 Calibration and Standardization
11.0 Procedure
12.0 Data Analysis and Calculations
13.0 Method Performance
14.0 Pollution Prevention
15.0 Waste Management
16.0 References
17.0 Tables, Diagrams, Flowcharts, and Validation Data

Starting with Section 11.0 Procedure, additional numbered sections may be inserted as required by the particular method.

Note: *Subsections within each of the 17 required sections do not need to correlate directly to the subsections included here. In other words, the information mentioned in 4.1.1 below might be covered in two or more subsections in a method.*

4.1 Scope and application

This section outlines the purpose, range, limitations, and intended use of the method, and identifies target analytes.

4.1.1 Define the purpose and intended use of the method. State what the method is based upon, noting any relationship of the method to other existing analytical methods. Indicate whether the method is associated with a sampling method.

4.1.2 List analytes that can be measured by the method, including each analyte's Chemical Abstracts Service Registry Number (CASRN). For pesticides, use "acceptable common names." The use of registered trade names is permitted.

4.1.3 Identify the matrix(ces) for which the method has been found satisfactory.

4.1.4 Indicate the level of validation to which the method has been subjected and the current status of the method.

Validation Parameters	Status
Interlaboratory Validated	Accepted Method
Peer Validated	Draft Method
Single lab validated	Proposed Method

4.1.5 Indicate the statistically determined method detection limit (MDL) and the analyte concentration range over which the method is applicable. State in what matrix(ces) the MDL was determined. If the MDL is not available, report an instrumental detection limit and define how it was derived. Indicate the minimum level (ML) and water quality criteria if appropriate to the analyte and method.

4.1.6 Describe method limitations, such as "This method is not applicable to saline water," or "This method is not intended for determination of metals at concentrations normally found in treated and untreated discharges from industrial facilities." Indicate any means of recognizing cases where the method may not be applicable to the sample under test.

4.1.7 List any restrictions that may apply, such as "This method is restricted to use by or under the supervision of analysts experienced in ..."

4.2 Summary of Method

This section provides an overview of the method procedure and quality assurance.

4.2.1 Outline, specifying amounts of sample and reagent, the procedure that is followed to determine the presence or absence of the listed analytes. Include any sample pretreatment, such as filtration or digestion. In this description, identify the basic steps involved in performing the method, but omit the details that are a necessary part of the complete statement of procedure.

4.2.1.1 For chemical methods, state the type of procedure (colorimetric, electrometric, volumetric, etc.) and describe the source of color, major chemical reaction, including pertinent chemical equations, etc. For instrumental methods, state the technique.

4.2.1.2 Use the passive voice, e.g., "Instrumental drift is corrected for by using internal standardization," rather than "Correct instrumental drift by using internal standardization."

4.2.2 Identify the determinative step in the method.

4.2.3 State in a summary fashion how quality is assured in the method.

4.2.4 List options to the method, if applicable.

4.3 Definitions

This section includes definitions of terms, acronyms, and abbreviations used in the method. If preferred, definitions may be provided in a glossary at the end of the method or manual. In this case, the Definitions section must still appear in the method, with a notation that definitions are provided in a glossary at the end of the method. Refer to the specific section number of the glossary.

4.3.1 Include an introductory statement as follows:

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

4.3.2 List units of weight and measure and their abbreviations or acronyms used in the method.

4.3.3 Alphabetically list and define terms, acronyms, and abbreviations used in the method. Where appropriate, include the purpose (e.g., The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample).

4.3.4 Include definitions of the terms *may, may not, must*, and *should*, as follows:

May–This action, activity, or procedural step is neither required nor prohibited.

Must not–This action, activity, or procedural step is prohibited.

Must–This action, activity, or procedural step is required.

Should–This action, activity, or procedural step is suggested but not required.

4.4 Interferences

This section identifies known or potential problems that may interfere during use of the method, and describes ways to reduce or eliminate interferences.

4.4.1 Describe any known or potential problem(s) (e.g., sample or equipment contamination, instrument noise) that may be encountered during the performance of the method and the source of the problem(s). Recommend techniques to avoid or minimize the problem(s) (e.g., ways to reduce sample or equipment contamination, or instrument noise).

4.4.2 Identify any substances, ions, or properties that are known to or likely to cause interference and the amounts that are known to or likely to interfere. Sometimes, this information can be obtained only by observation during the analysis. In such cases, include appropriate notes under "Procedure" or "Data Analysis and Calculations."

4.5 Safety

This section describes special precautions needed to ensure personnel safety during the performance of the method. Procedures described here should be limited to those which are above and beyond good laboratory practices. The section must contain information regarding specific toxicity of analytes or reagents.

4.5.1 Identify and warn analysts of potential hazards associated with using the method (e.g., toxicity or carcinogenicity of analytes or reagents, explosions, fire, radiation). Recommend techniques to minimize hazards where possible (e.g., performing operations in a hood or glove box).

4.5.2 Where the toxicity or carcinogenicity of each compound or reagent has not been precisely determined, include the following statement:

> The toxicity or carcinogenicity of each analyte or reagent has not been precisely determined; however, each chemical should be treated as a potential health hazard. It is recommended that prudent practices for handling laboratory chemicals, such as minimizing exposure, be employed.

4.5.3 Indicate the steps in the procedure at which hazards that could damage equipment may occur by use of the word CAUTION in bold face type,

followed by the details of the precautionary measures that must be taken. If any step in the procedure could result in personal injury or death, include the word WARNING in bold face type, followed by the details of the protective measures that must be taken.

4.5.4 Include the following statements:

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and 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 (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References ____.

4.6 Equipment and supplies

This section lists and describes all non-consumable supplies and equipment needed to perform the method.

4.6.1 Include the following statement as a note preceding the list of 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.*

4.6.2 Categorize and list required equipment and supplies by function (e.g., sampling equipment, equipment for glassware cleaning, equipment for calibration, equipment for sample extraction, etc.). Do not list common laboratory equipment, but do include special or modified forms of unusual sizes or numbers of common equipment that are required or that may require special preparation.

4.6.3 Describe the essential features of each required item. Include schematic drawings as needed to clarify or supplement apparatus descriptions.

4.6.4 Avoid the use of trademarks, brand names, trade names, or suppliers unless a specific manufacturer's product is required for a well-defined reason or the availability of the product is limited (e.g., the apparatus is unique or unusual). For example, when special types of glassware are required, such as heat-resistant, chemical-resistant, etc., state the significant characteristic desired rather than a trademark ("borosilicate glass" rather than Pyrex™ or Kimax™). If only a single source is known, that supplier may be identified.

4.6.5 Whenever a brand name is used, include "or equivalent" following the brand name to demonstrate that another product can be used.

4.6.6 Include any special glassware cleaning instructions.

4.6.7 List special facilities required, such as a special room for handling hazardous materials.

4.7 Reagents and standards

This section lists and describes all reagents and standards required to perform the method, and provides preparation instructions and/or suggested suppliers as appropriate.

4.7.1 List the name of the reagent and the necessary purity, followed by any descriptive terms. List reagents in a logical order (e.g., by order of occurrence or use, by group). The method should require that reagents be ACS Reagent Grade unless otherwise specified.

4.7.2 Spell out the full name of inorganic reagents when first used, and include within parentheses the exact chemical formula, showing its water of crystallization, etc. Subsequently, refer to inorganic compounds by formula if they can be specified clearly in this way. As exceptions, always spell out the word "water" and the names of substances in their elemental state (e.g., "lead" not "Pb," "oxygen" not "0_2").

4.7.3 Spell out organic, organometallic, or complex inorganic compounds. Chemical formulae are not necessary. Cite the CASRN to avoid ambiguity.

4.7.4 Avoid the use of trademarks and names of patented products. Use chemical names only, unless a specific product is required for a well-defined reason. The use of registered trade names is permitted.

4.7.5 Unique and unusual reagents can be named by brand. Whenever a brand name is used, include "or equivalent" following the brand name to demonstrate that another product can be used.

4.7.6 Specify the concentration of inorganic reagents in applicable terms, as follows:

Concentrated acids and bases	density
Dilute acids and bases	volume ratio, x + y (x volumes of reagent added to y volumes by water)
Nonstandardized solutions	normality, expressed decimally; or the equivalent of 1 mL of solution in terms of grams of a given element expressed as 1 mL = x.xx g of ...

4.7.7 Specify filter paper by describing the significant characteristic such as porosity, rate of filtering ash content, etc., or by reference to ASTM Specification D1100 for Filter Paper for Use in Chemical Analysis.

4.8 Sample collection, preservation, and storage

This section provides requirements and instructions for collecting, preserving, and storing samples.

4.8.1 Give detailed directions for collecting, filtering (if applicable), preserving, shipping, and storing samples.

4.8.2 Specify preservation and holding times with references to how they were established experimentally. If experimental data is not available provide guidelines. Guidelines should be consistent with preservation procedures and holding times specified in current EPA publications or regulations and with other methods for the same analytes if the method is to be submitted for promulgation as an EPA method.

4.9 Quality control

This section cites the procedures and analyses required to fully document the quality of data generated by the method. The required components of the field or laboratory quality assurance (QA) program and specific quality control (QC) analyses are described in this section. For each QC analysis, the complete analytical procedure, the frequency of required analyses, and interpretation of results are specified.

4.9.1 Include the following statements in the first subsection (Section 9.1):

> Each field sampling program or laboratory that uses this method is required to operate a formal quality assurance program (Reference ____). Laboratory or field performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

4.9.2 In the remainder of Section 9.1, outline the QC requirements that will be described in the section, and the purpose for each type of QC (e.g., blanks, matrix spikes/matrix spike duplicates, calibration verification).

4.9.3 Describe the procedure for matrix spikes, calculating percent recoveries, and calculating relative percent difference for duplicates.

4.9.4 Provide instructions for analysis of blanks, e.g., laboratory reagent blanks, method blanks.

4.9.5 Specify requirements for calibration verification.

4.9.6 Provide instructions for analysis of required precision and recovery standards QC standards.

4.9.7 Include requirements (or desirability) for analysis of quality control samples (QCS).

4.9.8 Include the following statement at the end of Section 9.0.

> Depending upon specific program requirements, field replicates and field spikes of the analytes of interest into samples may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

4.10 Calibration and standardization

This section describes the initial method/instrument calibration and standardization process, and required calibration verification. Corrective actions are described for cases when performance specifications are not met.

4.10.1 Specify operating conditions or refer to manufacturer's recommended operating conditions. If appropriate, specify a precalibration routine as needed to document instrument stability.

4.10.2 Give detailed instructions for the use of standards to prepare calibration curves or tables. Include the number of calibration standards, the need for blanks, the frequency of calibration checks, the critical range, etc.

4.10.3 Give detailed instructions for internal standardization, including number and concentration of internal standards.

4.10.4 Include instructions for calibration data storage.

4.11 Procedure

This section describes the sample processing and instrumental analysis steps of the method, and provides detailed instructions to analysts.

4.11.1 For methods used for determination of a method-defined analyte, include the following statement in the introductory portion of Section 11.0 Procedure.

> This method is entirely empirical. Precise and accurate results can be obtained only by strict adherence to all details.

Do not include this statement in methods for which the analyte is a chemical or physical parameter, the characteristics of which are known (e.g., oil and grease, COD, BOD).

4.11.2 Include in proper sequence detailed directions for performing the analysis.

4.11.2.1 Include steps that are essential to the process and avoid unnecessarily restrictive instructions.

4.11.2.2 Organize the procedure by type of activity, e.g., sample preparation, extraction, analysis.

4.11.2.3 Describe the procedure in the imperative mood, present tense, e.g., "Heat the sample aliquot," rather than "The sample aliquot should be heated." Comments and descriptive information that are not in the imperative mood may be included, as appropriate.

4.11.2.4 Write the text so that it is concise and easily understandable.

4.11.2.5 When alternative procedures are given, state which is preferred.

4.11.3 In chemical methods, specify the size of sample aliquot and indicate the required measurement accuracy. (There is no need to weigh a sample to five significant figures in a spectrophotometric method where the final absorbance measurement yields data with only three significant figures.)

4.11.4 Include "Notes" throughout the procedure to highlight critical points. Include notes of "WARNING" or "CAUTION" as appropriate to identify known or potential hazards to the analyst or equipment, respectively.

4.11.5 Indicate steps in which timing is critical, e.g., if a determination may not be interrupted overnight. For a color reaction, indicate how long the color is stable.

4.12 Data Analysis and calculations

This section provides instructions for analyzing data, and equations and definitions of constants used to calculate final sample analysis results.

4.12.1 Calculations–Provide directions for calculating the results of the analysis, including any equations.

4.12.1.1 Use the imperative mood, e.g., "Report results to three significant figures," rather than "Results should be reported to three significant figures."

4.12.1.2 Spell out names in the text (e.g., Total Kjeldahl Nitrogen) but use the abbreviations (e.g., TKN) in equations.

4.12.1.3 Define the symbols used in the equation immediately under the equation.

4.12.1.4 Use numerical values for any constants. Identify dilution factors, titration factors, etc.

4.12.2 Reporting results

4.12.2.1 Indicate the units in which the results are to be reported, i.e., mg/L, mg/kg, etc.

4.12.2.2 If the sample is a solid material such as a sediment or sludge, indicate whether results are to be reported as wet weight or dry weight.

4.12.2.3 Specify the number of significant figures to be reported.

4.12.2.4 Require that all values obtained by various QC procedures are reported along with the calculated results of the analysis.

4.12.3 Interpretation of results−Use this heading in place of Calculations when the results of the analysis must be expressed in descriptive form, relative terms, or abstract values. List and define the descriptive terms or classifications used.

4.13 Method performance

This section provides method performance criteria for the method, including precision/bias statements regarding detection limits and source/limitations of data produced using the method.

4.13.1 Explain how the method was validated. Provide a detailed description of method performance, including data on precision, bias, detection limits (including the method by which they were determined and matrices to which they apply), and statistical procedures used to develop performance specifications.

***Note**: This information can be provided through reference to the method validation study or NCASI Technical Bulletins.*

4.13.2 At a minimum, state single-operator precision and accuracy on reagent water. If other sample types have been investigated, also provide this information for them.

4.13.3 If a collaborative study has been completed, describe it and report the number of participating operators and laboratories, spike concentrations, level of replication, types of background waters, and any other significant aspects. If the study has been documented, cite the study report and include it in the References section. When citing reference documentation, the details of the study do not have to be included in this section.

4.14 Pollution prevention

This section describes aspects of the method that minimize or prevent pollution known to be or potentially attributable to the method.

4.14.1 Cite potential sources of pollution attributable to the method.

4.14.2 Recommend ways to minimize pollution.

4.15 Waste management

This section describes minimization and proper disposal of waste and samples.

4.15.1 Include the following statement as the first subsection:

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions

4.15.2 Provide instructions for sample and waste handling and disposal.

4.15.3 Include the following statement as the last subsection:

For further information on waste management, the EPA recommends consulting "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.

4.16 References

This section lists references for source documents and publications that contain ancillary information.

Note: *Each method should be a free-standing document, in which all information necessary for the method user to perform the method may be found. References within a method should be restricted to associated or source material. Procedural steps or instructions should not be referenced as being found elsewhere, but should be included in total within the method.*

4.16.1 Include references for other, related NCASI or EPA methods; and published studies/articles relating to method performance, techniques, or analytes, and health and safety.

4.16.2 List references in the order cited in the method, and assign each reference an identification number using Arabic numerals.

4.16.3 As a rule, do not list documents that are not readily accessible to the reader (e.g., unpublished theses, personal communications, private correspondence). If it is important to list these types of documents, identify where the reader may obtain a copy of the document.

4.17 Tables, diagrams, flowcharts, and validation data

This section contains all method tables and figures (diagrams and flowcharts), and may contain validation data referenced in the body of the method.

4.17.1 In addition to tables and figures, include additional useful information. Examples of such information include:

4.17.1.1 Notes on significance and interpretation of the method, used to amplify the statement in the text.

4.17.1.2 Development of equations used in the calculations.

4.17.1.3 Charts or supplementary information for computations.

4.18 Glossary

This optional section contains a glossary of terms, acronyms, abbreviations, and symbols used in the method. This information may appear in the Definitions section of the method (Section 3.0) or may be included in a glossary at the end of the method.

4.18.1 In the first subsection of the glossary, identify units of weight and measure used in the method and their abbreviations.

4.18.2 In the second subsection, define key terms and all acronyms used in the method.

4.18.2.1 List terms and acronyms alphabetically.

4.18.2.2 Definitions should appear only once. Where an acronym represents a term that is defined under its full name, spell out the acronym and ask the reader to look up the term for the definition.

Appendix A of Appendix B
Standard Acronyms, Abbreviations, and Symbols

ampere	amp
average	avg
calibration standard	CAL
centimeter	cm
cubic centimeter	cm^3
day	do not abbreviate
degree Celsius	^oC
diameter	dia
equation	Eq
figure	Fig. (only when followed by a numeral)
foot	ft
gallon	gal
gas chromatograph/chromatography	GC
gel permeation chromatograph/chromatography	GPC
gram	g
grams per Liter	g/L
greater than	>
high performance liquid chromatograph/chromatography	HPLC
high resolution GC	HRGC
high resolution MS	HRMS
hour	h
hydrogen ion concentration, negative logarithm of	pH
inch	in.
initial precision and recovery	IPR
inside diameter	ID
instrument detection limit	IDL
Kuderna-Danish concentrator	KD

kilogram	kg
laboratory control sample	LCS
laboratory duplicate	LD
laboratory fortified blank	LFB
laboratory fortified sample matrix	LFM
laboratory reagent blank	LRB
less than	<
linear dynamic range	LDR
liter	L
logarithm (common)	log
logarithm (natural)	log e or ln
mass spectrometer/spectrometry	MS
mass to charge ratio	m/z
matrix spike	MS
matrix spike duplicate	MSD
maximum	max
method detection limit	MDL
meter	m
microgram	ug
microgram per liter	ug/L
microliter	uL
milliequivalent	meq
milligram	mg
milligram per gram	mg/g
milligram per liter	mg/L
milligr am per milliliter	mg/mL
milliliter	mL
millimeter	mm
millimeter of mercury (pressure)	mm Hg
millisecond	ms
millivolt	mV

minimum	min
minimum level	ML
minute	min
molal	do not abbreviate
molar	M
molecular ion	M
mole	mol
month	do not abbreviate
most probable number	MPN
nanogram	ng
nanogram per liter	ng/L
nanometer	nm
normal	N
number	No. (only when followed by a numeral)
ongoing precision and recovery	OPR
ortho	o
outside diameter	OD
page	p.
pages	pp.
para	p
part per billion	ppb
part per million	ppm
part per quadrillion	ppq
part per trillion	ppt
per	/ (when used in expressions with unit symbols)
percent	%
pico (prefix)	p
picogram	pg
pound	lb
pounds-per-square inch gauge	psig
precision and recovery	PAR
quality assurance	QA

quality control	QC
quality control sample	QCS
quart	qt
reference	Ref
relative centrifugal force	RCF
relative percent difference	RPD
relative response	RR
relative standard deviation	RSD
response factor	RF
revolutions per minute	rpm
second	s
selected ion current profile	SICP
solid phase extraction	SPE
Soxhlet/Dean-Stark extractor	SDS
specific gravity	sp gr
micrometer	um
volt	V
volume (of a publication)	Vol. (only when followed by a numeral)
volume per unit volume	v/v
watt	W
weight per unit volume	w/v
year	do not abbreviate