SW-846 METHOD STYLE GUIDE
June 25, 2012

I. PURPOSE OF THIS STYLE GUIDE

This style guide is for use by developers of new methods for SW-846 and editors of existing SW-846 methods. Its use will help assure consistent method format and minimize editorial errors during the development and maintenance of SW-846. The guidelines will also help assure high quality conversions to PDFs during method preparation for electronic distribution (e.g., via the Internet).

This guide begins with a discussion of general method format and style guidelines, followed by specific directions for each section of an SW-846 method.

II. GENERAL METHOD FORMAT, CONTENT, AND STYLE GUIDELINES

The instructions to follow are guidelines -- not requirements. Do not follow a guideline if a method-specific situation dictates that a different approach will benefit method use. However, always follow these guidelines in the absence of such a situation or when in doubt about the most appropriate approach.

1. Section content and numbering

- Always follow the section numbering and content guidelines found in Sec. III of this style guide.

- Strive to present typical section information in the same order and level of detail for all methods, as available and applicable. Be especially consistent between methods involving the same analytical technologies.

- Avoid orphaned subsection numbers. For instance, do not precede a paragraph with "3.1.1" if a Sec. 3.1.2 does not follow.

- Avoid exceeding four levels of section numbers (e.g., avoid 9.3.3.3.5) by considering other approaches to organizing and presenting the information.

2. Grammar, style, and usage

- Except when otherwise specified in this guide, follow the American Chemical Society's (ACS) Style Guide with regard to grammar, style, and usage. The style adopted by ACS is for the most part taken from established authoritative sources, such as the GPO Style Manual and The Chicago Manual of Style; and in addition deals specifically with style and usage related to chemistry.

- Use an active voice whenever possible.

3. Chemical nomenclature

- Follow IUPAC rules for chemical nomenclature except when a deviation (e.g., use of common names or other nomenclature typically used by the RCRA regulations) is more appropriate. ACS Style Guide is recommended as a reference on chemical nomenclature.

4. Software

- Submit all method documents in MSWord (must be compatible with Microsoft Office Word 2003)

- If you cannot use MSWord, then at least use word processing software that can be easily converted to MSWord with minimal conversion errors.

5. Font

- Use Arial 11 point for text and Cambria Math 11 point for equations. Fonts can vary in tables, as long as the content is readable.

6. Margins

- Set the right and left margins at a spacing of one inch (1"). Set the top and bottom margins at one half (½") inch.

7. Justification

- Use left justification for method text alignment unless otherwise specified in this style guide.

8. Tabs and indents

- Do not tab major sections (e.g., 1.0, 2.0, etc.). Subsections (e.g., 1.1, 2.2, etc.) are first line tabbed. All subsequent subdivisions (e.g., 1.1.1, 2.2.2.2, etc.) are tabbed or indented to show their relative sub-categorization.

- Use the following left tab settings:

  1.0", 1.38", 1.88", 2.5", 3.25", and 4.13" (see the settings of this document at this point for what is meant by these settings)

  For example, using up to three levels:

9.0 QUALITY CONTROL

  9.1 Quality control related to the use of a test kit for RDX or HMX analysis.

    9.1.1 Follow the manufacturer's instructions for the quality control procedures specific to the test kit being used.

- The above tab settings accommodate up to five levels of numbered subsections, e.g., through 9.5.1.2.3. (However, please avoid exceeding four levels, see item no. 1 above.) If the section number includes a lot of double digits (e.g., 11.11.3.11), adjust the tab settings as necessary to allow sufficient spacing between the section number and section text.

- Use **tabs** to wrap the text of the second line of a section to the left of the first line.

  For example:

1.1 This method provides procedures for the gas chromatographic (GC) determination of organophosphorus (OP) compounds.

- Use **indents** when the text of the second line is not intended to wrap to the left of the first line.

- For third level and higher subsections, use indents to move the subsection number away from the left margin, followed by one tab immediately before the subsection number, and another tab after the number, and before the text. The use of the indent keeps the second line from wrapping all the way back to the left margin. For example:

    11.3.2 Take two drops of methyl blue and add it to a 50-mL vessel containing 5 mL of NAOH and fill to volume with sample aliquot. Stir the mixture briskly.

9. Spacing

- Triple space (two blank lines) before each major section (1.0, 2.0, etc.), double space between other sections. Single space within sections, as appropriate (sometimes double spacing within a section or paragraph is necessary to accommodate "notes," "warnings," equations, or other information.

- Use two spaces after periods and colons in sentences.

- Spacing in tables can vary, as long as the content is readable.

10. Capitalization

- Put the major section titles in all capital letters (e.g., 9.0 QUALITY CONTROL). Capitalize only the first letter of the titles of other subsections. For example:

    9.0 QUALITY CONTROL

        9.1 Surrogate recovery

11. Use of notes, warnings, and cautions

- Use the following conventions regarding the content and purpose of any notes, warnings, or cautions:

    WARNING: Provides information to prevent personal injury.

    CAUTION: Provides information to prevent damage to equipment or other significant occurrences to be avoided during application of the method.

    NOTE: Provides useful tips and background relating to the current topic.

- As illustrated above, present each of the above headings in all capital letters, underlined, with a colon, then an indent (with a "custom" tab setting of two spaces after the colon), such that the text after the colon is aligned even to the left.

- As necessary, modify the document section tabs to accommodate the indent for a "NOTE," "WARNING," or "CAUTION". Restore the default method tab after the note, warning, or caution.

- Indent notes, warnings, cautions only as far as the left of the preceding text; or NOT as far as the section number is tabbed, but to the point where the text wraps to the left. For example:

6.1 Columns

6.2 Drying column - 20-mm ID Pyrex® chromatographic column with Pyrex® glass wool at bottom and a PTFE stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex® glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

12. Keeping text together and page breaks

- To avoid the separation of section headings (including subsection headings, such as "9.1 Surrogate recovery") from the section text at a page break, use "keep text together" features provided by the word processing software.

- Avoid using hard page breaks in the text portion of the method.

- Always use hard page breaks between the last page of text (i.e., after the sentence under the title of Sec. 17.0) and the first table and all other subsequent pages to separate tables and figures.

13. Page numbering

- Position page numbers at the bottom center of the page as part of the footer. Begin the page number with the appropriate method number (including any letter suffix), followed by a hyphen, and then the page number starting with the number one (1). Place one space on either side of the hyphen. Use the automatic number sequencing in the software to accomplish this task. For example:

3562 - 1

14. Units

- Use the System Internationale (SI) units as the standard units of measurement.

- For the unit "micro", use the Greek symbol notation. This symbol (**μ**) can be located under Insert→Symbol (***μ***) in Microsoft Word 2007.

- Use the following standard types of units:

  mg/L (not μg/mL)
  ng/μL (not μg/mL)

- When using a number and a unit as an adjective to modify a noun, put a hyphen between the number and unit. However, do not insert a hyphen if the number and unit are instead used as a noun. For example: "Add 10 mL of distilled water to a 25-mL beaker, weigh a 10-g portion of the field blank sample and add 100 μL of the solution containing the nine internal standards diluted with 1.0 mL of acetone."

15. Equations

- Triple space between section text and an equation (bottom and top). Do not place the equations any closer to the text. Otherwise, PDF conversions compress the document material and may move the equations so close to the text that the equation cannot be fully read. Center the equations and set the height and width of the equation boxes large enough to view the whole equation when printed. Do not italicize equations. Use the equation editor in Word 2007 to enter equations in a Cabria Math 11 pt font.

For example:

Bottom line of preceding text here.

$$\%\ \text{dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

Top line of following text here.

Regarding file conversions from WordPerfect (.wpd) and/or Microsoft Word (.doc or .docx) to PDFs: Equations do not always convert well to PDF using Word 2007. It is usually best to use Acrobat Writer software, if available. As an alternative (for people who do not have access the full version of Adobe Acrobat), the equations are generally well preserved when the Word .docx file is printed as a file in PDF format through Word with a printer plugin such as PrimoPDF, rather than creating a PDF through the "Save as" dialog in Word.

- Be consistent with equation formats.

- Use units in the equation that are consistent with the method procedure and sample matrix.

- Define all equation variables.

- Do not number equations in a method.

16. Acronyms

- Prior to the first use of an acronym in a method, spell out the term it represents and then include the acronym in parentheses. For example:

  supercritical fluid extraction (SFE)

  Afterwards, use just the acronym, as appropriate.

- A proper noun is the name of a particular person (e.g., Dr. Paul Jones), place (e.g., New York), or thing (e.g., Table 1). Capitalize only proper nouns within a sentence (unless of course you are quoting a document title which includes the use of initial capitalization). Use of an acronym <u>does not</u> turn its full word into a proper noun. In other words, the conventional use of capitalized letters in an acronym does not mean that capitalized letters must be used in the full word -- unless the word is already a proper noun. For example:

  This is <u>correct</u>:

  "The use of supercritical fluid extraction (SFE) . . . "

This is incorrect, because "supercritical fluid extraction" **is not** a proper noun):

"The use of Supercritical Fluid Extraction (SFE) . . . "

However, this is correct, because "Office of Solid Waste" **is** a proper noun:

"The Office of Solid Waste (OSW)..."

17. Method and SW-846 manual references in the text of a method

- In general, do not refer to the method itself by its method number within its own text -- instead refer to it only as "this method."

- Usually, do not refer in the method text to "SW-846," as though the method is a separate document and not an integral part of the SW-846 manual. Instead, refer to "this manual." Exceptions apply in certain boilerplate statements which address use of the manual in general.

- Only use method numbers with their letter suffixes (e.g., 3500A) in the method number of the method title and page number. Do not include method number suffixes in the text of the methods, unless the method reference is part of a document title quote. For example, state "See Method 3510," even if the latest version of this method is "3510A". Otherwise, the text of many methods might have to be updated to reflect new suffixes each time a referenced method is revised. (See Sec. III below for information on the purpose of letter suffixes in method numbers.)

18. Section references in the text

- When referring to an SW-846 method section, capitalize and abbreviate the word "section," for example: Sec. 1.5

- When referencing sections in other methods, only go to the first level, e.g., 2.0, because the subsection content and numbering of any method may change in future updates. Also, for similar reasons, do not reference specific chapter sections, only reference the chapter number (exceptions to this may apply due to a boilerplate referral).

19. Document references in the text

- Cite references in the text by directing the reader to a specific reference listed in the references section (Sec. 16.0), using such phrases as "refer to" or "see". It does not matter whether "Reference" is spelled out or abbreviated as "Ref." -- as long as consistency is followed within the method. For example:

  For complete method performance data, see Reference 2.

20. Use of the terms "must (shall), should and may" and "required"

- Avoid use of the term "shall" -- use "must" instead as appropriate. Be careful nevertheless in the use of these terms, as they indicate a requirement.

- In general, follow the NACE method Style Manual for guidance regarding the proper use of the terms "must, should, and may." Specifically:

Use "must" to indicate mandatory instructions. (This means mandatory technology use instructions, not mandatory regulatory instructions. The instructions are considered "mandatory" because otherwise method application would be adversely affected.)

Use "should" to indicate that which is recommended.

Use "may" to indicate that which is considered fully optional (and which of course might depend on method performance goals and project-specific needs).

- Avoid unnecessary use of the terms "require" or "required." In general, reserve use of the word "require" when referring to project-specific criteria or regulatory requirements. Otherwise, replace "require" or "required" with such terms as "need" or "needed", "are necessary", etc. Also, for example, use the term "designated" instead of "required" as in "designated" volume instead of "required" volume.

21. Terms to use or avoid (exceptions may apply, dependent on content)

Correct: "alternative" acceptance limits
Incorrect: "tighter" acceptance limits (unless that is exactly what is intended)

Correct: "described" steps
Incorrect: "specified" steps (unless referring to what is written in a planning document -- those steps/criteria would be "specified")

Correct: "designated" volume
Incorrect: "specified" or “required” volume

Correct: "professional judgement" of the . . .
Incorrect: "discretion" of the . . .

Correct: "appropriate" calibration compound
Incorrect: "acceptable" calibration . . .

22. Quantitation Limit vs. Detection Limit

- Use the phrase "lower limit of quantitation" instead of "lower limit of detection" or "detection limit."

- Do not include or mention MDLs or EQLs, in the method. If necessary or useful, only include a generic description of anticipated method sensitivity for the matrix. The focus should be on actual measured performance, based on spike recoveries in the matrix of concern or of method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. Also, clearly note that the information is provided as guidance only, and that such limits are highly-matrix dependent and not always achievable. In some cases, such as in the immunoassay methods, the values quoted as "MDLs" are real measured values and thus should be in the method with a change in the terminology (e.g.,” the lower limit of quantitation”).

23. Footnotes

- Only use footnotes in the tables, avoid using them in the method text.

24. Other miscellaneous style guidelines

- **Negative ionic charge symbol** -- Use the math symbol (−), not a hyphen superscript.

- **Dot in chemical formulas** -- Use a typographical symbol that is not too large or too small (•).

- **Temperature** -- As directed by the ACS Style Guide, put a space right after the number and keep the degree symbol and "C" together.

- **Trade names for materials** -- Avoid use of trade names if something more generic is also appropriate. For example, use "polytetrafluoroethylene (PTFE) in place of "Teflon."

- **Measurement abbreviations for liter** -- Designate liter using an upper case "L" and milliliter as "mL".

- **Plurals of measurement abbreviations** -- Do not use plurals for abbreviated units of measure. For example: 50 mg *not* 50 mgs

- **Quotation marks** -- Use straight quotation marks (e.g., " *or* ') instead of smart quotes. That style is better suited to the serif-free Arial font.

- **En dash with numbered items** -- Use en dash (typed as a hyphen) with three or more numbered items. For example: 20 - 30 mL, References 3 - 5 . . .

- **Apostrophes** -- Do not use an apostrophe in plurals of chemical name acronyms. For example:

  Correct: PCBs
  Incorrect: PCB's

- **SW-846 chapter references** -- Specify the chapter number, even if the subject method is within the chapter. For instance, use the phrase "in Chapter Four" instead of "in this chapter."

- **Equipped with or fitted with** -- Be sure to include the phrase "equipped with" or "fitted with" instead of just "with" when referring to something attached to a piece of equipment.

  Incorrect: Transfer 25 mL of extract to a vial "with" a PTFE cap.
  Correct: Transfer 25 mL of extract to a vial "equipped with" a PTFE cap.

- **Abbreviations of time-related measurements** -- Abbreviate time-related measurements, e.g., use "hr" for hours, "min" for minutes.

- Do not put a hyphen in "coelution."

- **Serial Commas** -- Use serial commas when making lists to avoid confusion, e.g., "…a method blank, a matrix spike, a laboratory control sample, and a duplicate sample…"

- **Data should be used as a plural count noun**, not as a singular mass noun, e.g., "the data are more compelling", **not** "the data is more compelling. "

III. SECTION SPECIFIC GUIDELINES

This section of the style guide provides general guidance regarding method numbers, method titles, and the content of the 17 major sections of SW-846 methods. (See Attachment A for a one-page summary listing of the major sections.) It also provides general boilerplate sections that should appear in most SW-846 methods -- when appropriate. Please note that the directions in this section are guidelines and not rules. For example, other instructional "boilerplate" phrases, or revisions to those that appear in this guide, may be more appropriate to individual methods or groups of methods (e.g., based on similar technologies or steps used by the methods). This style guide does not include the many examples of such information. Therefore, developers of new methods should review recently published and technically similar methods for additional insight regarding section content, and consult with EPA for direction.

METHOD NUMBER AND TITLE

- Present the title in capital letters, underlined and centered.
- Use succinct method titles. The most important item, usually the analytes of concern, should be mentioned first. For example, begin with the analytes of concern followed by the matrix and the technology used by the method. An exception to that example, however, might be "SCREENING OF VOAs IN SOIL," where the fact that it is a screening method is the most important item.
- Do not begin the title with "ANALYSIS OF" or "DETERMINATION OF."
- Place the method number two lines above the title. (Method numbers are assigned by EPA based on the type of technology and analytes. See Attachment B to this style guide for the guidelines used by EPA.)

For example:

METHOD 8275

SEMIVOLATILE ORGANIC COMPOUNDS (PAHs AND PCBs)
IN SOILS/SLUDGES AND SOLID WASTES USING
THERMAL EXTRACTION/GAS CHROMATOGRAPHY/MASS SPECTROMETY (TE/GC/MS)

- Any method included for the first time in SW-846 will be "Revision 0" of that method. (Exception: A required, method-defined parameter (MDP), method will retain the revision level of its last version in the Third Edition.) A method formally increases in revision status each time it is revised and published as a final update to an SW-846 edition. During development of a new revision, the method revision number is increased once and that revision number is used when the method is final. Therefore, a method revision number does not increase as a result of each review within EPA's technical work group, or as it goes from a "draft" or "proposed" status to a published "final" update status. It only increases with each final publication, and its revision number is the same at "proposal" or any "draft" as it will be at final.

- Letter suffixes (e.g., A, B, C) to a method number identify the revision status of the method. New methods, i.e., Revision 0 methods, do not have a letter suffix. A suffix of "A" in a method number indicates Revision 1 (the method has been revised once and distributed as final). A suffix of "B" indicates Revision 2, etc. A method number containing the suffix "M" for "modified", e.g., Method 8015M, is not an EPA method. According to the SW-846 numbering system, the suffix "M" would stand for Revision 13 of the method, which is highly unlikely to be reached before issues of a new SW-846 edition.

- Can use "By" instead of "Using" in the title.

- Matrix of concern need not be in the title if would be universally understood (e.g., as with the SFE prep methods).

Title Boilerplate:

Place the following paragraphs between the title and Sec. 1.0 of all methods (triple space before and after):

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts formally trained in the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique, which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. Performance data included in this method are for guidance purposes only and must not be used as absolute quality control (QC) acceptance criteria for the purposes of laboratory QC or accreditation.

1.0 SCOPE AND APPLICATION

- In the first section, describe the purpose and technology of the method, including what types of analytes or attributes are being measured (e.g., this method is a colorimetric screening procedure that may be used to determine . . . in soil samples). Mention all applicable matrices.

- As appropriate, tabulate a list of analytes (put in a table even if only one analyte) validated by the method, by common name, with any common abbreviations, and the Chemical Abstract Service Registry number. Only include those analytes regulated under RCRA in this first table. Precede the table with this statement: The following RCRA compounds (or use the word "analytes" if "compounds" is not appropriate) have been determined by this method:" Do not say "can be" determined . . . For cross-method consistency, the header title for the target analytes column should be "Analytes", although this is not critical.

- Create a separate table for any other possible analytes (e.g., those not adequately validated for analysis using the subject method or those validated which are not RCRA analytes), set up exactly as in the previous table of analytes, but with a qualifying statement.

- Indicate any important relationships to other SW-846 methods, as applicable.

- Specify method limitations (e.g., what the method will not accomplish that the analyst may be seeking).

- As noted earlier in this style guide, do not address or include method detection or estimated quantitation limit discussions or data in Sec. 1.0 or anywhere else in the method. If necessary or useful, only include a generic description of method sensitivity, and clearly note that the information is provided as guidance only, and that such limits are highly-matrix dependent and not always achievable.

- In preparation or extraction methods, note that other solvent systems may be employed, and that for any solvent system used, including those mentioned in the method, one needs

to demonstrate adequate performance for the analytes of interest. (Also note this in Sec. 7.0 of the method, see example of such text given later in this document for Sec. 7.0.)

- Include any other method application information that would be particularly useful to the chemist during method selection (including, for example, particularly critical safety information, with a reference to Sec. 5.0 for details).

- Include boilerplate statements (see below) regarding intended method flexibility and required uses and regarding experience of analysts.

Sec. 1.0 boilerplate regarding method flexibility and required uses:

Generally in laboratory methods, include the next two paragraphs as the next to the last subsection under Sec. 1.0. The first sentence of the first paragraph may not be necessary for some methods (e.g., those not involving the use of base laboratory methods), and revise the "e.g." as necessary based on the subject method:

1.X Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for 1) guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies; and 2) the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by the Environmental Protection Agency (EPA) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives (DQOs) for the intended application.

For those methods which include the use of manufacturer kits and which allow less flexibility in material use, instead include the next two paragraphs (revise as appropriate for specific methods):

1.X Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Method 4000) and the manufacturer's instructions for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by the Environmental Protection Agency (EPA) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives (DQOs) for the intended application.

Another example of the boilerplate for methods without flexibility (taken from Method 8540):

1.X Prior to employing this method, analysts are advised to consult the manufacturer's instructions for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by the Environmental Protection Agency (EPA) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives (DQOs) for the intended application.

For inorganic preparatory methods:

1.X Prior to employing this method, analysts are advised to consult the determinative method that may be employed in the overall analysis for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for 1) guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies; and 2) the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by the Environmental Protection Agency (EPA) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objective (DQOs) for the intended application.

Sec. 1.0 boilerplate regarding analyst experience:

Include this boilerplate paragraph as the last subsection under Sec. 1.0. The first two examples of this paragraph is a generic approach and the third example illustrates a more specific approach. Example 2 might be the preferred to mention specific method instrumentation, e.g., GC, ICAP, etc.

Example 1: (as preferred for organic analyte methods):

1.X This method is restricted to use by, or under supervision of, appropriately experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

Example 2: (as preferred for inorganic analyte methods)

1.X This method is restricted to use by, or under supervision of, properly experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

Example 3:

1.X This method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use of <add method technology name here>. Each analyst must demonstrate the ability to generate acceptable results with this method.

Sec. 1.0 boilerplate regarding use of other solvent systems:

Include this boilerplate sentence in the subsection of Sec. 1.0 that identifies what solvent system was used for method validation, or which one is addressed directly in the method (identify at Sec. 7.X which subsection of Sec. 7.0 provides the complete boilerplate and more details regarding the use of solvent systems):

Other solvent systems may be employed; provided that adequate performance can be demonstrated for the analytes of interest (see Sec. 7.X).

Sec. 1.0 boilerplate for those methods that may not be appropriate for aqueous samples with high levels of suspended solids (edit as appropriate for the specific method):

1.X This method may not be appropriate for aqueous samples with high levels of suspended solids greater than 1%. However, if the particulate matter is not considered to be part of the sample composition based on specific project objectives and intended data usage, samples may be allowed to settle before measuring the aliquot to be extracted. If significant particulate matter is present and the total sample is of concern, then the sample should be treated as a multi-phase sample per Chapter Two.

2.0 SUMMARY OF METHOD

- Provide a brief summary of the method's major steps -- to the detail necessary to best prepare the analyst regarding what will be necessary during method application.

- Make this section complete enough for the analyst to anticipate possible interferences or other application problems.

- Do not include details of exact volumes or weights, etc.

3.0 DEFINITIONS

- Include a boilerplate referral to Chapter One, any other appropriate chapter (e.g., Chapter Three), and the manufacturer's instructions for definitions that may be applicable to the method.

  NOTE: There will NOT be a separate chapter of definitions in SW-846, remove any old boilerplates that referenced that chapter.

- This section can also include definitions of terms and acronyms particularly relevant to the method and those which may not be familiar to the reader. However, do not include particularly extensive lists of method-specific definitions (i.e., more than three definitions) -- instead include such long lists in a glossary at the end of the method (as a method appendix) and refer to it in this section.

Sec. 3.0 boilerplate:

For inorganic methods:

Refer to Chapter One, Chapter Three, and the manufacturer's instructions for definitions that may be relevant to this procedure.

For all other methods:

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

- Discuss known and potential problems and interferences that could affect method performance or an evaluation of the results.

- As appropriate, describe procedures that may be employed to prevent or minimize the problems. If such procedures are already included in Sec. 11.0 (Procedure), a reference to these subsections should be included in this section.

- If appropriate, include the boilerplate regarding demonstrating that materials used during analysis are free of interferents (see below).

- As applicable, refer the analyst to the base method for additional quality control guidance regarding potential interferences.

Sec. 4.0 boilerplate:

Include this boilerplate in most methods, when appropriate to method application. Do not include it in field test kit methods or similar applications.

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences during sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter <Three, for inorganic, or Four, for organic> for general guidance on glassware cleaning. Also refer to Method(s) <base method> for a discussion of interferences.

For inorganic and organic sample prep methods, replace the last sentence in the above with the following:

4.1 . . . Also refer to the determinative methods to be used for information regarding potential interferences.

5.0 SAFETY

- Discuss personnel health and safety issues specific to performance of the method and beyond the scope of routine laboratory practices. This includes information regarding specific toxicity of target analytes and reagents, special precautions to avoid harm, and any special protective equipment required for performing the method.

- Safety warnings may also occur in other sections of the method, for example, in the procedure at the step of concern. However, you must repeat in Sec. 5.0 all safety concerns found in any other section of the method.

- NOTE: **For inorganic methods, remove any referrals to Chapter Three for "additional safety guidelines." The current version (05/2012, Update V) does not provide any further information than what is already provided in the boilerplate below.**

- Add the appropriate boilerplate statement (see below) regarding safety.

Sec. 5.0 boilerplate:

Include as the first section in Sec. 5.0 for those methods that involve the handling of any chemicals:

5.1 This method does not address all safety issues associated with its use. The laboratory <or replace "laboratory" with "user" if the method is not a laboratory method> 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.

Include as the first section in Sec. 5.0 for those methods that do not involve the handling of any chemicals:

5.1 This method does not address all safety issues associated with its use. The laboratory <or replace "laboratory" with "user" if the method is not a laboratory method> is responsible for maintaining a safe work environment.

6.0 EQUIPMENT AND SUPPLIES

- List method-specific equipment and supplies (those other than reagents and standards), without mentioning a specific vendor whenever possible. Include the phrase "or equivalent" as appropriate when vendor-specific instrumentations or supplies are listed. If specific equipment is necessary based on method studies, clearly state what equipment and supplies were tested. If necessary, include sufficient information for locating and purchasing the correct equipment.

  Common laboratory apparatus, e.g., beakers, flasks, stirring bars, graduated cylinders, etc., should not be mentioned, unless there is a specific need for one with an unusual or non-standard characteristic, e.g., a specific chemical-resistant coating for a stirring bar, tinted glass flasks, Class A graduated cylinders, etc. All other apparatus should be mentioned in this section, e.g., pH meter, hot plate stirrer, analytical balance, etc. Generally, mention only the more expensive and unique equipment.

- If appropriate (see note before boilerplate), add the boilerplate (below) regarding how the mention of trade names is for illustrative purposes only (if such names are given in the method).

- If appropriate, also include the boilerplate (below) regarding glassware for solvent recovery, edited as appropriate to the specific method.

- Do not include more sensitivity specifications (significant figures) than necessary. For instance, a balance capable of weighing to "0.0001 g" is too sensitive of a specification for the weighing of 10-g samples. A specification of 0.01 g may be more appropriate.

Sec. 6.0 boilerplate regarding trade names:

Include this boilerplate in most methods, depending on the application and whether trade names for equipment are mentioned. However, do not include this boilerplate in immunoassay test kit methods or similar applications that depend on use of a specific commercial product:

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

Sec. 6.0 boilerplate regarding listing of common equipment:

The following should follow the previous boilerplate as a new single-sentence paragraph, or stand alone if the previous boilerplate does not exist. Do not give it a section number; treat it as an introductory paragraph:

This section does not list all common laboratory glassware (e.g., beakers and flasks) that might be used.

Sec. 6.0 boilerplate regarding a listed solvent recovery system:

Revise as appropriate to reflect the specific method application:

6.X Kuderna-Danish concentrator -- 500-mL, fitted with 10-mL concentrator tube and three-ball Snyder column. Other evaporation devices such as a Turbovap and N-evap may be used as appropriate for a given application. Users should consult the manufacturer's guidelines for operational requirements and instructions.

Include this boilerplate when appropriate after the above:

NOTE: A solvent recovery apparatus is recommended for the purpose of solvent recovery during the concentration procedures (Sec. <X.X>) using <identify the technology here, e.g., "Kuderna-Danish evaporative concentrators">. Incorporation of this apparatus may be required by Federal, State or local municipality regulations that govern air emissions of volatile organics. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction program. Solvent recovery is also a means of conforming with waste minimization and pollution prevention initiatives.

Sec. 6.0 boilerplate regarding gas chromatographs and accessories (revise as appropriate for the individual method):

6.X Gas chromatograph (GC)

An analytical system equipped with a gas chromatograph suitable for <e.g., on-column injection, split/splitless injection, direct aqueous injection, vacuum distillation sample introduction, etc.> and equipped with all necessary accessories, including analytical columns, suitable detectors, gases, syringes, and recorder/integrator or data system. A data system for measuring peak heights and/or peak areas is recommended. If a dual-column option is employed, the gas chromatograph must be equipped with two detectors. Refer to Method 8000 Sec. 6.0 for additional information regarded GC accessories.

6.X GC columns

The choice of GC columns will depend on the analytes of interest, the expected concentrations, and the intended use of the results. Refer to Method 8000 Sec. 6.0 for direction in selecting GC columns.

The columns listed in this section were the columns used either in developing the method or during subsequent updates of the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.X GC detectors

Detectors are the transducers that respond to components eluting from a GC column and produce the electrical signal used for quantitative determinations. SW-846 analyses in this manual are conducted using the detectors listed in Sec. 1.1 (of 8000D). Except where otherwise recommended by the instrument manufacturer, selective non-MS detectors should be maintained at least 20 °C above the highest oven temperature employed to prevent condensation and detector contamination. To prevent condensation between the GC and an MS detector, transfer

lines should be maintained at a temperature above the highest temperature of the oven program, or as specified by the instrument manufacturer

Sec. 6.0 boilerplate regarding high-performance liquid chromatographs (HPLC) (revise as appropriate for the individual method, below taken from Method 8318A, Feb 2007):

Additional sections will most likely be necessary. Add, subtract, and revise as needed for the individual method.

6.X High-performance liquid chromatograph (HPLC)

An analytical system equipped with a programmable solvent delivery system and all necessary accessories, including a high pressure injection valve, analytical column(s), mobile phase solvent degassing, etc. At a minimum, the solvent delivery system must be capable of handling a binary solvent system, and must be able to accurately deliver flow rates for the method.

The follow portions are taken from Method 8000D, 6/2012, Sec, 6. The full text has more details on troubleshooting and selection criteria:

6.X HPLC injectors

Liquids are essentially uncompressible, so a mechanical device is necessary that allows introduction of the sample into a high pressure flow without significant disruption in the flow rate and hydraulic pressure. Normally, a 6-port valve is used for this purpose. A sample loop is isolated from the flow of the mobile phase and filled with a sample extract. (Larger sample loops may be used to increase sensitivity; however, they may degrade chromatographic performance).

6.X HPLC pumps

The mobile phase used for HPLC should be accurately pressurized before it enters the injector. HPLC pumps are generally capable of delivering solvent at 5000 psi or above with excellent precision. Rate of delivery depends on the column used for the separation. Flow rates should be checked by collecting column effluent in a graduated cylinder for a designated time period. Most pumping systems are capable of changing solvent concentration during an analysis (i.e., gradient elution). Gradients are generated by either high pressure mixing of two streams between the pump and the injector or by proportional mixing of the solvents before they are pumped.

6.X HPLC Columns

HPLC columns are generally constructed of stainless steel tubing and are sealed with compression fittings. These columns should be constructed with minimum dead volume and a narrow particle size distribution. Manufacturers provide columns bonded with dissimilar functional groups (e.g., C_{18}, cyano, TMS) and have different percent carbon loading.

6.X HPLC detectors

Detectors are the transducers that respond to components eluting from a HPLC column and produce the electrical signal used for qualitative and quantitative determinations. SW-846 analyses are conducted using selective detectors or mass spectrometers listed in Sec. 1.1 (of 8000D). HPLC/MS involves the use of a sophisticated interface that separates target analytes

from the aqueous mobile phase. Examples include the thermospray (TSP), electrospray (ESP), and the atmospheric pressure chemical ionization (APCI) interfaces.

6.X Data systems

Raw chromatographic data have to be reduced in order to provide the quantitative information needed by analysts. Sophisticated data systems are strongly recommended for SW-846 chromatographic methods because the ability to store and re-plot chromatographic data is invaluable during data reduction and review. Organizations should select the system most suitable for their applications.

Sec. 6.0 boilerplate that may be appropriate for immunoassay methods:

6.1 Each test product will specify the apparatus and materials provided, as well as any additional apparatus and materials necessary for performance of the test. The immunoassay testing products of SW-846 immunoassay methods were submitted to EPA, evaluated by the Agency, and found to meet the performance specifications necessary for inclusion in SW-846. As additional testing products are evaluated by EPA and found to provide equivalent performance, information will be made available by the Office of Solid Waste regarding those testing products that are capable of meeting the performance specifications in the methods (see http://www.epa.gov/epaoswer/hazwaste/test/pdfs/kits.pdf). However, the methods will not be revised solely to include information on additional testing products. Descriptions and materials lists for products relevant to the methods should be given in the manufacturer's literature.

6.2 Most testing products provide the supplies specific to the immunoassay, including the tubes or plates containing the immobilized antibody, and the immunochemical reagents. Do not mix the equipment, supplies, and reagents from the testing products for different analytes or from the testing products from different manufacturers. Testing products contain immunochemical reagents that are evaluated by the manufacturer on a lot-specific basis. Do not mix the reagents from one lot with those from another lot unless expressly allowed by the manufacturer. Other equipment that may be required, but is not supplied with the testing product, includes common laboratory items such as precision pipetting devices, vortex mixers, etc.

7.0 REAGENTS AND STANDARDS

- Provide sufficient detail on necessary grades, the concentration, and the preparation of all reagents and standards to allow the work to be duplicated. Do not include lengthy discussions on common procedures.

  Exception regarding information on preparation of standard: If a standard must be prepared when it is about to be used (e.g., as in Method 8151), the description of standard preparation can appear at that location in the method (e.g., in Sec. 11.0), and a reference to that section should appear here in Sec. 7.0 as appropriate.

- List each chemical as follows: chemical name, concentration in parenthesis, and the formula. For example: Sodium hydroxide (2M), NaOH

- Be consistent with the standard or reagent name throughout the method (and the manual as possible), and include all standards and reagents mentioned by the method in any section.

- Keep a 1:1 correlation between the information in this section and the others, e.g., if the procedure calls for both 2N and 1N H_2SO_4, then include in this section both 2N and 1N H_2SO_4.

- As necessary, include specific information regarding the storage of the reagent or standard.

- List reagents before standards, particularly if the standards are to be prepared from the reagents. List the standards in descending order based on which are made from which, i.e., list the stock standards before the dilution standards. As compatible with these conditions, also match the listing order with the first appearance of the chemical in the procedure.

- Include boilerplate instructions (see below) regarding the reagent grade of chemicals and references to water, when appropriate. This boilerplate may not be appropriate for some methods. (In old methods, replace references to "ASTM Type II" water with "reagent." Also, because of the boilerplate for this section, reagents throughout the method need not be referred to as "analytical-grade.")

- In preparation or extraction methods, note that other solvent systems may be employed, provided that adequate performance can be demonstrated for the analytes of interest with any solvent employed, whether or not it is listed in the method.

- As applicable, use the phrase "elution" solvents, and not "eluting" solvents.

Sec. 7.0 reagent-grade chemical boilerplate:

Include the following paragraph when appropriate as the first section; this boilerplate may not be appropriate for some methods, such as field test kits.

7.1 Reagent-grade <add “or pesticide grade”, if applicable> chemicals, at a minimum, should be used in all tests. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where specifications are available. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent leaching of contaminants from plastic containers.

<Note to method editor: The last sentence of the previous boilerplate can sometimes be removed if the method doesn’t have to do with organic analysis>

Also include as appropriate:

7.2 Reagent water -- Reagent water must be interference free. All references to water in this method refer to reagent water unless otherwise specified.

Sec. 7.0 extraction solvent system boilerplate:

Include text similar to the following paragraphs (taken from Method 3546), edited as appropriate for methods involving solvent extraction systems:

7.X Extraction solvents

This method has been validated using a 1:1 mixture of hexane and acetone from matrices such as soil, glass fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy.

Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix, at the concentrations of interest. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the desired project-specific concentration levels. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

Many of the <revise as appropriate> solvent systems described below include the combination of a water-miscible solvent, such as acetone, and a water-immiscible solvent, such as methylene chloride or hexane. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water on the surface of the solid particles. The water-immiscible solvent extracts organic compounds with similar polarities. Thus, a non-polar solvent such as hexane is often used for non-polar analytes such as PCBs, while a polar solvent such as methylene chloride may be used for polar analytes. The polarity of acetone may also help extract polar analytes in mixed solvent systems.

All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use.

Sec. 7.0 paragraph regarding calibration standards, revised as appropriate organic methods (use "should" not "must", and reference Method 8000 for additional information).

7.X Calibration standards

A minimum of five different concentrations for each parameter of interest should be prepared . . . Calibration standards should be replaced after one or two months, or sooner if comparison with check standards indicates a problem. . . See Method 8000 for additional information on the preparation of calibration standards.

Sec. 7.0 paragraph regarding mixed-calibration standard solutions, revise as appropriate for inorganic methods (reference Methods 6010 and 6020 for additional information).

7.X Mixed-calibration standard solutions

Prepare by diluting stock standard solutions to levels in the linear range for the instrument. Use the same combination and concentrations of acids used in the preparation of the sample digestates. Store all mixed-calibration standards in an appropriate container and protect form light. Prior to preparing the mixed standards, each standard stock solution should be analyzed, separately, in order to determine possible spectral interferences and/or the presence of impurities. Standards which interfere with another analyte, or which are contaminated with another analyte, may not be included in the same calibration standard as that analyte. Refer to Method 6010 for recommendations in selecting the most appropriate stock standards (Sec. 7.0) to combine for the preparation of working-level, mixed-calibration standards.

Sec. 7.0 paragraph regarding blanks, revise as appropriate for spectroscopic methods or appropriate instrumentation (reference Methods 6010 and 6020 for additional information).

7.X Blanks

Three types of blanks are necessary for analysis: (1) the calibration blank, which is used in establishing the calibration curve; (2) the method blank, which is used to monitor for possible contamination resulting from the sample preparation procedure; and (3) the rinse blank, which is used to flush the system between all samples and standards. Refer to Method 6010 and 6020 for additional information and how to prepare the different types of blanks.

Sec. 7.0 paragraphs regarding surrogate standards, include and edit as appropriate organic methods (the text can vary based on the method, at the least include a sentence that other surrogates may be used, provided . . . etc.)

7.X Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. The following compounds are recommended as possible surrogates. Other surrogates may be used, provided that the analyst can demonstrate and document performance appropriate for the data quality needs of the particular application.

Sec. 7.0 paragraph regarding standard solutions; include and edit as appropriate to the method:

7.X Standard solutions

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards.

Sec. 7.0 caution to follow the above in methods which involve the analysis of explosives:

CAUTION: Calibration standards are commercially available from several sources including Supelco, AccuStandard, and Radian, as both solutions and neat materials. It is **highly recommended** that commercially-prepared stock standard solutions be obtained rather than neat materials be handled.

Sec. 7.0 boilerplate that may be appropriate for immunoassay methods:

7.2 As with the equipment and supplies, each commercially available testing product will supply or specify the reagents necessary for successful completion of the test. This includes the calibrators (standards) employed in the immunoassay. Detailed information on reagent requirements is given in the manufacturer's literature. As noted in Sec. 6.X, do not mix the equipment, supplies, and reagents from the testing products for different analytes or from the testing products from different manufacturers. Store all reagents and standards according to the manufacturer's instructions, and, where applicable, discard any that are past the expiration date assigned by the manufacturer.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- Provide any method-specific information on sample collection, preservation, shipment, and handling or storage. In general, this section should not deal with general field sampling information, nor refer to such guidance. However, include field sampling components in those methods for which such procedures are an integral part of the method, e.g., Method 5035/Appendix A.

- When appropriate, add a boilerplate referral (see below) to the method chapter for additional, or for any, guidance on sampling procedures that may be particular to the methods contained in that chapter.

Sec. 8.0 boilerplate:

Add this boilerplate right after the Sec. 8.0 title, without a section number:

Sample collection, preservation, and storage requirements may vary by EPA program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. Where such requirements are specified in the regulation, follow those requirements. In the absence of specific regulatory requirements, use the following information as guidance in determining the sample collection, preservation, and storage requirements.

Add this boilerplate when appropriate (i.e., if such guidance exists somewhere in the chapter):

See Chapter <no.>, "<put title of chapter here>" for sample collection and preservation instructions.

For organic prep methods (revise as appropriate):

See Sec. < X.X> of Method <XXXX>. Also see the introductory material to Chapter Four, "Organic Analytes," Method 3500, and the specific determinative methods to be employed.

Sample containers boilerplate for inorganic prep and other inorganic methods (if appropriate) (from 3015A of Update IV Feb 2007):

8.X All sample containers must be prewashed with acids, water, and metal-free detergents, if necessary, depending on the use history of the container. Plastic and glass containers are both suitable. For further information, see Chapter Three.

9.0 QUALITY CONTROL

- Describe method-specific quality control measures.

- Do not include general QC information that is redundant to that already contained in Chapter One.

- Do not include calibration information in Sec. 9.0.

- Add the first boilerplate referral (see below) to the SW-846 Chapter One for guidance on additional quality assurance and quality control protocols. When this boilerplate is not appropriate (e.g., for a field test kit method), then add the second boilerplate.

Sec. 9.0 boilerplate:

Include the following as the first section in all methods; revise as appropriate for methods not performed in the laboratory (see next boilerplate):

9.1 Refer to Chapter One for guidance on quality assurance (QA) and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results.

Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged <as described in Sec. X.X>. Use of instrument specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and quality control data should be maintained for reference or inspection.

Sec. 9.1 boilerplate example for non-laboratory methods (e.g., field test kits):

9.1 Follow the manufacturer's instructions for the quality control procedures specific to use of the testing product. Also, refer to Chapter One for additional guidance on quality assurance (QA) and QC protocols that may be applicable. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results.

Organic Determinative Methods Sec. 9.0 Boilerplate

The subsections to follow are examples of what might be included in many organic determinative laboratory methods. These paragraphs may be expanded or revised to meet the needs of the specific determinative method; however, the order of these concepts should be identical or very similar to what follows. Some of the differences in the QC sections may be because of differences in detectors; some may be more susceptible to interferences and less stable than others. If dealing with the same detectors, the QC should be similar. Once Method 8000 is finalized it will be the determining factor for what will ultimately be included in the QC sections of the other 8000 series methods:

PLEASE NOTE: A DISALLOWANCE OF BLANK CORRECTION WAS ADDED TO SEC. 9.5. and should be added to at least the determinative organic and inorganic methods as appropriate.

9.1 General Guidance

Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results.

Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged, as described in Sec. 9.6. Use of instrument specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 or 5000 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 3500, 3600, 5000 or 8000.

9.3 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of retention time windows, calibration verification, and chromatographic analysis of samples.

Note to method editor: Some methods also have method-specific information as subsections to this section. Current applicability of this information can be reviewed as each method is edited.

9.4 Initial demonstration of proficiency (IDP).

The initial demonstration of method proficiency must be performed by the laboratory prior to independently running an analytical method, and should be repeated if other changes occur (e.g., instrument repair, significant change in procedure, and change in analyst). Refer to Method 8000 Sec. 9.0 for additional information regarding instrument, procedure, and analyst IDPs. An IDP must consist of replicate reference samples from each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix taken through the entire preparation and analysis. If an autosampler is used to perform sample dilutions, prior to use, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions.

<Note to method editor: Some methods also have method-specific information about the demonstration as additional sentences or subsections. Current applicability of this information can be reviewed as each method is edited.>

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation, if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

The laboratory should not subtract the results of the method blank from those of any associated samples. Such "blank subtraction" may lead to negative sample results. If the method blank results do not meet the project-specific acceptance criteria and reanalysis is not practical, then the data user should be provided with the sample results, the method blank results, and a discussion of the corrective actions undertaken by the laboratory.

9.6 Sample QC for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch. The addition of surrogates to each field sample and QC sample when surrogates are used is also recommended. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, laboratories may use a matrix spike and a duplicate analysis of an unspiked field sample. If samples are not

expected to contain target analytes, then laboratories should use a matrix spike and matrix spike duplicate pair. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD.

<Note to method editor: Sometimes the above subsection is replaced with other text, as in Method 8261, which states that the surrogate use makes matrix spike samples unnecessary (Method 8261, a GC/MS method, is a special case as far as method handling is concerned -- the method developer included built-in recovery correction); or the subsection contains other method-specific information. These approaches will be reviewed as each method is edited.>

9.6.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample quality control procedures for preparation and analysis. In-house method performance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.7 Lower Limit of Quantitation (LLOQ) check standard

The laboratory must establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. LLOQ verification is recommended for each project application to validate quantitation capability at low analyte concentration levels. This verification may be accomplished by spiking either a clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix, free of target compounds at the LLOQ and processing through all preparation and determinative steps of the method. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated DQOs.

9.7.1 Determination of LLOQs using spiked clean control material represents a best-case scenario and does not evaluate potential matrix effects of real-world samples. For application of LLOQs on a project-specific basis with established DQOs, a representative matrix-specific LLOQ verification may provide a more reliable estimate of the lower quantitation limit capabilities.

9.7.1.1 A LLOQ check standard (not part of an initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at the predicted LLOQ concentration level(s). Alternatively, a representative sample matrix may be spiked with the analytes of interest at the predicted LLOQ concentration levels. The LLOQ check is carried through the same preparation procedures as the environmental samples and other QC.

9.7.1.2 Recovery of target analytes in the LLOQ check standard should be within established in-house limits, or other such project-specific acceptance limits, to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ±20% may be used for the LLOQ acceptance criteria. This acknowledges the poorer overall response at the low end of the calibration curve. Historically-based

acceptance criteria should be determined as soon as practical once sufficient data points have been acquired.

9.7.2 In-house acceptance criteria for recovery of the LLOQ check standard for a particular sample matrix can be calculated when sufficient data points exist. The laboratory should have a documented procedure for establishing in-house acceptance ranges; if the lower limit of the acceptance range is calculated to be <10%, it should be set to 10%. However, an alternative lower acceptance limit may be established by the laboratory or set at the project level through the DQOs in a QAPP.

<Note to method editor: Sometimes other subsections are included, which will be reviewed as each method is edited.>

9.7 Surrogate recoveries

If surrogates are used, the laboratory should evaluate surrogate recovery data from individual samples against the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

9.8 It is recommended that the laboratory adopt additional QA practices for use with this method. Specific practices that are most productive depend upon the needs of the laboratory, the nature of the samples, and project-specific requirements. Field duplicates may be analyzed to assess precision of the environmental measurements. When doubt exists over identification of a peak on the chromatogram, confirmatory techniques such as GC with a dissimilar column, element-specific detector, or mass spectrometer (selected ion monitoring or full scan) must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

Organic Prep Extraction Methods (35XX series) Sec. 9.0 Boilerplate:

Revise the following as appropriate for each method:

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results.

Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged. Use of instrument specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency (IDP) and lower limit of quantitation (LLOQ)

Each laboratory must perform an IDP with each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target

analytes in a clean matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 Sec. 9.0 for information on how to accomplish a demonstration of proficiency.

The laboratory shall establish the LLOQ as the lowest point of quantitation, which in most cases, is the lowest concentration in the calibration curve. LLOQ verification is recommended for each project application to validate quantitation capability at low analyte concentration levels. This verification may be accomplished with either clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix, free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated DQOs.

A LLOQ check standard (not part of an initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at the predicted LLOQ concentration level(s). Alternatively, a representative sample matrix may be spiked with the analytes of interest at the predicted LLOQ concentration levels. The LLOQ check is carried through the same preparation procedures as environmental sample and other QC samples.

Recovery of target analytes in the LLOQ check standard should be within established in-house limits, or other such project-specific acceptance limits, to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% may be used for the LLOQ acceptance criteria. This acknowledges the poorer overall response at the low end of the calibration curve. Historically-based LLOQ acceptance criteria should be determined as soon as practical once sufficient data points have been acquired.

9.3 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination.

9.4 Any method blanks, matrix spike samples, or replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.5 Each extraction batch of twenty or fewer samples should include a minimum of a method blank, a laboratory control sample (LCS), a matrix spike sample, and a matrix spike duplicate or laboratory duplicate sample.

9.6 Standard quality assurance practices should be used with this method as included in appropriate systematic planning documents and laboratory SOPs. All instrument operating conditions should be recorded.

9.7 Also refer to Method 3500 for QC procedures related to extraction and sample preparation and refer to the determinative methods to be used for determinative QC procedures.

9.8 As noted earlier, use of any extraction technique, including <add method-specific technology here>, should be supported by data that demonstrate the performance of the specific solvent system and operating conditions for the analytes of interest, at the levels of interest, in the sample matrix.

9.9 All field and QC samples should be spiked with an appropriate mix of surrogate compounds in order to track extraction efficiency.

9.10 Any QC samples should be subjected to exactly the same analytical procedures as those used on field samples.

Organic Clean-up Methods (36XX series) Sec. 9.0 Boilerplate (most recent update from Method 3620C Feb 2007):

Revise the following as appropriate for each method:

9.1 Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Also refer to Method 3600 for cleanup QC procedures, and Method 8000 and the specific determinative method to be used for information on determinative QC procedures.

9.3 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination.

9.4 The analyst must demonstrate that the compounds of interest are being quantitatively recovered by the cleanup technique before applying this method to actual samples. This test applies to both the column cleanup and cartridge cleanup procedures. A recovery check needs to be performed using standards of the target analytes at known concentration.

9.5 Any method blanks, matrix spike samples, or replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples. For sample extracts that are cleaned up using this method, the associated quality control samples should also be processed through this cleanup method.

Inorganic Method Sec. 9.0 Boilerplate:

The Agency is in the process of developing boilerplate for QC guidelines in the inorganic methods, as applicable. Language for the 30XX series has been completed and follows.

Otherwise, method writers should consider previous boilerplate given above for organic methods, and revise the text as appropriate for the subject inorganic analytical technology.

Remember to include in Sec. 9.0:

9.X The laboratory should not subtract the results of the method blank from those of any associated samples. Such "blank subtraction" may lead to negative sample results. If the method blank results do not meet the project-specific acceptance criteria and reanalysis is not practical, then the data user should be provided with the sample results, the method blank results, and a discussion of the corrective actions undertaken by the laboratory.

Sec. 9.0 boilerplate for sample digestion methods (edit as necessary for other 3000 series methods):

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance (QA) and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results.

Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged <as described in Sec. X.X>. Use of instrument specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency (IDP).

The initial demonstration of method proficiency must be performed prior to independently running an analytical method, and should be repeated if other changes occur (e.g., instrument repair, significant change in procedure, and change in analyst). An IDP must consist of replicate reference samples from each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix taken through the entire preparation and analysis.

9.2.1 Refer to the determinative method for information on preparing replicate reference samples for IDP.

9.3 Sample quality control for preparation and analysis

9.3.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair per analytical batch. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, then laboratories should use a matrix spike and matrix spike duplicate pair. The consideration as to which sample for a given batch is selected for QC analyses should be decided during the project planning process and documented in an approved sampling and analysis plan. The actual sample selected for QC analyses should be representative of the entire matrix composition for a given extraction batch, since data quality assumptions will likely be applied to all batch samples based on compliance to the stated data quality objectives and meeting the recommended precision and accuracy criteria. Therefore, it is inappropriate to combine dissimilar matrices in a single sample preparatory batch and expect to use a single set of QC samples. Sec. 9.3.3 provides guidance on establishing the concentration of the matrix spike compounds in the sample chosen for spiking.

The choice of analytes to be spiked should reflect the analytes of interest for the specific project. Thus, if only a subset of the list of target analytes provided in a determinative method is of interest, then these would be the analytes of interest for the project. In the absence of project-specific analytes of interest, it is suggested that the laboratory periodically change the analytes that are spiked with the goal of obtaining matrix spike data for most, if not all, of the analytes in a given determinative method. If these compounds are not target analytes for a specific project, or if other compounds are known to be of greater concern at a given site, then other matrix spike compounds should be employed.

CAUTION: The utility of the data for the matrix spike compounds depends on the degree to which the spiked compounds mimic the compounds already present in a field sample. Therefore, it is CRITICAL that any compounds added to a sample are added to the sample aliquot PRIOR TO any additional processing steps. It is also CRITICAL that the spiked compounds be in the same chemical form as the target compounds.

9.3.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume: e.g., reagent water for the water matrix or sand or soil for the solid matrix. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

9.3.3 The concentration of the matrix spike sample and/or the LCS should be determined as described in the following sections.

9.3.3.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit or action level, the spike should be at or below the regulatory limit or action level, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher.

9.3.3.2 If historical data are not available, it is suggested that an uncontaminated sample of the same matrix from the site be submitted for matrix

spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries.

9.3.3.3 If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the concentration of the matrix spike should be at the same concentration as the reference sample (Sec. 9.2.4), near the middle of calibration range, or approximately 10 times the quantitation limit in the matrix of interest. It is again suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.

9.3.4 Analyze these QC samples (the LCS and the matrix spikes or the optional matrix duplicates) following the procedures in the determinative method. Calculate and evaluate the QC data as outlined in the determinative method.

9.3.5 Blanks -- The preparation and analysis of method blanks and other blanks are necessary to track potential contamination of samples during the extraction and analysis processes.

9.4 The laboratory must also have procedures for documenting the effect of the matrix on method performance. Refer to each determinative method for specific guidance on developing method performance data.

9.5 Periodically, the accuracy of the temperature measurement system used to control the microwave equipment should be validated per Sec. 6.X.

9.6 **The following step is not necessary if using temperature feedback control.** -- Each day that samples are extracted, the microwave-power calibration should be verified by heating 1 kg of reagent water (at 22 ± 3 °C) in a covered, microwave-transparent vessel for 2 min at the setting for 490 W and measuring the water temperature after heating per Sec. 10.X. If the power calculated (according to Sec. 12.0) differs from 490 W by more than ± 10 W, the microwave settings should be recalibrated according to Sec. 10.0.

9.7 The choice of an acid or acid mixture for digestion will depend on the analytes of interest and no single acid or acid mixture is universally applicable to all analyte groups. Whatever acid or acid mixture is employed, including those specifically listed in this method, the analyst must demonstrate adequate performance for the analytes of interest, at the levels of interest. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

Sec. 9.0 boilerplate for inorganic determination methods (taken from Update V Methods 6010D and 6020B 05/2012)

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance (QA) and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those implementing the project and assess the results.

Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged <as described in Sec. X.X>. Use of instrument specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Methods 3005, 3010, 3015, 3031, 3040, 3050, 3051, 3052, 6800, and 7000 for QC procedures to ensure the proper operation of the various sample preparation techniques. Any more specific QC procedures provided in this determinative method will supersede those noted in the previously stated methods.

9.3 Instrument detection limits (IDLs)

Instrument detection limits are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified according to the guidance in Sec. 9.8. IDLs in µg/L can be estimated as the mean of the blank result plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project. An instrument log book should be kept with the dates and information pertaining to each IDL performed.

9.4 Initial demonstration of proficiency (IDP)

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination by generating data of acceptable precision and bias for target analytes in a clean matrix. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. It is recommended that the laboratory should repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation and/or procedures are made.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment that come into direct contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are digested and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If an interference is observed that would prevent the determination of the target analyte, determine the source and eliminate it, if possible, before processing the samples. The method blank should be carried through all stages of sample preparation and instrument determination procedures. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagent or chemicals prior to sample preparation if the source showed no prior problems.

However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6 Linear range

The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover within 10% of the true value and if successful establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run. If a liner range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.

9.7 Sample QC for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, bias, and sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike (MS), a laboratory control sample (LCS), and a duplicate sample in each analytical batch. Any method blanks, LCS, MS samples, and duplicate samples should be subjected to the same preparatory and instrument determination procedures as those used on actual samples (see Sec. 11.0). Refer to Methods 6010 and 6020 for information on the preparation and analysis of QC samples.

9.8 Lower limit of quantitation (LLOQ) check standard

9.8.1 The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. The LLOQ should be verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be ± 35% of the true value and RSD should be ≤ 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias. Refer to a scientifically valid and published method such as Chapter 9 of Quality Assurance of Chemical Measurements (Taylor 1987) or the Report of the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs (http://water.epa.gov/scitech/methods/cwa/det/index.cfm) for calculating precision and bias for LLOQ.

9.8.2 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than or equal to the desired regulatory action levels based on the stated project-specific requirements.

<Note to method editor: The next section follows the LLOQ section, sometimes not immediately, so take care in fixing the section numbers.>

9.9 If less than acceptable bias and precision data are generated for the matrix spike(s), the additional QC protocols in Sec. 9.9.1 and/or 9.9.2 should be performed prior to reporting concentration data for the elements in this method. At a minimum these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. If matrix interference effects are confirmed, then an alternative test method

should be considered or the current test method modified, so that the analysis is not affected by the same interference. The use of a standard-addition analysis procedure may also be used to compensate for this effect (refer to Method 7000).

9.9.1 Dilution test

If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within ± 20% of the original determination. If not, then a chemical or physical interference effect must be suspected. The matrix spike is often a good choice of sample for the dilution test, since reasonable concentrations of most analytes are present. Elements that fail the dilution test are reported as estimated values.

9.9.2 Post-digestion MS

If a high concentration sample is not available for performing the dilution test, then a post-digestion MS should be performed. The test only needs to be performed for the specific elements that failed original matrix spike limits, and only if the spike concentration added was greater than the concentration determined in the unspiked sample. Following preparation, which may include but is not limited to, pre-filtration, digestion, dilution and filtration, an aliquot, or dilution thereof, should be obtained from the final aqueous, unspiked-analytical sample, and spiked with a known quantity of target elements. The spike addition should be based on the indigenous concentration of each element of interest in the sample. The recovery of the post-digestion MS should fall within a ± 25% acceptance range, relative to the known true value, or otherwise within the laboratory-derived acceptance limits. If the post-digestion MS recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values.

<Note to method editor: The following section should appear later in Sec. 9.0, sometimes second to last or last.>

9.X It is recommended that the laboratory adopt additional QA practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze reference materials and participate in relevant performance evaluation (PE) studies.

10.0 CALIBRATION AND STANDARDIZATION

- If preferred, calibration and standardization information need not be separated from the procedure information in Sec. 11 and put into this section. Instead, simply refer to the text on calibration and standardization found in Sec. 11, Procedure. (At a minimum, this referral approach must be used for the organic methods.) Include the first boilerplate below.

- Use the second boilerplate below for methods to which calibration information does not apply.

- If calibration information will be included in Sec. 10, provide the information listed below. Keep instrument operating conditions in the procedure section (Sec. 11).

    - As appropriate, describe initial calibration procedures, with details on how to do them.

- Indicate acceptance limits for the calibration, or refer back to a base method if appropriate.

- Provide guidance on what to do if the relevant performance criteria are not met.

- As appropriate, describe calibration verification. At the least, include an indication of verification frequency.

Sec. 10.0 boilerplate when calibration information is kept in the procedure section:

See Sec 11.X for information on calibration and standardization.

Sec. 10.0 boilerplate for methods with no applicable calibration information, e.g., sample preparative or cleanup methods:

There are no calibration or standardization steps directly associated with this <sample extraction> procedure.

Sec. 10.0 boilerplate for organic preparation methods, as appropriate (from Method 3545):

There are no calibration or standardization steps directly associated with this sample extraction procedure, other than establishing the extraction conditions in Sec. 11.X.

11.0 PROCEDURE

- Provide detailed step-by-step instructions for using the method. Write in an active voice, as much as possible. Include a description of sample processing and instrumental or physical analysis steps. Include those steps that are essential to the procedure, and avoid unnecessary restrictive instructions.

- Strive for cross-method consistency in the presentation of procedures, especially for similar technologies.

Sec. 11.0 boilerplate (from Method 8041) regarding confirmation (for GC methods only, not necessary for GC/MS methods) -- to be in all non-specific chromatographic methods, including inorganic analyte methods, adapted to the specific method as necessary:

11.X Confirmation

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is necessary when the sample composition is not well characterized. Confirmatory techniques such as gas chromatography with a dissimilar column or a mass spectrometer should be used. See Method 8000 for information on confirmation of tentative identifications.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative result on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

When the dual-column approach is employed, the target phenols are identified and confirmed when they meet the identification criteria on both columns.

Boilerplate for determination of % dry weight boilerplate:

11.X Determination of percent dry weight

When sample results are to be calculated on a dry weight basis, a separate portion of sample for this determination should be weighed out at the same time as the portion used for analytical determination.

CAUTION: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

11.X.1 Immediately after weighing the sample aliquot to be digested, weigh an additional 5- to 10-g aliquot of the sample to the nearest 0.01 g into a tared crucible. Dry this aliquot overnight at 105 °C. Allow to cool in a desiccator before weighing.

11.X.2 Calculate the % dry weight as follows:

$$\%\ \text{dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

This oven-dried aliquot is not used for the extraction and should be appropriately disposed of once the dry weight is determined.

Sec. 11.0 boilerplate regarding retention times given in the method (this may also appear elsewhere in the method if retention times are referenced elsewhere):

11.X The retention times listed in Table X are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

Extract cleanup boilerplate for applicable methods (originally from Method 8081):

11.X Extract cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed, expected interferences coextracted from the sample, and the DQOs for the measurements. General guidance on the selection of cleanup methods is provided in Method 3600.

12.0 DATA ANALYSIS AND CALCULATIONS

- Describe quantitative and qualitative information for deriving final sample results from typical instrumental data. Otherwise (e.g., in organic analyte methods), include a reference to a base method or to Sec. 11.0 for the information (e.g., referral applied to avoid a disruption of the procedural flow).

- Include any of the various boilerplates provided below, as appropriate.

Sec. 12.0 boilerplate for referral to Sec. 11 (In organic methods, always put any data analysis and calculations information in Sec. 11.0 and use a referral in Sec. 12.0 to the appropriate subsection of Sec. 11.0):

See Sec. 11.X and base method <add "and base method" when applicable> for information on data analysis and calculations.

Or simply state:

See Sec. 11.X.

Sec. 12.0 boilerplate regarding units and dilutions:

12.X Results need to be reported in units commensurate with their intended use and all dilutions must be taken into account when computing final results.

In particular, add the following boilerplate to methods that specifically mention sample dilutions (from Method 6020B Update V).

12.X If dilutions were performed, apply the appropriate corrections to the sample values.

Sec. 12.0 boilerplate for methods like the 4XXX series or other field methods that rely on manufacturer's kits/instructions:

See the manufacturer's instructions regarding data analysis and data calculations.

Sec. 12.0 boilerplate for methods with no data calculation steps (e.g., preparative methods):

There are no determinative data analysis and calculation steps directly associated with this procedure. Follow the directions given in the determinative method.

Sec. 12.0 boilerplate approach for the prep methods:

There are no calculations explicitly associated with this <extraction> procedure. See the appropriate determinative method for the calculation of final sample results.

Sec. 12.0 boilerplate for referral to a base method:

See Method 8000 <or whatever is the appropriate base method> for information regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

- Provide summaries regarding sources of performance data examples, with brief descriptions of the studies and the results. Also, include references to the data sources and performance data found in tables of the method. (Important: List all of these data sources in the reference section (Sec. 16.0) and provide complete copies to EPA for the central method file.)

- Clearly indicate that the performance data are examples of what might be achieved and that the data are not intended to be used as acceptance criteria (also add the word "example" to referenced table titles, as appropriate).

- Do not address or include method detection limit discussions or refer to tables of such data anywhere in the method. If necessary or useful, only include a generic description of anticipated method sensitivity for the matrix. The focus should be on spike recovery performance and the calibration range. Also, clearly note that the information is provided as guidance only, and that such limits are highly-matrix dependent and not always achievable. In some cases such as in the immunoassay methods, the values quoted as "MDLs" are real measured values and thus should be left in the method with a change in the terminology.

- Include the boilerplates below, as appropriate.

Sec. 13.0 boilerplate applicable to most methods:

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data does not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. Performance data must not be used as absolute QC acceptance criteria for laboratory QC or accreditation.

Sec. 13.0 boilerplate for end of paragraphs referring to or discussing performance data studies and examples:

This data is provided for guidance purposes only.

Sec. 13.0 boilerplate for preparative methods without performance information:

13.X Refer to the appropriate determinative method for performance data examples and guidance.

Sec. 13.0 boilerplate for field test kits:

13.1 Performance data and related information are provided by the manufacturer in the package insert.

Sec. 13.0 boilerplates for kits that may be used in either the field or the laboratory:

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data does not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. Performance data must not be used as absolute QC acceptance criteria for laboratory QC or accreditation.

13.2 In the case of this method (which may be used in either the field or the laboratory), any test kits used must be able to meet the performance specifications for the intended application. However, required performance criteria for a particular testing product may be included in the manufacturer's instructions.

Sec. 13.0 boilerplate applicable to other methods without performance data (rare):

13.X Performance data examples and guidance for this method currently are not available.

14.0 POLLUTION PREVENTION

- At a minimum, add the boilerplate below regarding EPA's stance on pollution prevention.

- Include any method-specific aspects that minimize or prevent pollution.

Sec. 14.0 boilerplate:

Include as the first two subsections (before finalizing the 4th edition methods, ACS will be directly contacted regarding the long term availability of the publications mentioned in Sec. 14.2 and 15.0, and appropriate changes will be made to the sections to reflect the reply):

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. 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, the Agency recommends recycling as the next best option.

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*, a free publication available from the American Chemical Society (ACS), Committee on Chemical Safety, http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf.

15.0 WASTE MANAGEMENT

- At a minimum, add the boilerplate below regarding EPA's stance on laboratory waste management.

- If necessary, also include any method-specific aspects of laboratory waste management.

Sec. 15.0 boilerplate:

Include as the first paragraph or subsection (if more subsections follow):

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

For kits used in the field, add the following paragraph:

Field waste management procedures must also be consistent with Federal, State and local regulations.

16.0 REFERENCES

- List those method source documents (specifically those documents used as a text source during method writing and development), and publications that document method performance and are directly referenced by the method. This list should not include general educational references regarding the method technology or its analytes, or other such documentation that was not used during method development. If such indirect references are included in the method, list them as a final subsection to Sec. 13, Method Performance, preceded by the statement: "The following documents may provide additional guidance and insight on the performance and application of this method technology: . ."

- List the author(s)'s name(s) first, using the first initial then middle initial separated by a period, followed by the surname and a comma. The title follows the comma. Articles from magazines or journals, and reports and studies are enclosed in quotation marks, while books are not. A comma follows the title within the quotes, then publisher, research institute, or other publishing body's information is listed along with project number, and document date, separated by commas, where applicable. Journal names are written in italics (not applicable to the example given below.) For example:

  T. F. Jenkins, P. G. Thorne and M. E. Walsh, "Field Screening Method for TNT and RDX in Groundwater," US Army Cold Regions Research and Engineering Laboratory, Special Report, Hanover, New Hampshire 03755, 1994.

- All references must have dates. If a report is "in progress" or not yet published, a version must be dated and referenced as such -- and included in the method file. Do not simply put "in progress" or "to be published."

17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

- Directly following the Sec. 17.0 title, add the following brief boilerplate (revised as appropriate): The following pages contain the tables and figures referenced by this method.

  If the method has an appendix or a glossary before or after the tables and figures, include reference to it in the 17.0 boilerplate. For example:

  The following pages contain the appendix, tables, and figures referenced by this method.

- Follow the above statement by a hard page break, followed in turn by the tables and then the figures (with hard page breaks as appropriate). Always place tables before figures. In the event of a "natural" occurrence of a soft page break between tables or figures, replace it with a hard page break

- Include only those tables and figures which were mentioned and briefly described in the method. Place the tables or figures within Sec. 17.0 in the order referenced by the text. Do not include tables or figures within the method at other locations. (The only exceptions to including tables in the text are the table of analytes and the table of additional analytes that may appear in Sec. 1.0. However, those tables are not numbered.)

- Diagrams or figures should only include new or unusual equipment or aspects of the method.

- Completely identify sources of all figures and tabulated data. Do not include copyrighted figures or data; unless permission has been obtained (submit the record of permission to EPA for inclusion in the central method file). Include complete source citations in Sec. 16.0 of the method for the figures and table data.

- Do not include flow diagrams of procedure steps in the organic analysis methods. If preferred, procedural flow diagrams can be included at the end of other methods.

Table formatting

- Except in instances where clarity is compromised, omit table grid lines. Use a double line across the top and bottom of the table. Use a single line between the header row and the rows below. Identify the source of the information using "Data taken from Reference __" as a footnote to the table with proper document number from the references section.

  For example:

Element	10 mL HNO3 Digest	9 mL HNO3 + 3 mL HCL Digest	Total Analyte Concentration
Cd	3.40 ± 0.34	3.62 ± 0.17	3.45 ± 0.22
Ni	45.5 ± 5.9	42.2 ± 3.2	44.1 ± 3.0

  Data taken from Reference 12**.**

  This data is provided for guidance purposes only.

For retention time data table:

This data is provided for guidance purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method. See Method 8000 for additional information on establishing these parameters.

Table titles

- Number tables in a sequential manner. Center the title of each table at the top of the page, separated by a blank line below the top label of table number. Present the title in all capital letters. For example:

TABLE 1

CHROMATOGRAPHIC CONDITIONS FOR
1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLOROPROPANE (DBCP)

- For large tables, the font for the contents of the table (not the title) may be a size smaller than "11" if necessary. However, keep the material readable.

Figure titles

- Designate figures in a sequential manner. Begin with "FIGURE 1" centered at the top of the page, skip a line, and then provide the title of the figure. Present the title in all capital letters. For example:

FIGURE 1

CALIBRATION CURVE FROM A COMPETITIVE IMMUNOASSAY

Figure formatting

- Place “Figure taken from Reference __" somewhere on the figure page as a reference to the source of the figure, with proper reference number included.

- If you are a method developer submitting a method to EPA for consideration as an SW-846 method, do not embed figures in the text. Provide separate electronic copies and clear prints of the figures. The Agency will import the figures into the method documents in the format currently used for SW-846 methods.

ATTACHMENT A

ORDER OF PARTS AND SECTIONS IN SW-846 METHODS

METHOD NUMBER

TITLE

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, FLOW CHARTS, AND VALIDATION DATA

APPENDICES (e.g., GLOSSARY)

ATTACHMENT B

NUMBERING SYSTEM FOR SW-846 METHODS

Method No.	Method Type
0000 Series	Sampling Methods
001x	Air Sampling - Stack - Volatile Organics
002x	Air Sampling - Stack - Semivolatile Organics
003x	Air Sampling - Stack - Volatile Organics
004x	Air Sampling - Stack - Volatile Organics
005x	Air Sampling - Stack - Acid Gases
006x	Air Sampling - Stack - Metals
01xx	Air Sampling - Ambient
1000 Series	Certain Characteristics Methods (Also see 9XXX, e.g., for pH method)
10xx	Ignitability
11xx	Corrosivity
13xx	Extraction/Leaching Procedures
3000 Series	Sample Preparation Methods
30xx	Metals/Inorganics
302x	Mercury Species
311x	Arsenic Species
35xx	Organic Extraction or Dilution
36xx	Extract Cleanup
38xx	Organic Screening
4000 Series	Immunoassay Methods
40xx	Organic Analytes (Screening)
45xx	Metals/Inorganics (Screening)
46xx	Organic Analytes (Assay)
5000 Series	Volatile Organics/Combustion Preparative Methods
50xx	Volatile Organic Preparation/Sample Introduction
505x	Combustion Preparative Methods
6000 Series	Metals/Inorganic Determinative Methods
60xx	ICP Determinative
62xx	X-ray Determinative
65xx	Electrochemical Determinative
68XX	Isotope Dilution and Chromatographic Separation with Mass Spectrometry Detection Determinative
6850	LC/MS or LC/MS/MS for perchlorate
6860	IC/MS or IC/MS/MS for perchlorate
6870	IC/MS or IC/MS/MS for inorganic and organic arsenic species
7000 Series	Individual Metals/Inorganic Determinative Methods (Primarily AA with Some Other Techniques)

Method No.	Method Type
8000 Series	Organic Determinative Methods
80xx	GC Determinative/Various Detectors
81xx	GC Determinative/Various Detectors
82xx	GC Determinative/Mass Spec Detectors
83xx	HPLC Determinative/Various Detectors
832x	HPLC Determinative/Mass Spec Detectors
84xx	IR Determinative
85xx	UV/Vis Determinative
9000 Series	Miscellaneous Analytes and Tests Methods
901x	Cyanide
902x	Organic Halogen
903x	Sulfur Containing Anions
904x	pH
905x	Specific Conductance/Ion Chromatography (Anions) Determinative
906x	Nonspecific Organics (TOC, Phenolics)
907x	Oil and Grease/Chlorine in Used Oil
908x	Cation Exchange Capacity
909x	Land Disposal Restrictions Test
910x	Saturated Hydraulic Conductivity, Saturated Leachate Conductivity and Intrinsic Permeability
913x	Microbiological
92xx	Anions - Nitrate/Chloride
921x	Anions Determinative - Ion-Selective Electrode
93xx	Radionuclides
931x	Radioactivity