LABORATORY DOCUMENTATION REQUIRED FOR DATA EVALUATION

USEPA Region IX
Quality Assurance Office
San Francisco, California

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Appendices

Appendix A: Suggested Summary Forms for Common Organic Methods  A - 1
Suggested Summary Forms for Common Inorganic Methods  A - 2

Appendix B: References  B - 1
1.0 INTRODUCTION

In order for data to be used for decision-making purposes it is essential that it be of known and documented quality. Verification and validation of data requires that appropriate quality assurance and quality control (QA/QC) procedures be followed, and that adequate documentation be included for all data generated both in the laboratory and in the field.

The QA/QC documentation provided by any laboratory, in conjunction with sample results, allows for evaluation of the following indicators of data quality:

- Integrity and stability of samples;
- Instrument performance during sample analysis;
- Possibility of sample contamination;
- Identification and quantitation of analytes;
- Analytical precision; and
- Analytical accuracy.

General laboratory documentation requirements discussed in this document are formatted into two sections, organic and inorganic analyses. These specifications are intended to establish general, analytical documentation requirements that contract and subcontract laboratories should meet when generating data for USEPA Region IX.

However, project or contract requirements may supercede this document. In order to fulfill project specific objectives, laboratories may be required to supply additional documentation. Users should defer to project specific planning documents to determine if they are required to provide any additional information in deliverables.

Questions or comments concerning this document should be directed to Carl Brickner, Jr., USEPA Region IX Quality Assurance Office, at (415) 744-1536 or brickner.carl@epa.gov.
2.0 GENERAL DOCUMENTATION REQUIREMENTS

2.1 Data Package Format

The data package submitted to EPA should consist of five sections:

- Case narrative;
- Chain-of-Custody (COC) documentation;
- Summary of results for environmental samples;
- Summary of QA/QC results; and
- Raw data.

Summaries of data and results may be presented in a Contract Laboratory Program (CLP) type format or any equivalent that supplies the required information as stated below. All laboratory data qualifiers shall be defined in the deliverable.

In cases where the laboratory has varied from established methodologies, they are required to include the Standard Operating Procedures (SOPs) for those methods as an attachment to deliverables. Inclusion of SOPs in deliverables will aid in final review of the data by data reviewers and users.

2.2 Case Narrative

The case narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or their designee. The Case Narrative will consist of the following information:

- Client's sample identification and the corresponding laboratory identification;
- Parameters analyzed for each sample and the methodology used. EPA method numbers should be cited when applicable;
- Whether the holding times were met or exceeded;
- Detailed description of all analytical and/or sample receipt problems encountered;
- Discussion of reasons for any QA/QC sample result exceedences;
- Discussion of any manual integrations; and
- Observations regarding any occurrences which may adversely impact sample integrity or data quality.

2.3 Chain-of-Custody
Legible copies of all Chain-of-Custody forms for each sample shall be submitted in the data package. Copies of any internal laboratory tracking documents should also be included. It is anticipated that Chain-of-Custody forms and/or internal laboratory tracking documents will include the following information:

- Date and time of sampling and shipping;
- Sampler and shipper names and signatures;
- Type of sample (grab or composite);
- Analyses requested;
- Project, site, and sampling station names;
- Date and time of sample receipt;
- Laboratory sample receiver name and signature;
- Observed sample condition at time of receipt;
- Sample and/or cooler temperatures at time of receipt;
- Air bill numbers;
- Custody seal; and
- Sample numbers.
3.0 ORGANIC ANALYSES DOCUMENTATION REQUIREMENTS

3.1 Summary of Environmental Sample Results

The following information is to be included in the summary of sample results for each environmental sample.

- Client's sample identifications and corresponding laboratory identifications;
- Sample collection dates;
- Dates and times of sample extraction and/or analysis;
- Weights or volumes of sample used for extraction and/or analysis;
- Identification of instruments used for analysis;
- Gas Chromatography (GC) column and detector specifications;
- Dilution or concentration factor for the sample;
- Percent Difference between columns, if applicable;
- Percent Moisture or Percent Solids for soil samples;
- Method Detection Limits (MDLs) or sample Quantitation Limits (QLs);
- Analytical results and associated units; and
- Definitions for any laboratory data qualifiers used.

3.2 Summary of QA/QC Sample Results (as applicable)

The following QA/QC sample results shall be presented on QC summary forms. They shall also include the date and time of analysis. Additional summary forms may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

All summary forms shall, at a minimum, include in the header:

- Form Title;
- Site Name;
- Project Identifier (i.e., Case Number/Sample Delivery Group);
- Laboratory Name; and
- Sample Matrix.

3.2.1 Instrument Calibration (for each instrument used)

- GC/MS Tuning, if applicable

Report mass listings, ion abundance criteria, and percent relative abundances. List the instrument
identification (ID) and the date and time of analysis. Ensure that all ion abundances have been appropriately normalized.

- **Initial Calibration**

Report analyte concentrations of initial calibration standards and the date and time of analysis. List the instrument identification (ID), response factors (RF), relative response factors (RRF), or calibration factors (CF), percent relative standard deviation (%RSD), and retention time for each analyte. The initial calibration (IC) report must also include a sample identifier (ID), associated injection volume or quantity of sample analyzed, the acceptance criteria, such as minimum RF values, and associated maximum %RSD values.

- **Continuing Calibration**

Report the concentration of the calibration standard used for the continuing calibration and for the mid-level standard, and the date and time of analysis. List the instrument identification (ID), RF, RRF, CF, percent difference (%D), and retention time for each analyte.

- **Quantitation Limit or Contract Required Detection Limit Verification (if applicable)**

Report results for standards that are used to verify instrument sensitivity. Report the source for the verification standards. Report the concentration for the true value, the concentration found, the percent recovery, and control limits for each analyte analyzed. The date and time of analysis must also be reported.

3.2.2 **Method Blank Analysis**

List environmental samples and QC analyses associated with each method blank. Report concentrations of any analytes found in method blanks above the instrument detection limit.

3.2.3 **Surrogate Standard Recovery (if applicable)**

Report the name and concentration of each surrogate compound added. List percent recoveries of all surrogates
3.2.4 Internal Standard Summary (if applicable)

Report internal standard area counts of the associated calibration standard and retention times, include upper and lower acceptance limits. List internal standard area counts and retention times for all samples, method blanks, matrix spike/matrix spike duplicates and other QC analyses. Include the instrument identification (ID) and the date and time of analysis.

3.2.5 Compound Confirmation (if applicable)

Report retention times of each compound on both columns as well as retention time windows of the associated standard. In addition, report determined concentrations from each column and percent differences between results. List the instrument identification (ID) and the date and time of analysis. A summary should be generated for each sample, including dilutions and reanalyses, blanks, matrix spikes, and matrix spike duplicates.

3.2.6 Peak Resolution Summary (if applicable)

For primary and secondary columns report retention times of any target compounds and/or surrogates that coelute in the standards (ie. the Performance Evaluation Mixture for Contract Laboratory Program pesticides). Calculate and report the percent resolution between each pair of compounds which coelute. Include the instrument identification (ID), column ID, and the date and time of analysis.

3.2.7 Precision and Accuracy (if applicable)

- Matrix spike/matrix spike duplicate (MS/MSD) analysis

Report the name and concentration of each spiking compound. Samples are to be spiked with all specified compounds of potential concern. List sample results, spiked sample results, duplicate spiked sample results, percent recovery (%R) and the relative percent difference (RPD) between the MS and MSD (if applicable). Acceptance criteria that the
laboratory used for the analysis must also be presented.

- Laboratory duplicate analysis

Report the RPD between duplicate analyses, along with the associated acceptance criteria.

- Laboratory QC check sample analysis

Report the name and concentration of each spiking compound. List the QC check sample and duplicate (if applicable) results, percent recovery (%R), and the relative percent difference (RPD), if performed in duplicate. The acceptance criteria that the laboratory used for the analysis must also be presented.

### 3.2.8 Other QC Criteria

- Retention time windows determination

Report the retention time window for each analyte, for both primary and confirmation analyses.

- Compound identification

Report retention times and concentrations of each analyte detected in samples.

- MDL determination

List most recent method detection limits and dates determined.

- Additional method suggested QC parameters (ie. DDT/Endrin breakdown standards).

- Any Performance Evaluation (PE) samples associated with the environmental samples.

### 3.3 Raw Data

Legible copies of the raw data shall be organized systematically, each page shall be numbered, and a table of contents must be included with each package. Raw data for compound identification and quantitation must be sufficient to verify each result.
3.3.1 **Gas Chromatographic (GC) Analyses**

This section shall include legible copies of raw data for the following:

- Environmental samples arranged in sequential order by client sample number, include dilutions and reanalyses;
- Instrument calibrations; and
- QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for both primary and confirmation analyses are to be included.

Raw data for each analysis shall include the following:

- Appropriately scaled chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names). All chromatograms shall be scaled such that individual peaks can be readily resolved from any neighboring peaks;
- Appropriately scaled before and after manual integrations;
- Area print-outs or quantitation reports;
- Instrument analysis logs for each instrument used;
- Sample extraction and clean-up logs;
- Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including surrogates, internal standards, and spike solutions);
- Percent Moisture or Percent Solids for soil samples; and
- GC/MS confirmation, as applicable.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

3.3.2 **Gas Chromatographic / Mass Spectrometric (GC/MS) Analyses**

This section shall include legible copies of raw data for the following:

- Environmental samples arranged in sequential order by client sample number, include dilutions and reanalyses;
• Mass spectrometer tuning and mass calibration (BFB, DFTPP);
• Initial and continuing instrument calibrations; and
• QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

• Appropriately scaled chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names). All chromatograms shall be scaled such that individual peaks can be readily resolved from any neighboring peaks;
• Appropriately scaled before and after manual integrations;
• Ion scans and enhanced spectra of target analytes and tentatively identified compounds (TICs), with the associated best-match spectra;
• Area print-outs and quantitation reports;
• Instrument analysis logs for each instrument used;
• Sample extraction and clean-up logs;
• Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including surrogates, internal standards, and spike solutions); and
• Percent Moisture or Percent Solids for soil samples.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

3.3.3 High Performance Liquid Chromatographic Analyses

This section shall include legible copies of the raw data for the following:

• Environmental samples arranged in sequential order by client sample number, include dilutions and reanalyses;
• Instrument calibrations; and
• QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for both the primary and confirmation analyses are to be included.

Raw data for each analysis shall include the following:
• Appropriately scaled chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names). All chromatograms shall be scaled such that individual peaks can be readily resolved from any neighboring peaks;
• Appropriately scaled before and after manual integrations;
• Area print-outs or quantitation reports;
• Instrument analysis logs for each instrument used;
• Sample extraction and clean-up logs;
• Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including surrogates, internal standards, and spike solutions);
• Dual column confirmation, as applicable;
• Dual detector confirmation, as applicable;
• Wavelength confirmation, as applicable;
• Percent Moisture or Percent Solids for soil samples; and
• GC/MS confirmation, as applicable.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

3.3.4 Immunoassay Analyses

This section shall include legible copies of raw data for the following:

• Environmental samples arranged in sequential order by client sample number, include dilutions and reanalyses and
• QC analyses (i.e., method blanks, Laboratory Control Samples, etc.), as applicable.

Raw data for each analysis shall include the following:

• Copies of logbook pages for analyses that do not provide instrument print-outs and calculations used to derive reported sample concentrations;
• Spectrophotometric, colorimetric, or other measurements for all analyses;
• Sample preparation/extraction logs that include reagents used, standards referenced to standards preparation logs, manufacturer certificates of
analyses for standards, and volumes of reagents, preparation/extraction times, etc.; and

- Manufacturer directions and certification of test kits including expiration dates.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.
4.0 INORGANIC ANALYSES DOCUMENTATION REQUIREMENTS

4.1 Summary of Environmental Sample Results

The following information is to be included in the summary of sample results for each environmental sample.

- Client's sample identifications and corresponding laboratory identifications;
- Sample collection dates;
- Dates and times of sample digestion and/or analysis;
- Weights or volumes of sample used for digestion and/or analysis;
- Identification of instruments and analytical techniques used for analysis;
- Instrument specifications;
- Dilution or concentration factor for the sample;
- Percent Moisture or Percent Solids for soil samples;
- Instrument Detection Limits (IDLs), Method Detection Limits (MDLs), or sample Quantitation Limits (QLs);
- Analytical results and associated units; and
- Definitions for any laboratory data qualifiers used.

4.2 Summary of QA/QC Results

The following QA/QC sample results shall be presented on QC summary forms. They shall also include the date and time of analysis. Additional summary forms may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

All summary forms shall, at a minimum, include in the header:

- Form Title;
- Site Name;
- Project Identifier (i.e., Case Number/Sample Delivery Group);
- Laboratory Name; and
- Sample Matrix.

4.2.1 Instrument Calibration Verification (if applicable)

The order for reporting of calibration verifications for each analyte must follow the chronological order in which the standards were analyzed.

- Initial Calibration Verification
Report the source for the calibration verification standards. Report the concentration for the true value, the concentration found, the percent recovery, and control limits for each element analyzed. The date and time of analysis must also be reported.

- Continuing Calibration Verification

Report the source for calibration verification standards. Report the concentration for the true value, the concentration found, the percent recovery, and control limits for each element analyzed. The date and time of analysis must also be reported.

- Quantitation Limit or Contract Required Detection Limit Verification (if applicable)

Report results for standards that are used to verify instrument sensitivity. Report the source for the verification standards. Report the concentration for the true value, the concentration found, the percent recovery, and control limits for each element analyzed. The date and time of analysis must also be reported.

### 4.2.2 Blank Analysis

Report analyte concentrations above the instrument detection limits found in the initial calibration blanks (ICBs), continuing calibration blanks (CCBs), and in method/preparation blanks. The date and time of analysis must also be reported.

The order for reporting ICB and CCB results for each analyte must follow the chronological order in which the blanks were analyzed.

### 4.2.3 Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)/Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) Interference Check Samples (if applicable)

Identify the source for the interference check samples. For all analytes, spiked and unspiked, report true values, initial results, final results, calculated percent recoveries (%R), and control limits.
4.2.4 **Precision and Accuracy**

- **Matrix Spike (MS) analysis**

  Report concentrations of the unspiked sample result, the spiked sample result and the concentration of the spiking solution added to the predigestion spike for each analyte. Calculate and report the %R and list control limits. If performed in duplicate, provide the %R for the matrix spike duplicate (MSD) and relative percent difference (RPD).

- **Post Digestion Spike Analysis (if applicable)**

  In addition to matrix spikes, post-digestion spikes are often required by the method. Report concentrations of the unspiked sample results, spiked sample results, and the concentration of the spiking solution added. Calculate and report the %R and list control limits.

- **Laboratory Duplicate Analysis**

  Report concentrations of original and duplicate sample results. Calculate and report the RPD and list control limits.

- **Laboratory Control Sample**

  Identify the source for the laboratory control sample. Report the found concentration of the laboratory control sample and the true concentration for all analytes. Calculate and report the %R and list control limits.

4.2.5 **Other QC Criteria** (if applicable)

- **Method of Standard Additions (MSA)**

  This summary must be included when MSA analyses are required. Report absorbance values with corresponding concentration values. Report the final analyte concentration and list the associated correlation coefficient and control limits.

- **ICP-AES/ICP-MS Serial Dilution**

  Report initial and serial dilution results, associated %D, and control limits.
ICP-AES/ICP-MS Linear Dynamic Ranges

For each instrument and wavelength used, report the date on which linear ranges were established, the integration time, and the upper limit concentration.

ICP-AES Inter-element Correction (IEC) Factors

For each instrument and wavelength used, report the date on which inter-element correction factors were determined. List inter-element correction factors for Al, Ca, Fe, Mg and any other elements for which they have been determined. Include analytes affected and wavelengths to which IECs are applied.

ICP-MS Tune

For each instrument used, report elements tuned to, element masses, average of measured masses (in AMU), average peak widths at method required peak heights (in AMU), percent relative standard deviations, date, and time determined.

ICP-MS Internal Standards Relative Intensity Summary

For all samples, report client's sample identifications, percent relative intensities for all internal standards in samples, dates, and times determined.

IDL determination

List most recent IDLs for all analytes, methods used, and dates determined.

Any Performance Evaluation (PE) samples associated with the environmental samples.

4.3 Raw data

Legible copies of the raw data shall be organized systematically, each page shall be numbered, and a table of contents must be included with each package. Data should be organized sequentially by method and analysis date. Raw data for compound identification and quantitation must be sufficient to verify each result.

4.3.1 Inductively Coupled Plasma Atomic Emission Spectrometric (ICP-AES) Analyses
This section shall include legible copies of raw data for the following:

- Environmental sample results, include dilutions and reanalyses;
- Instrument calibrations; and
- QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

- Measurement print-outs for all instruments used;
- Emission intensities, absorbance units, or other measurements for all analyses;
- Sample preparation and digestion logs that include reagents used, standards referenced to standards preparation logs, volumes of reagents, digestion times, etc.;
- Instrument analysis logs for each instrument used;
- Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including internal standards and spike solutions);
- Wavelengths used for the analyses; and
- Percent Moisture or Percent Solids for soil samples.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

4.3.2 Inductively Coupled Plasma Mass Spectrometric (ICP-MS) Analyses

This section shall include legible copies of raw data for the following:

- Environmental sample results, include dilutions and reanalyses;
- Instrument calibrations; and
- QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

- Measurement print-outs for all instruments used;
- Emission intensities, area print-outs, absorbance units, or other measurements for all analyses;
Sample preparation and digestion logs that include reagents used, standards referenced to standards preparation logs, volumes of reagents, digestion times, etc.;

- Instrument analysis logs for each instrument used;
- Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including internal standards and spike solutions);
- Masses reported from or monitored during the analyses;
- Mass calibrations, resolution checks, instrument tunes, instrument stability checks, and/or any other precalibration routine data; and
- Elemental equations used for data calculations.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

### 4.3.3 Atomic Absorption (AA) and Atomic Emission (AE) Spectrometric Analyses

This section shall include legible copies of raw data for the following:

- Environmental sample results, include dilutions and reanalyses;
- Instrument calibrations; and
- QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

- Measurement print-outs for all instruments used or copies of logbook pages for analyses that do not provide instrument print-outs;
- Absorbance units, emission intensities, or other measurements for all analyses;
- Sample preparation and digestion logs that include reagents used, standards referenced to standards preparation logs, volumes of reagents, digestion times, etc.;
- Instrument analysis logs for each instrument used;
- Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including spike solutions);
Wavelengths used for the analyses; and
Percent Moisture or Percent Solids for soil samples.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

### 4.3.4 Ion Chromatographic (IC) Analyses

This section shall include legible copies of raw data for the following:

- Environmental sample results, include dilutions and reanalyses;
- Instrument calibrations; and
- QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

- Appropriately scaled chromatograms (label all analyte peaks with chemical names). All chromatograms shall be scaled such that individual peaks can be readily resolved from any neighboring peaks;
- Appropriately scaled before and after manual integrations;
- Area print-outs or quantitation reports;
- Instrument analysis logs for each instrument used;
- Sample preparation/extraction and clean-up logs;
- Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including spike solutions); and
- Dual column confirmation, as applicable.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

### 4.3.5 Titrimetric and Colorimetric Analyses

This section shall include legible copies of raw data for the following:

- Environmental sample results, include dilutions and reanalyses;
- Calibrations; and
• QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

• Copies of logbook pages for analyses that do not provide instrument print-outs and calculations used to derive reported sample concentrations;
• Titrant volumes, titration end-points, absorbance units, or other measurements for all analyses;
• Sample preparation and digestion logs that include reagents used, standards referenced to standards preparation logs, volumes of reagents, digestion times, sample volumes, solution normalities, etc.;
• Standards preparation logs and manufacturer certificates of analyses for standards, if applicable, sufficient to document traceability of all standards (including spike solutions); and
• Wavelengths used for the analyses.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.

4.3.6 Gravimetric Analyses

This section shall include legible copies of raw data for the following:

• Environmental sample results, include dilutions and reanalyses;
• Calibrations; and
• QC analyses (i.e., method blanks, Laboratory Control Samples, etc.).

Raw data for each analysis shall include the following:

• Copies of logbook pages for analyses that do not provide instrument print-outs and calculations used to derive reported sample concentrations;
• Weights, sample volumes, or other measurements for all analyses;
• Sample preparation and digestion logs that include reagents used, standards referenced to standards preparation logs, volumes of reagents, drying times, drying temperatures, etc.; and
• Standards preparation logs and manufacturer certificates of analyses for standards, if
applicable, sufficient to document traceability of all standards.

Note: Additional raw data may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.
# APPENDIX A

**SUGGESTED SUMMARY FORMS FOR COMMON ORGANIC METHODS**

<table>
<thead>
<tr>
<th>Method Numbers</th>
<th>Sample Results</th>
<th>GC/MS Tuning</th>
<th>Initial Calibration</th>
<th>Continuing Calibration</th>
<th>Peak Resolution, as applicable</th>
<th>Method Blank</th>
<th>Surrogate Standard, as applicable</th>
<th>Internal Standard, as applicable</th>
<th>Matrix Spike and Matrix Spike Duplicate, as applicable</th>
<th>Laboratory Duplicate, as applicable</th>
<th>Laboratory QC Check Sample, as applicable</th>
<th>Retention Time Windows</th>
<th>Compound Confirmation, as applicable</th>
<th>MDL Determination</th>
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*Note: Additional summary forms may be required for some methods. Therefore, when reporting data, laboratories should defer to specific method requirements.*
## APPENDIX A

**SUGGESTED SUMMARY FORMS FOR COMMON INORGANIC METHODS**

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<th>Sample Results</th>
<th>Initial and Continuing Calibration Verification</th>
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<th>Matrix Spike</th>
<th>Post Digestion Spike, as applicable</th>
<th>Laboratory Duplicate</th>
<th>Laboratory Control Sample</th>
<th>Method of Standard Additions, as applicable</th>
<th>ICP Serial Dilution, as applicable</th>
<th>ICP Linear Ranges</th>
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APPENDIX B – REFERENCES


