ACTIVITY/PROJECT SPECIFIC PROCEDURE

SP 12-9
CALIBRATION, USE, AND MAINTENANCE OF THE PERKIN ELMER OPTIMA 8300 DUAL VIEW (DV) INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY (ICP-AES)
Revision 6

Effective Date: September 29, 2020

Author: Heather Burton
Original signed by Heather Burton 9-29-2020

1.0 Purpose and Scope

This procedure prescribes the Sandia National Laboratories (SNL) Waste Isolation Pilot Plant (WIPP) process for the calibration, operation, and maintenance of the Perkin Elmer Dual View Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) (Model 8300 Optima DV) as part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project.

This procedure is applicable only for the Perkin Elmer ICP-AES. This document is concise, and is not meant to substitute for, or serve as a reproduction of the manufacturers’ instruction manuals for the ICP-AES (i.e., Hardware Assistance: Perkin Elmer, LLC, March, 2014. Software assistance: Syngistix guide May 2016 version 2). The user is responsible for reading and understanding the appropriate manuals (see references).

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

1.1 Instrument Description

When electrons are excited, they emit energy at specific wavelengths, in this way each element generates an individual wavelength signature. The energy emitted by an element is proportional to the concentration of the element in the solution. In many cases, elements can emit energies at multiple wavelengths. In this case the wavelength with the highest intensity/abundance should be chosen for analysis.

The ICP-AES is composed of two major components: the RF generator and the spectrometer. Samples must be in liquid form for analysis on the ICP-AES. The liquid sample is introduced into the nebulizer by a peristaltic pump. Humidified Argon gas is injected into the nebulizer along with the sample. This Argon/sample mist is then introduced into the torch assembly and the plasma flame. The radio frequency of the RF generator and the plasma work together to excite the introduced electrons of the analytes; as the electrons return to ground state they will emit photons of light at specific wavelengths. The spectrometer uses a grating to separate the light photons into a photomultiplier tube. The intensity of the light signifies the concentration of the element. The photomultiplier tube will convert the light
 photons into electrical signals. The Perkin Elmer Syngistix computer program can then convert the electrical signals produced at the specific wavelength to concentration when compared against a standard curve.

### 2.0 Implementation Actions

#### 2.1 Safety

The activities described in this SP shall conform with SNL Environmental Safety and Health programs (ES&H). This document does not address ES&H issues. Laboratory ES&H procedures and specific instrument hazards are described in laboratory-specific TWDs and JSAs, which can be found online.

#### 2.2 Responsibility

The Principal Investigator (PI), or designee, whose activities warrant the use of this procedure, is responsible for implementing the requirements of this procedure.

The Laboratory Technician, or designee, is responsible for performing the calibrations and measurements following the requirements of this procedure, documenting calibrations, and assuring that the latest revision of this document is followed.

#### 2.3 Controls

Controls are established by written procedures or instructions prepared in accordance with NP 5-1, Implementing Procedures, of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with NP 6-2, Document Control Process, of the Sandia National Laboratories WIPP Quality Assurance Program.

#### 2.4 Standards

Freshly-made standards should be prepared, as needed (not exceeding the expiration date), with the realization that concentration can change with age. The standards used for calibration and calibration checks, termed as derivative standards in this SP, are recommended to contain 0.2% of nitric acid (HNO₃) and a calculated amount of the primary standard using the following equation.

\[
C_1V_1 = C_2V_2
\]

Where:
- \(C_1\) is the Concentration (start) and \(V_1\) is the Volume (start).
- \(C_2\) is the Concentration (final) and \(V_2\) is the Volume (final).

The primary standards will be commercially obtained and traceable to NIST or other nationally recognized standards, and the certificates of the primary standards provided by the manufacturer will be submitted to the WIPP Record Center upon receipt by the person in charge of procurement of chemicals. The derivative standards shall be documented in the Scientific Notebook in accordance with NP 13-1, Control of Samples and Standards, and NP 20-2, Scientific Notebooks (see Appendix A). The primary standards will not be used past the expiration date listed on the container by the manufacturer. This also applies to the derivative standards and internal standard.
2.5 Quality Control

If the results of the calibration and QC samples are not within the acceptance/tolerance limits (described in subsequent sections), all samples shall be flagged on the data report and in the scientific notebook, and corrective action will be documented in the scientific notebook.

2.6 Calibration

Standard Curve

A calibration curve must be analyzed before each new analyte, with a minimum of a blank and three standards, which cover the range of expected measurements

- The linearity of the calibration curve should be better than or equal to 0.995 in terms of squares of linear correlation coefficients (R²).
  - If any sample concentration exceeds the highest concentration of the calibration standards, it must be diluted and re-analyzed. Alternatively, a new calibration including a standard to bracket the concentration of the sample will be performed and the sample re-analyzed. In the case that a sample is already used up when the concentration of the sample is higher than that of the highest concentration of the calibration standards, it is impossible to re-analyze the sample. In such cases, if the concentration of the sample is within 20% of the highest concentration of the calibration standards, the results are usable based on the fact that ICP-AES calibration curves are linear over several orders of magnitude in concentration. Otherwise, the results will not be used.

Recommended wavelengths for some elements are listed in Appendix B, these detection limits and wavelengths are for reference only. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Other recommended wavelengths are also listed in the Syngistix software.

Blank

A blank should be prepared by using ultra pure de-ionized water and 0.2% acid. A blank will be analyzed at the start of every standard curve, beginning of the run, end of the run, and every 10 samples.

NOTE: The majority of elemental analysis will follow a linear trend. However, some of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows it; however, the effective range must be checked and the second order curve fit should have a correlation coefficient of 0.995 or better. Third order fits are not acceptable.

2.7 QC Sample Criteria

- A calibration check standard and blank will be analyzed as samples immediately after the calibration curve. This calibration check standard and blank are standards used for establishing the calibration curve. These standards will be analyzed after every ten samples, and at the end of the analytical run. Results of check standards should agree with their respective expected values within 10%.
- The concentration of the blank should be lower than that of the lowest calibration standard.
- A BEC (Background Equivalent Concentration) test must be conducted before analysis and must be lower than 0.048 mg/L of the chosen analyte. (NOTE: the BEC test should always be negative)
- The concentration of the internal standard in samples and check standards should be within 30% of that of the internal standard target value which is set by the blank.
2.8 Instrument Start-up

1. Turn on the exhaust fan for the ICP-AES.
2. Check if there is enough liquid argon in the liquid Argon dewar. The pressure for argon should be between 80 and 120 psi.
3. Check if the recirculator for the ICP-AES is working properly (Temperature should be 15 oC and pressure should be 55 psi). If the recirculator is not working properly, do not use the ICP-AES. The manufacturer, Perkin Elmer, LLC, should be notified of the problem with the recirculator. The service contract of Sandia National Laboratories with Perkin Elmer, LLC covers the recirculator.
4. Check if there is enough rinse solution (DI water with 2% HNO₃) for rinsing.
5. Check if there is enough scandium internal standard.
6. Set the Argon regulator pressure to 80 PSI.

7. Click the Syngistix icon.

8. Click the ESI icon (this program runs the auto sampler).

9. Initialize the system by clicking the Diagnostics button on the Instrument Tab, then click Initialize.

10. Wait the specified time to warm-up indicated by the instrument software. Ensure that the auto sampler is connected to the instrument. If it did not, you may need to click the Instrument Tab/Devices/Initialize.

11. Ensure that there is proper flow in all of the lines to the nebulizer, including the Internal Standard line (if configured).

12. Check the pump tubing for the peristaltic pump. If the tubing is flattened, stretched out or damaged, replace it.

13. Turn on the plasma by clicking the plasma on button in the plasma control window.

14. Wait at least 45 minutes for the plasma to stabilize.

15. Perform Hg alignment: Under Instrument Tab/Spectrometer Control, click Hg Alignment. This is performed using only the rinse solution.

16. Perform radial view alignment: First click Radial View, then put the sample probe into 10 mg/L Mn standard solution, and then click Align View button. Then, rinse sample tube in rinse solution for approximately 1 minute.

17. Perform axial view alignment: First click Axial View, then put the sample probe into 1.0 mg/L Mn standard solution, and then click Align View button.

18. Click the Analysis Tab.

- Do Background Equivalent Concentration (BEC) test: Click Analysis Tab/Analysis Button. A new window will open containing a tab for manual operation or automatic operation. Click the Manual Tab.
- On the Analysis Tab, under Sample Info section click open. Open the BEC file.
- On the Analysis Tab, under Method, click open. Open the BEC Method file. On the previous window click Analyze Blank, which is the rinse solution (DI water with 2% HNO₃).
• Then, put the sample probe into 1.0 mg/L Mn standard solution, allow the solution to flow for ~1 minute and then click Analyze Standard.
• After the analysis for the Mn standard solution is finished, put the sample probe back into the rinse solution. Then, close the shutter (in Spectrometer Control panel), click Analyze Sample.
• After the analysis for the rinse solution is complete, put the mode of shutter into Auto. Click the exit (X) button to close Manual Operation mode. (This will automatically send the BEC test to the printer).

To ensure that the BEC test passed the QC criteria, go to Results Tab then Results to check the BEC value. The BEC value is defined as absolute value (however the BEC should always be negative). The BEC value should be lower than 0.048 mg/L Mn. This is the value recommended by the manufacturer. Record the BEC value in the ICP-AES log book. If the BEC value is higher than 0.048 mg/L Mn, repeat the BEC test until the BEC value is lower than 0.048 mg/L Mn.

19. On the Analysis Tab under sample Info enter sample names in a Sample Information File (.sif extension).
20. On the Analysis Tab under Method, Open the desired analyte method.
21. Click Analysis button and then the Automated Tab. Click the icon for saving the Results Data Set. Update the information with the Analyte to be ran, the date and initials of operator if desired.
22. Click Calibration to do the calibration. If the calibration meets the above calibration criteria (i.e., the linearity of the calibration curve should be 0.995 or better in terms of squares of linear correlation coefficients, $R^2$), proceed with the sample analysis.
23. Click Analyze Samples to analyze all samples. (NOTE: Before you click Analyze samples, ensure that the sample information is in a saved sample information file, including all check and blank positions.)

2.9 Instrument Shut-down

After the analysis is completed, do the following for shutting-down the instrument:

1. Click the Instrument Tab then the scheduler button.
2. A new window will open. Click Immediately on OK.
3. Click OK. (This will flush the sample introduction system, will extinguish the plasma and will remove the sample probe from the rinse solution.)
4. For long term shut-down Exit the software. If scheduling the instrument to initialize the next morning, leave the software open.
5. Release the clamp lever of the peristaltic pump and the tubing. This will increase the lifetime of the pump tubing.
6. For long term shut-down, turn down the Argon regulator pressure to ~40 PSI.
7. Turn off the exhaust fan for the ICP-AES.

Alternately, you can schedule the instrument to warm-up for the next analysis by the following:

1. Click the Instrument Tab, then the scheduler button.
2. A new window will open. Click Immediately on OK.
3. Click on Start-up and chose the day and time you’d like. (DO NOT click the Turn on Plasma and Pump button.)
4. Click OK. (This will flush the sample introduction system, will extinguish the plasma and will remove the sample probe from the rinse solution.)
5. Release the clamp lever of the peristaltic pump and tubing.
6. Make sure the plasma has been extinguished, then turn off the exhaust fan for the ICP-AES.

2.10 Sample Preparation

It is assumed that the user has at least basic knowledge of wet chemistry and knows how to use volumetric flasks to prepare and dilute samples. To prevent the sample introduction system from clogging, and to prevent the instrument from damage, samples with ionic strength ranging from 1.0 m to 5.0 m shall be diluted at least ten times. Samples with higher ionic strength shall be diluted at least twenty times.

The instrument is configured to continuously flow the internal standard solution in line with the peristaltic pump using a “T” adapter. With this, it is not necessary to prepare samples and standards with internal standard in the sample itself. The internal standard is recommended to be a 10 ppm scandium solution containing 0.2% HNO₃. Scandium counts must be 500,000 ± 100,000.

Use the following equation to calculate the desired amount of the primary scandium internal standard.

\[ C_1V_1 = C_2V_2 \]

\[ C_1V_1 = \text{Concentration (start) and Volume (start)} \]
\[ C_2V_2 = \text{Concentration (final) and Volume (final)} \]

**WARNING:** Many of the metal salts (e.g., arsenic, lead) are particularly hazardous substances. The primary route of exposure is ingestion since the salts are in solution. Wash hands thoroughly after handling.

2.11 Data Collection

Hard copies of the data collection and calibration records will be submitted to the WIPP Record Center (see Section 3 Records).

2.12 Maintenance

Please refer to Perkin Elmer, LLC, 2014.

2.13 References

- EPA Method 6010C, Revision 3, November 2000. *Inductively Coupled Plasma Atomic Emission Spectrometry*

3.0 Records

The following records, generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

**QA Record**
- Data printouts
- Scientific Notebook
4.0 Appendices

Appendix A: Form SP 12-9-1, Record of Preparation of ICP-AES Derivative Calibration Standards
Appendix B: Reference Wavelengths and Detection Limits
## Record of Preparation of ICP-AES Derivative Calibration Standards

<table>
<thead>
<tr>
<th>Element</th>
<th>Volume &amp; Concentration of Primary/Derivative Stock Standard</th>
<th>Volume of acid (HNO₃) mL</th>
<th>Final Volume mL</th>
<th>Final Concentration, mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Standard Name</th>
<th>Manufacturer/Reference</th>
<th>Lot#</th>
<th>Expiration Date</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Prepared by: ____________________________
Date Prepared: __________________________
Expiration Date: ________________________

**NOTE:** Label prepared standard with:
- Date prepared
- Initials of preparer
- SN identifier and page number where this preparation is recorded
- Expiration date if applicable
## Appendix B

### Reference Wavelengths and Detection Limits

**Recommended Wavelengths and Estimated Instrumental Detection Limits**

<table>
<thead>
<tr>
<th>Element</th>
<th>Wavelength (^a) (nm)</th>
<th>Estimated IDL (^b) (µg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>308.215</td>
<td>30</td>
</tr>
<tr>
<td>Antimony</td>
<td>206.833</td>
<td>21</td>
</tr>
<tr>
<td>Arsenic</td>
<td>193.696</td>
<td>35</td>
</tr>
<tr>
<td>Barium</td>
<td>455.403</td>
<td>0.87</td>
</tr>
<tr>
<td>Beryllium</td>
<td>313.042</td>
<td>0.18</td>
</tr>
<tr>
<td>Boron</td>
<td>249.678 (\times 2)</td>
<td>3.8</td>
</tr>
<tr>
<td>Cadmium</td>
<td>226.502</td>
<td>2.3</td>
</tr>
<tr>
<td>Calcium</td>
<td>317.933</td>
<td>6.7</td>
</tr>
<tr>
<td>Chromium</td>
<td>267.716</td>
<td>4.7</td>
</tr>
<tr>
<td>Cobalt</td>
<td>228.616</td>
<td>4.7</td>
</tr>
<tr>
<td>Copper</td>
<td>324.754</td>
<td>3.6</td>
</tr>
<tr>
<td>Iron</td>
<td>259.940</td>
<td>4.1</td>
</tr>
<tr>
<td>Lead</td>
<td>220.353</td>
<td>28</td>
</tr>
<tr>
<td>Lithium</td>
<td>670.784</td>
<td>2.8</td>
</tr>
<tr>
<td>Magnesium</td>
<td>279.079</td>
<td>20</td>
</tr>
<tr>
<td>Manganese</td>
<td>257.610</td>
<td>0.93</td>
</tr>
<tr>
<td>Mercury</td>
<td>194.227 (\times 2)</td>
<td>17</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>202.030</td>
<td>5.3</td>
</tr>
<tr>
<td>Nickel</td>
<td>231.604 (\times 2)</td>
<td>10</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>213.618</td>
<td>51</td>
</tr>
<tr>
<td>Potassium</td>
<td>766.491</td>
<td>See note c</td>
</tr>
<tr>
<td>Selenium</td>
<td>196.026</td>
<td>50</td>
</tr>
<tr>
<td>Silica (SiO(_2))</td>
<td>251.611</td>
<td>17</td>
</tr>
<tr>
<td>Silver</td>
<td>328.068</td>
<td>4.7</td>
</tr>
<tr>
<td>Sodium</td>
<td>588.995</td>
<td>19</td>
</tr>
<tr>
<td>Strontium</td>
<td>407.771</td>
<td>0.28</td>
</tr>
<tr>
<td>Thallium</td>
<td>190.864</td>
<td>27</td>
</tr>
<tr>
<td>Tin</td>
<td>189.980 (\times 2)</td>
<td>17</td>
</tr>
<tr>
<td>Titanium</td>
<td>334.941</td>
<td>5.0</td>
</tr>
<tr>
<td>Vanadium</td>
<td>292.402</td>
<td>5.0</td>
</tr>
<tr>
<td>Zinc</td>
<td>213.856 (\times 2)</td>
<td>1.2</td>
</tr>
</tbody>
</table>

---

Taken from EPA Method 6010C, Revision 3, November 2000. *Inductively Coupled Plasma Atomic Emission Spectrometry*
This work of authorship was prepared as an account of work sponsored by an agency of the United States Government. Accordingly, the United States Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so for United States Government purposes. Neither the National Technology and Engineering Solutions of Sandia, LLC., the United States Government, nor any agency thereof, nor any of their employees makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately-owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the National Technology and Engineering Solutions of Sandia, LLC., the United States Government, or any agency thereof. The views and opinions expressed herein do not necessarily state or reflect those of the National Technology and Engineering Solutions of Sandia, LLC., the United States Government or any agency thereof.


Parties are allowed to download copies at no cost for internal use within your organization only provided that any copies made are true and accurate. Copies must include a statement acknowledging Sandia's authorship of the subject matter.