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 NexIon 300D Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) as part of the laboratory geochemistry research activities in the support of Waste Isolation Pilot Plant (WIPP) projects.

This procedure is applicable only for the Perkin Elmer NexIon 300D ICP-MS. This document is concise, and is not meant to substitute for, or be a reproduction of the manufacturers’ instruction manuals for the ICP-MS. 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

The NexIon 300D ICP-MS is equipped with a collision cell and reaction cell to allow for lower detection limits and less polyatomic ion interference. The NexIon 300D is equipped with a Quadrupole Ion Deflector (QID), which filters the ions and focuses the ion beam, and also a Universal cell chamber that is specially designed to allow three different operating modes. The setup of these internal components is shown in Figure 1.

The NexIon has 3 operating modes: Standard, Collision (with Kinetic Energy Discrimination, KED) and Reaction Mode with Dynamic Reaction Cell, DRC (see Figure 2). The Standard Mode is used in the same way that normal ICP-MS instrument modes are and almost all ions are allowed to be processed by the instrument. The Collision Mode utilizes helium gas and is recommended for samples with low to medium interference (polyatomic and other). The Reaction Mode utilizes a reactive gas (Ammonia) and is recommended for samples with medium to high interferences.
Figure 1. *Internal components of the NexIon 300D ICP-MS.*

Figure 2. *Details of the three operating modes available on the NexIon 300D ICP-MS.*
The sample will be taken up by the auto sampler and processed by the in-line nebulizer. The nebulizer will sort the large droplets from the small droplets and only allow the small droplets through the spray chamber. The small droplets will be converted into a fine aerosol in the spray chamber. The sample aerosol will pass into the torch and through the argon plasma. The plasma will dry, atomize and ionize the sample. The sample will then enter a series of cones as shown in Figure 3 (Sampler cone, Skimmer cone, Hyperskimmer cone) which will filter out the majority of the photons, neutral and negative ions while also directing and narrowing the ion beam. Positively charged ions (and the remaining un-ionized material) will travel through the cones and enter the QID.

![Figure 3. Details of the unique Triple Cone Interface.](image)

In addition, these cones help to keep the instrument cleaner. After moving through the series of cones, the sample will enter the QID. The QID will turn the positively charged ions 90 degrees focusing the ion beam into the Universal Cell (see Figure 4). The Un-ionized material will continue in a straight line and be removed completely from the system.

![Figure 4. The Quadrupole Ion Deflector (QID) turns ions 90°, filtering off all neutrals for greater stability and consistency.](image)

The NexIon 300D Universal Cell can be operated in 3 different modes, as mentioned earlier.

**Standard Mode:**
- High Through-put
- Operates like a non-cell instrument
- For elements not requiring interference correction

**Collision Mode:**
- Uses non-reactive gas (Helium)
- Separates smaller analyte ions from larger interfering polyatomic ions
- For samples with low-moderate interferences

**Dynamic Reaction Mode:**
- For samples with high interferences
- Uses highly reactive gas (Ammonia) and removes interferences through chemical reactions
- Helps minimize formation of new polyatomic ions
The NexIon 300D is also equipped with a specialized Dual-Mode Detector. The detector can be operated in dual mode, pulse mode or analog mode. Pulse mode should be used if the analyte intensity to be measured is below 2 million cps. Analog should be used if sample analyte intensity is above 2 million cps. The decision as to which run mode to use is at the discretion of the investigator based upon the type of analysis to be conducted.

2.0 Implementation Actions

2.1 Safety

The activities described in this SP shall conform to 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 at the listed website: http://tdb.sandia.gov/WIPP_ESHDocs/

Working with Ammonia gas can be dangerous. When disconnecting the Ammonia tank, the checklist in Appendix B must be followed. This procedure should be done with one person performing the actions and one person checking off that the action has been completed. This will help to ensure a safe work environment while conducting this task.


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, will be prepared from the primary standards by using a few percentages of Optima Grade nitric acid (HNO₃) or HCl and a known concentration of an internal standard that is similar to the mass of the ion being measured. Multiple internal standards can also be added to one sample that cover a range of molecular masses (e.g., low amu, medium amu, large amu). The internal standards chosen should not be the same element that is being analyzed and should not be present in the sample. 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 Records 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 standards will not be used past the expiration date listed on the container by the manufacturer.

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 corrective action will be documented in the scientific notebook. The Blank, Calibration and QC samples must be within tolerance limits designated by this SP and in accordance with NP 16-1, Corrective Action.

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 0.995 in terms of squares of linear correlation coefficients ($R^2$).

  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-MS calibration curves are linear over several orders of magnitude in concentration. Otherwise, the results will not be used.

**BLANK**

The blank is considered acceptable if: the blank is less than the lowest calibration standard, or the lowest sample concentration measured is more than 5 times the measured blank concentration.

A blank will be analyzed at the start of the run, the end of the run and every 10 samples.

2.7 QC Sample Criteria

**CHECK STANDARD**

A calibration check standard will be analyzed as a sample immediately after the calibration. This calibration check standard is one of the standards used for establishing the calibration curve.

A check standard will be analyzed at the start of the run, the end of the run and every 10 samples.

- Results of standards should agree with their respective expected values within 10%. However, for check standards in the parts per trillion (ppt) range the results of standard checks should agree with their expected values to within 15%.
- A passing check standard should be present at the start of the run, the end of the run and every 10 samples.
• A Smart Tune Daily Performance test must be conducted before analysis and must pass all of
the specified criteria (see Section 2.10.1 for S10 autosampler and 2.10.2 for the seaFAST
autosampler)

**INTERNAL STANDARDS**

• The concentration of the internal standard in samples and check standards should be within
30% of the expected value.
• Each internal standard should be grouped in the NexIon software as described in the Method
Development section 2.11.
• It is suggested that the user prepare a stock of all the internal standards to be used at a
workable concentration (~1000 ppb) and then pipette the stock into all of the standards and
samples to be analyzed. (A pre-mixed solution at the proper concentration containing a range
of internal standards can be purchased if desired)

NOTE: A concentration of internal standard should be used to ensure an intensity of greater than
1 million counts (cps) +/- 500,000 (i.e., 10 ppb for Indium).

### 2.8 Sample and Solution 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, total dissolved solids (TDS) concentration
should be below 0.5% by weight (~5000 mg/L). Samples should be diluted accordingly.

**seaFAST**- If utilizing the seaFAST autosampler in preconcentration mode, extensive dilution rates are
not necessary. The seaFAST autosampler is designed to remove high matrix concentrations. Some
dilution may still be necessary, however. Use the lowest dilution possible to achieve the desired
analyte concentrations.

It is recommended that at least 10 mL of sample be prepared. Samples should be matrix-matched by
adding internal standards and the same acid that is introduced to calibration standards for
preservation purpose (usually Optima Grade HNO₃ or HCl).

**seaFAST**- It is not necessary to matrix match the samples when using the seaFAST autosampler.
The internal standard is added to the sample by the autosampler through the Eluent IS. See the
document: **SC-DX seaFAST S2 Method Guide PerkinElmer-NexIon** for more information on this.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after
handling.

**seaFAST Solution Preparation:**

**Buffer:**

To 300mL of ultrapure deionized water in a clean, 1L bottle, add 280 mL of glacial acetic acid and 280
mL of 29% ammonium hydroxide. Dilute to 1L in a grade A volumetric flask. (Note the recipe can
also be doubled or tripled as needed). Adjust the pH to 6.00 +/- 0.2 by adding small amounts of either
acetic acid or ammonium hydroxide. When checking the pH, pour a small amount of buffer into a
separate container so the pH probe never comes in contact with the buffer in the bottle/flask. Allow
the buffer to cool to approximately room temperature before the pH is taken.
Eluent:
Fill a 1L grade A volumetric flask approximately ¼ with DI water, add 106 mL of Optima HNO₃ and then fill the flask with DI water to the 1 L mark. (This recipe can be doubled or tripled as needed.)

Eluent IS:
Fill a 1L grade A volumetric flask approximately ¼ with DI water, add 106 mL of Optima HNO₃ and pipette appropriate volume of IS into the flask (for 10ppb concentration, pipette 1mL/1L). Then fill the flask to the 1L mark with DI water. (This recipe can be doubled or tripled as needed.)

Rinse Solution:
Rinse solution is a ~2% HNO₃ solution in DI water. For 1L, fill a grade a volumetric flask ¼ with DI water pipette 20mL of Optima HNO₃ then fill the flask to the 1L mark with DI water. (This recipe can be doubled or tripled as needed).

All of these solution recipes can be found in the document: SC-DX seaFAST S2 Method Guide PerkinElmer-NexION.

2.9 Instrument Start-up

2.9.1 Instrument Start-up Using the S10 Autosampler

1. Check to make sure that the LEV (exhaust fan) is functioning correctly (there is a light on the front of the instrument to verify the LEV operation, if the light is red, the LEV is not functioning, if the light is green the LEV is functioning)  
   NOTE: The instrument power and vacuum pump should remain on except during maintenance or long term shut-down. The chiller should be turned off only when the plasma is off.
2. Check all gas gauges to ensure proper settings
   - Argon (~90 PSI)
   - Helium (~40-45 PSI)
   - Ammonia (~9-12 PSI)
3. Turn on Chiller and re-attach the peristaltic pump tubing (not necessary for seaFAST autosampler see Section 2.15.2 of this SP). It is recommended that the tubing be changed after ~8 hours of run time.
4. Open the Syngistix Icon from the desktop.
5. To connect the Autosampler, click on the Devices Icon:
6. Select the autosampler that will be used: Autosampler Type box (S10 or ESI)
7. Click Initialize.
8. If the seaFAST autosampler is being used skip to Section 2.9.2 of this SP, if the S10 autosampler is being used continue in this section.
9. Click the Probe button from this window.
10. The following Screen will appear:

11. Click the Goto Rinse button (This will start the peristaltic pump and move it to the rinse solution location). For this step ensure that there is plenty 1-2% HNO3 rinse solution. You are now ready to light the plasma.

12. Click on the Instrument icon at the top of the screen:
13. Click on the Start Button next to the Plasma “Off” box.
14. After clicking this button the Plasma will light, wait ~40 minutes to allow the plasma to stabilize and then proceed to section 2.10 of this SP.

2.9.2 Instrument Start-up Using the seaFAST Autosampler

1. Click on the Syngistix icon:
2. Click on the Instrument icon:

3. Click on the Start Button next to the Plasma “Off” box

4. After clicking the start button the plasma will light, wait ~40 minutes to allow the plasma to stabilize.

5. Click on the ESI icon:

6. Ensure that all there is enough fluid in all necessary solutions for seaFAST autosampler: (i.e. Buffer, Eluent, Eluent IS, DI Water and 2% HNO₃ Rinse). See the document SC-DX seaFAST S2 Method Guide PerkinElmer-NexIon Pg.9 for preparation of these solutions.

7. The following screen will appear, when the ESI software opens. Click on Initialize.

8. Wait until the autosampler has completed initialization. The green writing at the bottom of the window will say “Autosampler Initialized”

9. Click on Calibrate-Calibrate Autosampler.

10. A new window will appear. Click on Go To Calibration Point.
11. A new window will appear. Follow the steps in the window to ensure that the autosampler probe is calibrated.

12. Once calibration is complete, click Go Home.

13. Exit out of this box.

14. From the main ESI screen, select Prime seaFAST and then press the play button (blue and white triangle).

15. Wait until the seaFAST autosampler has completed the priming procedure. The green writing at the bottom of the window will give the status of the priming. Once priming has begun it will be indicated as:

   ![Autosampler Initialized • Instrument Comm Port Opened • • Autosampler Position](image)

   FAST Sub-Method: C:\ProgramData\ESI\ESI SC\Methods\seaFAST\Prime_seaFAST

16. Verify that the box that says "FAST Control" is enabled/checked.

17. Once priming is complete continue to section 2.10.2.

   When the priming has completed, it will be indicated as such:
2.10 Daily Performance Check

2.10.1 Daily Performance Check Using the S10 Autosampler

A Smart Tune Daily Performance Check must be performed before any analysis can occur. A copy of this analysis should be included with the data print-out in the SN supplemental binder. This performance test must pass all of the listed criteria before an analysis can be performed:

Intensity Criterion: Be $9.0122 \geq 2,000$
Intensity Criterion: Mg $23.985 \geq 15,000$
Intensity Criterion: In $114.904 \geq 40,000$
Intensity Criterion: U $238.05 \geq 30,000$
Intensity Criterion: Bkgd $220 \leq 1.0$
Formula Criterion: CeO $155.9 / Ce 139.905 \leq 0.025$
Formula Criterion: Ce$^{++} 69.9527 / Ce 139.905 \leq 0.03$

If the initial Daily performance check fails, the SmartTune wizard is set-up to conduct a series of further calibrations in order to ensure optimal data collection. To begin the Daily Performance Check, follow these steps:

1. Click on the SmartTune Icon:
2. Click File Open SmartTune Daily. This will load the Standard Performance information in the SmartTune window for the S10 autosampler.
3. The following screen will appear (Ensure the box at the top of the window says SmartTune Manual):
4. Place a vial containing the SmartTune Solution (NexIon Set-up Solution), in autosampler position 1 and remove the cap.

5. Click Optimize at the bottom of the window. The autosampler will move to position 1 and the instrument will then perform a check to determine if the instrument is ready for operation. If the above listed criteria are not achieved, then the instrument will automatically continue on to conduct a: Torch Alignment, Nebulizer Gas Flow optimization and a QID STD/DRC optimization. After these calibrations are finished, the instrument will re-perform the STD Performance Check.

If the check passes, you may continue to section 2.11.

If the check fails again, begin trouble shooting and consult the ICP-MS manual.

2.10.2 Daily Performance Check Using the seaFAST Autosampler

A Smart Tune Daily Performance Check must be performed before any analysis can occur. A copy of this analysis should be included with the data print-out in the SN supplemental binder. This performance test must pass all of the listed criteria before an analysis can be performed. The passing criteria for a seaFAST STD Performance test is:

Intensity Criterion: Be 9.0122 ≥ 200
Intensity Criterion: In 114.904 ≥ 200,000
Intensity Criterion: U 238.05 ≥ 3000
Intensity Criterion: Bkgd 220 ≤ 1.0
Formula Criterion: Ce++ 69.9527 /Ce 139.905 ≤ 0.05
Formula Criterion: CeO 155.9 / Ce 139.905 ≤ 0.015

When utilizing a multiple element internal standard, certain elements such as Gallium may interfere with the Ce parameters in the STD Performance test. In that case it is allowable to utilize a customized Lab Performance test. The passing criteria for a customized Lab Performance test is:

Intensity Criterion: Be 9.0122 ≥ 200
Intensity Criterion: In 114.904 ≥ 200,000
Intensity Criterion: U 238.05 ≥ 3000
Intensity Criterion: Bkgd 220 ≤ 1.0
Formula Criterion: U++ 119 / U 238 ≤ 0.05
Formula Criterion: CeO 155.9 / Ce 139.905 ≤ 0.015

If the initial Daily performance check fails, the user can conduct a series of further calibrations manually. To begin the Daily Performance Check, follow these steps:
1. Go to the ESI software and select Direct Mode Only (uncheck the Preconcentration Mode Box). Ensure that the FAST Method File box updated correctly.

![SmartTune Icon]

2. Click on the SmartTune Icon:
3. Click File, Open and select seaFAST-SmartTune Daily.

![SmartTune Daily]

4. The following screen will appear (Ensure the box at the top of the window says SmartTune Manual):

![SmartTune Wizard]

5. Place a vial containing the SmartTune Solution (NexIon Set-up Solution), in autosampler position 9 and remove the cap.
6. Right Click STD Performance Check or Lab Performance Check (depending on the chosen Internal Standard solution) and select Quick Optimize. The autosampler will move to position 9 and the instrument will then perform a check to determine if the instrument is ready for operation.

If the above listed criteria are not achieved, then the user can choose to conduct a series of other optimizations such as: Torch Alignment, Nebulizer Gas Flow optimization, QID STD/DRC optimization etc. After these calibrations are finished, re-do the Performance Check to see if the listed criteria can be met.

7. Once the listed criteria have been met, proceed to section 2.11.2.

2.11 Method Development

2.11.1 Method Development Using the S10 Autosampler

This SP will serve to teach the user only the basic steps to begin a method for analysis. More in depth information about method development can be found in the ICP-MS manual.

1. To begin, click the method Icon:

2. The last method used, will appear on the screen. You will then want to open the method you wish to run. To do this click File…Open…

NOTE: If you would like to make a new method, click file…New

Select the method that you wish to open.
3. Click on the **Timing Tab**. Enter the analytes that you wish to analyze, by clicking the box in the Analyte column. A periodic table will appear that will allow you to select the analyte that you wish to analyze and the respective mass to be analyzed. Some analytes have numerous masses, it is usually best to select the analyte mass that has the highest abundance and the least interferences. (NOTE: this window may appear behind other open windows, so you may need to minimize other windows to find it.)

4. The analytes in the run must be grouped along with an Internal Standard. To make a group, select all of the analytes to go into the group, then click on the arrow below the Method icon and select “Define Group”.

5. To set the Internal Standard, select the analyte that will be the internal standard for a group, and click the arrow below the Method icon and select Set Internal Std.

6. The Timing Tab is also where you set the mode of analysis (Standard, KED, or DRC). Most analyses can be completed in Standard mode. However, if polyatomic ions are present DRC mode may need to be used.

7. Next, click the **Processing Tab**. This screen is where you will set the Detector type (Pulse, Analog or Dual). If you do not know what intensity your analyte will be, make sure you set this parameter to DUAL. All other values can remain at the default settings.
8. Next Click on the **Calibration Tab**. In this tab you will need to fill in concentrations for the analytes that you designated on the Timing tab. You will also need to designate the units for the concentrations.

<table>
<thead>
<tr>
<th>Std</th>
<th>Analyte</th>
<th>Mass (amu)</th>
<th>Curve Type</th>
<th>Sample Units</th>
<th>Std 1</th>
<th>Std 2</th>
<th>Std 3</th>
<th>Std 4</th>
<th>Std 5</th>
<th>Std 6</th>
<th>Std 7</th>
<th>Std 8</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>In</td>
<td>114.904</td>
<td>Simple Linear</td>
<td>ppb</td>
<td></td>
<td></td>
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<tr>
<td>2</td>
<td>Cs</td>
<td>132.905</td>
<td>Simple Linear</td>
<td>ppb</td>
<td>50</td>
<td>100</td>
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<td>200</td>
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</table>

9. Next click on the **Sampling Tab**. In this tab, you will fill in the names that you would like for each standard as well as the Auto Sampler location for each standard.

<table>
<thead>
<tr>
<th>Preselected Name</th>
<th>Time (sec)</th>
<th>Speed (µl/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample Flush</td>
<td>60</td>
<td>-48.0</td>
</tr>
<tr>
<td>Road Delay</td>
<td>30</td>
<td>-35.0</td>
</tr>
<tr>
<td>Analysis</td>
<td>170</td>
<td>-24.0</td>
</tr>
</tbody>
</table>

10. Finally, click on the **Report Tab** and fill in how you would like to process the data.
11. Your method is now set-up. Save it and continue to Section 2.12.

2.11.2 Method Development Using the ESI seaFAST Autosampler

The seaFAST autosampler is designed to run in two different modes: Direct mode and Preconcentration mode. The mode of operation should be selected based on the sample matrix and analyte to be analyzed. The following chart shows which elements should be analyzed in which mode.

For our instrument set-up, it is recommended that all elements shown, for Direct mode analysis only (Blue) be analyzed using the S10 autosampler, because these elements are usually at much higher concentrations and can be analyzed normally. Elements in Green can be analyzed using Preconcentration mode or Direct mode however, it is likely Preconcentration mode will be necessary
to achieve the desired results. The seaFAST is designed for determination of ultra-trace metals in undiluted seawater, making it perfect for samples containing trace analyte concentrations and high background matrices.

1. Details of the seaFAST method set-up portion of this procedure can be found in the document: SC-DX seaFAST S2 Method Guide PerkinElmer-NexION.
2. Go to the ESI software and select Preconcentration Mode and de-select Direct Mode.

3. Click on the Method Icon and then go to File-Open. Choose a seaFAST S method in the seaFAST method folder. Ensure to choose a method for the correct mode you are planning to run (i.e. preconcentration mode, direct mode, preconcentration +direct mode). Once the method is open, select File-Save As and rename the method for what you are planning to analyze.

4. Before editing the method in any way, note the value in the Est. Sample Time box:

This is the value that you will try to achieve for your final Est. Sample Time at the end of your method. To achieve this value you will edit the dwell time of the analytes in the method.
5. Insert empty rows into the method for the elements and Internal Standard you wish to analyze. To add the element click on the box below the column that says Analyte (in the row you just created). There will be a periodic table window that appears. (NOTE: this window may appear behind other open windows, so you may need to minimize other windows to find it.) From the Periodic Table window, click on the element you wish to insert into the method and select the desired mass to be analyzed. It is usually best to choose the mass with the highest abundance and least interferences.

6. Before removing the unwanted elements from the method, ensure to match the Mode, Cell Gas flow and RPq seen in the elements to be deleted to your new elements that you entered. **Note:**for DRC mode, the IS Cell Gas A value is 0.01 point lower than the elements in the method, and the element called END is 0.01 point higher than the analytes to be analyzed. This is an important trend to follow. You may adjust the cell gas value but ensure that you follow this trend regardless of the flow value. The seaFAST autosampler must be ran in either KED or DRC mode. It cannot be used in Standard mode for preconcentration analysis. This is because the gas value has to be slowly increased throughout the method and the gas value cannot be changed in Standard mode. All elements in each mode must have identical cell gas flow settings (for multiple element analysis).

7. Select all of the elements to be removed from the method, right click the mouse and select delete.
8. Once these elements have been deleted, you will now notice that the Est. Sample Time has significantly changed from the original value. We want to get it back to the original value. To do this, adjust the Dwell Time for the analyte of interest until the Est. Sample Time reads similar to the original number.

9. Ensure that you have defined the group for your analysis. To do this, select all of the elements for analysis and the Internal Standard. Then click the arrow below the Method icon and select Define Group. Set the internal standard by selecting the internal standard, then clicking the arrow below the Method icon and selecting Set Internal Standard.

10. Follow steps 7-11 of Section 2.11.1 this SP, to complete the remaining tabs. Your method is now set-up.

2.12 Sample Analysis Using the S10 Autosampler and ESI seaFAST Autosampler

1. To begin setting up an analysis run go back to the main screen and click the Sample icon:
2. Click File…Open… and select the sample file that you would like to open. (You may also select File…New)

3. Type in the Sample Names, Autosampler Locations, Measurement Action and Method to be used for the analysis. In the first sample of the Analysis make sure to select Run Blank, Stds. and Sample for the Measurement Action. See the example below.

4. To begin the analysis, select all of the samples for the run and click on the Build Run List… button.

5. An analysis window will appear that contains the run that you selected. In this window, ensure that your standards, blank and the selected run appear. (If the standards do not appear, a Calibration Curve will not be analyzed).
6. Click Analyze Batch. (Before clicking this button, make sure that all of the samples, Blank and Standards are in their designated Autosampler positions and the caps are removed.)

2.13 Shut-down and Maintenance

2.13.1 Instrument Shut-Down Using the S10 Autosampler

The following shut-down procedure is recommended:

1. While the plasma is still lit, flush system for 5 minutes with 1-2% HNO3.
2. While the plasma is still lit, flush tubing with DI water and put instrument into idle state by doing the following:
   a. Open Device Control window, Peristaltic tab
   b. Type 24 (or 48; see d below) into the Speed field
   c. Click the counter-clockwise arrow
   d. Run the DI water for 5 (or 2.5 if you choose 48 at b) minutes
3. Click the Instrument Icon.
4. Click the Stop button next to the Plasma On box.
5. Turn off the Chiller.
6. Unlatch peristaltic pump tubing and release the tension

2.13.2 Instrument Shut-Down Using the ESI seaFAST Autosampler

1. Click the instrument Icon.
2. Click the Stop button next to the Plasma On box.
3. Turn off the chiller.

2.14 Data Collection

Hard copies of the data collection and calibration records will be submitted to the WIPP Records Center (see Section 3.0 Records). Electronic copies will be stored on the computer hard-drive until the project is completed or results are published.

1. Click the Report Option Icon:
2. To change the format of the report, click File…Open… and select the desired Report format.
3. Click the Report View icon to ensure that this is the report that you want, and then click print.
   (NOTE: for information on Report options refer to the ICP-MS manual)

2.15 Maintenance

2.15.1 Maintenance for ICP-MS and S10 autosampler

Refer to the Maintenance Guide on the desktop of the ICP-MS computer for maintenance issues. A Maintenance log can also be found on the desktop for use if desired. The software for the instrument, keeps a running maintenance schedule for all of the components for the ICP-MS. To access this schedule click the Instrument icon:

Then click the Maintenance Tab. The following screen is an example of the Maintenance Schedule window. All users of the ICP-MS are responsible for keeping up the maintenance of the ICP-MS and should be aware of these maintenance deadlines. A non-QA maintenance logbook is located near the ICP-MS for users to update, as maintenance is completed (example: changing of peristaltic pump tubing, optimizations and cleaning of cones, see Chapter 4 of the Maintenance Guide for detail on cone cleaning). It is recommended that the user refer to this logbook before instrument operation. It is recommended that the user check the peristaltic tubing before operation for flattening or stretching and replace it when necessary (a good rule of thumb is after 8 hours of run time).
2.15.2 Maintenance ESI seaFAST autosampler

The following tasks are suggested maintenance for the ESI seaFAST autosampler.

- Check the internal components of the valves every six months for signs of corrosion or wear. Replace valves, rotors and/or stators as necessary.

- If the system is not to be used for more than 1 week:
  - Ensure that columns are conditioned. If a complete cycle has completed, then the columns are conditioned with buffer. If not, ensure that the syringes are full and run the submethod “SF_Condition Columns.”
  - Remove the columns and cap them with CTFE ¼-28 plugs for storage
  - Install ¼-28 unions in place of the columns
  - Place all solution lines into Eluent solution or another bottle of cleaning acid
  - Run at least one priming sequence
  - Place all solution lines into the DI water bottle.
  - Run at least two priming sequences.

2.16 References


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 12-31-1, Record of Preparation of ICP-MS Derivative Calibration Standards
Appendix B: Form SP 12-31-2, Record of Preparation of ICP-MS Derivative Calibration Standards
Using the seaFAST Autosampler
Appendix C: Ammonia Gas Purge Checklist
### Appendix A

**Record of Preparation of ICP-MS Derivative Calibration Standards**

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<th>Volume of acid (HNO₃) mL</th>
<th>Volume &amp; Concentration of Internal Standard Stock</th>
<th>Final Volume mL</th>
<th>Final Concentration μg/L</th>
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Date Prepared: __________________________ 
Expiration Date: _______________________
## Appendix B

### Record of Preparation of ICP-MS Derivative Calibration Standards Using the seaFAST Autosampler

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<tr>
<th>Element</th>
<th>Volume &amp; Concentration of Primary/Derivative Stock Standard</th>
<th>Volume of acid (HNO₃) mL</th>
<th>Volume of DI Water mL</th>
<th>Final Volume mL</th>
<th>Final Concentration μg/L</th>
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Prepared by: ________________________________
Date Prepared: ________________________________
Expiration Date: ________________________________
Appendix C
Ammonia Gas Purge Checklist

Outside Gas Cabinet

Inside Gas Cabinet
### Ammonia Gas Purge Checklist

<table>
<thead>
<tr>
<th>Task #</th>
<th>Task Description</th>
<th>Task Complete (X)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>INSIDE CABINET: Open gas cabinet window and close valve on ammonia tank (#1)</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>INSIDE CABINET: Close the blue LPI valve that is the Ammonia supply to ICP-MS (#2)</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>INSIDE CABINET: Open the LPV valve to vent the Ammonia to the LEV system (#3)</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>INSIDE CABINET: wait ~30 seconds or until the pressure gauge reads 0 and then close LPV valve (#3)</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>OUTSIDE CABINET: Close the Argon supply valve to the instrument (#4)</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>OUTSIDE CABINET: Open the Argon supply valve to the gas cabinet (#5)</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>INSIDE CABINET: Open the Argon supply valve (PGI) inside the gas cabinet (#6)</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>INSIDE CABINET: Increase the pressure on the Ammonia regulator until the pressure relief valve pops (this should occur around 25-30 PSI)</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>INSIDE CABINET: Re-set the Ammonia regulator pressure to the set-point (~7 +/- 2 PSI), wait ~30 seconds</td>
<td></td>
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<tr>
<td>10</td>
<td>INSIDE CABINET: Open the LPV valve (#3) to vent the gas to the LEV</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>INSIDE CABINET: Close the LPV (#3) valve and wait ~ 10 seconds.</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>INSIDE CABINET: Repeat steps 10 and 11 (10 times)</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>INSIDE CABINET: Close the LPV (#3) valve and then close the PGI (#6) valve</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>INSIDE CABINET: Disconnect the empty Ammonia tank</td>
<td></td>
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<tr>
<td>15</td>
<td>INSIDE CABINET: Connect a full Ammonia tank</td>
<td></td>
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<tr>
<td>16</td>
<td>INSIDE CABINET: Ensure that the PGI (#6) valve is closed, open the Ammonia tank valve and wait ~10 seconds.</td>
<td></td>
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<tr>
<td>17</td>
<td>INSIDE CABINET: Open the (#3) valve slowly, then close the (#3) valve and wait ~10 seconds (Repeat this step 5 times).</td>
<td></td>
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<tr>
<td>18</td>
<td>INSIDE CABINET: Make sure the #3 valve is closed, then open the LPI (#2) valve</td>
<td></td>
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<tr>
<td>19</td>
<td>INSIDE CABINET: Ensure that the Ammonia regulator is set to the proper PSI 7 +/- 2.</td>
<td></td>
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</table>

Person Conducting Tasks Signature: _____________________________

Person Conducting Verification Signature: _____________________________
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