	Alaska Department of Environmental Conservation Environmental Health Laboratory 5251 Dr. MLK Jr. Ave., Anchorage, AK 99507 Standard Operating Procedure
SOP Title:	**Determination of Trace Metals in Biota, Soils, and Sediments by EPA 6020A**
Version/Revision Date:	12/2012
Supersedes:	SOP-Chemistry-008 EPA 6020A version 01-2007
Reference Method:	USEPA, Method 6020A, Revision 1, February 2007

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Periodic Review:

Signature	**Title**	**Date**

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1.0 Scope and Application

This procedure is applicable to the quantitative determination of sub-µg/L concentrations of a large number of elements in various matrices.

2.0 Summary of Method

2.1 Prior to analysis, samples must be prepared in accordance with the EPA method appropriate for analysis. Soils, sediments, biota, and wastes are prepared using microwave digestion method EPA 3051A or hot block digestion method EPA 3050B. Other preparatory methods may be applicable depending on the matrix and/or project requirements.

2.2 This method measures the ions produced by a radio-frequency inductively coupled plasma (ICP). Analyte species originating in the acidified water sample or sample digest are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer (MS). The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences are assessed and valid corrections applied. Interference correction includes compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 Health and Safety Warnings

3.1 Before performing this SOP, the pertinent MSDS for each chemical should be read.

3.2 Gloves, eye protection glasses, and lab coat should be worn at all times while performing sample extraction steps or handling samples or extracts

3.3 Concentrated nitric acid presents various hazards. It is moderately toxic and extremely irritating to skin and mucous membranes. Use this reagent in a fume hood whenever possible. If eye or skin contact occurs, flush with large volumes of water. All expired stock and working standards must be disposed of by current ADEC EHL protocol.

3.4 Analytical plasma sources emit radiofrequency radiation in addition to intense UV radiation. Suitable precautions should be taken to protect personnel from such hazards. The inductively coupled plasma should only be viewed with proper eye protection for UV emissions.

4.0 Interferences

4.1 Isobaric elemental interferences.

4.1.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio.

4.1.2 Correction factors for isobaric interferences have been identified by the instrument manufacturer (PerkinElmer) and are incorporated into the ICP/MS data system software.

4.1.3 All masses which might affect data quality must be monitored. Isobaric polyatomic ion interference must be recognized and appropriate corrections made to the data.

4.2 Physical interferences.

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies.

4.3 Memory interferences.

Memory interferences or carry-over can occur when there are large concentration differences between samples or standards which are analyzed sequentially.

5.0 Personnel Qualifications

Analysts independently performing this method must complete an Initial Demonstration of Capability (IDC). Analysts who have not completed an IDC may perform the method only under the direct supervision of the Chemistry Supervisor or other qualified staff.

6.0 Sample Collection, Handling, and Preservation

6.1 For the determination of elements in fish tissues, biologists from International Pacific Halibut Commission (IPHC), Alaska Department of Fish and Game (ADF&G), National Oceanic and Atmospheric Administration (NOAA), commercial and/or native fishermen, or other designees will collect samples from predetermined locations. Fillets are homogenized, as per ADEC EHL SOP-Chemistry-005 Fish Tissue Processing. The homogenized tissue is stored in wide mouth jars in the freezer at ≤ -10°C until the day before analysis. It is then thawed in the refrigerator until weighed out. The unused portion is returned to the freezer.

6.2 For the determination of total recoverable elements in soil or other solid samples, samples are stored at 4°C. The typical sample size is 50g, but may be smaller.

6.3 Digests, processed as per ADEC EHL SOP-Chemistry-029 Acid Digestion of Biota by EPA 3051A, are stored at room temperature for at least 30 days after analysis, or until such time as it is determined that no further analysis is needed. Once confirmed that no more analysis will be needed, the samples may be disposed.

7.0 Equipment and Supplies

7.1 PerkinElmer Model ELAN DRC II ICP/MS or equivalent.

7.2 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.

7.3 Analytical balance with capability to measure to 0.0001g for use in weighing solids, preparing standards, and determining dissolved solids in digests or extracts.

7.4 Calibrated pipettes, 50-1000µL, or equivalent.

7.5 An assortment of high quality disposable pipet tips appropriate for the pipet used.

7.6 Labware.

7.6.1 50mL conical tubes, volume checked at 50mL ± 0.2mL per lot by laboratory (PerkinElmer catalog #B0193234 or equivalent).

7.6.2 15mL conical tubes, volume checked at 10mL ± 0.2mL, 5mL ± 0.2mL, and 2mL ± 0.2mL per lot by laboratory (VWR catalog #21008-216 or equivalent).

7.6.3 Narrow-mouth storage bottles, plastic with screw closure.

7.6.4 Squeeze bottle for 1% nitric acid (HNO_3).

8.0 Reagents and Standards

8.1 Reagent water - All references to reagent grade water in this SOP refer to ASTM Type I water (ASTM D1193) or Milli-Q water.

8.2 Reagent Acids.

Nitric acid, Ultrex II Ultrapure Reagent HNO_3, J.T. Baker, VWR catalog number JT6901-2 or equivalent.

8.3 1% Nitric Acid (HNO_3) solution.

Add 20mL concentrated HNO_3 solution to approximately 1500mL Milli-Q (reagent) water. Mix well and dilute to 2000mL with Milli-Q (reagent) water.

8.4 Standards.

8.4.1 Fish Monitoring.

8.4.1.1 Calibration Standard, AccuStandard catalog #ICP-MS-200.8-Cal-1R or equivalent.

8.4.1.2 Calibration Standard, AccuStandard catalog #ICP-MS-200.8-Cal2-1 or equivalent.

8.4.1.3 6020 Tuning Solution (1ppb ELAN 6100DRC setup solution), Inorganic Ventures catalog #6020TS or equivalent.

8.4.1.4 6020 Internal Standard, Inorganic Ventures catalog # 6020ISS or equivalent.

8.4.1.5 1000µg/mL Magnesium (Mg), Inorganic Ventures catalog #CGMG1-1 or equivalent.

8.4.1.6 1000µg/mL Indium (In), Inorganic Ventures catalog #CGIN1-1 or equivalent.

8.4.1.7 1000µg/mL Uranium (U), Inorganic Ventures catalog #CGU1-1 or equivalent.

8.4.1.8 1000µg/mL Cerium (Ce), Inorganic Ventures catalog #CGCE1-1 or equivalent.

8.4.1.9 1000µg/mL Barium (Ba), Inorganic Ventures catalog #CGBA1-1 or equivalent.

8.4.1.10 Initial Calibration Verification (ICV) Standard, Inorganic Ventures catalog #WW-MSCAL-2 or equivalent.

8.4.1.11 Interference Check Solution (ICS), Inorganic Ventures catalog #6020ICS-0A or equivalent.

8.4.2 Full List.

8.4.2.1 Calibration Stock Standard, AccuStandard catalog #ICP-MS-200.8-Cal-1R or equivalent.

8.4.2.2 1000µg/mL Iron (Fe) Standard, AccuStandard catalog #ICP-MS-27N- 0.1X-1 or equivalent.

8.4.2.3 100µg/mL Tin (Sn) Standard, AccuStandard catalog #ICP-MS-63N- 0.01X-1 or equivalent.

8.4.2.4 100µg/mL Lithium (Li) Standard, AccuStandard catalog #ICP-MS-30N- 0.01X-1 or equivalent.

8.4.2.5 6020 Tuning Solution (1ppb ELAN 6100DRC setup solution), Inorganic Ventures catalog #6020TS or equivalent.

8.4.2.6 6020 Internal Standard, Inorganic Ventures catalog #6020ISS or equivalent.

8.4.2.7 1000µg/mL Magnesium (Mg), Inorganic Ventures catalog #CGMG1-1 or equivalent.

8.4.2.8 1000µg/mL Indium (In), Inorganic Ventures catalog #CGIN1-1 or equivalent.

8.4.2.9 1000µg/mL Uranium (U), Inorganic Ventures catalog #CGU1-1 or equivalent.

8.4.2.10 1000µg/mL Cerium (Ce), Inorganic Ventures catalog #CGCE1-1 or equivalent.

8.4.2.11 1000µg/mL Barium (Ba), Inorganic Ventures catalog #CGBA1-1 or equivalent.

8.4.2.12 Initial Calibration Verification (ICV1) Standard, Inorganic Ventures catalog #WW-MSCAL-2 or equivalent.

8.4.2.13 Initial Calibration Verification (ICV2) Standard, Inorganic Ventures catalog #2008CAL-1 or equivalent.

8.4.2.14 1000µg/mL Iron (Fe) Standard, Inorganic Ventures catalog # CGFE1-1 or equivalent.

8.4.2.15 1000µg/mL Tin (Sn) Standard, Inorganic Ventures catalog # CGSN1-1 or equivalent.

8.4.2.16 1000µg/mL Lithium (Li) Standard, Inorganic Ventures catalog # CGLI1-1 or equivalent.

8.4.2.17 Interference Check Solution (ICS), Inorganic Ventures catalog #6020ICS-0A-1 or equivalent.

8.5 Recommended Calibration.

8.5.1 Fish Monitoring.

8.5.1.1 6020 Internal Standard (6020ISS/Ge) Working Solution.

In a 15mL conical tube, dilute 0.1mL (100µL) of 1000ppm Germanium (Ge) to 5mL with 1% HNO_3 for a 20ppm final concentration. Add 5mL of 6020ISS for a final volume of 10mL.

8.5.1.2 200ppb Calibration Standard.

Dilute 1.0mL (1000µL) of Calibration Stock Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 50mL with 1% HNO_3.

8.5.1.3 100ppb Calibration Standard (CCV).

Dilute 0.5mL (500µL) of Calibration Stock Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 50mL with 1% HNO_3.

8.5.1.4 10ppb Calibration Standard.

Dilute 0.05mL (50µL) of Calibration Stock Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 50mL with 1% HNO_3.

8.5.1.5 1µg/mL (ppm) Calibration Intermediate Standard.

Dilute 1.0mL of Calibration Stock Standard to 10mL with 1% HNO_3.

8.5.1.6 0.5ppb Calibration Standard (LLICV/LLCCV).

Dilute 0.025mL (25µL) of 1µg/mL (ppm) Calibration Intermediate Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 50mL with 1% HNO_3.

8.5.1.7 Log the Calibration Standards (8.5.1.1 to 8.5.1.6) into Element under Laboratory Standards.

8.5.2 Full List.

8.5.2.1 6020 Internal Standard (6020ISS/Ge) Working Solution.

In a 15mL conical tube, dilute 0.1mL (100µL) of 1000ppm Ge to 5mL with 1% HNO_3 for a 20ppm final concentration. Add 5mL of 6020ISS for a final volume of 10mL.

8.5.2.2 200ppb Calibration Standard.

Dilute 1.0mL of 10µg/mL (ppm) Calibration Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 10mL with 1% HNO_3.

8.5.2.3 100ppb Calibration Standard (CCV).

Dilute 0.5mL (500µL) of 10µg/mL (ppm) Calibration Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 10mL with 1% HNO_3.

8.5.2.4 10ppb Calibration Standard.

Dilute 0.05mL (50µL) of 10µg/mL (ppm) Calibration Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to10mL with 1% HNO_3.

8.5.2.5 1µg/mL (ppm) Calibration Intermediate Standard.

Dilute 1.0mL of Calibration Stock Standard to 10mL with 1% HNO_3.

8.5.2.6 0.5ppb Calibration Standard (LLICV /LLCCV).

Dilute 0.025mL (25µL) of 1µg/mL (ppm) Intermediate Calibration Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 10mL with 1% HNO_3.

8.6 6020 Tuning Solution.

10ppb 6020 Tuning Solution.

Dilute 0.05mL (50µL) of 6020 Turning Solution to 50mL with 1% HNO_3.

8.7 Daily Performance Check Solution.

8.7.1 Daily Performance Stock Solution (1ppm Mg, In, U, & Ce; 10ppm Ba).

Dilute 0.1mL (100µL) 1000µg/mL Mg, 0.1mL (100µL) 1000µg/L In, 0.1mL (100µL) 1000µg/mL U, 0.1mL (100µL) 1000µg/mL Ce, and 1.0mL of 1000µg/mL Barium to 100mL with 1% HNO_3.

8.7.2 Daily Performance Solution (1ppb Mg, In, U, & Ce; 10ppb Ba).

Dilute 0.1mL (100µL) Daily Performance Stock Solution to 100mL with 1% HNO_3.

8.7 Initial Calibration Verification (ICV).

8.7.1 Fish Monitoring.

Initial Calibration Verification (ICV).

Dilute 0.25mL (250µL) of ICV1 Standard and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 50mL with 1% HNO_3.

8.7.2 Full List.

8.7.2.1 20ppm Sn/Li Working Standard.

Dilute 0.2mL (200µL) of 1000µg/mL Sn and 0.2mL (200µL) of 1000µg/mL Li to 10mL with 1% HNO_3.

8.7.2.2 ICV Solution.

Dilute 0.25mL (250µL) of ICV1, 0.25mL (250µL) of ICV2, 0.25mL (250µL) of 1000µg/mL Fe, 0.25mL (250µL) of 20ppm Sn/Li Working Standard, and 0.25mL (250µL) of the 6020ISS/Ge Working Solution to 50mL with 1% HNO_3.

8.8 Interference Check Solution (ICS).

8.8.1 ICSA (IFA1).

Dilute 0.1mL (100µL) ICS to 10mL with 1% HNO_3 and mix.

8.8.2 ICSAB (IFB1).

Dilute 0.1mL (100µL) ICS and 0.1mL (100µL) Calibration Standard to 10mL with 1% HNO_3.

9.0 Instrument Setup, Calibration, and Standardization

9.1 Open the ELAN software.

9.2 Verify the argon pressure is at least 80 psi.

9.3 Change the sample and drain tubing if necessary. Click on the Devices icon and adjust the Peristaltic pump speed to "Fast". Verify that the tubing is rolling properly, press "Stop", and lock in the tubing.

9.4 Turn on the recirculator.

9.5 Go to the Instrument icon and click on the Front Panel button to open the ELAN control panel. The System Status bar should indicate "Ready" if all system hardware is operating properly and the plasma can be ignited. If the indicator says "Not Ready" or "Comm. error", refer to the operator manual to determine which system component is causing the fault.

9.6 Click on the plasma Start button to ignite the plasma. The ELAN initiates the ignition process, which requires a little over a minute to complete. The progress of the ignition sequence is shown in the ignition sequence status bar.

9.7 After the plasma ignites, place the sample probe into Milli-Q water and allow the instrument to warm up for approximately 30 minutes. If the plasma does not ignite, refer to the manual to troubleshoot and correct the problem.

9.8 To perform a tuning check of the ICP-MS, select the SmartTune icon. A SmartTune Wizard window will appear. Go to File and open "FishDefault.swz". Highlight "Optimization" on the list inside the SmartTune window. Verify the files on the right-hand side of the window match Figure 1. Create and load a new dataset using the current date (ex: 051711a).

9.9 Aspirate the tuning solution. Click on Mass Calibration and Resolution. Check that the criteria on the right side match Figure 2. Right click Mass Calibration and Resolution and select Quick Optimize.

When the sample is done, check the SmartTune Wizard – Summary and SmartTune – Details reports to verify that the mass calibration and resolution passed (mass values are ±0.1amu of the actual mass values and the resolution is less than 0.9 ± 0.1amu).

9.10 Select the Method icon and load the EPA 6020A 1Daily PerformanceA.mth. Click the Sample icon, Manual tab, and Analyze sample. The Daily Performance Report will automatically print. Confirm that the Net. Intens. RSD of all analytes (Li, Co, In, & Tl) is less than 5%. If this parameter is not met, re-perform the Mass Calibration and Resolution and/or consult the operator manual.

9.11 Aspirate the Daily Performance Check solution. Open the SmartTune Wizard and select Daily Performance Check. The Method File should be "C:\Elandata\Method\Daily Performance.mth". Right-click to Quick Optimize. After the optimization is complete the SmartTune Wizard – Summary and SmartTune – Details report will automatically print. Evaluate the Daily Performance Check to determine if suggested criteria are met. The Daily Performance Report will automatically print. Confirm that the Net. Intens. RSD of Mg, In, and U are less than 3%. If necessary, Quick Optimize the Nebulizer Gas Flow [NEB], Lens Voltage, and Auto Lens Calibration while analyzing the daily performance check solution. Save the results of each optimization under the Optimize icon and Manual Adjust. Reanalyze the Daily Performance Check solution by selecting Daily Performance Check, Quick Optimize under the SmartTune icon. If the parameters of the Daily Performance Check still do not meet suggested criteria, consult the operator manual.

9.12 Load "EPA 6020a fish.mth" for fish tissue analysis or "EPA 6020a Full List.mth" for full list under the Method tab. Select the Report tab on the right side of the screen.

9.12.1 In the Report View Section, check the Send to Printer box and load "EPA6020a quant comprehensiveR1.rop" for fish tissue or "6020a quant comprehensive (full list).rop" for full list in the Report Options Template.

9.12.2 Check the Send to File box in the Report to File box and select "EPA6020a quant comprehensiveR1.rop" for fish tissue or "6020a quant comprehensive (full list).rop" for full list as the Report Options Template. Update the Report Filename to the current date (ex: g:\eh\eh-lab\data capture\icpms_datafiles\ 051711a.rep). Select all boxes (Include Titles, Use Delimiter, and Use Separator) in the Report Format section. Choose Append in the File Write Option box. Select "file" and save method.

9.13 Click the Sample icon and batch tab. Save the sample batch with today's date (ex: 051711a, 051711b, etc). Enter the A/S Location and Sample ID for the samples to be analyzed. To calibrate the system select Run Blank, Stds, and Sample under Measurement Action (*) for the first sample. For additional samples select Run Sample for Measurement Action (*). In the Method (*) column choose either "EPA 6020a fish.mth" for fish tissue analysis or "EPA 6020a Full List.mth" for full list. Select Sample for Sample Type (*). After all changes have been made, save the sample batch.

9.14 Select the green R on the left side of the screen to update the analysis files.

9.14.1 Method.

Load the appropriate method for analysis ("EPA 6020a fish.mth" for fish tissue analysis or "EPA 6020a Full List.mth" for full list).

9.14.2 Dataset.

Create a New dataset using the current date (ex: 051711a). Load the dataset with the current date.

9.14.3 Sample.

The sample name should be sample batch with the current date (ex: 051711a, 051711b, etc).

9.14.4 Report Template.

The report template is "EPA6020a quant comprehensiveR1.rop" for fish tissue or "6020a quant comprehensive (full list).rop" for full list.

9.14.5 Tuning.

Select default.tun for tuning.

9.14.6 Optimization.

Choose default.dac for optimization.

9.14.7 Calibration.

Click on the CalibView icon and open a previous calibration and save it as the current date (ex: 051711a, 051711b, etc).

9.14.8 Polyatomic.

Select elan.ply for polyatomic.

9.15 Place the calibration standard tubes into the positions indicated in EPA 6020A quantitative page.

9.16 Return to the Sample icon and batch tab. Highlight the samples to be analyzed, select Build Run List and print.

9.17 Secure autosampler tubing and lock into place. Select Method icon, click the Sampling tab, press the Probe button, choose Go To Rinse, and place the sample probe in the autosampler.

9.18 Verify that the standards and samples are in the correct location on the autosampler and click Analyze Samples. Save tune file.

9.19 If there is difficulty meeting the linear calibration algorithm with $R \geq 0.998$, the instrument will require recalibration and/or maintenance.

10.0 Sample Preparation

10.1 Prepare samples in accordance with EPA methods 200.8 or EPA 3005 through 3052, depending on the matrix and/or project requirements.

10.2 Method Blank (MB, non-digested water samples only) and Calibration Blank.

Fill 50mL of 1% HNO_3 into a sample tube. Refer to the appropriate EPA method's MB preparation procedure for other matrices.

10.3 Continuing Calibration Verification Standard (CCV).

100ppb Calibration Standard (8.5.2.3).

11.0 Sample Analysis

11.1 Follow instrument warm up and set up procedure described in section 9.0.

11.2 Set up sample queue (similar to the example in Table 1) and analyze sequence.

11.3 Confirm that all quality control requirements stated in Table 2 are met.

11.4 If there is difficulty meeting the QC requirements, the instrument will require recalibration and/or maintenance.

11.5 Shutdown Procedure.

11.5.1 Immerse the sample probe in 1% HNO_3 for 10 minutes; then rinse in water for 5 minutes.

11.5.2 Take the sample probe out of solution, place on autosampler rack, and wait until spray chamber is dry (no more bubbles leave the bottom of the spray chamber).

11.5.3 In the ELAN software, click on "Instrument", and turn off plasma by pressing "Stop" in the Plasma box.

11.5.4 Unclamp and loosen the sample and drain tubing.

11.5.5 Shut off the recirculator.

11.5.6 If running overnight, the Scheduler can be used to rinse and shutdown the instrument. Consult ELAN Online help for scheduling and automating tasks.

12.0 Data Analysis and Calculations

12.1 Under the main Element window, click on "Laboratory", and select **Data Entry/Review**. An "Enter/Edit Data" window will appear. Click on the batch number. Select the **Data Entry** tab, then **Create** to upload the data into the window below. Select the **Data Tool** button.

12.2 Select PE ELAN-REP (*.rep) from the file type drop-down box in the top right hand corner. Double click the file to be uploaded under *G:\EH\Eh-Lab\Data Capture\ICPMS_DataFiles.* The samples will appear in the upper left window. Highlight samples and QC's analyzed, click **Include**, and select **Done**. **Merge** the files.

12.3 Name any unnamed samples as they are in the Element batch under the **Instrument Data** tab. Click the **Refresh** button. Click on **Save**, choose the Element folder *C:\ELMNT\UserFiles*, and select the "gg" spreadsheet. Answer "Yes" to replace the existing data. Click **Done** and close the Data Tool window.

12.4 In the **Enter/Edit Data window**, change the analyst initials from ZZZ to the actual analyst's initials by clicking the Analyst column header, right-clicking, and selecting "Fill cells". Use the drop-down menu to select the analyst's initials.

12.5 Click **Save** in the Enter/Edit Data window to save the merged files. Click done. Under the Data Review tab, select **Query.**

12.6 The test results will now appear. Select the status column, right click and update the status to Analyzed. Lock the page by clicking on the Lock column header and selecting Lock. Click the Printer icon to print the Data Review report.

12.7 Calculations.

12.8.1 Sample Concentration.

$$\text{Concentration (mg/kg)} = \frac{\mu g}{L} \times \frac{mg}{1000\mu g} \times \frac{L}{1000mL} \times \frac{50mL}{\text{Sample Weight (g)}} \times \frac{1000g}{1kg} \times D_f$$

where:

D_f = Dilution factor. If no dilution was made, D = 1.

12.8.2 Percent Drift (%D).

$$\%\ \text{Drift} = \frac{|C_c - C_t|}{C_t} \times 100\%$$

where:

C_c= Calculated concentration

C_t= Theoretical concentration

12.8.3 LCS Percent Recovery.

$$\%\ \text{Recovery} = \frac{C_s}{C_a} \times 100\%$$

where:

$C_s =$ Observed spike concentration

$C_a =$ Spike Level

12.8.4 Duplicate Relative Percent Difference (RPD).

$$RPD = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \times 100\%$$

where:

X_1 = Concentration of sample

X_2 = Concentration of duplicate

13.0 Data and Records Management

13.1 Final results are reported in mg/kg.

13.2 Assemble a data package consisting (in order):

13.2.1 Final report from Element;

13.2.2 Data Review Checklist;

13.2.3 Non Conformance Details report(s) (if applicable);

13.2.4 Element Preparation Bench Sheet;

13.2.5 Element analysis sequence;

13.2.6 ELAN DRC II Sample Run List;

13.2.7 Calibration curve; and

13.2.8 ELAN DRC II EPA6020 quantitative comprehensive report for each sample and QC sample. Then,

13.2.9 Place data package in a sea foam green end tab classification folder. Print a label indicating the Element sequence number, Element batch number, and method performed and affix to the end tab of the folder.

13.3 Submit data package to peer or supervisor for second level review. Upon successful review, the second level reviewer updates the Element batch status to reviewed, and files the data package in the central files.

14.0 Quality Control

14.1 Method QC requirements are summarized in Table 2.

14.2 Initial Demonstration of Capability (IDC).

14.2.1 An IDC must be conducted by each new analyst.

14.2.2 Four passing LCS's must be provided to validate an analyst for this method.

14.3 Method Detection Limit (MDL).

14.3.1 An MDL determination must be conducted annually.

14.3.2 An MDL determination should also be conducted when significant changes in instrument occur, or when a new instrument is purchased for the analysis.

14.3.3 If appropriate, the IDC (14.2), MDL, and LLQC (14.5) determinations may be combined.

14.4 Instrument Detection Limits (IDLs).

14.4.1 IDLs should be determined at least every three months or at a project-specific designated frequency.

14.4.2 IDLs in µg/L are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.

14.5 Lower Limit of Quantitation Check (LLQC) Sample.

14.5.1 The lower limits of quantitation should be established for all isotope masses utilized for each type of matrix analyzed, each preparation method used, and for each instrument. These limits are considered the lowest reliable laboratory reporting concentrations and should be established from the lower limit of quantitation check sample and then confirmed using either the lowest calibration point or from a low-level check standard.

14.5.2 The LLQC sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the sample concentration with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ±30% of their true value. This check should be used to both establish and confirm the lowest quantitation limit.

15.0 References

15.1 USEPA, Methods 6020A, Revision 1, February 2007.

15.2 ELAN Online Help.

15.3 PerkinElmer ELAN Version 3.0 Software Guide, 2003.

15.4 ELAN DRC II Hardware Guide, 2005.

15.5 AS-93 Autosampler for Atomic Spectrometry User's Guide, P/N 0993-5278 or equivalent.

15.6 Element DataSystem, Version 6 or equivalent.

15.7 Element DataSystem Data Tool, Version 3.590 or equivalent.

Table 1
Example Element Sequence

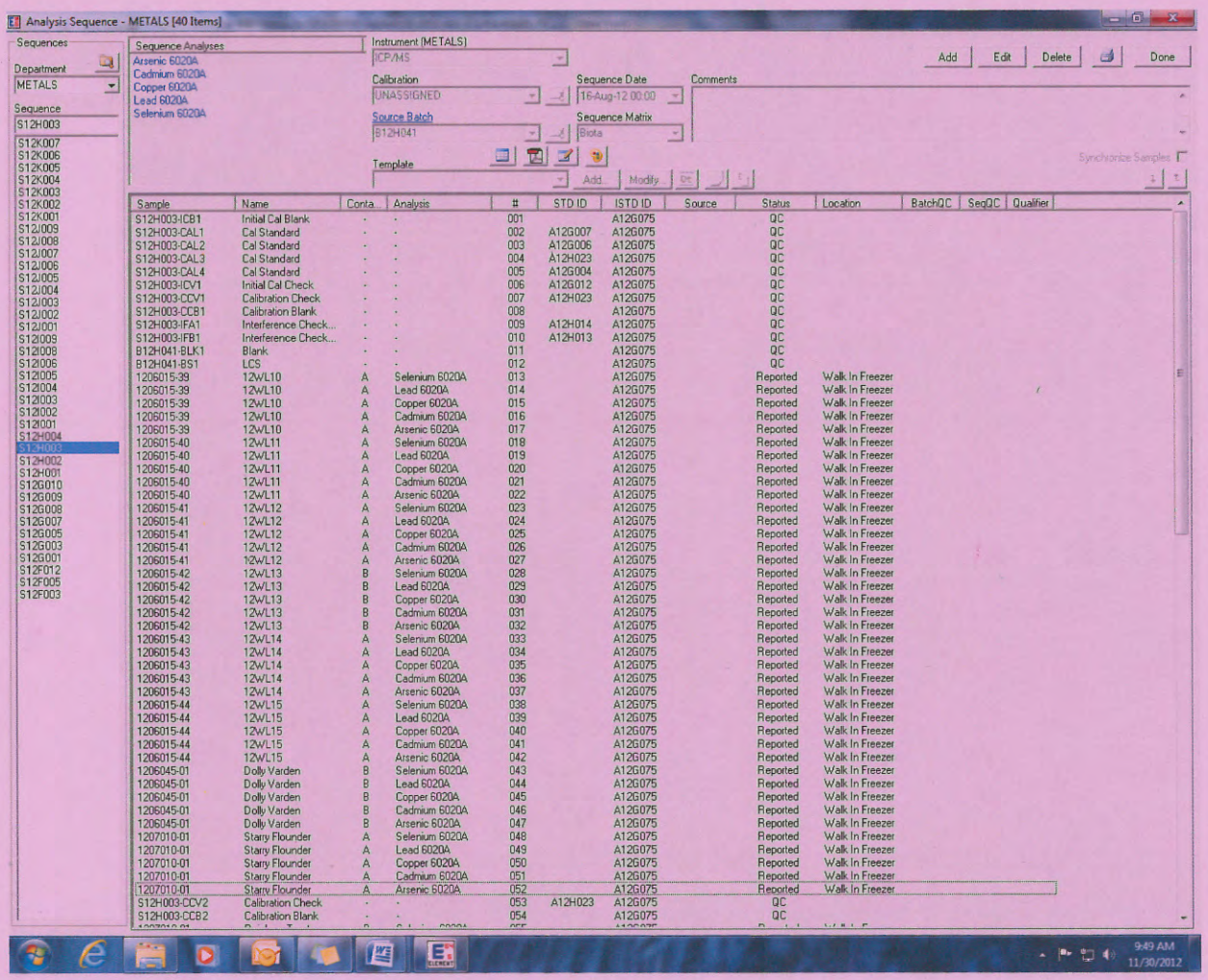

Table 1
Example Element Sequence (cont.)

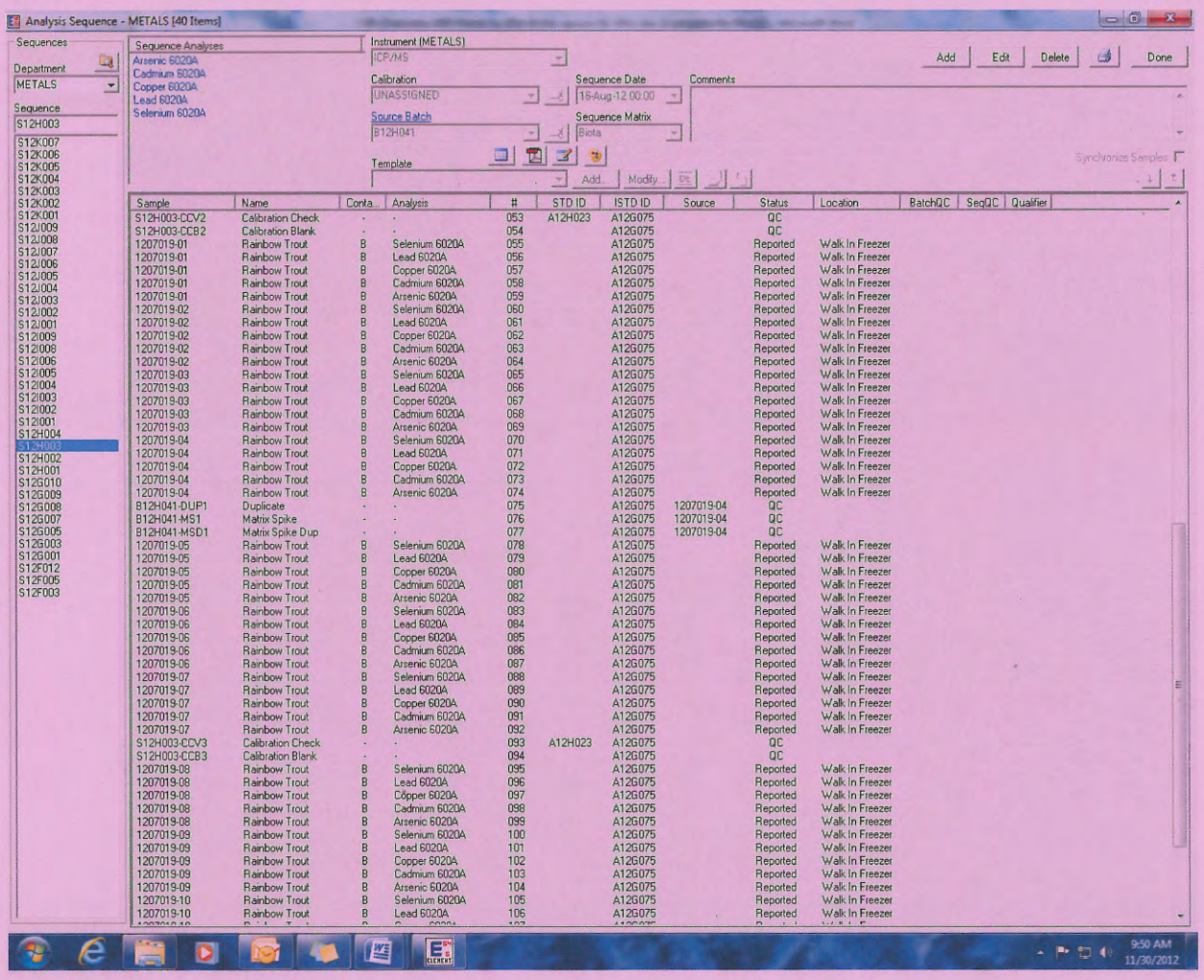

Table 1
Example Element Sequence (cont.)

Sample	Name	Conta.	Analysis	#	STD ID	ISTD ID	Source	Status	Location	BatchQC	SeqQC	Qualifier
1207019-02	Rainbow Trout	B	Cadmium 6020A	063		A12G075		Reported	Walk In Freezer			
1207019-02	Rainbow Trout	B	Arsenic 6020A	064		A12G075		Reported	Walk In Freezer			
1207019-03	Rainbow Trout	B	Selenium 6020A	065		A12G075		Reported	Walk In Freezer			
1207019-03	Rainbow Trout	B	Lead 6020A	066		A12G075		Reported	Walk In Freezer			
1207019-03	Rainbow Trout	B	Copper 6020A	067		A12G075		Reported	Walk In Freezer			
1207019-03	Rainbow Trout	B	Cadmium 6020A	068		A12G075		Reported	Walk In Freezer			
1207019-03	Rainbow Trout	B	Arsenic 6020A	069		A12G075		Reported	Walk In Freezer			
1207019-04	Rainbow Trout	B	Selenium 6020A	070		A12G075		Reported	Walk In Freezer			
1207019-04	Rainbow Trout	B	Lead 6020A	071		A12G075		Reported	Walk In Freezer			
1207019-04	Rainbow Trout	B	Copper 6020A	072		A12G075		Reported	Walk In Freezer			
1207019-04	Rainbow Trout	B	Cadmium 6020A	073		A12G075		Reported	Walk In Freezer			
1207019-04	Rainbow Trout	B	Arsenic 6020A	074		A12G075		Reported	Walk In Freezer			
B12H041-DUP1	Duplicate	-	-	075		A12G075	1207019-04	QC				
B12H041-MS1	Matrix Spike	-	-	076		A12G075	1207019-04	QC				
B12H041-MSD1	Matrix Spike Dup	-	-	077		A12G075	1207019-04	QC				
1207019-05	Rainbow Trout	B	Selenium 6020A	078		A12G075		Reported	Walk In Freezer			
1207019-05	Rainbow Trout	B	Lead 6020A	079		A12G075		Reported	Walk In Freezer			
1207019-05	Rainbow Trout	B	Copper 6020A	080		A12G075		Reported	Walk In Freezer			
1207019-05	Rainbow Trout	B	Cadmium 6020A	081		A12G075		Reported	Walk In Freezer			
1207019-05	Rainbow Trout	B	Arsenic 6020A	082		A12G075		Reported	Walk In Freezer			
1207019-06	Rainbow Trout	B	Selenium 6020A	083		A12G075		Reported	Walk In Freezer			
1207019-06	Rainbow Trout	B	Lead 6020A	084		A12G075		Reported	Walk In Freezer			
1207019-06	Rainbow Trout	B	Copper 6020A	085		A12G075		Reported	Walk In Freezer			
1207019-06	Rainbow Trout	B	Cadmium 6020A	086		A12G075		Reported	Walk In Freezer			
1207019-06	Rainbow Trout	B	Arsenic 6020A	087		A12G075		Reported	Walk In Freezer			
1207019-07	Rainbow Trout	B	Selenium 6020A	088		A12G075		Reported	Walk In Freezer			
1207019-07	Rainbow Trout	B	Lead 6020A	089		A12G075		Reported	Walk In Freezer			
1207019-07	Rainbow Trout	B	Copper 6020A	090		A12G075		Reported	Walk In Freezer			
1207019-07	Rainbow Trout	B	Cadmium 6020A	091		A12G075		Reported	Walk In Freezer			
1207019-07	Rainbow Trout	B	Arsenic 6020A	092		A12G075		Reported	Walk In Freezer			
S12H003-CCV3	Calibration Check	-	-	093	A12H023	A12G075		QC				
S12H003-CCB3	Calibration Blank	-	-	094		A12G075		QC				
1207019-08	Rainbow Trout	B	Selenium 6020A	095		A12G075		Reported	Walk In Freezer			
1207019-08	Rainbow Trout	B	Lead 6020A	096		A12G075		Reported	Walk In Freezer			
1207019-08	Rainbow Trout	B	Copper 6020A	097		A12G075		Reported	Walk In Freezer			
1207019-08	Rainbow Trout	B	Cadmium 6020A	098		A12G075		Reported	Walk In Freezer			
1207019-08	Rainbow Trout	B	Arsenic 6020A	099		A12G075		Reported	Walk In Freezer			
1207019-09	Rainbow Trout	B	Selenium 6020A	100		A12G075		Reported	Walk In Freezer			
1207019-09	Rainbow Trout	B	Lead 6020A	101		A12G075		Reported	Walk In Freezer			
1207019-09	Rainbow Trout	B	Copper 6020A	102		A12G075		Reported	Walk In Freezer			
1207019-09	Rainbow Trout	B	Cadmium 6020A	103		A12G075		Reported	Walk In Freezer			
1207019-09	Rainbow Trout	B	Arsenic 6020A	104		A12G075		Reported	Walk In Freezer			
1207019-10	Rainbow Trout	B	Selenium 6020A	105		A12G075		Reported	Walk In Freezer			
1207019-10	Rainbow Trout	B	Lead 6020A	106		A12G075		Reported	Walk In Freezer			
1207019-10	Rainbow Trout	B	Copper 6020A	107		A12G075		Reported	Walk In Freezer			
1207019-10	Rainbow Trout	B	Cadmium 6020A	108		A12G075		Reported	Walk In Freezer			
1207019-10	Rainbow Trout	B	Arsenic 6020A	109		A12G075		Reported	Walk In Freezer			
1208019-01	Sleeper Shark	A	Selenium 6020A	110		A12G075		Reported	Walk In Freezer			
1208019-01	Sleeper Shark	A	Lead 6020A	111		A12G075		Reported	Walk In Freezer			
1208019-01	Sleeper Shark	A	Copper 6020A	112		A12G075		Reported	Walk In Freezer			
1208019-01	Sleeper Shark	A	Cadmium 6020A	113		A12G075		Reported	Walk In Freezer			
1208019-01	Sleeper Shark	A	Arsenic 6020A	114		A12G075		Reported	Walk In Freezer			
S12H003-CCV4	Calibration Check	-	-	115	A12H023	A12G075		QC				
S12H003-CCB4	Calibration Blank	-	-	116		A12G075		QC				

Table 2
Quality Control Requirements

Description	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Daily	$r \geq 0.998$	Re-analyze initial calibration.
Low Level Initial Calibration Verification (LLICV)	Immediately following ICAL	QC % R 70-130%	Recalibrate.
Initial Calibration Verification (ICV)	Immediately following LLICV	QC % R 90-110%	Recalibrate.
Initial Calibration Blank (ICB)	Immediately following ICV	≤½MRL	Investigate source of contamination and correct the problem. Recalibrate after contamination source eliminated.
Interference Check Sample A (ICSA)	Immediately following ICB	Should be run every 12 hours during active analysis of samples.	Evaluate system for elemental and molecular-ion isobaric interferences. Recalibrate after corrective action completed.
Interference Check Sample B (ICSB)	Immediately following ICSA	Should be run every 12 hours during active analysis of samples.	Evaluate adequacy of inter-element correction factors. Recalibrate after corrective action completed.
Low Level Continuing Calibration Verification (LLCCV)	Every 10 injections **and** at end of analytical sequence	QC % R 70-130%	Re-analyze LLCCV. If second re-analysis of LLCCV fails criteria, then recalibrate. Samples analyzed prior to failing CCV must be reanalyzed for elements that failed criteria.

Table 2
Quality Control Requirements (cont.)

Description	Frequency	Criteria	Corrective Action
Continuing Calibration Verification (CCV)	Every 10 injections **and** at end of analytical sequence	QC % R 90-110%	Re-analyze CCV. If second re-analysis of CCV fails criteria, then recalibrate. Samples analyzed prior to failing CCV must be reanalyzed for elements that failed criteria.
Continuing Calibration Blank (CCB)	Follows every CCV	≤½MRL	Re-analyze CCB. Investigate source of contamination and correct the problem. Recalibrate after contamination source eliminated. Re-analyze samples or qualify results for elements that failed CCB criteria.
Method Blank (MB)	Ever preparation batch or 20 samples, whichever is less	≤MRL	If samples < RL, no action. If samples > RL and level in MB is < 5% of the amount found in samples, report data with "B" qualifier. If samples > RL but < 5% of amount found in samples, re-extract and re-analyze batch for elements with blank contamination.
Laboratory Control Sample (LCS)	Ever preparation batch or 20 samples, whichever is less	Limits established by control charts. If no limits established, then use advisory limits %R = 80%-120%.	If samples ND and LCS %Rec > upper control limit, no corrective action. Otherwise, re-digest and re-analyze batch for elements with out of control LCS recoveries.

Table 2
Quality Control Requirements (cont.)

Description	Frequency	Criteria	Corrective Action
Duplicate	Every preparation batch or 20 samples, whichever is less.	%RPD ≤ 20% for analyte values greater than 100x the RL.	Qualify data. Possible non-homogeneous sample.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Every preparation batch or 20 samples, whichever is less	Limits established by control charts. If no limits established, then use advisory limits %R = 75%-125%.	Prepare and analyze post digestion spike addition.
Post Digestion Spike Addition (PS)	Analyze if MS/MSD requirements are unacceptable.	%R = 80%-120%	Dilute sample and re-analyze to compensate for matrix effect.
Dilution (DIL)	Analyze if PS requirements are unacceptable.	Results must agree within ±10% for analyte values greater than 100x the RL	Possible matrix interference. Evaluate need for method of standard additions.
Internal Standard (IS)	Every standard and sample	IS intensity ≥70% of original calibration blank	Dilute sample and re-analyze to compensate for matrix effect.

Table 3
ELAN Smart Tune Wizard

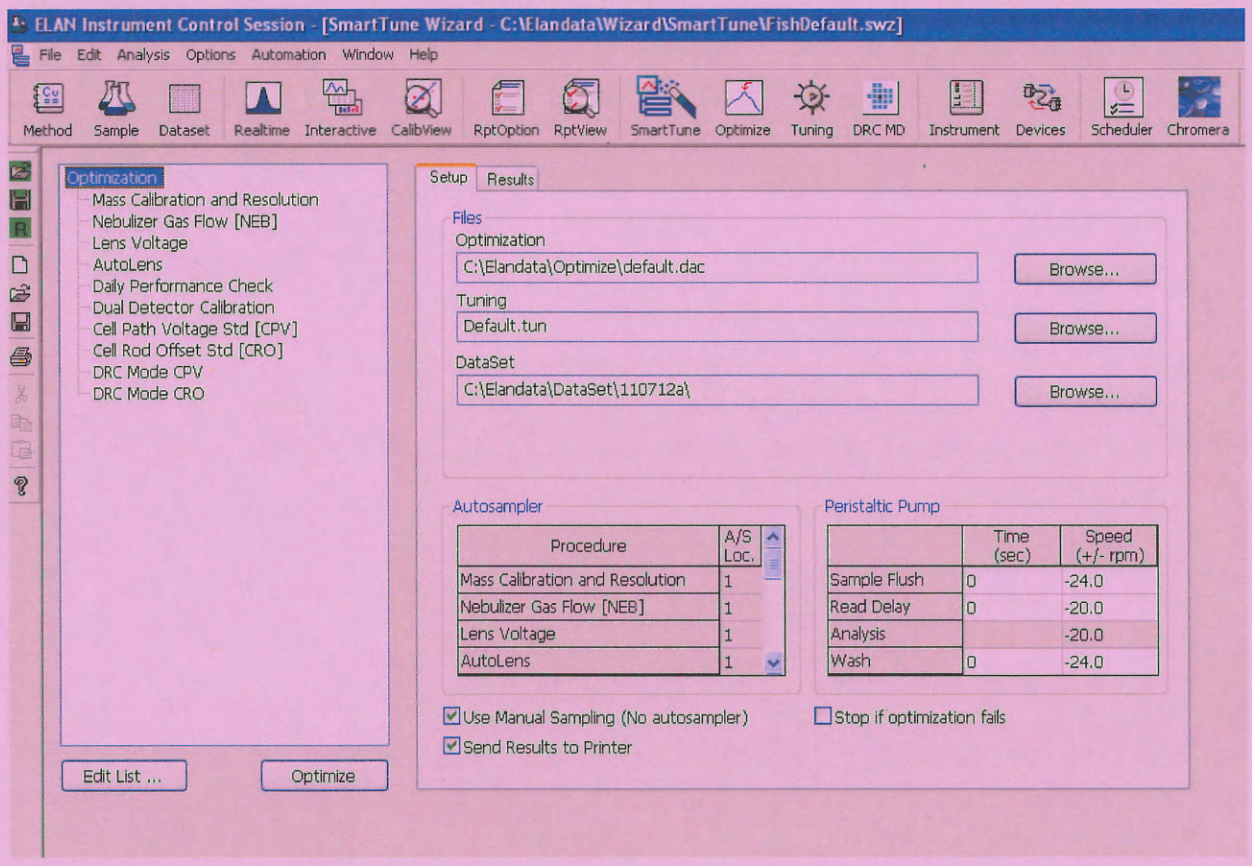

Table 4
Element Data Tool Analyte Cross Table - Fish Monitoring

Edit Cross Table

Table

Analytes [29] | Units |

CSInstrument	CSAnalyte	ELMAnalysis	ELMAnalyte	DataRange	StaticDiln	ISTD
ELAN	As [75]	Arsenic 6020A	Arsenic	9999999		
ELAN	Cd [114]	Cadmium 6020A	Cadmium	9999999		
ELAN	Cu [63]	Copper 6020A	Copper	9999999		
ELAN	Pb [208]	Lead 6020A	Lead	9999999		
ELAN	Se [82]	Selenium 6020A	Selenium	9999999		

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Table 5
Element Data Tool Analyte Cross Table – Full List

Edit Cross Table

Table

Analytes [39] | Units |

CSInstrument	CSAnalyte	ELMAnalysis	ELMAnalyte	DataRange	StaticDiln	ISTD
ELAN	Al [27]	Aluminum 6020A	Aluminum	9999999		
ELAN	Sb [121]	Antimony 6020A	Antimony	9999999		
ELAN	As [75]	Arsenic 6020A	Arsenic	9999999		
ELAN	Ba [138]	Barium 6020A	Barium	9999999		
ELAN	Be [9]	Beryllium 6020A	Beryllium	9999999		
ELAN	Cd [114]	Cadmium 6020A	Cadmium	9999999		
ELAN	Cr [52]	Chromium 6020A	Chromium	9999999		
ELAN	Co [59]	Cobalt 6020A	Cobalt	9999999		
ELAN	Cu [63]	Copper 6020A	Copper	9999999		
ELAN	Fe [56]	Iron 6020A	Iron	9999999		
ELAN	Pb [208]	Lead 6020A	Lead	9999999		
ELAN	Li [7]	Lithium 6020A	Lithium	9999999		
ELAN	Mn [55]	Manganese 6020A	Manganese	9999999		
ELAN	Mo [98]	Molybdenum 6020A	Molybdenum	9999999		
ELAN	Ni [60]	Nickel 6020A	Nickel	9999999		
ELAN	Se [82]	Selenium 6020A	Selenium	9999999		
ELAN	Ag [107]	Silver 6020A	Silver	9999999		
ELAN	Tl [205]	Thallium 6020A	Thallium	9999999		
ELAN	Th [232]	Thorium 6020A	Thorium	9999999		
ELAN	Sn [118]	Tin 6020A	Tin	9999999		
ELAN	U [238]	Uranium 6020A	Uranium	9999999		
ELAN	V [51]	Vanadium 6020A	Vanadium	9999999		
ELAN	Zn [66]	Zinc 6020A	Zinc	9999999		

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