



100 Majestic Way, Bangor, PA 18013 / [www.biospectra.us](http://www.biospectra.us)

ANALYTICAL METHOD OF ANALYSIS:  
DETERMINATION OF ICH Q3D ELEMENTAL  
IMPURITIES + IRON BY INDUCTIVELY COUPLED  
PLASMA MASS SPECTROMETRY (ICP-MS) IN  
SODIUM DECANOATE

## TABLE OF CONTENTS

1. PURPOSE.....	3
2. SCOPE .....	3
3. RESPONSIBILITIES .....	3
4. REFERENCES .....	3
TABLE 1: LIMITS FOR SODIUM DECANOATE (10 GRAM/DAY EXPOSURE).....	4
5. MATERIALS AND EQUIPMENT .....	5
TABLE 2: REFERENCE STANDARDS.....	5
6. PROCEDURE.....	6
TABLE 3: INTERMEDIATE STANDARD .....	7
TABLE 4: 0.5J CALIBRATION STANDARD.....	8
TABLE 5: 1.5J CALIBRATION STANDARD.....	9
TABLE 6: 2.0J CALIBRATION STANDARD.....	10
TABLE 7: CALIBRATION BLANK .....	11
TABLE 8: TEMPERATURE CONTROLLED MICROWAVE DIGESTION PROGRAM .....	11
7. INSTRUMENT PROCEDURE.....	13
TABLE 9: EXAMPLE SAMPLE ANALYSIS SEQUENCE.....	13
TABLE 10: ICP-MS PARAMETERS.....	14
TABLE 11: LINEAR RANGE AND CORRESPONDING TUNE MODE.....	15
8. REPORTING.....	15

## 1. PURPOSE:

- 1.1. To provide a procedure for the assessment of Elemental Impurities + Iron via the NexION 350X S/N 85VN5093001 ICP-MS. This procedure was assessed as a full quantitative option-1 procedure as per validation report BSI-RPT-0866 v1.0 and follows the validation parameters for quantitation procedures as outlined in USP <233>.
- 1.2. Elements under validated for this test method are as follows:
  - 1.2.1. Class 1: Hg, As, Cd, and Pb
  - 1.2.2. Class 2A: Co, V, and Ni
  - 1.2.3. Class 2B: Tl, Au, Pd, Ir, Os, Rh, Ru, Se, Ag, and Pt
  - 1.2.4. Class 3: Li, Sb, Sn, Ba, Mo, Cu, and Cr
  - 1.2.5. Class 4: Fe

## 2. SCOPE:

- 2.1. Applies to Sodium Decanoate and related products manufactured at BioSpectra.
- 2.2. Applies to the NexION 350X S/N 85VN5093001 ICP-MS located in the Quality Control (QC) Laboratory at the BioSpectra Bangor, PA facility.

## 3. RESPONSIBILITIES:

- 3.1. QC management or other qualified designated individual, is responsible for the control, implementation, training, and maintenance of this method.
- 3.2. The QC Staff is responsible for complying with the requirements of this procedure
- 3.3. If any abnormalities are determined during routine use of the ICP-MS or during calibration, the QC Managers shall be promptly notified. If necessary, the ICP-MS will be serviced and recalibrated by Perkin Elmer before being approved for use.

## 4. REFERENCES:

- 4.1. BSI-PRL-0454, Analytical Method Validation Protocol: Determination of ICH Q3D Elemental Impurities by ICP-MS in Sodium Decanoate
- 4.2. BSI-RPT-0866, Analytical Method Validation Report: Determination of ICH Q3D Elemental Impurities by ICP-MS in Sodium Decanoate
- 4.3. BSI-SOP-0303, NexION 350X ICP-MS SOP
- 4.4. BSI-SOP-0304, NexION 350X ICP-MS Care and Maintenance SOP
- 4.5. BSI-SOP-0426, Operation and Maintenance of CEM Mars 6 Digestion Microwave SOP
- 4.6. ICH Guideline for Elemental Impurities Q3D Current
- 4.7. NexION Operation with Syngistix Software Guide
- 4.8. USP <730> Plasma Spectrochemistry
- 4.9. USP <1730> Plasma Spectrochemistry—Theory and Practice
- 4.10. USP <232>, <233>

**TABLE 1: LIMITS FOR SODIUM DECANOATE (10 GRAM/DAY EXPOSURE)**

<b>Element</b>	<b>ICH Class</b>	<b>Parenteral PDE Limits (µg/day)</b>	<b>0.3J LOQ (µg/g) in sample</b>	<b>0.5J Target (µg/g) in sample</b>	<b>1.0J Target (µg/g) in sample</b>	<b>1.5J Target (µg/g) in sample</b>
As	1	15	0.45	0.75	1.5	2.25
Cd	1	2.0	0.06	0.10	0.20	0.30
Hg	1	3.0	0.09	0.15	0.30	0.45
Pb	1	5.0	0.15	0.25	0.50	0.75
Co	2A	5.0	0.15	0.25	0.50	0.75
Ni	2A	20	0.60	1.0	2.0	3.0
V	2A	10	0.30	0.50	1.0	1.5
Tl	2B	8.0	0.24	0.40	0.80	1.2
Se	2B	80	2.4	4.0	8.0	12
Ag	2B	10	0.30	0.50	1.0	1.5
Au	2B	100	3.0	5.0	10	15
Pd	2B	10	0.30	0.50	1.0	1.5
Ir	2B	10	0.30	0.50	1.0	1.5
Os	2B	10	0.30	0.50	1.0	1.5
Pt	2B	10	0.30	0.50	1.0	1.5
Rh	2B	10	0.30	0.50	1.0	1.5
Ru	2B	10	0.30	0.50	1.0	1.5
Ba	3	700	21	35	70	105
Sb	3	90	2.7	4.5	9.0	13.5
Li	3	250	7.5	12.5	25	37.5
Mo	3	1500	45	75	150	225
Cu	3	300	9.0	15	30	45
Sn	3	600	18	30	60	90
Cr	3	1100	33	55	110	165
Fe	4	*50	1.5	2.5	5.0	7.5

\*PDE calculated based on customer specification.

**5. MATERIALS AND EQUIPMENT:**

## 5.1. Equipment

- 5.1.1. Analytical Balance
- 5.1.2. NexION 350X ICP-MS S/N 85VN5093001
- 5.1.3. CEM Mars 6 Digestion Microwave System
- 5.1.4. Micropipettes, Rainin or Eppendorf

## 5.2. Reagents

- 5.2.1. Nitric Acid, Trace metals grade or equivalent
- 5.2.2. Hydrochloric Acid, Trace metals grade or equivalent
- 5.2.3. Sulfuric acid, Trace metals grade or equivalent
- 5.2.4. Deionized (DI) water (Type 1 Ultrapure)
- 5.2.5. Thiourea, 99+ % grade
- 5.2.6. NexION Setup and KED Setup Solution

## 5.3. Consumable Supplies

- 5.3.1. SCP Digitubes<sup>®</sup> 15 mL, 50 mL and 100 mL
- 5.3.2. Pipette Tips of various sizes
- 5.3.3. SiliaPrep MB SPE Cartridges, Silica-Based AMPA, 500 mg, 4 mL, 40 - 63  $\mu\text{m}$ , 60  $\text{\AA}$

## 5.4. Personnel

- 5.4.1. All personnel that executed the protocol are trained on ICP-MS or are considered Subject Matter Experts. This test method will be assigned a mark as read training to QC analysts involved with the execution.

**TABLE 2: REFERENCE STANDARDS**

Identification**	Manufacturer	Concentrations / Elements
Pharma-CAL Standard Parenteral STD# 1 IA 140-131-201*	SCP Science	Ag (10 $\mu\text{g/mL}$ ), As (15 $\mu\text{g/mL}$ ), Cd (2 $\mu\text{g/mL}$ ), Co (5 $\mu\text{g/mL}$ ), Hg (3 $\mu\text{g/mL}$ ), Ni (20 $\mu\text{g/mL}$ ), Pb (5 $\mu\text{g/mL}$ ), Se (80 $\mu\text{g/mL}$ ), Tl (8 $\mu\text{g/mL}$ ), V (10 $\mu\text{g/mL}$ )
USP232/ICH Q3D Parenteral STD# 2 IA 140-131-215*	SCP Science	Au (100 $\mu\text{g/mL}$ ); Ir, Os, Pd, Pt, Rh, & Ru (10 $\mu\text{g/mL}$ )
Pharma-CAL Standard Parenteral STD# 3 IA 140-131-221*	SCP Science	Ba (700 $\mu\text{g/mL}$ ), Cr (1,100 $\mu\text{g/mL}$ ), Cu (300 $\mu\text{g/mL}$ ), Li (250 $\mu\text{g/mL}$ ), Mo (1,500 $\mu\text{g/mL}$ ), Sb (90 $\mu\text{g/mL}$ ), Sn (600 $\mu\text{g/mL}$ )
Iron Stock Standard	Perkin Elmer	Fe (1,000 $\mu\text{g/mL}$ )
Pharma-CAL Custom Standard AQ0-086-125* (Internal Standard)	SCP Science	Be, Sc, Y, Re (10 $\mu\text{g/mL}$ ); Te (25 $\mu\text{g/mL}$ ); Ge, Tb, Bi (5 $\mu\text{g/mL}$ )

\* SCP Science catalog numbers ending in 1 denote 125 mL bottle sizes and catalog numbers ending in 5 denote 500 mL bottle sizes.

\*\* Additional standards/custom standards can be used as long as the concentration remains the same in final preparations.

## 6. PROCEDURE:

- 6.1. All standards will be prepared volumetrically from stock solutions purchased from certified vendors. If the vendor supplied stock standard is within 2% of the nominal value as per the certificate of analysis, then the nominal value will be used to calculate the concentration of the standard. If the stock standard certificate of analysis value is greater than or less than 2% of the nominal value, then the certificate of analysis value will be used for the stock standard concentration.
- 6.2. Acid Digestion Mix
- 6.3. [2:1] Nitric Acid (HNO<sub>3</sub>): Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>) (Prepare same *day*)
  - 6.3.1. Caution: Combining nitric acid and sulfuric acid generates excessive heat. Never seal cap tightly before solution has completely cooled.
  - 6.3.2. To prepare, add 50 mL of nitric acid to a 100 mL Digitube<sup>®</sup> and then slowly add 25 mL of sulfuric acid. Solution can be placed in a cold-water bath to aid cooling.
    - 6.3.2.1. Scale as necessary for use (Prepare same day).
- 6.4. Internal Standard/Complexing Solution
  - 6.4.1. Weigh approximately 1.0 gram of Thiourea into a 50 mL Digitube<sup>®</sup>.
  - 6.4.2. Add approximately 20 mL of deionized water and mix to dissolve.
  - 6.4.3. Filter solution through a SiliaPrep Cation Solid Phase Extraction (SPE) cartridge into a separate 50 mL digitube.
  - 6.4.4. Add 2.5 mL of Internal Standard Intermediate followed by 25 mL of hydrochloric acid.
  - 6.4.5. Dilute to a final volume of 50 mL with deionized water and mix well.
  - 6.4.6. Scale proportionally as needed for use.
- 6.5. 2% Thiourea Solution
  - 6.5.1. Weigh approximately 1.0 gram of Thiourea into a 50 mL Digitube<sup>®</sup>.
  - 6.5.2. Add approximately 20 mL of deionized water and mix to dissolve.
  - 6.5.3. Filter solution through a SiliaPrep Cation Solid Phase Extraction (SPE) cartridge into a separate 50 mL digitube.
  - 6.5.4. Dilute to a final volume of 50 mL with deionized water and mix well.
  - 6.5.5. Scale proportionally as needed for use.

## 6.6. Intermediate Standard Preparation

- 6.6.1. Prepare a standard solution containing the elements listed in Table 3, using the standards STD#1 IA, STD#2 IA, STD#3 IA, and additional single source iron stock standard.
- 6.6.2. Prepare by adding stock standards to a 15 mL Digitube®.
- 6.6.3. Dilute to approximately 8 mL using deionized water and add hydrochloric acid.
- 6.6.4. Dilute to final volume using DI Water and mix well.

TABLE 3: INTERMEDIATE STANDARD

Identification	Element	Stock Identification	Amount Added (mL)	HCl (mL)	Final Volume (mL)	Final Concentration (µg/mL)
Intermediate Standard	As	STD# 1 IA 140-131-201*	1.0	1.0	10	1.5
	Cd					0.20
	Hg					0.30
	Pb					0.50
	Co					0.50
	Ni					2.0
	V					1.0
	Tl					0.80
	Se					8.0
	Ag					1.0
	Au	STD# 2 IA 140-131-215*	1.0			10
	Pd					1.0
	Ir					1.0
	Os					1.0
	Pt					1.0
	Rh					1.0
	Ru					1.0
	Ba	STD# 3 IA 140-131-221*	1.0			70
	Sb					9.0
	Li					25
Mo	150					
Cu	30					
Sn	60					
Cr	110					
Fe	1,000 µg/mL Fe Std	0.050	5.0			

\* SCP Science catalog numbers ending in 1 denote 125 mL bottle sizes and catalog numbers ending in 5 denote 500 mL bottle sizes.

## 6.7. 0.5J Calibration Standard Preparation

- 6.7.1. Prepare a solution containing the elements listed in Table 4 below in 5.33% HNO<sub>3</sub>, 2.67% H<sub>2</sub>SO<sub>4</sub>, 1.0% HCl and 0.04% (400 µg/mL) Thiourea.
- 6.7.2. Add 0.050 mL of intermediate standard to separate 50 mL Digitube<sup>®</sup> followed by addition of approximately 35 mL of deionized water.
- 6.7.3. Add 4.0 mL of Acid Mixture then dilute to 45 mL using deionized water.
- 6.7.4. Add 1.0 mL of internal standard/complexing solution and dilute to volume using deionized water.
- 6.7.5. Do not allow intermediate standard to contact concentrated acids while preparing solutions. (Standards are stable for 24 hours)

**TABLE 4: 0.5J CALIBRATION STANDARD**

Identification	Element	Intermediate Standard (mL)	Acid Mix (mL)	Internal Standard/Complexing Solution (mL)	Final Volume (mL)	Final Concentration (µg/L)
<b>0.5J Calibration Standard</b>	As	0.050	4.0	1.0	50	1.5
	Cd					0.20
	Hg					0.30
	Pb					0.50
	Co					0.50
	Ni					2.0
	V					1.0
	Tl					0.80
	Se					8.0
	Ag					1.0
	Au					10
	Pd					1.0
	Ir					1.0
	Os					1.0
	Pt					1.0
	Rh					1.0
	Ru					1.0
	Ba					70
	Sb					9.0
	Li					25
Mo	150					
Cu	30					
Sn	60					
Cr	110					
Fe	5.0					

The information contained herein is the confidential property of BioSpectra. The recipient is responsible for its safe-keeping and the prevention of unauthorized appropriation, use, disclosure and copying.



## 6.8. 1.5J Calibration Standard Preparation

- 6.8.1. Prepare a solution containing the elements listed in Table 5 below in 5.33% HNO<sub>3</sub>, 2.67% H<sub>2</sub>SO<sub>4</sub>, 1.0% HCl and 0.04% (400 µg/mL) Thiourea.
- 6.8.2. Add 0.150 mL of intermediate standard to separate 50 mL Digitube<sup>®</sup> followed by addition of approximately 35 mL of deionized water.
- 6.8.3. Add 4.0 mL of Acid Mixture then dilute to 45 mL using deionized water.
- 6.8.4. Add 1.0 mL of internal standard/complexing solution and dilute to volume using deionized water.
- 6.8.5. Do not allow intermediate standard to contact concentrated acids while preparing solutions. (Standards are stable for 24 hours)

**TABLE 5: 1.5J CALIBRATION STANDARD**

Identification	Element	Intermediate Standard (mL)	Acid Mix (mL)	Internal Standard/Complexing Solution (mL)	Final Volume (mL)	Final Concentration (µg/L)
<b>1.5J Calibration Standard</b>	As	0.150	4.0	1.0	50	4.5
	Cd					0.60
	Hg					0.90
	Pb					1.5
	Co					1.5
	Ni					6.0
	V					3.0
	Tl					2.4
	Se					24
	Ag					3.0
	Au					30
	Pd					3.0
	Ir					3.0
	Os					3.0
	Pt					3.0
	Rh					3.0
	Ru					3.0
	Ba					210
	Sb					27
	Li					75
Mo	450					
Cu	90					
Sn	180					
Cr	330					
Fe	15					

The information contained herein is the confidential property of BioSpectra. The recipient is responsible for its safe-keeping and the prevention of unauthorized appropriation, use, disclosure and copying.

## 6.9. 2.0J Calibration Standard Preparation

- 6.9.1. Prepare a solution containing the elements listed in Table 6 below in 5.33% HNO<sub>3</sub>, 2.67% H<sub>2</sub>SO<sub>4</sub>, 1.0% HCl and 0.04% (400 µg/mL) Thiourea.
- 6.9.2. Add 0.200 mL of intermediate standard to separate 50 mL Digitube<sup>®</sup> followed by addition of approximately 35 mL of deionized water.
- 6.9.3. Add 4.0 mL of Acid Mixture then dilute to 45 mL using deionized water.
- 6.9.4. Add 1.0 mL of internal standard/complexing solution and dilute to volume using deionized water.
- 6.9.5. Do not allow intermediate standard to contact concentrated acids while preparing solutions. (Standards are stable for 24 hours)

**TABLE 6: 2.0J CALIBRATION STANDARD**

Identification	Element	Intermediate Standard (mL)	Acid Mix (mL)	Internal Standard/Complexing Solution (mL)	Final Volume (mL)	Final Concentration (µg/L)
<b>2.0J Calibration Standard</b>	As	0.200	4.0	1.0	50	6.0
	Cd					0.80
	Hg					1.2
	Pb					2.0
	Co					2.0
	Ni					8.0
	V					4.0
	Tl					3.2
	Se					32
	Ag					4.0
	Au					40
	Pd					4.0
	Ir					4.0
	Os					4.0
	Pt					4.0
	Rh					4.0
	Ru					4.0
	Ba					280
	Sb					36
	Li					100
Mo	600					
Cu	120					
Sn	240					
Cr	440					
Fe	20					

The information contained herein is the confidential property of BioSpectra. The recipient is responsible for its safe-keeping and the prevention of unauthorized appropriation, use, disclosure and copying.

## 6.10. Calibration Blank

- 6.10.1. Prepare a solution containing 5.33% HNO<sub>3</sub>, 2.67% H<sub>2</sub>SO<sub>4</sub>, 1.0% HCl and 0.04% (400µg/mL) Thiourea as described in Table 7 below.
- 6.10.2. To a separate 50 mL Digitube<sup>®</sup>, add approximately 35 mL of DI Water.
- 6.10.3. Add 4.0 mL of Acid Mixture then dilute to 45 mL using DI Water.
- 6.10.4. Add 1.0 mL of internal standard/complexing solution and dilute to volume using DI Water.
- 6.10.5. Do not allow Internal Standard Solution to contact concentrated acids.

TABLE 7: CALIBRATION BLANK

Description	Acid Mix (mL)	Internal Standard/Complexing Solution (mL)	Final Volume (mL)
Cal Blank	4.0	1.0	50

## 6.11. Method Blank Preparation

- 6.11.1. Add 4.0 mL of Acid Digestion Mixture into a clean 20 mL digestion vessel, place a plug on the vessel, and properly torque the vessel cap. Place the vessel in the microwave carousel then digest and complete preparation according to Section 6.12 below.

## 6.12. Sample Preparation

- 6.12.1. Samples are stable for 24 hours after preparation time.
- 6.12.2. Weigh approximately 100 mg of sample into a clean 20 mL digestion vessel and add 4.0 mL of Acid Digestion Mixture.
- 6.12.3. Properly torque vessel cap and place in microwave carousel. Digest and complete preparation according to Section 6.12 below.

## 6.13. Microwave Digestion Procedure

- 6.13.1. Refer to BSI-SOP-0426 for general usage guidelines of the Mars 6 Microwave Digestion System.
- 6.13.2. Prepare at least one method blank per digestion run. Method blank is prepared in the same manner as the sample without the addition of the sample (see above).
- 6.13.3. If less than 4 total vessels are to be digested, add additional “dummy” samples so that no fewer than 4 vessels are used in a single digestion. “dummy” samples can be discarded after digestion is complete.
- 6.13.4. Digest the sample vessels using the program listed in Table 8.

TABLE 8: TEMPERATURE CONTROLLED MICROWAVE DIGESTION PROGRAM

Power (Watts)	Percent Power	Ramp (Minutes)	Temperature (°C)	Hold (Minutes)
1800	100	15:00	150	10:00
1800	100	6:00	175	15:00

- 6.13.5. After digestion, place the vessels into an ice bath and allow the vessels to cool for approximately 40 minutes. Before opening, turn the vessels sideways and slowly rotate in order to collect the condensation on the inside of the vessel walls.
- 6.13.6. Quantitatively transfer the vessel contents into a 50 mL Digitube<sup>®</sup> containing approximately 5 mL of deionized water and 1.0 mL of Internal Standard/Complexing Solution. Rinse the bottom of the plug into the 50 mL Digitube<sup>®</sup> using deionized water.

- 6.13.7. Extract any remaining volatile elements by adding 15 mL of a pre-mixed solution of 0.500 mL of 2% Thiourea diluted to 15 mL using deionized water to the digestion vessel. Add this directly to the 50 mL Digitube®.
- 6.13.8. Rinse the vessel an additional two more times using deionized water and transfer each rinse to the 50 mL Digitube®. Dilute to a final volume of 50 mL using deionized water and mix well.
- 6.14. Isobaric Overlap Corrections
- 6.14.1. An isobaric interference results from equal mass isotopes of different elements present in the sample solution. Analysis sequences that are processed utilizing multi-element standards will require the use of correction equations to compensate for known isobaric overlaps originating from the elemental standard and sample. The following correction equations should be used:

**KED Mode:**

$$M_c(58) = M_u(58) \times 1 - M_{(rm)}(57) \times 0.13208$$

$$M_c(98) = M_u(98) \times 1 - M_{(rm)}(99) \times 0.14655$$

$$M_c(106) = M_u(106) \times 1 - M_{(rm)}(111) \times 0.09766$$

$$M_c(108) = M_u(108) \times 1 - M_{(rm)}(111) \times 0.06953$$

$$M_c(120) = M_u(120) \times 1 - M_{(rm)}(125) \times 0.01273$$

$$M_c(123) = M_u(123) \times 1 - M_{(rm)}(125) \times 0.12588$$

$$M_c(190) = M_u(190) \times 1 - M_{(rm)}(195) \times 0.00036$$

$$M_c(192) = M_u(192) \times 1 - M_{(rm)}(195) \times 0.02315$$

$$M_c(196) = M_u(196) \times 1 - M_{(rm)}(202) \times 0.005023$$

The correction equations can be derived from the following equation:

$$M_c = M_u - [M_{(rm)} \times (A_{(ie)}/A_{(rm)})]$$

**Where:**

$M_c$  = Corrected Count Rate for the analyte

$M_u$  = Uncorrected count rate for the analyte

$M_{(rm)}$  = Count Rate of Reference Mass (rm) for the Interfering Element

$A_{(ie)}$  = Percent Abundance of Interfering Element (ie) at the analyte mass

$A_{(rm)}$  = Percent Abundance of Interfering Element at the Reference Mass (rm)

**Example:**

$$M_c(58) = M_u(58) \times 1 - M_{(rm)}(57) \times (0.28 / 2.12)$$

- 6.14.2. All correction coefficients were calculated based on the Agilent Technologies 2016 Relative Isotopic Abundance Table.
- 6.14.3. Multiplier used in the correction equation may differ slightly from the multiplier used in the Syngistix instrument method due to rounding.

**7. INSTRUMENT PROCEDURE:**

- 7.1. Perform the ICP-MS daily performance check prior to beginning the analytical sequence. Refer to NexION 350X ICP-MS SOP BSI-SOP-0303 for Daily Check procedures.
- 7.2. A calibration curve of no less than two standards and a blank must be used. The calibration correlation coefficient (R) must be  $\geq 0.99$ .
- 7.3. Set up the sequence as per Table 9.
- 7.4. Confirm the calibration by analyzing the 1.5J standard after the calibration. The calibration check must recover  $\pm 20\%$  of the calculated theoretical concentration for multi-element analysis and  $\pm 10\%$  for single element determinations.
- 7.5. The check standard must be verified after the calibration. A re-analysis of the check standard will be performed a minimum of once every 10 samples and at the end of the analytical run.
- 7.6. The drift between the bracketing standard checks must be NMT 20% for each Target element.
- 7.7. The sample concentration is calculated as:

$$\text{Conc. } (\mu\text{g/g}) = \frac{\text{Solution Conc. } (\mu\text{g/L}) \times \text{Solution vol. (L)} \times \text{Dilution Factor}}{\text{Sample Mass (g)}}$$

**TABLE 9: EXAMPLE SAMPLE ANALYSIS SEQUENCE**

<b>ID</b>	<b>Type</b>	<b>Level</b>
Cal Blank	Cal Blank	Level 1
0.5J Cal Std	Cal Std	Level 2
1.5J Cal Std	Cal Std	Level 3
2.0J Cal Std	Cal Std	Level 4
Cal Blank Check	QC Check	N/A
1.5J Check Std 1	QC Check	N/A
Method Blank	Sample	N/A
Sample(s) 10 or less	Sample	N/A
1.5J Check Std 2	QC Check	N/A

## 7.8. Instrument Setup and Parameters

- 7.8.1. Instrument settings are only listed as guidelines. Settings may be changed in order to accommodate changes in sample matrix or hardware configurations.
- 7.8.2. The AMS-II makeup gas must be engaged during analysis using a minimum dilution gas ratio of 15%.
- 7.8.3. The elements arsenic, iron, and selenium can be analyzed using hydrogen reaction gas in order to remove poly atomic interferences. A hydrogen DRC flow rate of approximately 4 mL/min should be used.
- 7.8.4. The instrument method is stored under the Approved Test Method Folder labelled as "Sodium\_Decanoate\_EI.mth" for elemental impurities testing.
- 7.8.5. The internal standard recovery can vary in the samples and final bracketing check standard. This is caused by the sample matrix differing greatly from the standards and a suppressive or enhancement effect occurs depending on ionization energy potential.

**TABLE 10: ICP-MS PARAMETERS**

<b>ICP-MS System</b>	Perkin Elmer NexION350X Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Syngistix Software
<b>Sweeps/Readings</b>	20
<b>Replicates</b>	3
<b>Nebulizer Gas</b>	Argon
<b>Collision Gas</b>	Helium
<b>Reaction Gas</b>	Hydrogen
<b>Dilution Gas</b>	Argon
<b>Sample and Skimmer Cone</b>	Platinum
<b>Sample Rinses</b>	Rinse-1: 60 sec at 45 rpm 5.0% HNO <sub>3</sub> , 2.5% HCl, with 0.04% Thiourea or as applicable to mitigate carry over

TABLE 11: LINEAR RANGE AND CORRESPONDING TUNE MODE

Isotope	Internal Standard	Mode	Linear Range (µg/L)	Isotope	Internal Standard	Mode	Linear Range (µg/L)
7Li	9Be	STD	15-100	113Cd	125Te	KED	0.12-0.80
51V	45Sc	KED	0.60-4.0	118Sn	125Te	KED	36-240
52Cr	45Sc	KED	66-440	119Sn	125Te	KED	36-240
53Cr	45Sc	KED	66-440	120Sn	125Te	KED	36-240
56Fe	72Ge	H <sub>2</sub> DRC	3.0-20	121Sb	125Te	KED	5.4-36
57Fe	72Ge	KED	3.0-20	123Sb	125Te	KED	5.4-36
58Ni	72Ge	KED	1.2-8.0	135Ba	159Tb	KED	42-280
59Co	72Ge	KED	0.30-2.0	137Ba	159Tb	KED	42-280
60Ni	72Ge	KED	1.2-8.0	138Ba	159Tb	KED	42-280
62Ni	72Ge	KED	1.2-8.0	188Os	209Bi	KED	0.60-4.0
63Cu	72Ge	KED	18-120	189Os	209Bi	KED	0.60-4.0
65Cu	72Ge	KED	18-120	190Os	209Bi	KED	0.60-4.0
75As	72Ge	H <sub>2</sub> DRC	0.90-6.0	191Ir	209Bi	KED	0.60-4.0
75As	72Ge	KED	0.90-6.0	192Os	209Bi	KED	0.60-4.0
77Se	89Y	H <sub>2</sub> DRC	4.8-32	193Ir	209Bi	KED	0.60-4.0
78Se	89Y	H <sub>2</sub> DRC	4.8-32	194Pt	185Re	KED	0.60-4.0
95Mo	89Y	KED	90-600	195Pt	185Re	KED	0.60-4.0
97Mo	89Y	KED	90-600	196Pt	185Re	KED	0.60-4.0
98Mo	89Y	KED	90-600	197Au	209Bi	KED	6.0-40
99Ru	125Te	KED	0.60-4.0	199Hg	185Re	KED	0.18-1.2
101Ru	125Te	KED	0.60-4.0	200Hg	185Re	KED	0.18-1.2
103Rh	125Te	KED	0.60-4.0	202Hg	185Re	KED	0.18-1.2
105Pd	125Te	KED	0.60-4.0	203Tl	209Bi	KED	0.48-3.2
106Pd	125Te	KED	0.60-4.0	205Tl	209Bi	KED	0.48-3.2
107Ag	125Te	KED	0.60-4.0	206Pb	209Bi	KED	0.30-2.0
108Pd	125Te	KED	0.60-4.0	207Pb	209Bi	KED	0.30-2.0
109Ag	125Te	KED	0.60-4.0	208Pb	209Bi	KED	0.30-2.0

## 8. REPORTING

- 8.1. Any result below the 0.3J target concentration will be reported as less than the corresponding LOQ value listed in Table 1. Results above the LOQ concentration will be reported in µg/g and to 2 significant figures. Report the average result for multiple isotopes of the same element that are above the LOQ concentration.