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ANALYTICAL METHOD VALIDATION REPORT:  
QUANTIFICATION OF SODIUM BY INDUCTIVELY  
COUPLED OPTICAL EMISSION SPECTROMETRY  
(ICP-OES) IN SODIUM DECANOATE

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**1. PURPOSE:**

- 1.1. The purpose of this validation report is to establish documented evidence that the test protocol, BSI-PRL-0467 v. 1.0, for Sodium Quantification in Sodium Decanoate performs according to BioSpectra Analytical Method Validation Master Plan and USP requirements.

**2. SCOPE:**

- 2.1. Applies to Sodium Decanoate and related products manufactured at BioSpectra.
- 2.2. Applies to the Avio 500 S/N 081S1905062 ICP-OES located in the Quality Control (QC) Laboratory at the BioSpectra Bangor, PA facility.
- 2.3. This report applies to the protocol validation for sodium quantification in sodium decanoate, by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) performed at Biospectra Inc.

**3. REFERENCES:**

- 3.1. BSI-PRL-0467, Sodium Quantification Method Validation Protocol
- 3.2. BSI-SOP-0362, Avio 500 ICP-OES SOP
- 3.3. BSI-SOP-0436, Analytical Method Validation Master Plan
- 3.4. USP <730> Plasma Spectrochemistry
- 3.5. USP <1730> Plasma Spectrochemistry—Theory and Practice

**4. BACKGROUND:**

- 4.1. This validation was executed as a Category I Quantitative Analytical Method.
- 4.2. The test protocol validation report includes the following parameters:
  - 4.2.1. Specificity
  - 4.2.2. Linearity and Range
  - 4.2.3. Accuracy
  - 4.2.4. Precision (Repeatability)
  - 4.2.5. Intermediate Precision (Ruggedness)
  - 4.2.6. Standard and Sample Solution Stability

**5. MATERIALS AND EQUIPMENT:**

<b>TABLE 1: EQUIPMENT</b>				
<b>Type</b>	<b>Supplier</b>	<b>Model</b>	<b>Serial Number</b>	<b>Cal. Due</b>
Analytical Balance <sup>1</sup>	Sartorius	MSE224S	36707108	04/2022 10/2022
Automatic Pipette <sup>1</sup>	Rainin	E4-XLS (20-200 µL)	C016314640	06/30/22 12/31/22
Automatic Pipette <sup>1</sup>	Rainin	E4-XLS (100-1000 µL)	C016314969	06/30/22 12/31/22
Automatic Pipette <sup>1</sup>	Rainin	E4-XLS (0.5-5 mL)	C023506909	06/30/22 12/31/22
ICP-OES <sup>1</sup>	Perkin Elmer	Avio 500	081S1905062	09/2022 09/2023
Digestion Microwave <sup>1</sup>	CEM	Mars 6	MY2255	09/22/22 09/08/22
Deionized Water System <sup>1</sup>	Millipore	IQ-7005/ Element POD	F9SA14284H	06/2022 06/2023

<sup>1</sup>Additional calibration due dates from third experiment for intermediate precision as all equipment was serviced between experiments.

<b>TABLE 2: REAGENTS</b>					
<b>Type</b>	<b>Grade</b>	<b>Supplier</b>	<b>Catalog Number</b>	<b>Lot Number</b>	<b>Expiration</b>
70% Nitric Acid	Trace Metal	VWR	87003-261	1121060	07/12/23
70% Nitric Acid	Trace Metal	VWR	87003-261	1122020	02/16/24
Sulfuric Acid	Trace Metal	Fisher	A510-P212	3119020	05/21/22
Sulfuric Acid	Trace Metal	Spectrum	S1136	3121040	06/30/24
Deionized water	Type 1 Ultrapure	In-House	N/A	N/A	N/A

**5.1. Consumable Supplies**

5.1.1. SCP Digitubes® 15 mL, 50 mL and 100 mL

5.1.2. Pipette Tips of various sizes

<b>TABLE 3: REFERENCE STANDARDS</b>				
<b>Identification</b>	<b>Manufacturer</b>	<b>Lot Number</b>	<b>Expiration</b>	<b>Concentrations / Elements</b>
Manganese Stock Standard N9303783	Perkin Elmer	25-71MNY1	10/30/22	Mn (1,000 µg/mL)
Scandium Stock Standard N9303798	Perkin Elmer	25-96SCY1	01/30/23	Sc (1,000 µg/mL)
Sodium Stock Standard 140-061-111	SCP Science	S200709003	03/29/22	Na (10,000 µg/mL)
Sodium Stock Standard N9304124	Perkin Elmer	AU18-172NAY1	08/30/23	Na (10,000 µg/mL)
Yttrium Stock Standard N9303810	Perkin Elmer	25-126YY1	02/28/23	Y (1,000 µg/mL)

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**6. PROCEDURE:**

- 6.1. All standards were prepared volumetrically from stock solutions purchased from certified vendors. If the vendor supplied stock standard was within 2% of the nominal value as per the certificate of analysis, then the nominal value was used to calculate the concentration of the standard. If the stock standard certificate of analysis value was greater than or less than 2% of the nominal value, then the certificate of analysis value was used for the stock standard concentration.
- 6.2. Acid Digestion Mixture (Acid Mix)  
 [2:1] Nitric Acid (HNO<sub>3</sub>) : Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>)
- 6.2.1. Added 50 mL of nitric acid to a 100 mL Digitube<sup>®</sup> and then slowly added 25 mL of sulfuric acid. Scaled as required.
- 6.2.2. Solution was placed in a cold-water bath to aid cooling and was prepared day of use.
- 6.3. Internal Standard Solution
- 6.3.1. Added 100 µL of 1,000 µg/mL Scandium Stock Standard and 100 µL of 1,000 µg/mL Yttrium Stock Standard into a 15 mL Digitube<sup>®</sup>.
- 6.3.2. Diluted to a final volume of 10 mL using deionized water and mixed well.
- 6.3.3. Scaled proportionally as needed for use.
- 6.4. 1,000 µg/mL Sodium Intermediate Stock Standard
- 6.4.1. Added 1.0 mL of 10,000 µg/mL Stock Sodium Stock Standard into a 15 mL Digitube<sup>®</sup>.
- 6.4.2. Diluted to a final volume of 10 mL using deionized water and mixed well.
- 6.4.3. Scaled proportionally as needed for use.
- 6.5. Calibration Standard Preparation
- 6.5.1. Prepared calibration blank and calibration standards, as per Table 4 below, using the intermediate and internal standard solution prepared above. Added intermediate standard to separate 50 mL Digitubes<sup>®</sup> followed by addition of internal standard. Added DI water to approximately 45 mL then added nitric acid. Diluted to final volume using DI Water.

TABLE 4: CALIBRATION STANDARDS						
Identification	Element	Intermediate Standard (mL)	Internal Standard (mL)	Nitric Acid (mL)	Final Volume (mL)	Final Concentration (µg/L)
Cal Blank	Na	0.000	0.100	2.5	50	0.0
1.0 ppm Std		0.050				1.0
2.5 ppm Std		0.125				2.5
5.0 ppm Std		0.250				5.0
10 ppm Std		0.500				10

## 6.6. Method Blank Preparation

6.6.1. Pipetted 4.0 mL of Acid Digestion Mixture into a clean 20 mL digestion vessel, placed a plug on the vessel, and properly torqued the vessel cap. Placed vessel in the microwave carousel then digested and completed preparation according to Section 6.8 below.

## 6.7. Sample Preparation

6.7.1. Weighed approximately 100 mg of sample into a clean 20 mL digestion vessel and added 4.0 mL of Acid Digestion Mixture.

6.7.2. Placed a plug on the vessels and properly torqued vessel cap before placing in the microwave carousel. Digested and completed sample preparation according to Section 6.8 below.

## 6.8. Microwave Digestion Procedure

6.8.1. Referred to BSI-SOP-0426 for general usages and guidelines for the Mars 6 Microwave Digestion System.

6.8.2. Prepared at least one method blank per digestion run. Method blank was prepared in the same manner as the samples without the addition of actual sample.

6.8.3. Digested the samples using the program listed in Table 5 below.

<b>Power (Watts)</b>	<b>Percent Power</b>	<b>Ramp (Minutes)</b>	<b>Temperature (°C)</b>	<b>Hold (Minutes)</b>
1800	100	15:00	150	10:00
1800	100	6:00	175	5:00

6.8.4. After digestion, the digestion vessels were placed in an ice bath and allowed the vessels to cool for approximately 40 minutes. Before opening, the vessels were turned sideways and slowly rotated in order to collect the condensation on the inside walls of the vessels.

6.8.5. Quantitatively transferred the vessel contents into a 50 mL Digitube® containing 5 mL of deionized water. Rinsed the bottom of the plug into the same 50 mL Digitube® using deionized water.

6.8.6. Rinsed the vessel an additional two times with deionized water and transferred each rinse to the 50 mL Digitube®. Diluted to final volume of 50 mL using deionized water and mixed well.

6.8.7. Performed an additional dilution for each sample by pipetting 1.0 mL of the digested sample into another 50 mL Digitube®.

6.8.8. Pipetted 100 µL of internal standard and diluted to 45 mL using deionized water.

6.8.9. Added 2.5 mL of nitric acid then diluted to final volume of 50 mL using deionized water and mixed well.

**7. INSTRUMENT PROCEDURE:**

- 7.1. Performed the ICP-OES daily performance check prior to beginning the analytical sequence. Refer to Avio 500 ICP-OES SOP DCN BSI-SOP-0362 for Daily Check procedures.
- 7.2. A calibration curve of no less than two standards and a blank was used. The calibration correlation coefficient (R) was  $\geq 0.995$ .
- 7.3. Set up the sequence as per Table 6.
- 7.4. Confirmed the calibration by analyzing the 2.5 ppm standard after the calibration. The calibration check must recover  $\pm 10\%$  of the calculated theoretical concentration for single element determinations.
- 7.5. The check standard was verified after each calibration. A re-analysis of the check standard was to 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 10% for each Target wavelength.
- 7.7. The sample concentration is calculated as:

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

<b>TABLE 6: EXAMPLE SAMPLE ANALYSIS SEQUENCE</b>		
<b>ID</b>	<b>Type</b>	<b>Level</b>
Cal Blank	Cal Blank	Level 1
1.0 ppm Std	Cal Std	Level 2
2.5 ppm Std	Cal Std	Level 3
5.0 ppm Std	Cal Std	Level 4
10 ppm Std	Cal Std	Level 5
Cal Blank Check	QC Check	N/A
2.5 ppm Check Std 1	QC Check	N/A
Method Blank	Sample	N/A
Sample(s) 10 or less	Sample	N/A
2.5 ppm 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 Gas Flows for Plasma, Auxiliary, and Nebulizer were set at 12 mL/min, 0.20 mL/min, and 0.60 mL/min, respectively.

<b>TABLE 7: ICP-OES PARAMETERS</b>	
<b>ICP-OES System</b>	Perkin Elmer Avio 500 Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) with Syngistix Software
<b>Points per Peak</b>	3
<b>Replicates</b>	3
<b>Viewing Distance</b>	15.0
<b>Nebulizer Gas</b>	Argon
<b>Shear Gas</b>	Compressed Air + Nitrogen
<b>Sample Rinse</b>	Rinse-1: 30 sec at 1.0 mL/min 5.0% HNO <sub>3</sub> (or as applicable to mitigate carry over)

<b>TABLE 8: LINEAR RANGE AND CORRESPONDING MODE</b>			
<b>Element</b>	<b>Mode</b>	<b>Wavelength (nm)</b>	<b>Linear Range (µg/mL)</b>
Na	Axial	330.237	1.0-10
		588.995	
		589.592	



## 7.9. Linearity and Range

7.9.1. The ICP-OES linearity study included standards equivalent to the following concentrations: 1.0 ppm, 2.5 ppm, 4.0 ppm, 5.0 ppm, 7.5 ppm, and 10 ppm. Each standard was prepared in triplicate and analyzed against the calibration curve using the calibration standards prepared above in Section 6.5. The average standard recovery for each level of the three replicates was then determined.

### 7.9.1.1. Acceptance Criteria:

7.9.1.1.1. The mean standard recovery for sodium at each of the linearity levels must be in the range of 80% -120%.

7.9.2. The linearity study was further analyzed by plotting signal versus concentration along with performing statistical regression analysis based on theoretical concentration versus observed concentration for linearity standards at each wavelength. The  $R^2$  values for the linear trendlines of concentrations versus signal was greater than 0.995 for all wavelengths. All wavelengths showed greater than 0.995  $R^2$  values for regression analysis as well. All further data analysis is shown in supporting information of report.

Description	Intermediate Standard (mL)	Internal Standard (mL)	Nitric Acid (mL)	Final Volume (mL)
Cal Blank	N/A	0.100	2.5	50
1.0 ppm Std	0.050	0.100	2.5	50
2.5 ppm Std	0.125	0.100	2.5	50
4.0 ppm Std	0.200	0.100	2.5	50
5.0 ppm Std	0.250	0.100	2.5	50
7.5 ppm Std	0.375	0.100	2.5	50
10 ppm Std	0.500	0.100	2.5	50

Wavelength »	Na 589.592	Na 330.237	Na 588.995
1.0 ppm Mean	101	99	100
2.5 ppm Mean	94	96	95
4.0 ppm Mean	96	102	98
5.0 ppm Mean	97	101	98
7.5 ppm Mean	98	97	99
10 ppm Mean	101	99	100

All wavelengths meet Linearity acceptance criteria of 80% - 120%.

## 7.10. Accuracy

- 7.10.1. The theoretical concentration of sodium in sodium decanoate is approximately 11.84% or 118,400 ppm. The specification of sodium was set 90%-110% recovery or between 106,500 and 130,200 ppm. Accuracy was performed on 80% to 120% range of the theoretical concentration to evaluate the effectiveness to quantify sodium in sample solutions.
- 7.10.2. Three (N=3) samples were prepared for analysis at five separate levels. The triplicate sample preparation of the 100% preparation was used for recovery calculations. The solutions were analyzed by ICP-OES as per the protocol and by a single analyst.

$$\% \text{ Recovery} = \frac{\text{Concentration of replicate} \times 100}{\text{Theoretical concentration Based on 100\% Preparation}}$$

### 7.10.2.1. Acceptance Criteria

- 7.10.2.1.1. The mean recovery at each of the four alternative preparation levels must be in the range of 95.0% to 105.0% from the 100% preparation level.

### 7.10.3. Spike Recovery Sample Preparation

- 7.10.3.1. Weighed the appropriate amount of sample into a clean 20 mL digestion vessel.
- 7.10.3.2. Added 4.0 mL of Acid Digestion Mix then properly torque cap and placed vessel in microwave carousel. Digested samples according to Section 6.8.
- 7.10.3.3. After digestion, vessels were placed in an ice bath for approximately 40 minutes. Before transferring, the vessels were turned sideways and slowly rotated to collect condensation on the inside of the vessel walls.
- 7.10.3.4. Quantitatively transferred the contents into a 50 mL Digitube® containing 5 mL of deionized water.
- 7.10.3.5. Rinsed the vessel an additional two times using deionized water and transferred each rinse to the 50 mL Digitube®. Diluted to final volume of 50 mL and mixed well.
- 7.10.3.6. Performed an additional dilution by pipetting 1.0 mL of the digested sample into another 50 mL Digitube® along with 100 µL of internal standard then diluted to 45 mL using deionized water.
- 7.10.3.7. Added 2.5 mL of nitric acid then diluted to final volume of 50 mL using deionized water.

<b>Description</b>	<b>Sample Amount (mg)</b>	<b>Internal Standard (mL)</b>	<b>Nitric Acid (mL)</b>	<b>Final Volume (mL)</b>
<b>Method Blank</b>	N/A	0.100	2.5	50
<b>80% Preparation</b>	80	0.100	2.5	50
<b>90% Preparation</b>	90	0.100	2.5	50
<b>100% Preparation</b>	100	0.100	2.5	50
<b>110% Preparation</b>	110	0.100	2.5	50
<b>120% Preparation</b>	120	0.100	2.5	50

<b>TABLE 12: ACCURACY RESULTS</b> (Mean percent recovery of triplicate preparations)			
<b>Wavelength »</b>	<b>Na 589.592</b>	<b>Na 330.237</b>	<b>Na 588.995</b>
80% Prep	97.7	97.4	98.0
90% Prep	98.7	101.1	98.9
110% Prep	100.8	99.4	100.5
120% Prep	100.6	101.4	100.3

All wavelengths meet accuracy acceptance criteria of 95.0% - 105.0%.

#### 7.11. Specificity

- 7.11.1. Specificity was demonstrated by using a method blank and a calibration blank for ICP-OES analysis. The method blank was prepared during the accuracy portion and prepared according to the protocol as this is not a duplicate preparation to that of the calibration blank.
- 7.11.2. The solutions were analyzed as per the analytical method and the intensities of the method blank, calibration check blank, 1.0 ppm standard, and 10 ppm standard are reported to show no matrix interference is observed at all three wavelengths. Intensities are corrected based on the calibration blank intensities in the software. Specificity chromatograms are attached in supporting documents to show intensities of the calibration standards and blanks to show no interferences present.
- 7.11.2.1. Acceptance Criteria:
- 7.11.2.1.1. The lack of a significant interference or by any other element in the spiked blank solution or the solution matrix itself will indicate the specificity of the method.

<b>TABLE 13: SPECIFICITY RESULTS</b>			
<b>Wavelength »</b>	<b>Na 589.592</b>	<b>Na 330.237</b>	<b>Na 588.995</b>
Blank (CPS)	8474	187	12861
Method Blank (CPS)	4110	34	13967
1.0 ppm Std (CPS)	426034	611	689169
10 ppm Std (CPS)	5091391	4504	7820196

## 7.12. Precision

## 7.12.1. Repeatability

7.12.2. All solutions for the Repeatability test were prepared by a single analyst.

7.12.3. Six sample solutions were prepared at the 100% Preparation Concentration as shown in Section 6.7 above. For ICP-OES analysis, the three 100% preparation sample solutions from the accuracy section were also included in the analysis for the precision portion.

7.12.4. The concentrations are reported in ppm in calibration units based on the calibration curve, which is a factor of 25,000 less than the actual amount of sodium present in the 100% target preparation.

## 7.12.4.1. Acceptance Criteria:

7.12.4.1.1. The %RSD for the 100% preparation must be NMT 5.0% for each wavelength.

<b>TABLE 14: PRECISION RESULTS</b> (Mean concentration of N=6 100% preparations)			
<b>Wavelength »</b>	<b>Na 589.592</b>	<b>Na 330.237</b>	<b>Na 588.995</b>
100% Mean Concentration	4.436	4.527	4.532
%RSD	0.2	2.6	1.1

All wavelengths meet Precision RSD% acceptance criteria of NMT 5.0%.

## 7.13. Intermediate Precision (Ruggedness)

7.13.1. A second analyst, on a different day from the performance of the Repeatability experiment, prepared and analyzed the Intermediate Precision solutions. The second analyst prepared and analyzed an additional set of Intermediate Precision solutions on a second different day. These additional sample solutions were prepared at the 100% preparation level as shown in Section 6.7 for ICP- OES analysis for each additional dataset (this fulfilled a total of three experiments as described in USP <730> as “different day” and “different analyst”).

7.13.2. The concentrations are reported in ppm in calibration units based on the calibration curve, which is a factor of 25,000 less than the actual amount of sodium present in the 100% target preparation.

## 7.13.2.1. Acceptance Criteria:

7.13.2.1.1. The %RSD for the 100% preparation the three datasets (N=18) must be NMT 8.0% for each wavelength.

7.13.2.1.2. The check standard failed system suitability for Na 330.237 wavelength and the results were deemed invalid for this wavelength.

<b>TABLE 15: RUGGEDNESS RESULTS</b> (Mean concentration of N=18 100% preparations)			
<b>Wavelength »</b>	<b>Na 589.592</b>	<b>Na 330.237</b>	<b>Na 588.995</b>
100% Mean Concentration	4.590	4.692	4.679
%RSD	5.2	5.9	5.2

All wavelengths meet the Ruggedness %RSD acceptance criteria of NMT 8.0%.

#### 7.14. Sample and Standard Stability

7.14.1. The 1.0 ppm, 5.0 ppm, and 10 ppm calibration standards were analyzed as samples against calibration curves constructed from freshly prepared calibration standards at T=1 day, T=3 days, and T=8 days from the date of preparation or T=0.

7.14.2. One sample solution prepared at the target 100% preparation from the Accuracy experiment was used for sample stability. The sample solution was analyzed against calibration curves constructed from freshly prepared calibration standards at time points T=1-day, T=3 days, and T=8 days (days from the date of preparation).

##### 7.14.2.1. Acceptance Criteria:

7.14.2.1.1. The recovery of each wavelength must be within the range of 90% to 110% recovery of the T = 0 results for the calibration standards.

7.14.2.1.2. The recovery of each wavelength must be within the range of 90% to 110% recovery of the T = 0 results for the sample solution.

7.14.2.1.3. The check standard failed system suitability for Na 330.237 wavelength at T=1 day stability and the results were deemed invalid for this wavelength.

Wavelength »	Na 589.592			Na 330.237			Na 588.995		
	T=1	T=3	T=8	T=1	T=3	T=8	T=1	T=3	T=8
1.0 ppm Std <sup>1</sup>	100	103	103	68	95	66	100	102	104
5.0 ppm Std	96	102	102	90	101	88	96	102	102
10 ppm Std	96	101	102	99	106	100	96	101	102
100% Prep Sample	97	102	100	96	100	92	97	101	100

<sup>1</sup>Na 330.237 failed low for the 1.0 ppm calibration stability at T=1 and T=8. All other wavelengths met the acceptance criteria of 90% to 110% recovery for stability solutions for standards and samples.

## 8. DEVIATIONS:

8.1. In the protocol, solution stability was to be performed at T=1 day, T=3 days, and T=5 days. Solution stability was performed at T=8 days instead of T=5 days. This is justified due to the company being closed for holidays and the length of stability was increased by an additional three days.

8.2. Wavelength 330.237 failed system suitability for check standard outside of 90-110% during precision and intermediate precision studies. The results for this wavelength were considered invalidated and the wavelength will not be included in the final test method.

8.3. The method validation protocol (BSI-PRL-0467) was mistakenly designed as a Category II Impurity test, as opposed to a Category I Assay. Since the sodium present in Sodium Decanoate is not an impurity, this report has been updated to reflect the following changes in acceptance criteria to harmonize with USP <730>:

8.3.1. Accuracy & Robustness acceptance criterion changed from 80-120% to 95.0-105.0%. This acceptance criterion was still met.

8.3.2. Precision acceptance criterion changed from NMT 10% to NMT 5.0%. This acceptance criterion was still met.

8.3.3. Intermediate Precision criterion changed from NMT 15% to NMT 8.0%. This acceptance criterion was still met.

8.3.3.1. Due to wording in USP <730> referring to a minimum of “three experiments” with regard to Intermediate Precision, an additional six precision samples were prepared and analyzed by the second analyst on 09/22/22. The Intermediate Precision acceptance criterion is still met with N=18 measurements.

8.3.4. General correlation coefficient requirements for calibration changed from NLT 0.99 to NLT 0.995. This requirement was met for all portions of this method validation.

**9. CONCLUSION:**

- 9.1. The test method for sodium quantification in sodium decanoate has been validated. The Method was found to be:
  - 9.1.1. Specific: The calibration blank and method blank did not show any significant interferences for all analyzed wavelengths.
  - 9.1.2. Linear: 1.0 ppm through 10 ppm of calibration standards. Mean percent recovery ranged from 94% to 101%. Na 330.237 wavelength did not meet system suitability and will be invalidated.
  - 9.1.3. Accurate: From 80% to 120% preparation of target preparation with mean percent recoveries ranging from 97.7% to 101.4%. All masses analyzed met acceptance criteria within the specified range, but Na 330.237 wavelength did not meet system suitability for QC check recovery and will be invalidated. Na 330.237 wavelength results not included.
  - 9.1.4. Precise: Closeness of agreement demonstrated between six sample preparations by percent RSDs of 0.2% and 1.1%. Na 330.237 wavelength results not included.
  - 9.1.5. Rugged: Satisfactory precision was demonstrated between two sets of six sample preparations performed on different days and by different analysts. The percent RSDs were 5.2% for both the Na 589.592 and Na 588.995 wavelengths.
  - 9.1.6. Stable: With respect to stability of solutions, the sample solution for target preparation was shown to be stable for 8 days for all validated wavelengths analyzed using this protocol. The calibration standard preparations are shown to be stable for 8 days.
  - 9.1.7. Na 589.592 wavelength will be noted to be the preferred wavelength and reportable result in the final test method. Both 589.592 and 588.995 are shown to be effective through the validation of the protocol, so 588.995 will be noted to be used for confirmation purposes.

**10. NOTEBOOK REFERENCE:**

TABLE 17: NOTEBOOK REFERENCE		
STUDY		NOTEBOOK REFERENCE
Specificity		ICPOES1 pages 18-20
Linearity and Range		ICPOES1 pages 16-17
Accuracy and Precision		ICPOES1 pages 18-20
Intermediate Precision (Ruggedness)		ICPOES1 pages 21-24 ICPOES4 pages 42-44
Solution Stability	Day-0	ICPOES1 pages 18-20
	Day-1	ICPOES1 pages 21-24
	Day-3	ICPOES1 pages 25-26
	Day-8	ICPOES1 pages 27-28