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ANALYTICAL METHOD VALIDATION REPORT:
IRON AND LEAD DETECTION BY INDUCTIVELY
COUPLED PLASMA OPTICAL EMISSION
SPECTROSCOPY (ICP-OES) IN POTASSIUM
BROMIDE

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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-0568 v. 1.0, for Iron and Lead Detection in Potassium Bromide (KBr) products, performs according to USP and BioSpectra requirements.
 - 1.1.1. Elements under USP <232> will be considered and are as follows:
 - 1.1.1.1. Class 1: Pb
 - 1.1.1.2. Class 4: Fe

2. SCOPE:

- 2.1. Applies to Potassium Bromide products manufactured at BioSpectra with a product code of KBRO-2201 and/or KBRO-2301.
- 2.2. Applies to the Avio 500 S/N 081S1905062 ICP-OES located in the Laboratory Department at the BioSpectra Bangor, PA facility.
- 2.3. This report applies to the protocol validation for Iron and Lead Detection in Potassium Bromide, by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) performed at BioSpectra Inc.

3. REFERENCES:

- 3.1. BSI-PRL-0568, Analytical Method Validation Protocol: Iron and Lead Detection by ICP-OES for Potassium Bromide
- 3.2. BSI-SOP-0362, Avio 500 ICP-OES SOP
- 3.3. BSI-SOP-0436, Analytical Method Validation Master Plan
- 3.4. ICH Guideline for Elemental Impurities Q3D
- 3.5. USP <730> Plasma Spectrochemistry
- 3.6. USP <1730> Plasma Spectrochemistry—Theory and Practice
- 3.7. USP <232>, <233>

4. BACKGROUND:

- 4.1. This validation was executed using a 100% Target concentration of 10 ppm in order to meet the product codes listed in the scope section above. The protocol was performed as per USP General Chapters <232> and <233>, Elemental Impurities –Procedures, Validation of Quantitative Procedures. (See Table 1)
- 4.2. The test protocol validation report includes the following parameters:
 - 4.2.1. Specificity
 - 4.2.2. Linearity and Range
 - 4.2.3. Limit of Quantification (LOQ)
 - 4.2.4. Accuracy by “Spiked Recovery”
 - 4.2.5. Precision (Repeatability)
 - 4.2.6. Intermediate Precision (Ruggedness)
 - 4.2.7. Standard and Sample Solution Stability

TABLE 1: LIMITS FOR FINISHED GOODS					
Elements	ICH Class	30% LOQ (µg/g) in sample	50% Target (µg/g) in sample	100% Target (µg/g) in sample	150% Target (µg/g) in sample
Pb	1	3.0	5.0	10	15
Fe	4	3.0	5.0	10	15

5. MATERIALS AND EQUIPMENT:

TABLE 2: EQUIPMENT				
Type	Supplier	Model	Serial Number	Cal. Due
Analytical Balance	Sartorius	MSE224S	36707108	10/2022
Automatic Pipette	Rainin	E4-XLS (20-200 µL)	C016314640	12/31/22
Automatic Pipette	Rainin	E4-XLS (100-1000 µL)	C016314969	12/31/22
Automatic Pipette	Rainin	E4-XLS (0.5-5 mL)	C023506909	12/31/22
ICP-OES	Perkin Elmer	Avio 500	081S1905062	09/2022, 09/2023 ¹
Deionized water system	Millipore	IQ-7005/ Element POD	F9SA14284H	06/2023

¹ICP-OES instrument was serviced for annual PM/OQ during validation.

TABLE 3: REAGENTS					
Type	Grade	Supplier	Catalog Number	Lot Number	Expiration
70% Nitric Acid	Trace Metal	VWR	87003-261	1122020	02/16/24
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

TABLE 4: REFERENCE STANDARDS					
Identification	Part Number	Manufacturer	Lot Number	Expiration	Element/Concentration
Lead Stock Standard	N9300175	Perkin Elmer	25-53PBY1	11/30/22	Pb (1,000 µg/mL)
Iron Stock Standard	N9303771	Perkin Elmer	26-37FEY1	10/30/23	Fe (1,000 µg/mL)
Scandium Stock Standard	N9303798	Perkin Elmer	25-96SCY1	01/30/23	Sc (1,000 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. Internal Standard Solution Preparation
- 6.2.1. Added 0.500 mL of Sc (1,000 µg/mL) and 0.500 mL of Y (1,000 µg/mL) to a 50 mL Digitube®.
- 6.2.2. Diluted to a final volume of 50 mL with deionized water and mixed well.
- 6.2.3. Scaled proportionally as needed for use.
- 6.3. Intermediate Standard Preparation
- 6.3.1. Prepared a standard solution containing the elements listed in Table 5 using the individual single source 1,000 µg/mL stock standards. Prepared by adding stock standards to a 15 mL Digitube®. Added DI water to approximately 8 mL then added nitric acid. Diluted to volume using DI Water.

TABLE 5: INTERMEDIATE STANDARD						
Identification	Element	Stock Identification	Amount Added (mL)	Nitric Acid (mL)	Final Volume (mL)	Final Concentration (µg/mL)
Intermediate Standard	Fe	1,000 µg/mL Fe Std	0.500	1.0	10	50
	Pb	1,000 µg/mL Pb Std	0.500			50

6.4. 50 ppb Calibration Standard Preparation

6.3.2. Prepared a solution containing the elements listed in Table 6 below in 5.0% HNO₃. Added intermediate standard to a separate 50 mL Digitube[®] followed by addition of approximately 35 mL of DI Water. Added nitric acid then diluted to 45 mL using DI Water. Added internal standard solution and diluted to volume using DI Water. Intermediate standards were not allowed to contact concentrated acids while preparing solutions.

TABLE 6: 50 PPB CALIBRATION STANDARD						
Identification	Element	Intermediate Standard (mL)	Nitric Acid (mL)	Internal Standard Solution (mL)	Final Volume (mL)	Final Concentration (µg/L)
50 ppb Calibration Standard	Fe	0.050	2.50	1.0	50	50
	Pb					50

6.5. 150 ppb Calibration Standard Preparation

6.3.3. Prepared a solution containing the elements listed in Table 7 below in 5.0% HNO₃. Added intermediate standard to a separate 50 mL Digitube[®] followed by addition of approximately 35 mL of DI Water. Added nitric acid then diluted to 45 mL using DI Water. Added internal standard solution and diluted to volume using DI Water. Intermediate standards were not allowed to contact concentrated acids while preparing solutions.

TABLE 7: 150 PPB CALIBRATION STANDARD						
Identification	Element	Intermediate Standard (mL)	Nitric Acid (mL)	Internal Standard Solution (mL)	Final Volume (mL)	Final Concentration (µg/L)
150 ppb Calibration Standard	Fe	0.150	2.50	1.0	50	150
	Pb					150

6.6. 200 ppb Calibration Standard Preparation

6.3.4. Prepared a solution containing the elements listed in Table 8 below in 5.0% HNO₃. Added intermediate standard to a separate 50 mL Digitube[®] followed by addition of approximately 35 mL of DI Water. Added nitric acid then diluted to 45 mL using DI Water. Added internal standard solution and diluted to volume using DI Water. Intermediate standards were not allowed to contact concentrated acids while preparing solutions.

TABLE 8: 200 PPB CALIBRATION STANDARD						
Identification	Element	Intermediate Standard (mL)	Nitric Acid (mL)	Internal Standard Solution (mL)	Final Volume (mL)	Final Concentration (µg/L)
200 ppb Calibration Standard	Fe	0.200	2.50	1.0	50	200
	Pb					200

6.7. Calibration Blank

6.3.5. Prepared a solution containing 5.0% HNO₃ as per Table 9 below. Internal Standard Solution was not allowed to contact concentrated acids. To a separate 50 mL Digitube[®], added approximately 35 mL of DI Water. Added nitric acid then diluted to 45 mL using DI Water. Added Internal Standard Solution and diluted to volume using DI Water.

TABLE 9: CALIBRATION STANDARD			
Description	Nitric Acid (mL)	Internal Standard Solution (mL)	Final Volume (mL)
Cal Blank	2.50	1.0	50

6.8. Method Blank Preparation

6.3.6. Added approximately 35 mL of deionized water to a 50 mL Digitube[®].

6.3.7. Added 2.50 mL of nitric acid and swirled to mix.

6.3.8. Added deionized water to approximately 45 mL and then transferred 1.0 mL of Internal Standard Solution.

6.3.9. Diluted to a final volume of 50 mL using deionized water and mixed well.

6.9. Potassium Bromide Sample Preparation

6.3.10. Weighed approximately 500 mg of the sample into a 50 mL Digitube[®].

6.3.11. Added 20 mL of deionized water and swirled to dissolve sample.

6.3.12. Added 2.50 mL of Nitric Acid and swirled again to mix.

6.3.13. Added deionized water to approximately 45 mL and then transferred 1.0 mL of Internal Standard Solution.

6.3.14. Diluted to a final volume of 50 mL with deionized water and mixed thoroughly.

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) for the calibration curve was to be ≥ 0.99 .
- 7.3. Set up the sequence as per Table 10.
- 7.4. Confirmed the calibration by analyzing the 150 ppb standard after the calibration. The calibration check was to recover $\pm 10\%$ of the calculated theoretical concentration.
- 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 bracketing standard checks were to recover $\pm 10\%$ of the calculated theoretical concentration. Additionally, the drift (calculated as absolute difference) between the bracketing standard checks was to be NMT 10% for each target wavelength.
- 7.7. The sample concentration is calculated as:

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

ID	Type	Level
Cal Blank	Cal Blank	Level 1
50 ppb Cal Std	Cal Std	Level 2
150 ppb Cal Std	Cal Std	Level 3
200 ppb Cal Std	Cal Std	Level 4
Cal Blank Check	QC Check	N/A
150 ppb Check Std 1	QC Check	N/A
Method Blank	Sample	N/A
Sample(s) 10 or less	Sample	N/A
150 ppb 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. Table 11 depicts the settings used for the entirety of the method validation.
- 7.8.2. The gas flows for Plasma, Auxiliary, and Nebulizer were set at 12 mL/min, 0.20 mL/min, and 0.70 mL/min, respectively.

ICP-OES System	Perkin Elmer Avio 500 Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
Points per Peak	4
Replicates	3
Viewing Distance	15.0
Nebulizer Gas	Argon
Shear Gas	Compressed Air + Nitrogen
Sample Rinses	Rinse-1: 30 sec at 1.0 mL/min 5.0% HNO ₃ (or as applicable to mitigate carry over)

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Element	Internal Standard	Mode	Wavelength	Linear Range (µg/L)
Pb	Sc 357.253	Axial	220.353	30-200
Fe	Sc 357.253	Axial	238.204	30-200
			239.562	
			259.939	

7.9. Linearity and Range

7.9.1. The ICP-OES linearity study included the following standards across the linear range in Table 12 and were prepared according to Table 13: (30 ppb, 50 ppb, 100 ppb, 150 ppb, and 200 ppb). Each standard was prepared in triplicate and analyzed against the calibration curve described in Section 6.4 to Section 6.6. The average standard recovery for each level of the three replicates was then determined. Each replicate was plotted against standard concentration to obtain linearity plot where statistical regression analysis was performed. Results for mean standard recovery are shown in Table 14.

7.9.1.1. Acceptance Criteria:

7.9.1.1.1. The mean standard recovery for each element at each of the spike levels must be in the range of 70% - 150%.

7.9.2. Figures 1 through 4 show the plots of signal versus concentration for the four wavelengths along with the linear regression trendlines and correlation coefficients. All four plots show a correlation NLT 0.999. Statistical analysis was performed on the theoretical concentration versus actual concentration of each replicate at all five levels. Table 15 shows values from the regression analysis including residual sum of squares, standard error, and R^2 values.

Description	Intermediate Standard (mL)	Nitric Acid (mL)	Internal Standard Solution (mL)	Final Volume Deionized Water (mL)
Cal Blank Reference	N/A	2.50	1.0	50
30 ppb Standard	0.030	2.50	1.0	50
50 ppb Standard	0.050	2.50	1.0	50
100 ppb Standard	0.100	2.50	1.0	50
150 ppb Standard	0.150	2.50	1.0	50
200 ppb Standard	0.200	2.50	1.0	50

Element	Wavelength (nm)	30 ppb Mean	50 ppb Mean	100 ppb Mean	150 ppb Mean	200 ppb Mean
Pb	220.353	103	102	101	101	102
	238.204	101	101	102	101	101
Fe	239.562	101	100	102	101	101
	259.939	102	101	103	102	102

All wavelengths meet Linearity acceptance criteria of 70% - 150%.

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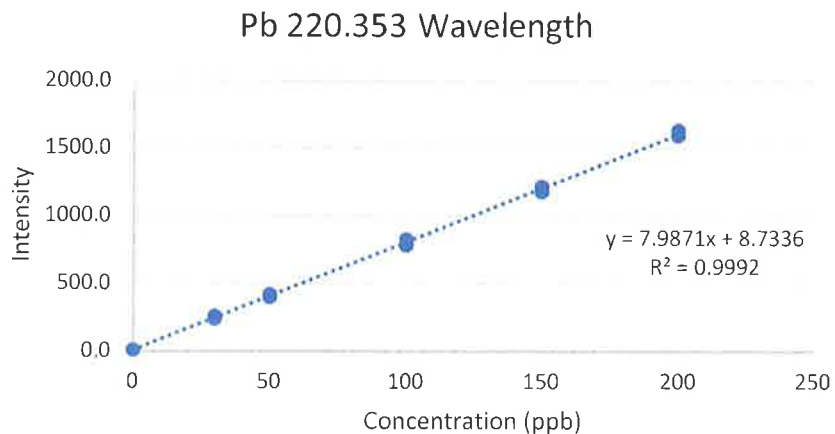


Figure 1: Linearity plot, Pb 220.353 signal versus concentration.

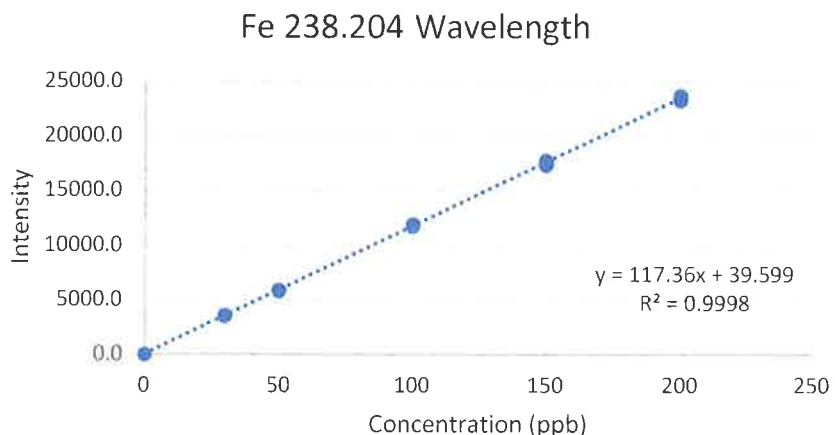


Figure 2: Linearity plot, Fe 238.204 signal versus concentration.

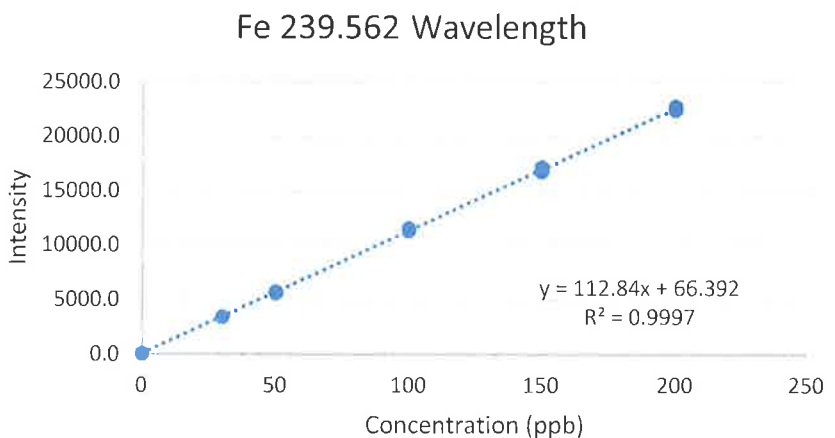


Figure 3: Linearity plot, Fe 239.562 signal versus concentration.

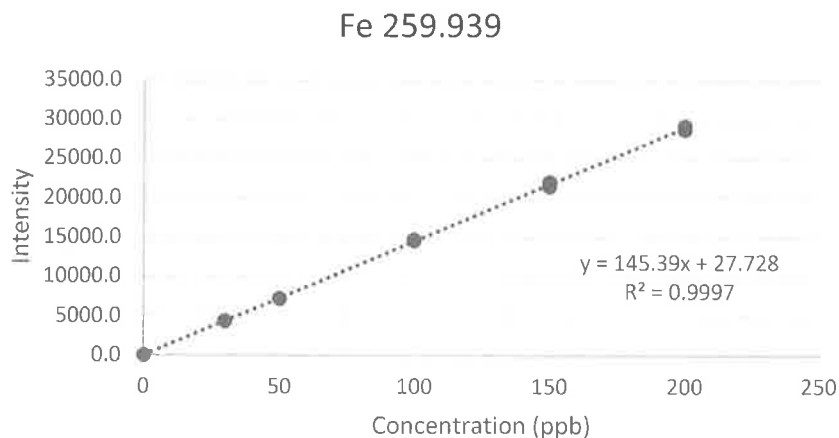


Figure 4: Linearity plot, Fe 259.939 signal versus concentration.

TABLE 15: STATISTICAL REGRESSION RESULTS				
Element	Wavelength (nm)	R² Value	Standard Error	Residual Sum of Squares
Pb	220.353	0.9991	2.1187	58.3564
Fe	238.204	0.9997	1.1255	16.4678
	239.562	0.9997	1.1832	18.1995
	259.939	0.9997	1.2200	19.3499

7.10. Accuracy

7.10.1. Three (N=3) unspiked samples were prepared for analysis. The unspiked sample preparations were used for spike recovery calculations. Samples were prepared in triplicate at three spiking levels (50%, 100%, and 150% of the Target Concentration) as shown in Table 1. The solutions were analyzed by ICP-OES, as per the method, by a single analyst. Table 17 shows accuracy results for potassium bromide.

$$\% \text{ Recovery} = \frac{(\text{Conc. of spiked replicate} - \text{Average Conc. of 3 unspiked samples}) \times 100}{\text{Expected spiked concentration}}$$

7.10.1.1. Acceptance Criteria

7.10.1.1.1. The mean spike recovery for each element at each of the three spike levels must be in the range of 70% - 150%.

7.10.2. Spiked Reference (Unspiked) Solution Preparation

7.10.2.1. Prepare as per section 6.9.

7.10.3. Spike Recovery Sample Preparation – Potassium Bromide

7.10.3.1. Weighed approximately 500 mg of sample into a 50 mL Digitube®.

7.10.3.2. Pipetted appropriate intermediate standard spike amount as per Table 16.

7.10.3.3. Added approximately 20 mL of deionized water and swirled to dissolve sample.

7.10.3.4. Pipetted 2.50 mL of nitric acid and swirled to mix solution.

7.10.3.5. Added deionized water to 45 mL and transferred 1.0 mL of Internal Standard Solution.

7.10.3.6. Diluted to a final volume of 50 mL with deionized water and mixed well.

7.10.3.7. Prepared spiked sample solutions in triplicate and three preparations of unspiked sample solutions.

Description	Sample Amount (mg)	Intermediate Standard Spike (mL)	Nitric Acid (mL)	Internal Standard Solution (mL)	Final Volume (mL)
Method Blank	N/A	N/A	2.50	1.0	50
Unspiked	500	N/A	2.50	1.0	50
30% Spiked Sample	500	0.030	2.50	1.0	50
50% Spiked Sample	500	0.050	2.50	1.0	50
100% Spiked Sample	500	0.100	2.50	1.0	50
150% Spiked Sample	500	0.150	2.50	1.0	50

Element	Wavelength (nm)	50% Mean	100% Mean	150% Mean
Pb	220.353	93	95	96
	238.204	92	94	94
Fe	239.562	94	95	94
	259.939	93	94	93

All wavelengths meet accuracy acceptance criteria of 70% - 150%.

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7.11. Specificity

- 7.11.1. Specificity was demonstrated by using a calibration blank, spiked calibration blanks, and method blank for ICP-OES analysis. The calibration blank was prepared as per the analytical method protocol. A separate blank was spiked with a mixed standard solution which produced a spiked solution at a concentration equivalent to the 50 ppb and 200 ppb standards used for calibration.
- 7.11.2. The solutions were analyzed as per the analytical method and the intensities for the calibration blank, method blank, and the 50 ppb and 200 ppb calibration standards are reported in Table 18 below.
- 7.11.3. The spectrum overlay for the calibration blank, method blank, and three calibration standards are shown below in Figure 5 and Figure 6 to depict no spectral interferences are observed in the four wavelengths.
- 7.11.4. The instrument method uses corrected mean intensity for reporting, so the method blank and calibration standards are the corrected intensities compared to the calibration blank.
 - 7.11.4.1. Acceptance Criteria:
 - 7.11.4.1.1. The lack of a significant interference (as demonstrated by the spike recovery of 70% to 150%) or by any other element in the spiked blank solution or the solution matrix itself will indicate the specificity of the method.

TABLE 18: SPECIFICITY RESULTS					
Element	Wavelength (nm)	Blank (CPS)	50 ppb STD (CPS)	200 ppb STD (CPS)	Method Blank (CPS)
Pb	220.353	-90	402	1577	9
	238.204	-106	5893	23122	41
Fe	239.562	-410	5759	22273	67
	259.939	-60	7307	28558	9

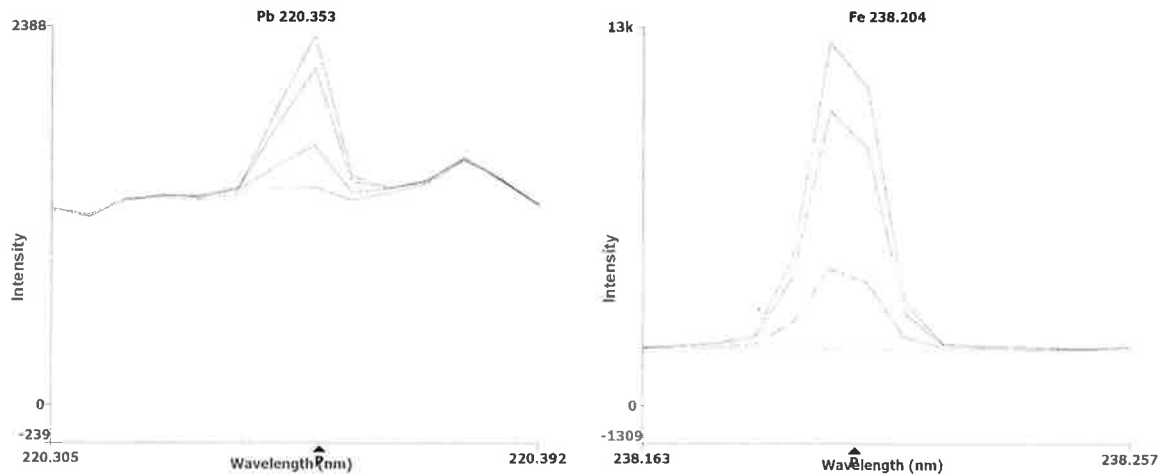


Figure 5: Specificity overlay for Pb 220.353 and Fe 238.204

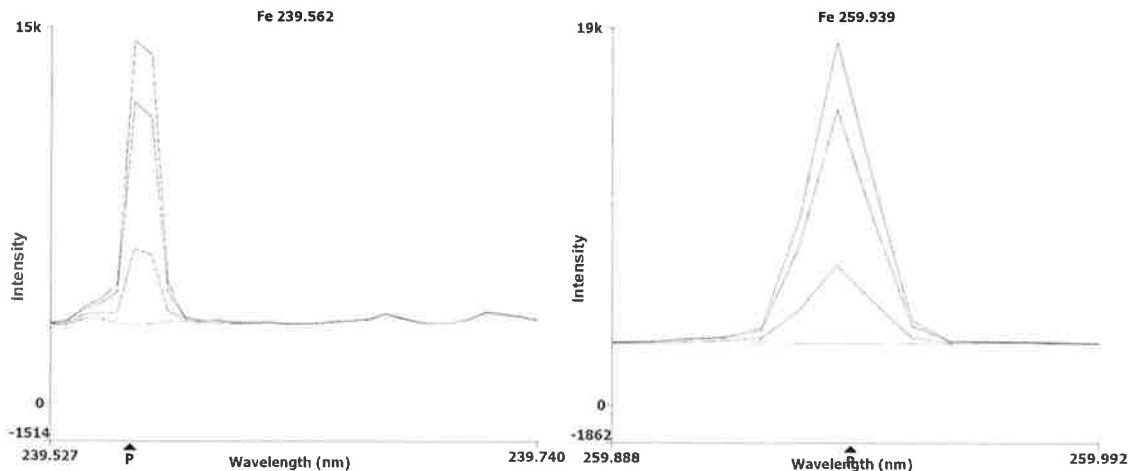


Figure 6: Specificity overlay for Fe 239.562 and Fe 259.939

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% Target Concentration as shown in Table 1. For ICP-OES analysis, the 100% Target Concentration spiked samples used in the accuracy study were also used for the precision study. Table 19 shows results for precision study.

7.12.3.1. Acceptance Criteria:

7.12.3.1.1. The %RSD for the spike recovery concentration must be NMT 20% for each wavelength.

TABLE 19: PRECISION RESULTS FOR POTASSIUM BROMIDE (Mean recovery concentration of 6 preparations)			
Element	Wavelength (nm)	100% Mean Recovery Conc. N=6 (µg/g)	% RSD N=6
Pb	220.353	9.557	2
Fe	238.204	9.326	1
	239.562	9.397	1
	259.939	9.276	1

All wavelengths meet Precision %RSD acceptance criteria of NMT 20%.

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. Six sample solutions were prepared at the 100% Target Concentration level found in Table 1 for ICP-OES analysis (this fulfilled two events as “different day” and “different analyst”). Results for ruggedness are reported in Table 20 below.

7.13.1.1. Acceptance Criteria:

7.13.1.1.1. The %RSD for the spike recovery concentration from both analysts (N=12) must be NMT 25% for each wavelength.

TABLE 20: RUGGEDNESS RESULTS FOR POTASSIUM BROMIDE (Mean recovery concentration of 6 preparations)			
Element	Wavelength (nm)	100% Mean Recovery Conc. N=6 (µg/g)	% RSD N=6
Pb	220.353	9.336	3
Fe	238.204	9.625	3
	239.562	9.701	3
	259.939	9.573	3

All wavelengths meet Ruggedness %RSD acceptance criteria of NMT 25%.

7.14. Limit of Quantitation (LOQ)

7.14.1. The limit of quantitation (LOQ) is demonstrated from spike recovery performed at the 30% Target Concentration spiking levels as shown in Table 1.

7.14.2. Results for the LOQ study are reported in Table 21 below.

7.14.3. Samples were prepared in triplicate following Section 7.10.3 and using amounts listed for 30% spiked samples in Table 16 above.

7.14.3.1. Acceptance Criteria:

7.14.3.1.1. The mean percent spike recovery for each element at the 30% Target Concentration spiking levels must be in the range of 70% - 150%.

TABLE 21: LIMIT OF QUANTITATION RESULTS (Mean percent recovery of 3 preparations)		
Element	Wavelength (nm)	LOQ % Recovery
Pb	220.353	96
Fe	238.204	91
	239.562	93
	259.939	92

All wavelengths meet the LOQ %RSD acceptance criteria of 70% - 150%.

7.15. Sample and Standard Stability

7.15.1. The 50 ppb and 200 ppb calibration standards were analyzed as samples against calibration curves constructed from freshly prepared calibration standards at T=1 (1 day from the date of preparation) and T=7 (7 days from the date of preparation).

7.15.2. Spiked sample solution prepared at the 100% Target Concentration level in Table 1 from the Precision experiments was used for sample stability. The spiked sample solution was analyzed against calibration curves constructed from freshly prepared calibration standards at time points T=1 (1 day from the date of preparation) and T=7 (7 days from the date of preparation).

7.15.3. Table 22 reports the percent recovery for the 50 ppb and 200 ppb calibration standards along with replicate 1 of potassium bromide 100% target sample solution, from the precision study, at the two timepoints below.

7.15.3.1. Acceptance Criteria:

7.15.3.1.1. The recovery of each element must be within the range of 80% to 120% recovery of the T = 0 results for the calibration standard.

7.15.3.1.2. The recovery of each element must be within the range of 80% to 120% recovery of the T = 0 results for the spiked sample solution.

TABLE 22: SAMPLE AND STANDARD STABILITY (% RECOVERY)							
Element	Wavelength (nm)	50 ppb Std T=1 (%)	50 ppb Std T=7 (%)	200 ppb Std T=1 (%)	200 ppb Std T=7 (%)	KBr Sample T=1 (%)	KBr Sample T=7 (%)
Pb	220.353	104	102	101	94	98	92
Fe	238.204	100	101	101	98	109	97
	239.562	100	101	101	99	108	94
	259.939	100	100	101	99	109	96

All wavelengths meet the acceptance criteria of 80% to 120% recovery for stability solutions for standards and sample.

8. DEVIATIONS:

8.1. Pb 224.688 wavelength was included in the instrument method and analyzed for each study. This wavelength failed system suitability of NLT 0.99 correlation coefficient for each run. All data was not included in the report since system suitability was not met and therefore this wavelength will be omitted from final test method. This is deemed acceptable as Pb 220.353 wavelength met all acceptance criteria for the entire validation.

9. CONCLUSION:

- 9.1. The test method for Iron and Lead Detection in Potassium Bromide has been validated. The Method was found to be:
- 9.1.1. Specific: The calibration and method blanks did not show any significant interference for all analyzed wavelengths.
 - 9.1.2. Linear: 30 ppb to 200 ppb for the standard solution mean percent recovery ranged from 100% to 103%. All wavelengths showed NLT 0.999 for correlation coefficient when plotting concentration versus intensity. All wavelengths met acceptance criteria.
 - 9.1.3. Sensitive: LOQ recoveries were within 91% to 96% for potassium bromide spiked samples. All wavelengths met acceptance criteria.
 - 9.1.4. Accurate: From 50% to 150% of target concentration level with mean percent recoveries ranging from 92% to 96%. All wavelengths analyzed met acceptance criteria within the specified range.
 - 9.1.5. Precise: Closeness of agreement demonstrated between six sample preparations by percent RSDs were either 1% or 2%. All wavelengths met acceptance criteria.
 - 9.1.6. Rugged: Satisfactory precision was demonstrated between two sets of six sample preparations performed on different days and by different analysts. All wavelengths were 3% for percent RSDs and met acceptance criteria.
 - 9.1.7. Stable: With respect to stability of solutions, the sample solutions for potassium bromide were shown to be stable for 7 days. The working standard preparations are shown to be stable for 7 days as well.
 - 9.1.8. All wavelengths met all validation parameters with the exception of Pb 224.688, which all results are disregarded due to system suitability failure. In order to simplify the method printouts and reduce the amount of total wavelength analyzed, the final test method will include the following wavelengths: Pb 220.353 and Fe 259.939. The analytical testing method may be revised to include other wavelengths if necessary in the future.

10. NOTEBOOK REFERENCE:

TABLE 23: NOTEBOOK REFERENCE		
STUDY		NOTEBOOK REFERENCE
Specificity		ICPOES4/ pages 33-36
Linearity		ICPOES4/ pages 33-36
Accuracy/ Precision/LOQ by "Spiked" recovery		ICPOES4/ pages 33-36
Intermediate Precision (Ruggedness)		ICPOES4/ pages 37-39
Solution Stability	Day-0	ICPOES4/ pages 33-36
	Day-1	ICPOES4/ pages 37-39
	Day-7	ICPOES4/ pages 40-41

Signature Manifest

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Change Request

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