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CHEMICAL BIOLOGICAL CENTER

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Inductively Coupled Plasma Mass Spectrometry: Sample Analysis of Arsenic and Selenium

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14. ABSTRACT:
Members of the Forensic Analytical Center at the U.S. Army Combat Capabilities Development Command Chemical Biological Center (Aberdeen Proving Ground, MD) were requested to analyze samples that were suspected of containing arsenic and/or selenium using inductively-coupled plasma mass spectrometry (ICP–MS). The capability to process samples for specific elemental components allows for scientists to further analyze the samples for specific chemical warfare materials.

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PREFACE

The work described in this report was started in April 2018 and completed in August 2018.

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CONTENTS

FIGURES

TABLES

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY: SAMPLE ANALYSIS OF ARSENIC AND SELENIUM

1. INTRODUCTION

Members of the Forensic Analytical Center (FAC) at the U.S. Army Combat Capabilities Development Command (CCDC) Chemical Biological Center (Aberdeen Proving Ground, MD) developed an analytical method for investigating samples using inductively coupled plasma mass spectrometry (ICP–MS). Specifically, the aqueous samples were analyzed for arsenic and selenium. ICP–MS has the capability to detect elements from subpart-per-trillion to percent-level concentrations. This capability to process samples for specific elemental components allows scientists to do further sample testing, if needed, to screen for additional possible suspect chemical warfare agents.

2. BACKGROUND

A central mission for CCDC Chemical Biological Center FAC members is to develop and validate quantitative and qualitative analytical methods, with a focus on chemical agents. Forensic capabilities were originally developed at CCDC Chemical Biological Center to support U.S. obligations under the Chemical Weapons Convention, whereby the United States was required to procure sampling and analysis expertise to verify treaty compliance. As required for treaty compliance, the FAC is accredited to the ISO/IEC 17025 standard that refers to laboratory operations and quality control.[1] To demonstrate its high standards in chemical and biological agent detection and analysis, CCDC Chemical Biological Center FAC members maintain elite status in international proficiency testing sponsored by the Organisation for the Prohibition of Chemical Weapons (The Hague, Netherlands).

Toxic chemicals are constantly being developed by malicious elements to cause harm. The use of chemical agents has become highly publicized. Specific arsenical chemical weapons include Lewisite, Clark I, Clark II, and Adamsite. Scientists have discussed different pathways to produce Se variants of these arsenical compounds.[2]

The Agilent 7900 ICP–MS system (Agilent Technologies; Santa Clara, CA) has an argon plasma and Ultra High Matrix Introduction accessory that operates at more than 100 times the traditional matrix limit for ICP–MS technology.[3] The samples are routinely prepared using 3% nitric acid. Because dichloromethane (DCM) or cyclohexanol is used for sample extraction when the sample is a solid material, a small amount of the solvent ends up in the 3% nitric acid solution. This requires the same solvent concentration to be used in the calibration standards that are used for the corresponding sample.

3. MATERIALS AND METHODS

3.1 Materials

Laboratory-grade chemicals from any supplier are appropriate for this procedure. The following chemicals were used:

- liquid argon (dewar);
- helium (tank);
- hydrogen (tank);
- 18 MΩ or double-distilled water that was free of metals;
- concentrated HNO_3 of a minimum trace-metal grade or grade that was suitable for the intended detection level; and
- tuning solution of 1 ppm Ce, Co, Li, Tl, and Y.

The following common laboratory equipment and supplies were used:

- analytical balance with readability of 0.001 mg,
- disposable plastic droppers,
- polypropylene (PP) bottles,
- PP graduated cylinders or equivalent,
- PP Falcon tubes of 15 and 50 mL or equivalent,
- pipetters and disposable tips ranging from 10 µL to 10 mL,
- a waste container, and
- laboratory cleaning wipes.

The following specialized equipment and supplies were used:

- 7900 series ICP–MS system (part no. G8403A; Agilent Technologies);
- ICP–MS MassHunter workstation (part no. G7215C with #003; Agilent Technologies);
- Cetac ASX-500 autosampler (part no. G3286A; Cetac Technologies; Omaha, NE);
- 1000 µg/mL As in 3% HNO_3 (CAS no. 7440-38-2; part no. CGAS1; Inorganic Ventures; Christiansburg, VA); and
- 1000 µg/mL Se in 3% HNO_3 (CAS no. 7782-49-2; part no. CGSE(4)1; Inorganic Ventures).

3.2 Methods

3.2.1 Preparation of the Matrix Solution

All stock solutions and the dilutions from those stock solutions must be remade every 3 months unless a stability study is performed that shows a longer storage time is acceptable. All solutions must be stored at ambient temperatures.

Solutions were prepared as follows:

- a PP, 1 L volumetric flask or high-density polyethylene (HDPE) bottle was approximately half-filled with water;
- 30 mL of concentrated nitric acid was added;
- the mixture was swirled; and
- the final volume or weight was adjusted to 1 L (volumetric flask) or 1000 g (HDPE bottle) by adding water, respectively, for a 3% solution.

3.2.2 Preparation of Stock Standard Solutions

The standard solutions (Tables 1 and 2) were made in 15 mL PP tubes. Dilutions of the standard solutions were prepared by pipetting the solvent of diluted nitric acid into a tube and then pipetting the standard solution. For the samples that required dichloromethane or cyclohexanol, 100 µL of the respective solvent was added as the last step to each sample tube. Note that the solvent must match the acid composition and concentration that was used for the sample preparation. Ideally, the standards and samples will have the same percentages of acids in them. This allows for the same amount of sample to be introduced from the nebulizer to the detector and thereby minimizes matrix effects.

The indicated amount of stock solution was added to the tube with a volumetric pipette. The remaining volume of solvent (3% HNO_3) was pipetted to generate a total volume of 10 mL. A 100 µL volume of the respective solvent was added to 5 mL of each sample to the samples that required dichloromethane or cyclohexanol as the last step before analysis was performed. The samples were vortexed vigorously after the dichloromethane or cyclohexanol was added before they were placed on the autosampler.

Table 1. Dilution Instructions for As Calibration Standards

Solution Name	Final Concentration (µg/mL)	Amount Added to 15 mL PP Tube		
		Solute		3% HNO_3 (mL)
		Name	Amount (mL)	
Level 1	10	As	0.100	9.9
Level 2	0.5	Level 1	0.5	9.5
Level 3	0.2	Level 1	0.2	9.8
Level 4	0.1	Level 1	0.1	9.9
Level 5	0.05	Level 2	1.0	9.0
Level 6	0.02	Level 3	1.0	9.0
Level 7	0.01	Level 4	1.0	9.0
Level 8	0.005	Level 5	1.0	9.0
MDL	0.001	Level 7	1.0	9.0

MDL is method detection limit.

Table 2. Dilution Instructions for Se Calibration Standards

Solution Name	Final Concentration (µg/mL)	Amount Added to 15 mL PP Tube		
		Solute		3% HNO_3 (mL)
		Name	Amount (mL)	
Level 1	10	As	0.100	9.90
Level 2	0.25	Level 1	0.25	9.75
Level 3	0.1	Level 1	0.1	9.9
Level 4	0.05	Level 1	0.05	9.95
Level 5	0.025	Level 2	1.0	9.0
Level 6	0.01	Level 3	1.0	9.0
Level 7	0.005	Level 4	1.0	9.0
MDL	0.001	Level 6	1.0	9.0

MDL is method detection limit.

3.2.3 Quality Assurance Samples

For the method blank, a sample of H_2O (from the same lot) was processed through the entire sample preparation procedure with each batch of samples. The method blank was then analyzed with the samples.

For the reagent blank, a sample of 3% HNO_3 was run after each continuing calibration verification sample and each sample to ensure that no carryover occurred.

3.2.4 Sample Analysis

To prepare the instrument for analysis, necessary performance verification was completed in accordance with WI-091-ICP-MS-7900.[4] Fresh 3% HNO_3 solutions were added to the 15 mL PP tubes, which served as rinses, and a set was required between each sample. Tubing was changed as necessary. The calibration curve samples, unknown samples, and rinse samples were loaded into the autosampler, and the sequence was run.

4. RESULTS AND DISCUSSION

4.1 Calibration Curves

Qualitative analyses of the analytes in the solutions were determined by the presence of a signal for the most abundant isotope of a given element. Interferences of major isotopes were minimized by running the collision reaction cell in no-gas (no. 1), helium gas (no. 2), and hydrogen gas (no. 3) modes. In this instance, the ions were analyzed under helium mode. Quantitative analysis of analytes in solution was performed by generating at least a five-point calibration curve and forcing the intercept through zero. Equation 1 was used to convert the signal into a concentration using the slope and intercept of the calibration curve:

$$Y = \frac{(A/S) - B}{M} \tag{1}$$

where Y is the measured concentration, A is the signal of the analyte, S is the signal of the standard, B is the y intercept, and M is the slope of the calibration curve.

The measured concentration was converted to a final concentration by multiplying by a dilution factor as shown:

$$C_f = C_M \times (V_f / V_s) \tag{2}$$

where C_f is the final concentration, C_M is the measured concentration, V_f is the final volume of the solution analyzed, and V_s is the volume of the sample.

The concentrations used in this study were optimized to produce linear regression fits for the calibration curves. The goodness of the linear fits are summarized in Table 3 and Figures 1–3.

Table 3. Concentration Range for Each Analyte

Analyte		Concentration Range (ppb)
No.	Isotope	
1	^{33}As	1–500
2	^{34}Se	1–250

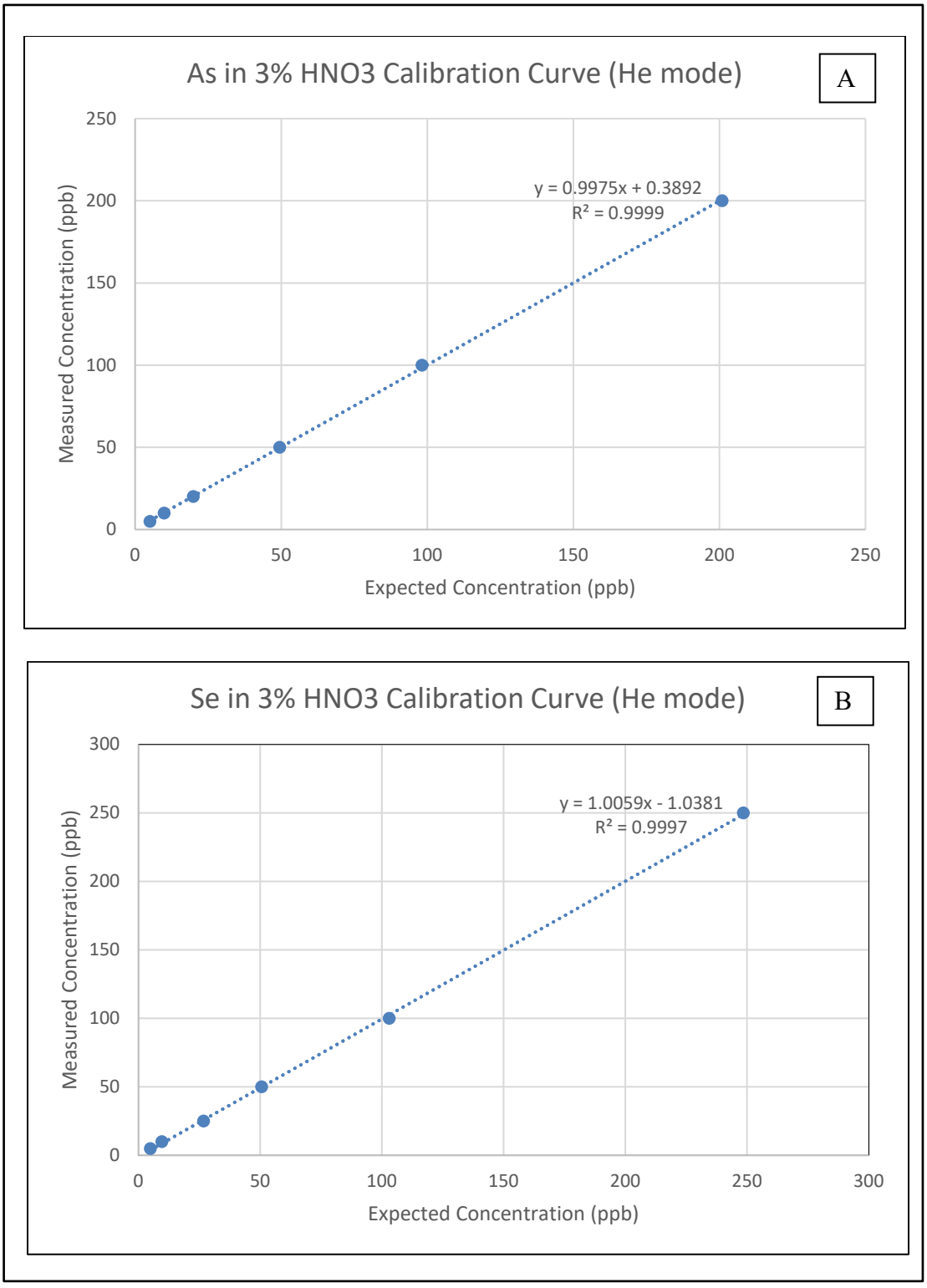

Figure 1. Isotopes (A) ^{33}As and (B) ^{34}Se in 3% nitric acid calibration curves.

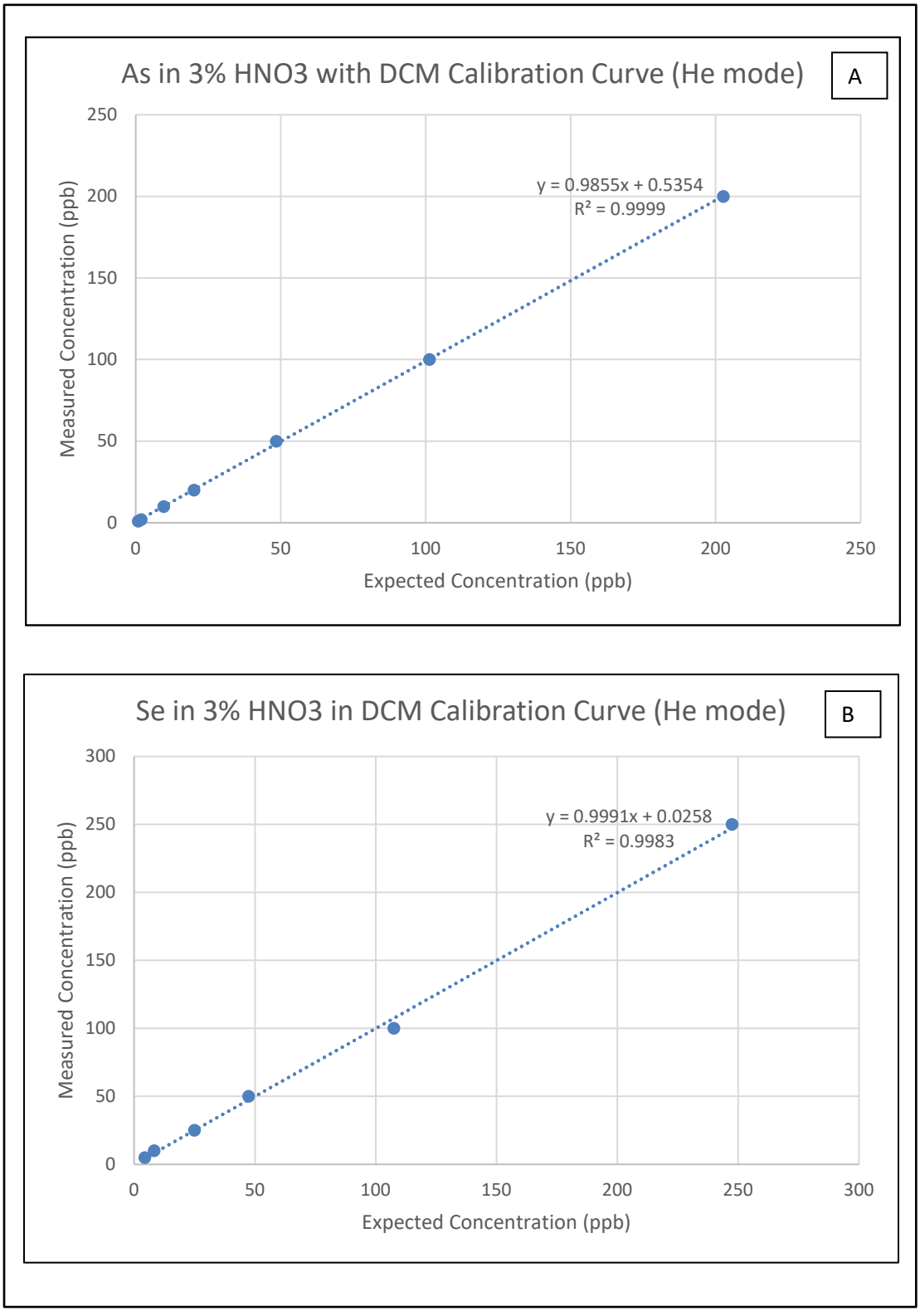

Figure 2. Isotopes (A) ^{33}As and (B) ^{34}Se in 3% nitric acid with dichloromethane calibration curves.

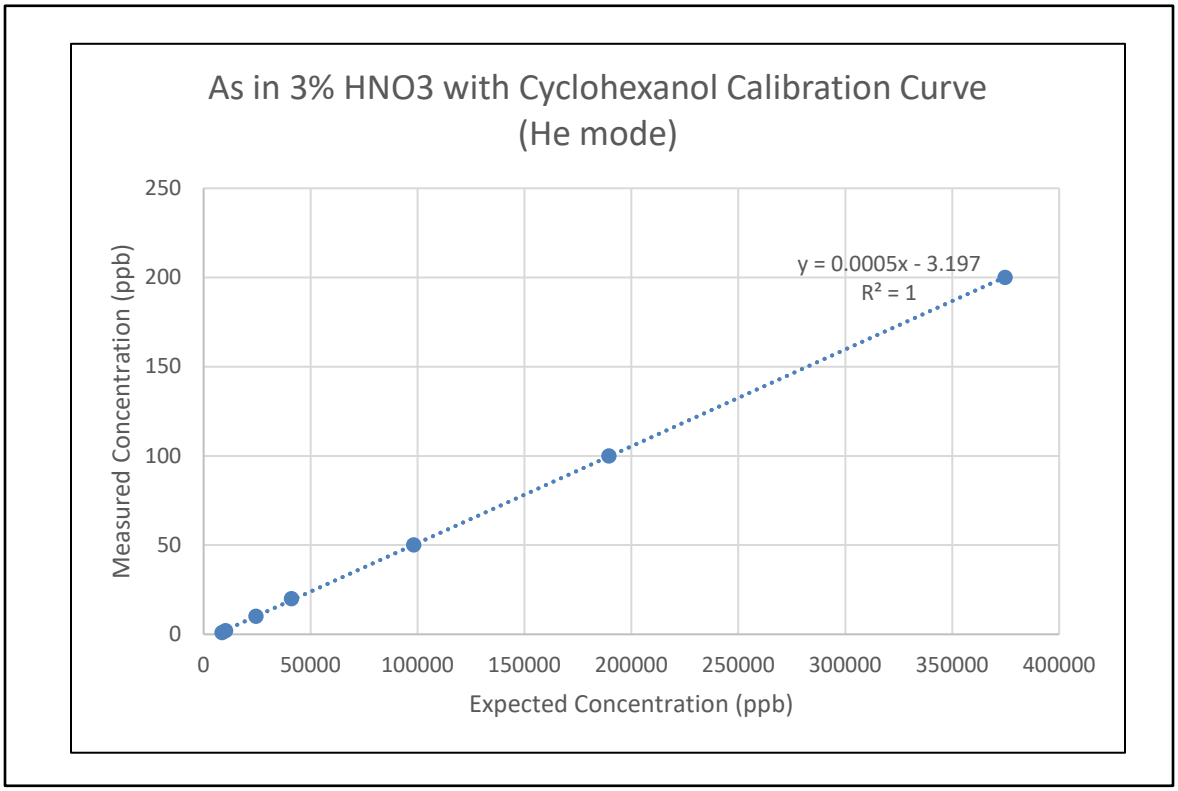

Figure 3. ^{33}As in 3% nitric acid with cyclohexanol calibration curves.

4.2 Relative Percent Differences

The precision of the analysis of the samples is determined by calculating the relative percent difference between the measured and expected concentrations:

$$\left(\frac{C_M - C_E}{\text{Average } (C_M + C_E)}\right) \times 100\% \quad (3)$$

where C_M is the concentration measured by the instrument, and C_E is the expected concentration.

Each ion had a minimum of five valid points in the calibration curve (Table 2).

4.3 Method Detection Limits (MDLs)

An MDL was performed as described in Chapter 1 of Test Method 6020A, Rev. 1.[5] Five replicate solutions with all of the analytes were prepared by spiking the matrix with the analyte at a concentration equal to three to five times the estimated MDL. The solutions were analyzed in random order. Each element was spiked at 0.001 ppm. The MDL was determined by multiplying the appropriate one-sided 99% *t* statistic (2.57 for six measurements) by the standard deviation (SD) of the replicate measurements:

$$\text{MDL} = 2.57 \times \text{SD} \quad (4)$$

As shown in Table 4, the MDLs for the analytes was 1 ppb.

Table 4. MDLs for All Analytes

Sample ID: Isotope in 3% HNO_3	SD	MDL (ppb)
^{33}As	0.3844	1
^{34}Se	0.1295	
^{33}As with DCM	0.2366	
^{34}Se with DCM	0.3459	
^{33}As with cyclohexanol	0.4390	

4.3.1 Precision and Accuracy (P&A) Study

Three solutions at each calibration level were prepared and analyzed in random order. This procedure was repeated on a second day to prepare a separate batch of standards at each level for a total of six analyses at each calibration level. Concentrations of the tested calibration levels are listed in Tables 5–9.

Table 5. Concentrations of As in 3% Nitric Acid Calibration Solutions Used for P&A Levels

Level	Concentration (ppb)		
	Target	Day 1	Day 2
1	5	8.587	5.078
2	10	13.639	9.969
3	20	23.707	19.907
4	50	52.304	49.953
5	100	99.555	98.229
6	200	199.004	200.906

Table 6. Concentrations of Se in 3% Nitric Acid Calibration Solutions Used for P&A Levels

Level	Concentration (ppb)		
	Target	Day 1	Day 2
1	5	4.939	5.810
2	10	9.690	11.416
3	25	26.778	19.801
4	50	50.654	56.559
5	100	103.100	97.319
6	250	248.465	250.208

Table 7. Concentrations of As in 3% Nitric Acid with DCM Calibration Solutions Used for P&A Levels

Level	Concentration (ppb)		
	Target	Day 1	Day 2
1	1	0.015	0.884
2	2	1.947	1.850
3	10	10.235	9.677
4	20	20.052	20.091
5	50	50.352	48.470
6	100	100.415	101.256
7	200	198.724	202.608
8	500	500.3885	498.862

Table 8. Concentrations of Se in 3% Nitric Acid with DCM Calibration Solutions Used for P&A Levels

Level	Concentration (ppb)		
	Target	Day 1	Day 2
1	5	4.499	4.179
2	10	8.252	8.833
3	25	25.020	22.317
4	50	47.431	45.465
5	100	107.624	94.907
6	250	247.542	253.275

Table 9. Concentrations of As in 3% Nitric Acid with Cyclohexanol Calibration Solutions Used for P&A Levels

Level	Concentration (ppb)		
	Target	Day 1	Day 2
1	1	0.684	2.647
2	2	1.495	3.390
3	10	9.644	11.696
4	20	18.668	21.358
5	50	50.889	51.639
6	100	102.408	101.497
7	200	200.595	203.252
8	500	499.254	498.139

The relative standard deviation (RSD) of the three solutions at each calibration level was calculated using eq 4:

$$\text{RSD} = \left(\frac{\text{SD of replicates}}{\text{average result}}\right) \quad (5)$$

Percent recoveries were ≥90% for As and Se (Tables 10–14).

Table 10. ICP-MS Data for the As in Nitric Acid P&A Study (He Mode)

	Recovered Concentration (ppb)					
Level	**1**	**2**	**3**	**4**	**5**	**6**
Spiked Conc. (ppb)	**5**	**10**	**20**	**50**	**100**	**200**
Replicate 1	9.05	15.08	29.79	53.51	108.68	208.54
Replicate 2	9.88	14.95	25.46	55.41	108.56	212.50
Replicate 3	10.06	14.48	29.55	55.77	108.65	220.30
Replicate 4	5.33	10.34	20.84	51.91	100.78	210.61
Replicate 5	5.17	10.57	20.75	51.75	100.67	205.87
Replicate 6	5.58	10.6	21.56	52.73	104.91	210.94
Mean Value	7.51	12.67	24.66	53.51	3.88	4.90
Mean % Recovery	150.23	126.70	123.29	107.03	105.38	105.73
SD	2.39	2.38	4.25	1.73	3.88	4.90
%RSD	31.75	6.13	10.93	4.45	9.97	12.59

Conc. is concentration.

Table 11. ICP-MS Data for the As in Nitric Acid with DCM P&A Study (He Mode)

	Recovered Concentration (ppb)					
Level	**1**	**2**	**3**	**4**	**5**	**6**
Spiked Conc. (ppb)	**5**	**10**	**20**	**50**	**100**	**200**
Replicate 1	5.10	10.55	19.51	48.74	106.73	189.66
Replicate 2	5.44	9.33	20.35	50.34	94.56	196.75
Replicate 3	5.22	9.76	19.93	49.00	94.65	195.52
Replicate 4	5.28	10.34	21.16	49.63	99.86	198.11
Replicate 5	5.66	10.82	20.49	51.66	99.49	201.88
Replicate 6	5.76	10.44	21.01	52.87	100.52	209.21
Mean Value	5.41	10.21	20.41	50.38	99.3	198.52
Mean % Recovery	108.21	102.05	102.05	100.75	99.30	99.26
SD	0.26	0.55	0.63	1.61	4.50	6.58
%RSD	4.78	5.43	3.08	3.20	4.53	3.31

Conc. is concentration.

Table 12. ICP-MS Data for the Se in Nitric Acid P&A Study (He Mode)

	Recovered Concentration (ppb)					
Level	**1**	**2**	**3**	**4**	**5**	**6**
Spiked Conc. (ppb)	**5**	**10**	**20**	**50**	**100**	**200**
Replicate 1	5.04	10.71	25.41	49.47	109.31	261.95
Replicate 2	5.65	10.81	27.72	55.20	104.27	269.85
Replicate 3	5.80	10.20	27.62	52.61	104.86	260.40
Replicate 4	4.99	10.19	25.32	54.20	100.89	259.78
Replicate 5	4.98	10.76	28.35	51.37	101.72	256.28
Replicate 6	5.32	9.72	25.57	47.96	111.11	278.07
Mean Value	5.30	10.40	26.66	51.50	105.36	264.39
Mean % Recovery	105.94	103.98	106.66	103.60	105.36	105.75
SD	0.36	0.43	1.38	2.77	4.08	8.07
%RSD	6.74	4.18	5.16	5.35	3.88	3.05

Conc. is concentration.

Table 13. ICP-MS Data for the Se in Nitric Acid with DCM P&A Study (He Mode)

	Recovered Concentration (ppb)					
Level	**1**	**2**	**3**	**4**	**5**	**6**
Spiked Conc. (ppb)	**5**	**10**	**25**	**50**	**100**	**250**
Replicate 1	4.89	9.27	28.74	46.75	97.17	244.1
Replicate 2	4.35	10.34	24.56	49.76	100.14	239.43
Replicate 3	4.41	9.21	24.68	51.93	101.70	245.80
Replicate 4	5.05	7.92	23.3	44.95	91	218.51
Replicate 5	4.5	9.48	21.98	42.63	92.27	220
Replicate 6	4.24	9.16	23.09	44.63	90.8	223
Mean Value	4.57	9.23	24.39	45.78	95.51	231.81
Mean % Recovery	94.47	92.3	97.57	93.55	95.51	92.72
SD	0.32	0.78	2.35	3.48	4.81	12.64
%RSD	7.06	8.42	9.65	7.44	5.03	5.45

Conc. is concentration.

Table 14. ICP-MS Data for the As in Nitric Acid with Cyclohexanol P&A Study (He Mode)

	Recovered Concentration (ppb)					
Level	**1**	**2**	**3**	**4**	**5**	**6**
Spiked Conc. (ppb)	**2**	**10**	**20**	**50**	**100**	**200**
Replicate 1	4.2198	12.7938	22.5071	51.4366	–	218.3807
Replicate 2	3.9586	13.4145	23.7769	56.193	104.1062	219.0493
Replicate 3	3.9558	13.3154	23.2226	51.689	101.5837	–
Replicate 4	–	7.6084	18.3445	27.2045	100.075	284.8141
Replicate 5	–	8.3162	18.0636	51.6534	89.9991	203.492
Replicate 6	0.03752	8.6632	17.9994	52.2379	105.5164	201.8105
Mean Value	3.0	10.7	20.7	48.4	100.3	225.5
Mean % Recovery	152.1465	106.8528	103.2618	96.8048	100.2561	112.7547
SD	2.0074	2.7561	2.7884	10.5388	6.1134	34.1175
%RSD	152.1465	106.8528	103.2618	96.8048	100.2561	112.7547

Conc. is concentration.
– is not applicable.

4.3.2 Measurement of Uncertainty

The measurements of uncertainty for the three solutions at each calibration level were determined in accordance with CCDC Chemical Biological Center FAC work instruction WI-098.[4] The major sources contributing to the measurement of uncertainty were errors from the calibration curve and process repeatability. These sources were measured during the method validation and then combined to determine the overall uncertainty at 95% confidence (Table 15).

Table 15. Measurement of Uncertainty

Analyte in 3% Nitric Acid	Concentration (µg/mL)	Uncertainty (%)
As	0.001	12.0
	*0.005	31.75
	0.01	18.81
	0.02	17.24
	0.05	3.23
	0.1	3.68
	0.2	72.32
As with DCM	0.001	7.0
	*0.005	4.78
	0.01	5.43
	0.02	3.08
	0.05	3.20
	0.1	4.53
	0.2	3.31
	0.5	3.36
Se	0.001	4.0
	*0.005	4.78
	0.01	5.43
	0.025	3.08
	0.05	3.20
	0.1	4.53
	0.25	3.31
Se with DCM	0.001	11.0
	*0.005	6.74
	0.01	4.18
	0.025	5.16
	0.05	5.35
	0.1	3.88
	0.25	3.05
As with cyclohexanol	0.002	5.16
	0.01	27.46
	0.02	7.17
	0.05	27.08
	0.1	15.71
	0.2	87.68

*Denotes practical quantitation limit identified through P&A and measurement uncertainty.

5. CONCLUSIONS

ICP–MS is a viable method for qualitative and quantitative analysis of two analytes in aqueous solutions. Calibration curves were valid for a minimum of five points for all analytes. MDLs were calculated for arsenic and selenium. Measurements of uncertainty were also calculated for As and Se at each calibration level in each sample matrix.

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ACRONYMS AND ABBREVIATIONS

DCM	dichloromethane
CCDC	U.S. Army Combat Capabilities Development Command
FAC	Forensic Analytical Center
HDPE	high-density polyethylene
ICP–MS	inductively coupled plasma mass spectrometry
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
PP	polypropylene
P&A	precision and accuracy
RSD	relative standard deviation
SD	standard deviation

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