Berry and Plant Tissue Monitoring Near the Eagle Mine and Humboldt Mill

Marquette County, Michigan

Superior Watershed Partnership

Community Environmental Monitoring Program

Year One Monitoring Results and Analysis

April 14, 2016

www.swpcemp.org

www.superiorwatersheds.org

SUPERIOR WATERSHED PARTNERSHIP AND LAND TRUST

TABLE OF CONTENTS

Berry and Plant Tissue Monitoring Study Near the Eagle Mine and Humboldt Mill

Introduction

The Community Environmental Monitoring Program (CEMP) of the Eagle Mine, located in Marquette County, Michigan, began in 2012 based on community concerns regarding potential environmental and cultural impacts associated with mining operations. The CEMP is implemented by two community-based organizations; the Superior Watershed Partnership (SWP) and the Community Foundation of Marquette County (CFMC) and is defined and governed by formal agreements between these organizations and Eagle Mine LLC, a subsidiary of Lundin Mining. The CEMP is designed to build a comprehensive and accurate picture of any environmental impacts that may be a result of Eagle Mine's operations at the mine site, the Humboldt Mill, and along the designated transportation route. The CEMP is independent, transparent, and based on the highest scientific standards. Monitoring results of Eagle Mine's environmental performance are made available to the public on the CEMP website www.swpcemp.org.

During 2015, CEMP monitoring was expanded to evaluate concerns raised by the Keweenaw Bay Indian Community (KBIC) and other community members regarding potential impacts from mining operations on nearby edible plant species and species of high cultural value. Fruit bearing plants located near the Eagle Mine and Humboldt Mill were identified as a priority for the study. They included; blueberry, juneberry, chokecherry, pin cherry, raspberry, blackberry, strawberry, thimbleberry, cranberry, juniper berry, and wild rice. Specific objectives of the monitoring program included:

- Locate and identify edible and culturally-important plant species in the immediate vicinity/adjacent to the Eagle Mine and Humboldt Mill sites.
- Obtain plant tissue (berries, leaves, and/or roots) for analysis at a certified laboratory.
- Evaluate concentrations of various metals observed in tissue samples and compare them to US Environmental Protection Agency's (US EPA) oral tolerable intake values (TDI), and the Food and Drug Administration's (FDA) recommended Daily Values (DV).
- Use data/results to monitor short-term and long-term changes that may indicate impacts from mining activities.

Methods

Study methods and activities included the following:

1) *Selection of test and control site locations including culturally important plant species*
   Monitoring locations consisted of two test sites that were established within a 2.0 mile radius of the Eagle Mine and the Humboldt Mill. Following reconnaissance efforts, a third site (control site) was established in an area that is unlikely to be impacted by environmental contaminants related to mining activities (Figure 1). All test sites were documented, mapped, and categorized according to species sampled.

2) *Plant tissue collection from test and control sites*
   Plant tissue specimens were identified in the field and collected by experienced SWP/CEMP staff in accordance with guidelines established by White Water Associates, Inc. Plant species identified and collected included: blueberry (*Vaccinium spp.*) fruit, blueberry plant tissue (stems, leaves, and roots), and raspberry (*Rubus idaeus*) fruit . Sample collection occurred during the peak fruit maturation period for each species. Blueberries and blueberry plant tissue samples were collected between July 17 and July 23, 2015, while raspberries were collected August 11. 2015. Additional raspberry and blueberry plants were collected on September 22, 2015 to provide additional material for analyses of monitoring parameters (mercury and uranium) that were not included in earlier analyses.

   A total of nine samples were collected. Blueberry fruit, blueberry plant tissue, and raspberry fruit were collected from: 1) sites within a two mile radius of the Eagle Mine, 2) sites within a two mile radius of

the Humboldt Mill, and 3) the control site. Actual sample sites within the designated two mile radius varied according to the distribution of each species. At each collection site, approximately 25 grams of each sample was collected for analysis. Samples were collected while wearing clean nitrile gloves, placed in labelled plastic bags, and stored on ice. Samples were shipped overnight to White Water Associates, Inc. for general chemistry and trace metal analysis. Additional material (~100 grams/sample) was collected for mercury and uranium analyses.

3) *Laboratory analysis of plant tissue*
Analytical services were conducted by White Water Associates, Inc. using analytical QA/QC and reporting protocols of NELAP, USEPA, CLP, DoD QSM, SW846, 40 CFR Part 136, ASTM, Standard Methods, and in-house Standard Operating Procedures (SOPs).

Parameters and analytical methods for each sample type are listed in Tables 1 and 2. Plant tissue was dried and ground with a mortar and pestle before undergoing the sample digestion process and subsequent sample analysis. Detailed digestion process protocols as provided by White Water Associates, Inc. are provided in Appendix A.

Results

Analytical test results for each sample site are noted in Tables 1 and 2. Sample parameters were considered to be present if detected at values greater than the method detection limit (MDL) and results were considered to be non-detect (i.e. not present) if below the MDL. Non-detect sample results are reported as less than the MDL (e.g. <1.0) in Tables 1 and 2. The MDL is a value derived from a statistical calculation in which the lab is 99% confident that the result is greater than zero. The laboratory also calculates a value known as the method quantitation limit (MQL). The MQL is higher than the MDL and is a value in which precision and accuracy limits can be reliably achieved (greater than 99% confidence). In the event that a sample result is detected above the MDL, but is not greater than the MQL, the sample result is considered estimated because the laboratory can only say with 99% confidence that the result is greater than zero. In these situations, the sample result is denoted with a "J" on Tables 1 and 2 indicating that it is an estimated value. Sample results greater than the MQL do not require any qualification since the laboratory can reliably test to those levels. The MDLs and MQLs for each parameter and sample type are summarized in Appendix B

Also listed in Tables 1 and 2 are the US Environmental Protection Agency's (US EPA) oral tolerable intake values (TDI) and the Food and Drug Administration's (FDA) recommended Daily Values (DV). Tolerable daily intake refers to the estimated amount of a potentially harmful substance in food or drinking water that can be ingested daily over a lifetime without appreciable health risk. The Daily Value provides the suggested amount of each nutrient that an adult consuming a 2,000 calorie diet should incorporate into their daily diet.

While metal concentrations varied among sample types and locations, 24 of the parameters tested recorded greater levels at one or more test sites (Eagle Mine or Humboldt Mill) than at the control site. Of those parameters, Copper, Nickel, Nitrate/Nitrite, Potassium, and total Kjeldahl Nitrogen recorded elevated levels among **all** test site samples (Table 3, Figure 2). Nineteen parameters were elevated in one or more but **not all** of the sample groups (blueberries, blueberry plant, raspberries). Conversely, seven of the parameters tested for were not detected among any of the three sample groups. Parameters included: Antimony, Arsenic, Beryllium, Cadmium, Lead, Molybdenum, and Thallium (Table 4).

Based on the metal concentrations and the established TDIs for each parameter, Tables 5 and 6 depict the amount of blueberries and raspberries (in both kilograms and cups of berries) that would need to be ingested on a daily basis over a lifetime in order to exceed the TDI. These values were calculated using the following formulas:

Kg needed to ingest to exceed TDI = (TDI x 70 kg) / (Parameter test result)
Equivalent in cups of blueberries = (kg needed to exceed TDI) / (0.140 kg)
Equivalent in cups of raspberries = (kg needed to exceed TDI) / (0.125 kg)

In the above formulas, 70 kg, equal to approximately 154 pounds, was used to represent the mass of the average adult, while 0.140 and 0.125 kg were used to represent the average mass of one cup of blueberries or raspberries, respectively.

Table 7 illustrates the known essential, beneficial, and nonessential metals as indicated in the US EPA's Metals Risk Assessment. Of the nonessential metals listed, Aluminum, Barium, Silver, and Strontium recorded greater levels at one or more test sites than at the control site for one or more sample groups (Tables 3 and 7).

The sample set collected in 2015 provides baseline data regarding metal concentrations in blueberry and raspberry plants and berries collected near the Eagle Mine and Humboldt Mill sites. Referencing these data in future years may help to support whether or not bioaccumulation of metals is occurring in these plant species. Due to a small sample size and various other potential confounding factors including regional variation, species specific differences, insufficient length of study, and alternate sources of pollution, any differences noted between control and test samples cannot be reliably attributed to mining activities at this time.

TABLES

Table 1. Laboratory test results for blueberry fruit and blueberry plants listed by parameter and sample location.

		Test Result - Blueberry Fruit (mg/kg)*			Test Result - Blueberry Plant (mg/kg)				
PARAMETER	**METHOD**	**MINE SITE (7/22/15)**	**MILL SITE (7/17/15)**	**CONTROL SITE (7/23/15)**	**MINE SITE (7/22/15)**	**MILL SITE (7/17/15)**	**CONTROL SITE (7/23/15)**	**Oral Tolerable Daily Intake Values (TDI)** mg/kg-d**	**DAILY VALUE*** (mg)**
Aluminum	200.7	21 **J**	16 **J**	<8.9	110	410	62	1	
Ammonia-N	350.1	<130	<160	<110	<54	<56	<46		
Antimony	200.7	<1.8	<1.9	<1.8	<2	<2	<1.9	0.0004	
Arsenic	200.7	<1.8	<1.9	<1.8	<2	<2	<1.9	0.0003	
Barium	200.7	32	20	7.5	130	160	59	0.2	
Beryllium	200.7	<0.036	<0.037	<0.036	<0.04	<0.04	<0.037	0.002	
Boron	200.7	9 **J**	6 **J**	6 **J**	23	30	48	0.2	
Cadmium	200.7	<0.091	<0.093	<0.089	<0.1	<0.1	<0.093	0.000057	
Calcium	200.7	2100	1400	840	7100	10000	6200		1000
Chromium	200.7	0.94	1.2	0.66	2.6	4	2.3	0.00003	0.12
Cobalt	200.7	0.25 **J**	<0.19	0.26 **J**	<0.2	0.35 **J**	0.27 **J**		
Copper	200.7	4.7	5.7	4.6	10	9	6.9	0.04	2
Iron	200.7	35	24	17	150	650	72	0.7	18
Lead	200.7	<0.91	<0.93	<0.89	<1	<1	<0.93		
Lithium	200.7	<0.6	<0.56	<0.54	0.91 **J**	1.4 **J**	0.65 **J**	0.02	
Magnesium	200.7	630	630	510	1900 **J**	3100	2100		400
Manganese	200.7	260	160	130	1.1	1.4 **J**	1.7 **J**	0.0000143	2
Molybdenum	200.7	<0.3	<0.28	<0.27	<0.3	<0.3	<0.28	0.005	0.075
Mercury (9/22/15)	7471B	-	-	-	<0.02	<0.023	<0.034		
Nickel	200.7	0.93 **J**	2.6 J	<0.89	1.6 **J**	1.3 **J**	<0.93	0.02	
Nitrate/Nitrite-N	4500-NO3-F	110 **J**	220	82	57	3900	52		
Potassium	200.7	5900	5300	3300	4300	5300	3100		3500
Selenium	200.7	<1.8	<1.9	2 **J**	2.1 **J**	2.6 **J**	<1.9	0.005	0.07
Silver	200.7	<0.27	<0.28	<0.27	0.94 **J**	1.6	0.89 **J**	0.005	
Sodium	200.7	<45	<46	<45	<50	<50	<46		2400
Strontium	200.7	2.2	3.6	2	6.2	15	6.2	0.6	
Sulfate	4500-SO4-E	2800	<2400	2200 **J**	<3200	5500 **J**	3100 **J**		

		Test Result - Blueberry Fruit (mg/kg)*			Test Result - Blueberry Plant (mg/kg)				
PARAMETER	**METHOD**	**MINE SITE (7/22/15)**	**MILL SITE (7/17/15)**	**CONTROL SITE (7/23/15)**	**MINE SITE (7/22/15)**	**MILL SITE (7/17/15)**	**CONTROL SITE (7/23/15)**	**Oral Tolerable Daily Intake Values (TDI)** mg/kg-d**	**DAILY VALUE*** (mg)**
Sulfur (mg/L)	200.7	667	692	519	639	986	228		
Thallium	200.7	<1.8	<1.9	<1.8	<2	<2	<1.9	0.00007	
Total Kjeldahl Nitrogen (s)	351.2	4400	2800	2500	8000	7400	4400		
Total Phosphorus	365.4	760	440	450	960	890	700		
Uranium (9/22/15)	6010C	-	-	-	<9.5	<11.3	<17.1	0.0002	
Vanadium	200.7	<0.36	<0.37	<0.36	<0.4	1.3 **J**	<0.37	0.001	
Zinc	200.7	11	11	10	29	35	33	0.3	15

* Test results for Sulfur measured in mg/L

** Provided by the US EPA. Tolerable daily intake refers to the estimated amount of a potentially harmful substance in food or drinking water that can be ingested daily over a lifetime without appreciable health risk.

*** Provided by the FDA. Based on 2,000 calorie diet.

Dashed lines indicate parameters that were not analyzed.

"**J**" Indicates that the quantitation is an estimated value because the result is less than the sample method quantitation limit (MQL) but greater than the method detection limit (MDL)

All parameters that were not detected in a sample are listed as < the respective MDL.

Table 2. Laboratory test results for raspberry fruit listed by parameter and sample location.

PARAMETER	METHOD	Test Result - Raspberry Fruit (mg/kg)			Oral Tolerable Daily Intake Values (TDI)* mg/kg-d	DAILY VALUE** (mg)
		MINE SITE (8/11/15)	MILL SITE (8/11/15)	CONTROL SITE (8/11/15)		
Aluminum	200.7	<9.6	<9.8	<10	1	
Ammonia-N	350.1	<130	130 **J**	<140		
Antimony	200.7	<2	<2	<2	0.0004	
Arsenic	200.7	<2	<2	<2	0.0003	
Barium	200.7	14	12	13	0.2	
Beryllium	200.7	<0.04	<0.04	<0.04	0.002	
Boron	200.7	15	15	14	0.2	
Cadmium	200.7	<0.1	<0.1	<0.1	0.000057	
Calcium	200.7	2000	2500	3000		1000
Chromium	200.7	0.81	0.87	0.87	0.00003	0.12
Cobalt	200.7	<0.2	<0.2	<0.2		
Copper	200.7	9.3	6.7	5.4	0.04	2
Iron	200.7	54	44	51	0.7	18
Lead	200.7	<1	<1	<1		
Lithium	200.7	<0.58	<0.59	<0.62	0.02	
Magnesium	200.7	1800	1900	1700		400
Manganese	200.7	320	98	39	0.0000143	2
Molybdenum	200.7	<0.29	<0.29	<0.31	0.005	0.075
Mercury* **	7471B	0.024	<0.028	0.033		
Nickel	200.7	2.4 **J**	1.5 **J**	1.1 **J**	0.02	
Nitrate/Nitrite-N	4500-NO3-F	78 **J**	380	73		
Potassium	200.7	12000	13000	10000		3500
Selenium	200.7	<3	3.8 **J**	<3	0.005	0.07
Silver	200.7	<0.3	<0.3	<0.3	0.005	
Sodium	200.7	<48	51 **J**	<52		2400
Strontium	200.7	4.9	6.6	5.7	0.6	
Sulfate	4500-SO4-E	3200 **J**	2800 **J**	3400 **J**		
Thallium	200.7	<2	<2	<2	0.00007	
Total Kjeldahl Nitrogen	351.2	8700	10000	4600		
Total Phosphorus	365.4	1600	2000	1700		
Uranium* **	6010C	<10.6	<13.5	<16.8		
Vanadium	200.7	<0.6	<0.6	<0.6	0.001	
Zinc	200.7	39	43	26	0.3	15

* Source: US EPA.

** Source: FDA. Based on 2,000 calorie diet.

***Samples included plant tissue (not berries) obtained on 9/22/15

"**J**" Indicates that the quantitation is an estimated value because the result is less than the sample method quantitation limit (MQL) but greater than the method detection limit (MDL)

All parameters that were not detected in a sample are listed as < the respective MDL.

Table 3. Parameters where samples obtained from test sites (Eagle Mine and Humboldt Mill) recorded greater level(s) than control samples.

Check Indicates that the Test Sample Recorded a Level Greater than the Control Sample						
	Blueberries		Blueberry Plant		Raspberries	
Parameter	Mine	Mill	Mine	Mill	Mine	Mill
Aluminum	x	x	x	x		
Ammonia - N						x
Barium	x	x	x	x	x	
Boron	x				x	x
Calcium	x	x	x	x		
Chromium	x	x	x	x		
Cobalt				x		
Copper	x	x	x	x	x	x
Iron	x	x	x	x	x	
Lithium			x	x		
Magnesium	x	x		x	x	x
Manganese	x	x			x	x
Nickel	x	x	x	x	x	x
Nitrate/Nitrite-N	x	x	x	x	x	x
Potassium	x	x	x	x	x	x
Selenium			x	x		x
Silver			x	x		
Sodium						x
Strontium	x	x		x		x
Sulfate	x			x		
Sulfur (mg/L)	x	x	x	x	-	-
Total Kjeldahl Nitrogen	x	x	x	x	x	x
Total Phosphorus	x		x	x		x
Zinc	x	x		x	x	x

Gold indicates elevated levels (greater than control) for all test samples

Dashed lines indicate parameters that were not analyzed

Table 4. Parameters not detected at sample sites.

PARAMETERS NOT DETECTED AT SAMPLE SITES (MINE, MILL, CONTROL) FOR EACH SAMPLE SET			
Parameter	**Blueberries**	**Blueberry Plant**	**Raspberries**
Aluminum			x
Ammonia-N	x	x	
Antimony	x	x	x
Arsenic	x	x	x
Beryllium	x	x	x
Cadmium	x	x	x
Cobalt			x
Lead	x	x	x
Lithium	x		x
Mercury	-	x	
Molybdenum	x	x	x
Silver	x		x
Sodium	x	x	
Thallium	x	x	x
Uranium*	-	x	x
Vanadium	x		x

*Raspberry Plant Tissue (not berries)
Green indicates parameters not detected in all three sample sets
Dashed lines indicate parameters that were not analyzed

Table 5. Mass of blueberries (in kilograms and cups) required to exceed US EPA designated Tolerable Daily Intake Values (TDIs).

Parameter	US EPA Oral Tolerable Daily Intake Values (TDI), mg/kg-d***	Blueberry Fruit								
		Test Result Mine Site (mg/kg)	kg needed to ingest to exceed TDI*	Equivalent in cups of berries**	Test Result Mill Site (mg/kg)	kg needed to ingest to exceed (TDI)*	Equivalent in cups of berries**	Test Result Control Site (mg/kg)	kg needed to ingest to exceed TDI*	Equivalent in cups of berries**
Aluminum	1	21	3.33	23.8	16	4.38	31.25	ND	-	-
Barium	0.2	32	0.44	3.1	20	0.70	5.00	7.5	1.87	13.3
Boron	0.2	9	1.56	11.1	6	2.33	16.67	6	2.33	16.7
Calcium	NL	2100	-	-	1400	-	-	840	-	-
Chromium	0.00003	0.94	0.002	0.02	1.2	0.002	0.01	0.66	0.003	0.02
Cobalt	NL	0.25	-	-	ND	-	-	0.26	-	-
Copper	0.04	4.7	0.60	4.3	5.7	0.49	3.51	4.6	0.61	4.3
Iron	0.7	35	1.40	10.0	24	2.04	14.58	17	2.88	20.6
Magnesium	NL	630	-	-	630	-	-	510	-	-
Manganese	0.0000143	260	0.000004	0.00003	160	0.00001	0.00004	130	0.00001	0.0001
Nickel	0.02	0.93	1.51	10.8	2.6	0.54	3.85	ND	-	-
Nitrate/Nitrite-N	NL	110	-	-	220	-	-	82	-	-
Potassium	NL	5900	-	-	5300	-	-	3300	-	-
Selenium	0.005	ND	-	-	ND	-	-	2	0.18	1.3
Strontium	0.6	2.2	19.09	136.4	3.6	11.67	83.33	2	21.00	150.0
Sulfate	NL	2800	-	-	ND	-	-	2200	-	-
Total Kjeldahl Nitrogen	NL	4400	-	-	2800	-	-	2500	-	-
Total Phosphorus	NL	760	-	-	440	-	-	450	-	-
Zinc	0.3	11	1.91	13.6	11	1.91	13.64	10	2.10	15.0

NL = Not Listed
ND = Not Detected

* Based on body weight of 70 kg (approximately 154 lb). Calculated using the formula: (TDI x 70 kg)/Test Result.
** Based on 1 cup of blueberries = 140 grams = 0.140 kg. Calculated using the formula: (kg needed to exceed TDI)/(0.140 kg).
*** Tolerable daily intake refers to the estimated amount of a potentially harmful substance in food or drinking water that can be ingested daily over a lifetime without appreciable health risk.

Table 6. Mass of raspberries (in kilograms and cups) required to exceed US EPA designated Tolerable Daily Intake Values (TDIs).

Parameter	US EPA Oral Tolerable Daily Intake Values (TDI), mg/kg-d***	Test Result - Raspberry Fruit								
		Test Result Mine Site (mg/kg)	kg needed to ingest to exceed TDI*	Equivalent in cups of berries**	Test Result Mill Site (mg/kg)	kg needed to ingest to exceed TDI*	Equivalent in cups of berries**	Test Result Control Site (mg/kg)	kg needed to ingest to exceed TDI*	Equivalent in cups of berries**
Ammonia-N	NL	ND	-	-	130	-	-	ND	-	-
Barium	0.2	14	1.00	8.0	12	1.17	9.3	13	1.08	8.6
Boron	0.2	15	0.93	7.5	15	0.93	7.5	14	1.00	8.0
Calcium	NL	2000	-	-	2500	-	-	3000	-	-
Chromium	0.00003	0.81	0.003	0.02	0.87	0.002	0.02	0.87	0.002	0.02
Copper	0.04	9.3	0.30	2.4	6.7	0.42	3.3	5.4	0.52	4.1
Iron	0.7	54	0.91	7.3	44	1.11	8.9	51	0.96	7.7
Magnesium	NL	1800	-	-	1900	-	-	1700	-	-
Manganese	0.0000143	320	0.000003	0.00003	98	0.00001	0.0001	39	0.00003	0.0002
Nickel	0.02	2.4	0.58	4.7	1.5	0.93	7.5	1.1	1.27	10.2
Nitrate/Nitrite-N	NL	78	-	-	380	-	-	73	-	-
Potassium	NL	12000	-	-	13000	-	-	10000	-	-
Selenium	0.005	ND	-	-	3.8	0.09	0.7	ND	-	-
Sodium	NL	ND	-	-	51	-	-	ND	-	-
Strontium	0.6	4.9	8.57	68.6	6.6	6.36	50.9	5.7	7.37	58.9
Sulfate	NL	3200	-	-	2800	-	-	3400	-	-
Total Kjeldahl Nitrogen	NL	8700	-	-	10000	-	-	4600	-	-
Total Phosphorus	NL	1600	-	-	2000	-	-	1700	-	-
Zinc	0.3	39	0.54	4.3	43	0.49	3.9	26	0.81	6.5

NL = Not Listed
ND = Not Detected

* Based on body weight of 70 kg (approximately 154 lb). Calculated using the formula: (TDI x 70 kg)/Test Result.
** Based on 1 cup of raspberries = 125 grams = 0.125 kg. Calculated using the formula: (kg needed to exceed TDI)/(0.125 kg)
*** Tolerable daily intake refers to the estimated amount of a potentially harmful substance in food or drinking water that can be ingested daily over a lifetime without appreciable health risk.

Table 7. US EPA's Metals Risk Assessment - Metals Classified by their Known Essentiality

Metals Classified by their Known Essentiality					
	Essential (Known requirement for Health and Function)		Beneficial (But Not Known to be Essential)		Nonessential (and not known to be beneficial)
Metal	Plants	Animals	Plants	Animals	
Aluminum					x
Antimony					x
Arsenic				x	
Barium					x
Beryllium					x
Cadmium					x
Chromium		x			
Cobalt		x	x		
Copper	x	x			
Lead					x
Manganese	x	x			
Mercury					x
Molybdenum	x	x			
Nickel	x	x			
Selenium		x	x		
Silver					x
Strontium					x
Thallium					x
Vanadium				x	
Zinc	x	x			

Source: US EPA's Metals Risk Assessment (http://www.epa.gov/sites/production/files/2013-09/documents/metals-risk-assessment-final.pdf)

Gold indicates nonessential metals that recorded greater levels at test sites than at the control site for one or more sample groups

FIGURES

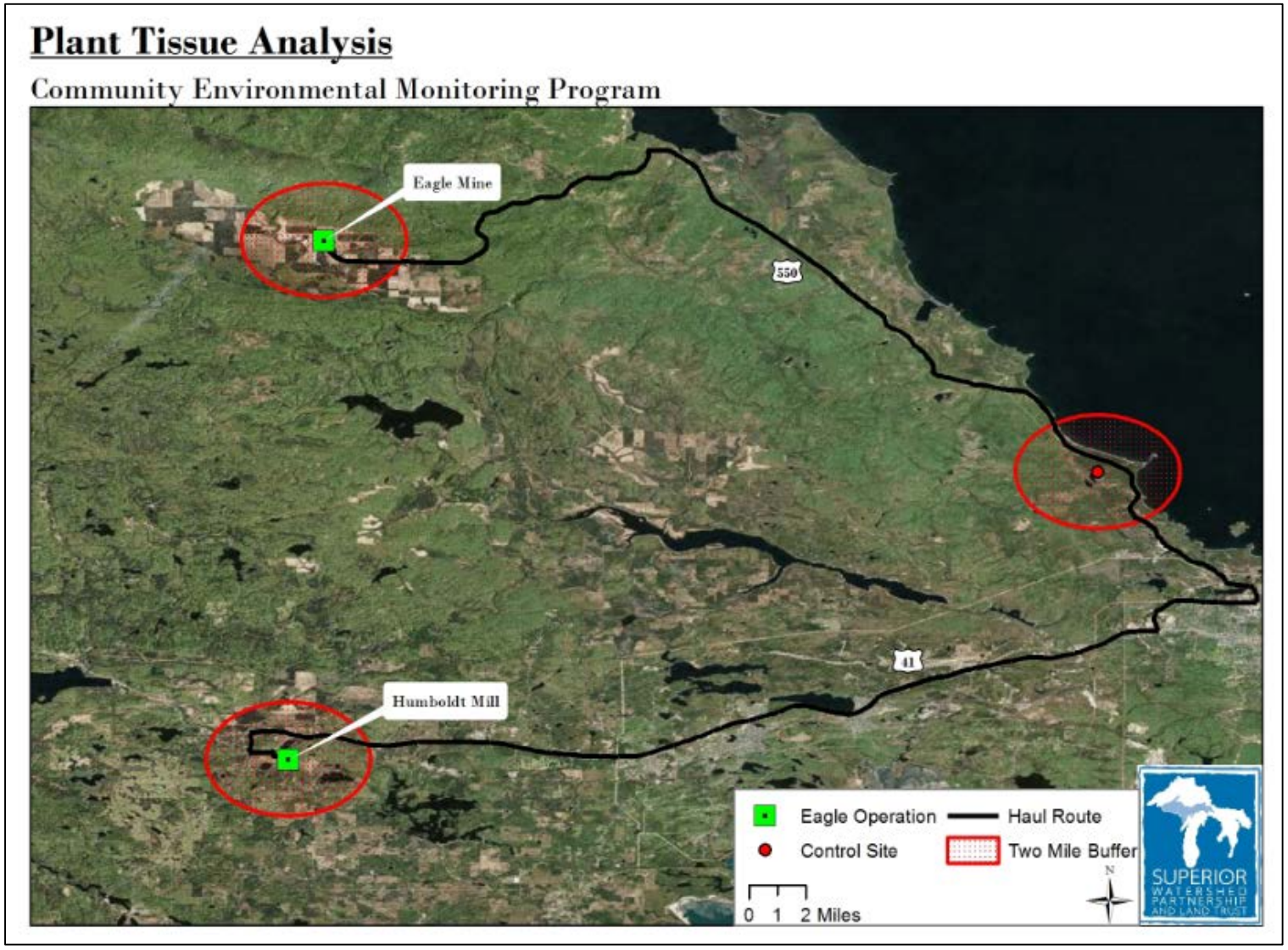

Figure 1. Test and control sample sites.

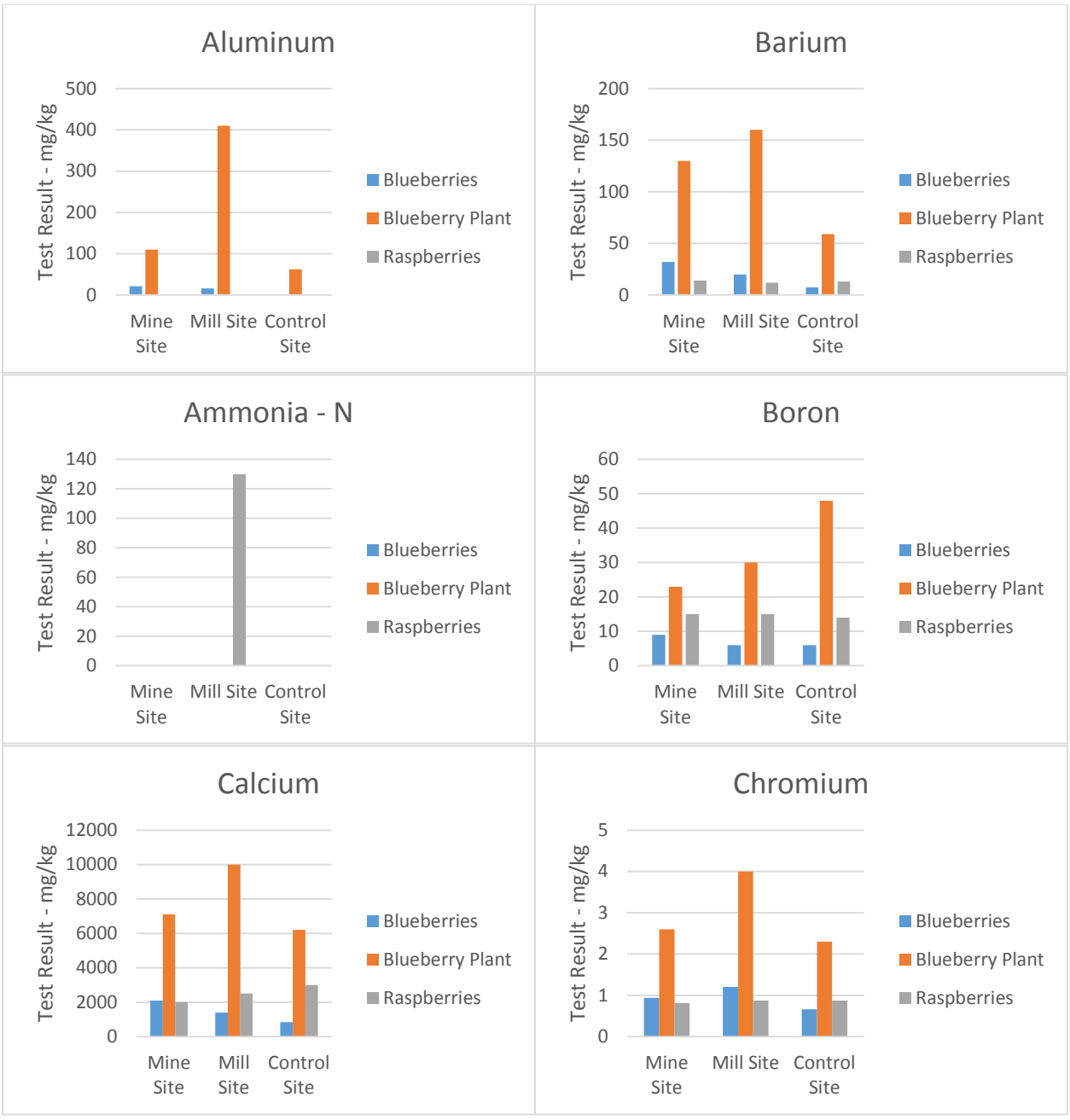

Figure 2 a-f. Bar graphs comparing test results (mg/kg) for all three sample types (blueberries, blueberry plant, and raspberries) at all sample locations (mine site, mill site, control site) for Aluminum, Barium, Ammonia, Boron, Calcium, and Chromium (respectively).

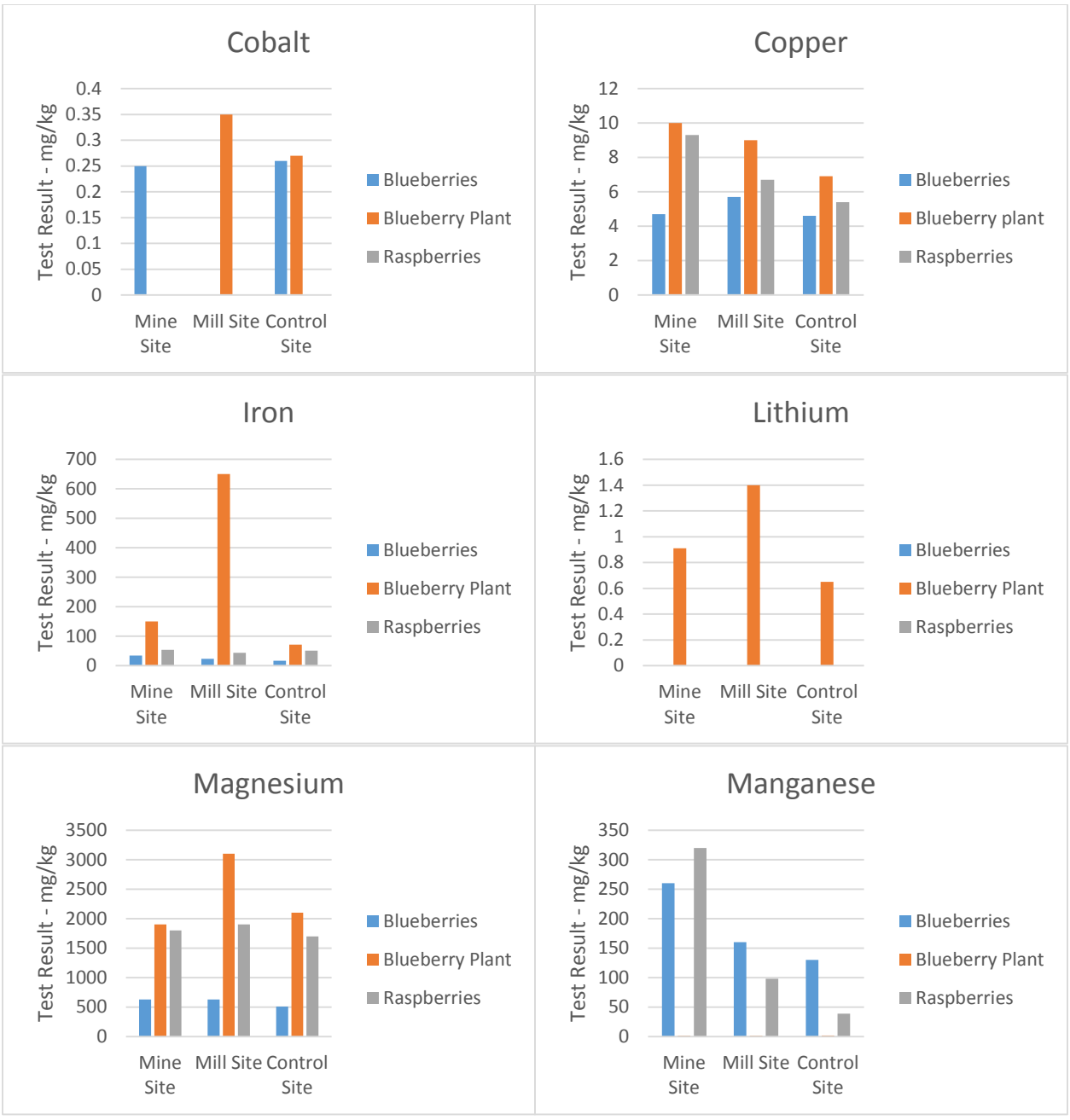

Figure 2 g-l. Bar graphs comparing test results (mg/kg) for all three sample types (blueberries, blueberry plant, and raspberries) at all sample locations (mine site, mill site, control site) for Cobalt, Copper, Iron, Lithium, Magnesium, and Manganese (respectively).

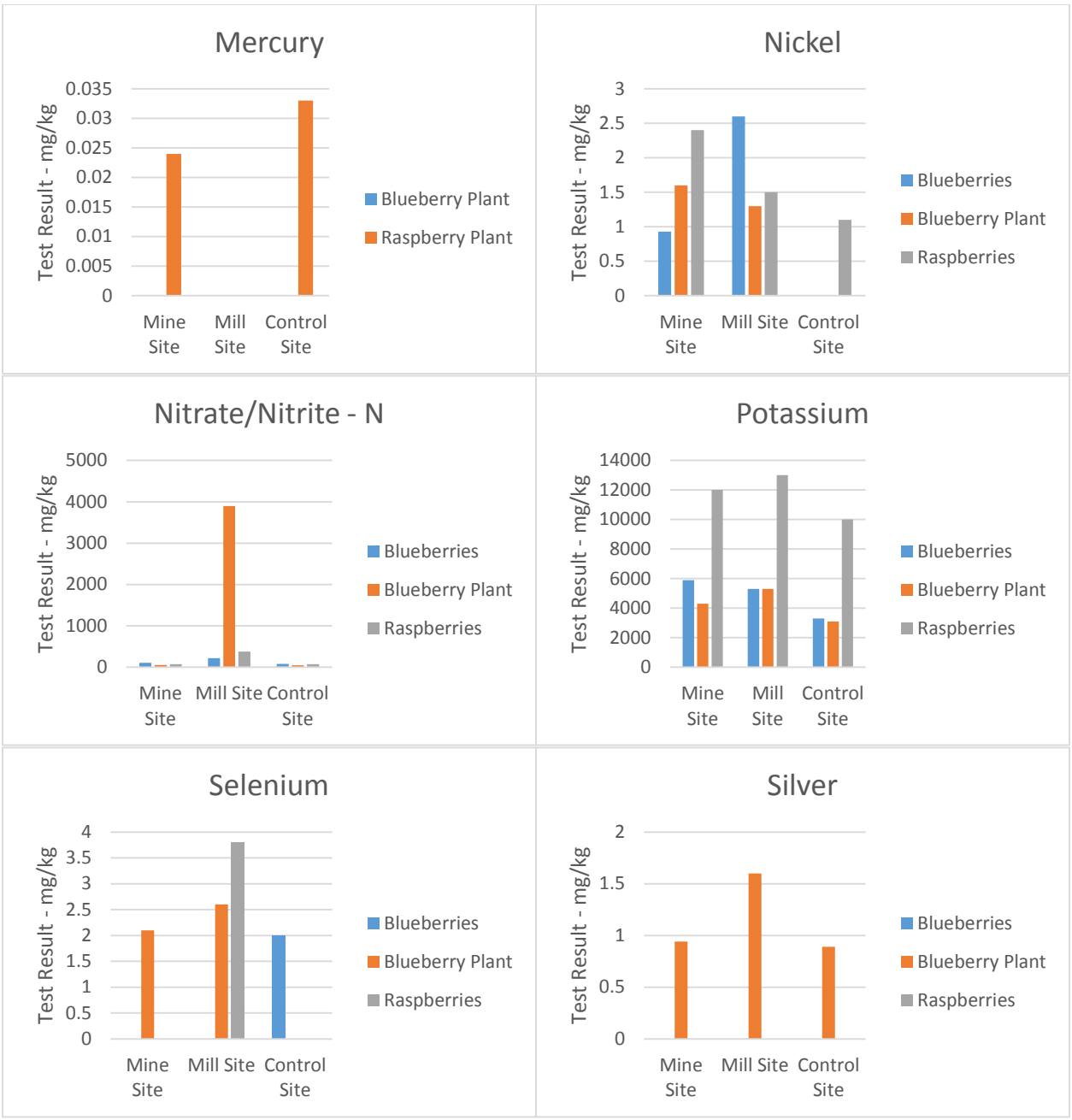

Figure 2 m-r. Bar graphs comparing test results (mg/kg) for all three sample types (blueberries, blueberry plant, and raspberries) at all sample locations (mine site, mill site, control site) for Mercury, Nickel, Nitrate/Nitrite, Potassium, Selenium, and Silver (respectively).

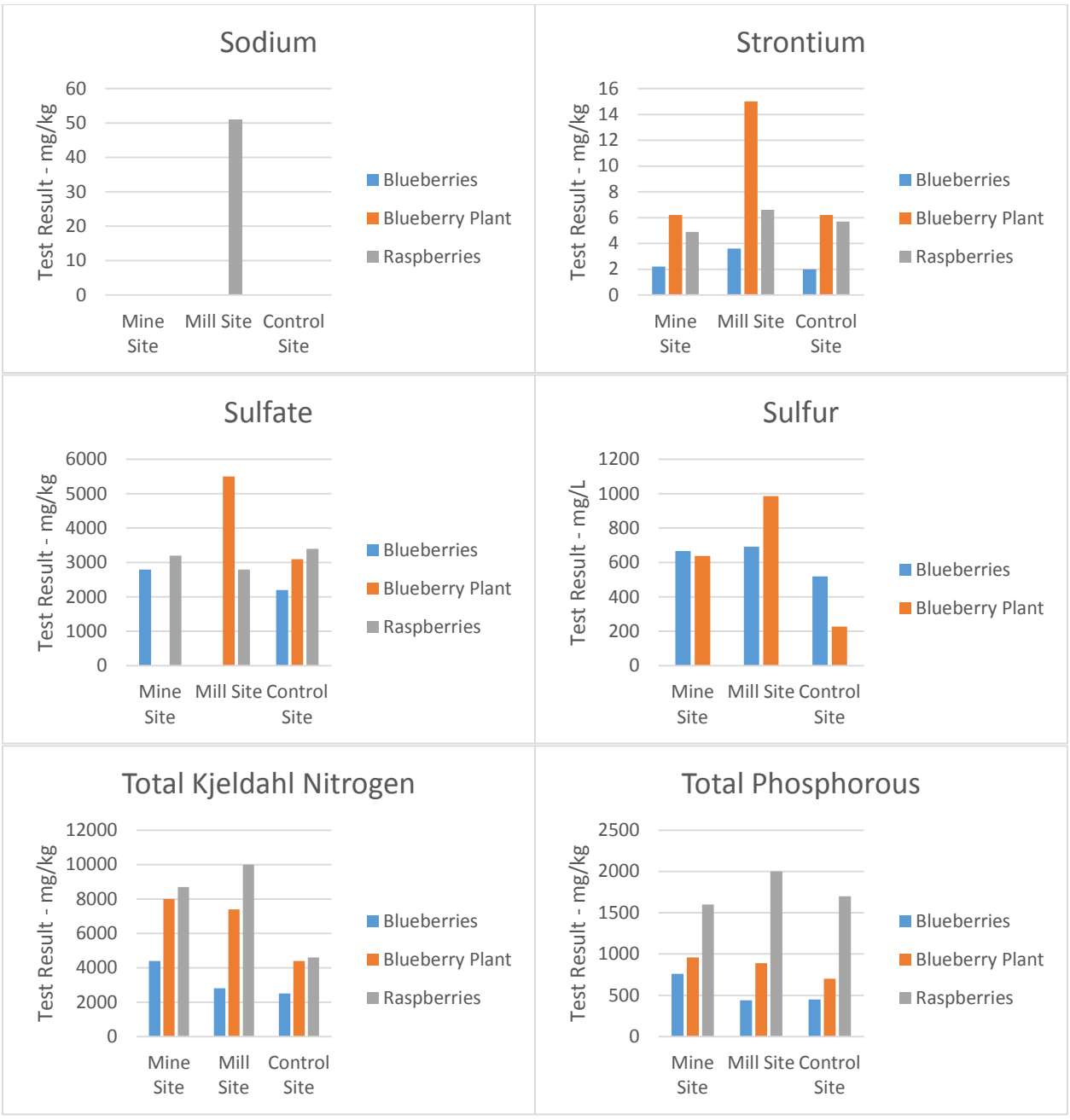

Figure 2 s-x. Bar graphs comparing test results (mg/kg) for all three sample types (blueberries, blueberry plant, and raspberries) at all sample locations (mine site, mill site, control site) for Sodium, Strontium, Sulfate, Sulfur, Total Kjeldahl Nitrogen, and Total Phosphorous.

Figure 2. y-z. Bar graphs comparing test results (mg/kg) for all three sample types (blueberries, blueberry plant, and raspberries) at all sample locations (mine site, mill site, and control site) for Vanadium and Zinc (respectively).

APPENDIX A

Acid Digestion Protocols used by White Water Associates, Inc. for Plant Tissue Analysis

8.1.5. Method 3050B: Total Recoverable Metals in Soil, Sludge, Tissue, and Solid Waste

8.1.5.1. This method is an acid digestion for the preparation of sediments, sludges, tissue and soil samples for ICP-OES and ICP-MS analysis.

8.1.5.2. In an aluminum pan, crucible, or beaker, pre-dry a representative sample overnight. For hot block method, transfer 0.5 grams of a dried sample into a pre-cleaned polyethylene digestion vessel. For this subsampling procedure, we will avoid a non-representative sample by not targeting a specific weight.

8.1.5.3. Add matrix spike analytes at this time to the sample(s) selected for spiking. Prepare a sufficient number of duplicate samples or duplicate spikes for digestion (see the quality control section of this procedure for required frequency of matrix spikes and duplicates).

8.1.5.4. Add 5 ml of 1:1 nitric acid, mix the slurry, and cover with a reflux cap. Heat the sample to 95oC+5oC and reflux for 25 minutes without boiling. Allow the sample to cool, add 2.5 ml of concentrated nitric acid, replace reflux cap and continue for 30 minutes. If brown fumes are generated, repeat this step until no brown fumes are given off. Allow the solution to evaporate to approximately 2.5 ml without boiling. Maintain a covering of solution over the bottom of the vessel at all times.

8.1.5.5. After the sample has cooled, add 1.0 ml of DI water and 1.5 ml of 30% H2O2 and heat until effervescence subsides. Cool and continue to add 30% H2O2 in 0.5 ml aliquots with warming until effervescence is minimal or sample appearance no longer changes. DO NOT ADD MORE THAN A TOTAL OF 5.0 ml of H2O2. Care must be taken upon additions of H2O2. Some samples are more reactive than others and may foam over causing sample loss.

8.1.5.6. Continue heating the acid-peroxide digestate until the volume has been reduced to approximately 2.5 ml. Maintain a covering of solution over the bottom of the digestion vessel at all times.

8.1.5.7. Add 5.0 ml concentrated HCl to the sample digest and cover with a reflux cap. Continue at 95oC+5oC for 15 minutes.

8.1.5.8. Bring volume back to 50 ml with DI water. Sample may be filtered if suspended material is present.

8.1.5.9. Prepare and digest an MB and LCS with Ottawa sand for each batch of samples digested.

10.2.4. Method 3050B: Total Recoverable Metals in Soil, Sludge, and Solid Materials

10.2.4.1. This method is an acid digestion for the preparation of sediment, sludge, soil and other solids samples for ICP-OES analysis.

10.2.4.2. Metals digested for ICP-OES by this method are: Al, As, Sb, Se, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Ag, Na, Tl, V, and Zn.

10.2.4.3. As In an aluminum pan or equivalent, pre-dry a representative sample overnight. For hot block method, transfer 0.50gm of a dried sample into a pre-cleaned polyethylene digestion vessel. For this subsampling procedure, we will avoid a non-representative sample by not targeting a specific weight.

10.2.4.4. Add matrix spike analytes at this time to the sample(s) selected for spiking. Prepare a sufficient number of duplicate samples or duplicate spikes for digestion (see the quality control section of this procedure for required frequency of matrix spikes and duplicates).

10.2.4.5. Add 5 mls of 1:1 nitric acid, mix the slurry, and cover with a reflux cap. Heat the sample to 95oC+5oC and reflux for 25 minutes without boiling. Record the start time in the digestion log book along with the starting hot block temperature. Allow the sample to cool, add 2.5mLs of concentrated nitric acid, replace reflux cap and continue for another 30 minutes. If brown fumes are generated, repeat this step until no brown fumesare given off. Allow the solution to evaporate to approximately 2.5mL without boiling. Maintain a covering of solution over the bottom of the vessel at all times.

10.2.4.6. After the sample has cooled, add 1.0mL of DI water and 1.5mLs of 30% H2O2 and heat until effervescence subsides. Cool and continue to add 30% H2O2 in 0.5-mL aliquots with warming until effervescence is minimal or sample appearance no longer changes. DO NOT ADD MORE THAN A TOTAL OF 5.0mLs of H2O2. Care must be taken upon additions of H2O2. Some samples are more reactive than others and may foam over causing sample loss.

10.2.4.7. Continue heating the acid-peroxide digestate until the volume has been reduced to approximately 2.5mLs. Maintain a covering of solution over the bottom of the digestion vessel at all times.

10.2.4.8. Add 5.0mLs concentrated HCL to the sample digest and cover with a watch reflux cap. Reflux at 95oC+5oC for 15 minutes. Record the stop time along with the ending hotblock temperature reading in the digestion logbook.

10.2.4.9. Bring volume back to 50ml with DI water. Sample may be filtered if suspended material is present.

10.2.4.10. Prepare a laboratory digested blank (MB) and LCS for matrix and batch of 20 samples digested. NOTE: To improve samples solubility and recoveries of antimony, barium, lead, and silver, refer to section 7.5 of Method 3050B, SW

APPENDIX B

Sample Methods, MDL, and MQL Values Used in Analyses

B.1 Sample method, MDL, and MQL values used for blueberry analysis

		BLUEBERRY SAMPLES					
		Mine Site Sample		Mill Site Sample		Control Site Sample	
PARAMETER	METHOD	MDL	MQL	MDL	MQL	MDL	MQL
Aluminum	200.7	9.1	36	9.3	37	8.9	36
Ammonia-N	350.1	130	260	160	320	110	220
Antimony	200.7	1.8	5.5	1.9	5.6	1.8	5.4
Arsenic	200.7	1.8	5.5	1.9	5.6	1.8	5.4
Barium	200.7	0.018	0.091	0.018	0.093	0.018	0.089
Beryllium	200.7	0.036	0.18	0.037	0.19	0.036	0.18
Boron	200.7	4	10	4	9	4	9
Cadmium	200.7	0.091	0.27	0.093	0.28	0.089	0.27
Calcium	200.7	3.6	9.1	3.7	9.3	3.6	8.9
Chromium	200.7	0.073	0.27	0.074	0.28	0.071	0.27
Cobalt	200.7	0.18	0.91	0.19	0.93	0.18	0.89
Copper	200.7	0.27	0.91	0.28	0.93	0.27	0.89
Iron	200.7	0.9	2.7	0.9	2.8	0.9	2.7
Lead	200.7	0.91	2.7	0.93	2.8	0.89	2.7
Lithium	200.7	0.6	3	0.56	2.8	0.54	2.7
Magnesium	200.7	1.8	9.1	1.9	9.3	1.8	8.9
Manganese	200.7	0.05	0.09	0.05	0.09	0.04	0.09
Molybdenum	200.7	0.3	1	0.28	0.93	0.27	0.89
Nickel	200.7	0.91	2.7	0.93	2.8	0.89	2.7
Nitrate/Nitrite-N	4500-NO3- F	72	140	78	160	60	120
Potassium	200.7	9.1	36	9.3	37	8.9	36
Selenium	200.7	1.8	9.1	1.9	9.3	1.8	8.9
Silver	200.7	0.27	1.1	0.28	1.1	0.27	1.1
Sodium	200.7	45	150	46	150	45	140
Strontium	200.7	0.02	0.06	0.02	0.06	0.02	0.05
Sulfate	4500-SO4- E	2200	4300	2400	4700	2000	4100
Sulfur	200.7	20	50.1	19.8	49.4	19.7	49.2
Thallium	200.7	1.8	5.5	1.9	5.6	1.8	5.4
Total Kjeldahl Nitrogen	351.2	45	130	54	150	47	130
Total Phosphorus	365.4	32	130	38	150	33	130
Vanadium	200.7	0.36	1.8	0.37	1.9	0.36	1.8
Zinc	200.7	0.91	2.7	0.93	2.8	0.89	2.7

B.2 Sample method, MDL, and MQL values used for blueberry plant analysis

		BLUEBERRY PLANT SAMPLES					
		Mine Site Sample		Mill Site Sample		Control Site Sample	
PARAMETER	METHOD	MDL	MQL	MDL	MQL	MDL	MQL
Aluminum	200.7	10	40	10	40	9.3	37
Ammonia-N	350.1	54	110	56	110	46	92
Antimony	200.7	2	6	2	6	1.9	5.6
Arsenic	200.7	2	6	2	6	1.9	5.6
Barium	200.7	0.02	0.1	0.02	0.1	0.018	0.093
Beryllium	200.7	0.04	0.2	0.04	0.2	0.037	0.19
Boron	200.7	4	10	4	10	4	9
Cadmium	200.7	0.1	0.3	0.1	0.3	0.093	0.28
Calcium	200.7	4	10	4	10	3.7	9.3
Chromium	200.7	0.08	0.3	0.08	0.3	0.074	0.28
Cobalt	200.7	0.2	1	0.2	1	0.19	0.93
Copper	200.7	0.3	1	0.3	1	0.28	0.93
Iron	200.7	1	3	1	3	0.9	2.8
Lead	200.7	1	3	1	3	0.93	2.8
Lithium	200.7	0.6	3	0.6	3	0.56	2.8
Magnesium	200.7	2	10	2	10	1.9	9.3
Manganese	200.7	0.5	2	0.5	2	0.46	1.9
Molybdenum	200.7	0.3	1	0.3	1	0.28	0.93
Mercury	7471B	0.02	0.059	0.023	0.068	0.034	0.1
Nickel	200.7	1	3	1	3	0.93	2.8
Nitrate/Nitrite-N	4500-NO3- F	27	54	27	54	23	47
Potassium	200.7	10	40	10	40	9.3	37
Selenium	200.7	2	10	2	10	1.9	9.3
Silver	200.7	0.3	1.2	0.3	1.2	0.28	1.1
Sodium	200.7	50	160	50	160	46	150
Strontium	200.7	0.02	0.06	0.02	0.06	0.02	0.06
Sulfate	4500-SO4- E	3200	6500	3200	6500	2800	5600
Sulfur	200.7	20	49.9	19.9	49.7	19.5	48.8
Thallium	200.7	2	6	2	6	1.9	5.6
Total Kjeldahl Nitrogen	351.2	98	280	94	270	33	96
Total Phosphorus	365.4	14	56	13	54	12	48
Uranium	6010C	9.5	167	11.3	200	17.1	301
Vanadium	200.7	0.4	2	0.4	2	0.37	1.9
Zinc	200.7	1	3	1	3	0.93	2.8

B.3 Sample method, MDL, and MQL Values used for raspberry analysis

		RASPBERRY SAMPLES					
		Mine Site Sample		Mill Site Sample		Control Site Sample	
PARAMETER	METHOD	MDL	MQL	MDL	MQL	MDL	MQL
Aluminum	200.7	9.6	38	9.8	39	10	42
Ammonia-N	350.1	130	260	130	260	140	280
Antimony	200.7	2	6	2	6	2	6
Arsenic	200.7	2	6	2	6	2	6
Barium	200.7	0.03	0.2	0.03	0.2	0.03	0.2
Beryllium	200.7	0.04	0.2	0.04	0.2	0.04	0.2
Boron	200.7	4	10	4	10	4	10
Cadmium	200.7	0.1	0.3	0.1	0.3	0.1	0.3
Calcium	200.7	3.8	9.6	3.9	9.8	4.2	10
Chromium	200.7	0.12	0.3	0.12	0.3	0.12	0.3
Cobalt	200.7	0.2	1	0.2	1	0.2	1
Copper	200.7	0.3	2.5	0.3	2.5	0.3	2.5
Iron	200.7	1	2.9	1	2.9	1	3.1
Lead	200.7	1	3	1	3	1	3
Lithium	200.7	0.58	2.9	0.59	2.9	0.62	3.1
Magnesium	200.7	1.9	9.6	2	9.8	2.1	10
Manganese	200.7	0.05	0.1	0.05	0.1	0.05	0.1
Molybdenum	200.7	0.29	0.96	0.29	0.98	0.31	1
Mercury*	7471B	0.0047	0.021	0.028	0.085	0.031	0.093
Nickel	200.7	1	3	1	3	1	3
Nitrate/Nitrite-N	4500-NO3- F	64	130	64	130	70	140
Potassium	200.7	48	190	49	200	52	210
Selenium	200.7	3	10	3	10	3	10
Silver	200.7	0.3	2.5	0.3	2.5	0.3	2.5
Sodium	200.7	48	150	49	160	52	170
Strontium	200.7	0.02	0.06	0.02	0.06	0.02	0.06
Sulfate	4500-SO4- E	1900	3800	1900	3800	2100	4200
Thallium	200.7	2	6	2	6	2	6
Total Kjeldahl Nitrogen	351.2	100	260	100	260	56	140
Total Phosphorus	365.4	70	320	70	320	77	350
Uranium*	6010C	10.6	187	13.5	239	16.8	297
Vanadium	200.7	0.6	2	0.6	2	0.6	2
Zinc	200.7	1	3	1	3	1	3

* Samples included plant tissue, not berries.

Image 1. CEMP Field Technician collecting fruit/plant specimens at a test site.