Cleaner Air Oregon Level 4 Risk Assessment Workplan

Cascade Steel Rolling Mills McMinnville, Oregon

Prepared for:

Oregon Department of Environmental Quality

August 2, 2024

BRIDGEWATER GROUP, INC.

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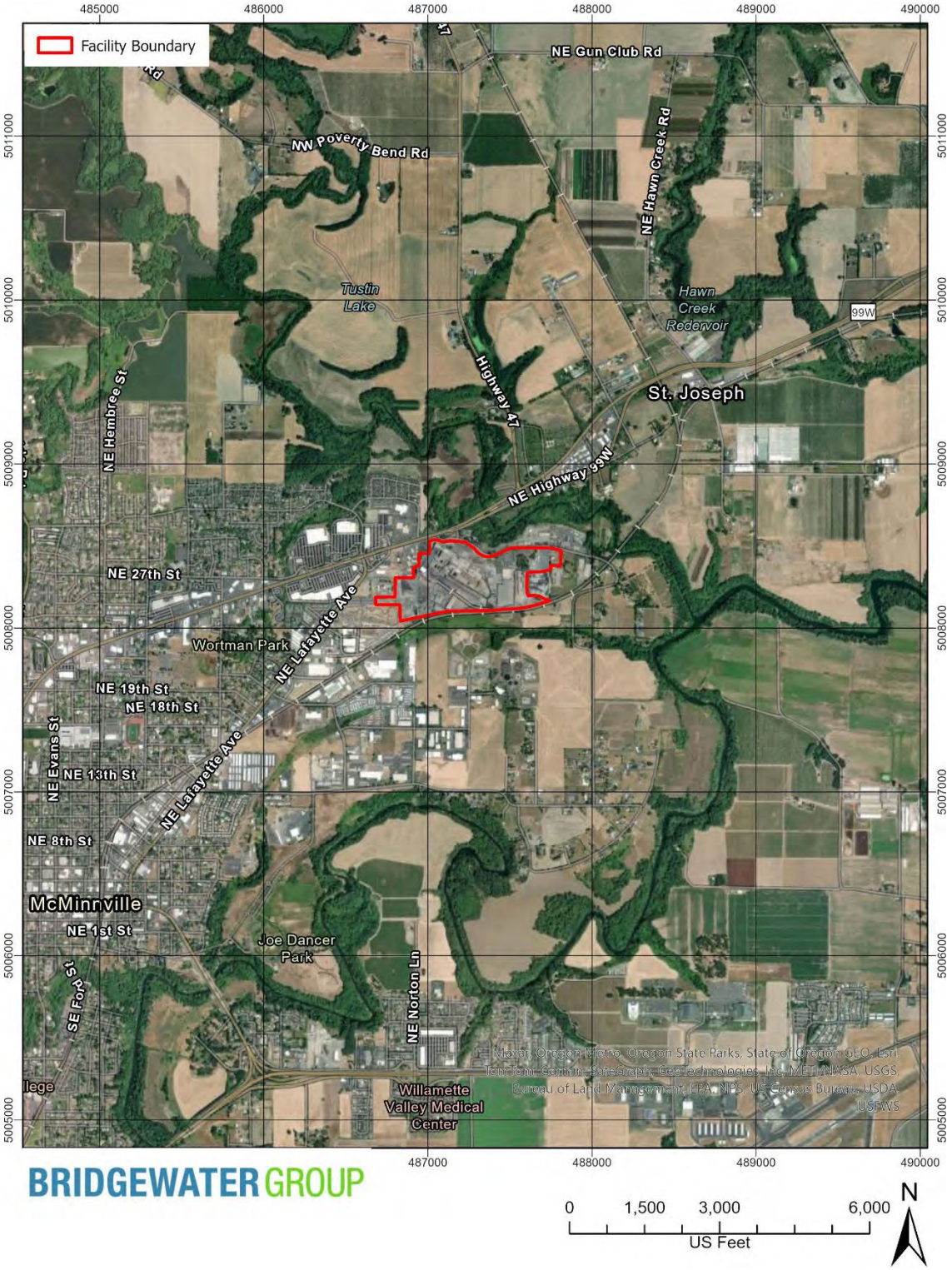
1.0 Introduction

Cascade Steel Rolling Mills, Inc. (CSRM) operates a steel manufacturing facility located at 3200 N Highway 99 in McMinnville, Oregon (source number 202528). The site is shown in Figure 1-1 and is located at a latitude of N 45° 13′ 43″ and longitude of W123° 9′ 49″, which corresponds to Universal Transverse Mercator (UTM – NAD 83) Zone 10 coordinates of 487,156 meters Easting by 5,008,356 meters Northing.

CSRM melts ferrous scrap metal to produce steel products, predominately consisting of reinforcing bar (rebar) for the construction industry, but also including flat and round merchant bar for steel fabrication and various other finished products. The steel mill was founded in 1968 and now consists of a melt shop, a rolling mill, and supporting operations. The 85-acre facility is served by truck and rail.

CSRM was called into the Cleaner Air Oregon (CAO) program on February 7, 2022. CAO regulates emissions of toxic air contaminants from facilities based on comparing a calculated risk value to certain risk action levels (RALs) defined by Oregon Department of Environmental Quality (ODEQ) regulations. The risk assessment procedure is defined under OAR-340-245-0050. CAO is a multi-step process, involving the development of an air toxics emissions inventory, dispersion modeling, and a risk assessment. Over the past several years, CSRM has conducted numerous source tests of various emission sources to better characterize their emissions and has submitted multiple versions of their emissions inventory (May 9, 2022; October 10, 2022; February 13, 2023; July 3, 2023; August 9, 2023; October 9, 2023; November 14, 2023; and most recently on March 1, 2024). On April 5, 2024, the ODEQ approved CSRM's March 1, 2024 emission inventory. A CAO modeling protocol was prepared and submitted to ODEQ on May 5, 2024. The modeling protocol outlined the methods, assumptions, and datasets that will be used to calculate the off-site air concentrations for use in the risk assessment. CSRM is planning to conduct a Level 4 risk assessment. This document is the CSRM's Level 4 Risk Assessment Workplan, which outlines the methods, assumptions, and datasets that will be used to estimate the potential cancer risks and non-cancer hazards posed by CSRM's emissions.

Figure 1-1. Site Location



2.0 Source Description

2.1 Process Description

Figure 2-1 shows the process flow diagram for CSRM. CSRM receives clean scrap via rail or truck. The scrap is unloaded and sorted into one of two storage piles areas (main and secondary). Scrap is loaded into charge buckets and transferred to the Electric Arc Furnace (EAF), which melts the scrap to produce molten steel. The molten steel is poured into a ladle from a bottom tap. Melted nonferrous scrap constituents, which are lighter than the molten metal, float to the top of the EAF vessel and are decanted off into a slag pit. The chemistry of the molten metal is fine-tuned in the ladle furnace (LF) through the addition of alloys and other compounds. After the final chemistry and temperature adjustments are made, the ladle is moved to the casting area. The molten metal is poured from the ladle into the tundish, which is a reservoir above the continuous caster molds. Molten metal funnels from the tundish into a continuous caster into a series of five molds. The solidified metal billets exiting the bottom of the mold are cut into appropriate lengths. After cooling, the scale is removed and the billets are transferred to the Rolling Mill where they are reformed into bars, smooth rods, rebar coils, wire rod and bar-length products. Figure 2-2 shows the conceptual site model for the CAO process from TEUs to exposure routes. TEU's are described in the modeling protocol.

Figure 2-1. Process Flow Diagram

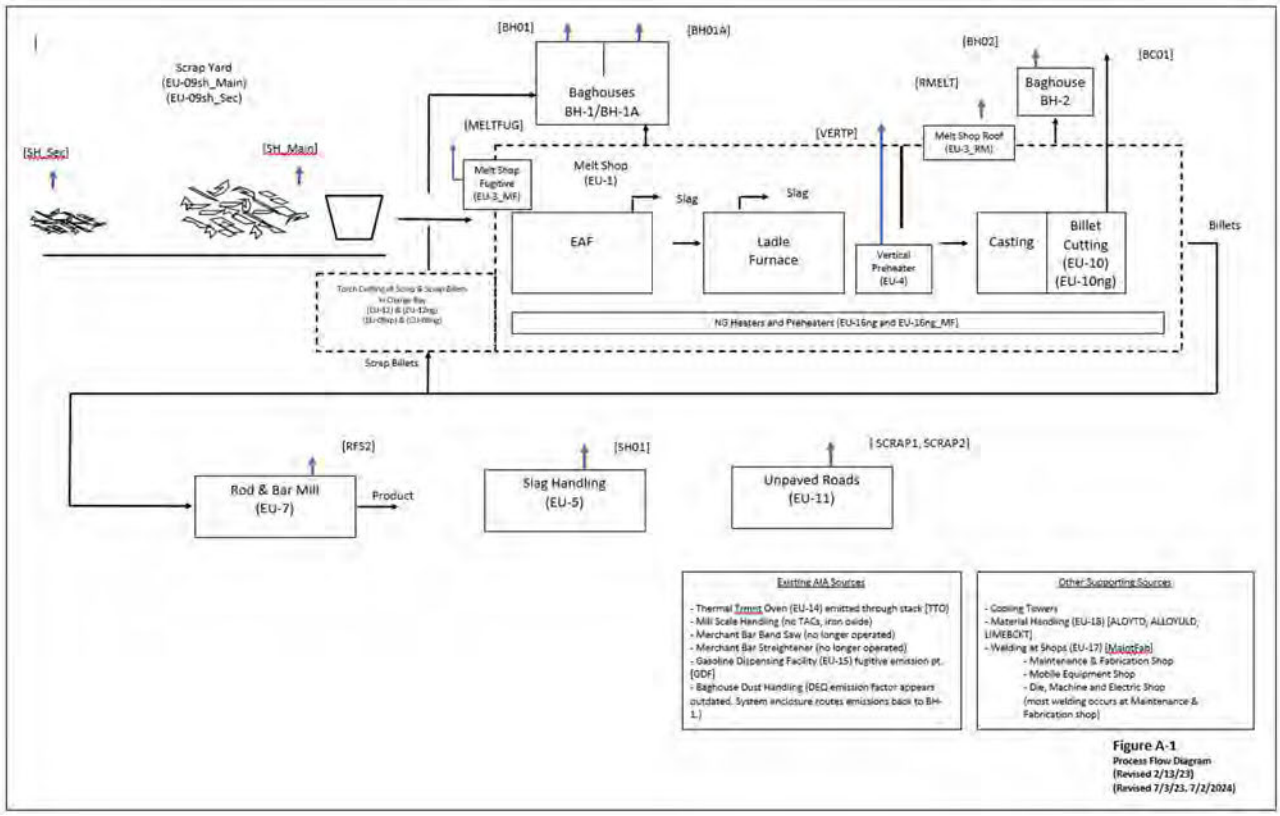


Figure 2-2. CAO Conceptual Site Model

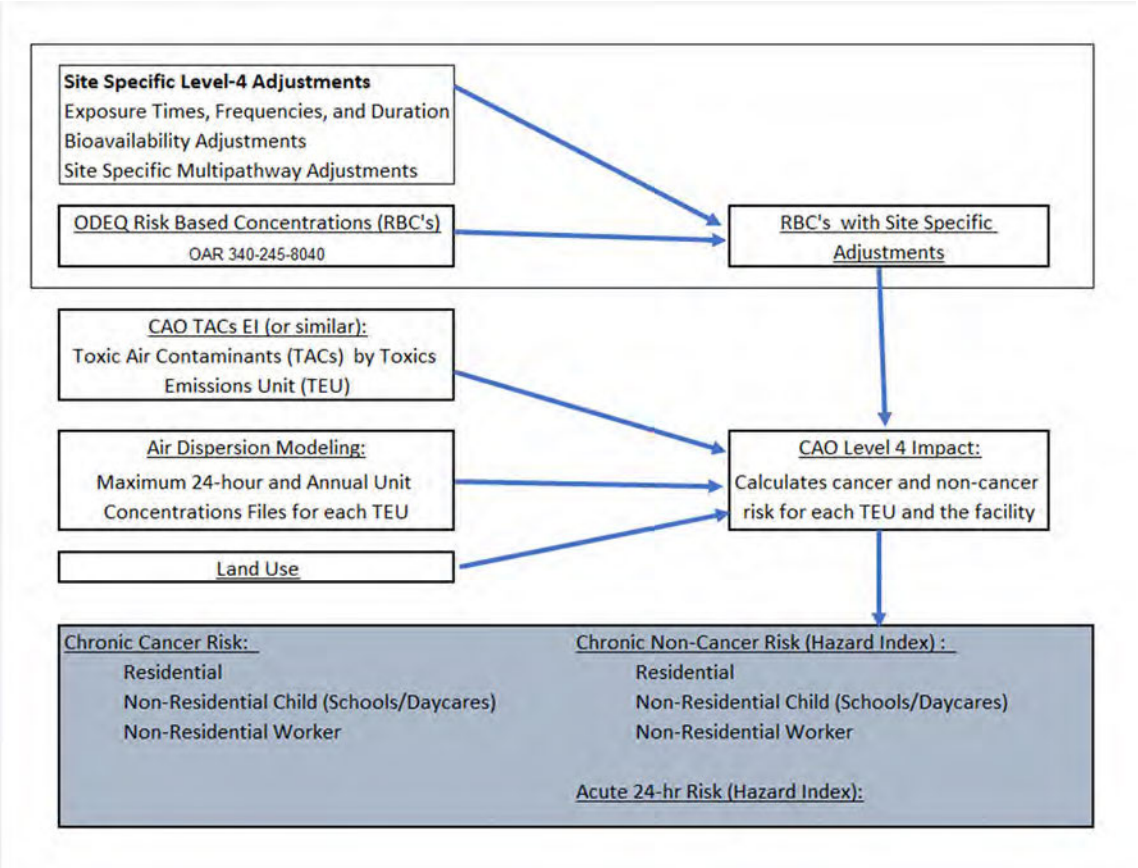
EI TEU	Input Material	Process	Control Device	Emission Point	Model ID	Pollutants	Pathway
Scrap Handling and Preparation							
EU-09ng: Natural Gas Combustion for Cutting							
	Natural Gas	NG Combustion	Baghouse	EU-09ng-SC_BH01	BH01	NG Comb.	Inhalation
				EU-09ng-SC_BH01A	BH01A		
				EU-09ng-SC_MELTFUG	MELTFUG		
EU-09sc Scrap Cutting							
	Scrap	Cutting	Baghouse	EU-09sp-SC_BH01	BH01	EL/MP Metals	
				EU-09sp-SC_BH01A	BH01A		
				EU-09sp-SC_MELTFUG	MELTFUG		
EU-09sh Scrap Handling							
	Scrap	Moving to and from piles	Water Spray	EU-09sh_Main (daily)	SH_MAINV	EL/MP Metals	Inhalation, multipathway
				EU-09sh_Main (annual)	SH_MAINA		
				EU-09sh_Sec (daily)	SH_SECV		
				EU-09sh_Sec (annual)	SH_SECA		
EU-18 Material Handling							
	SiMn	Truck dump of SiMn to bunker	Water Spray	EU-18_ATDSiMn (daily)	ALLTD1V	EL/MP Metals	
				EU-18_ATDSiMn (annual)	ALLTD1C		
	FeMn	Truck dump of FeMn to bunker	Water Spray	EU-18_ATDFeMn (daily)	ALLTD2V	EL/MP Metals	
				EU-18_ATDFeMn (anual)	ALLTD2C		
	SiMn	Transfer SiMn to feeder		EU-18_AULDSiMn	ALLULD	EL/MP Metals	
	FeMn	Transfer FeMn to feeder		EU-18_AULDFeMn	ALLULD	EL/MP Metals	
	Lime	Lime addition to charge bucket		EU-18_LIMEBCKT	LBCKT	Silica	Inhalation
Meltshop Operations, Melting and Pouring							
EU-01 Meltshop							
	Scrap	EAF Melting and pouring	Baghouse	EU-1_BH01	BH01	Gases	Inhalation
	SiMn			EU-1_BH01A	BH01A	HF, Fluorides	
	FeMn			EU-1_BH02	BH02	Dioxins/Furans	
	Lime					PCB	Inhalation, multipathway
EU-03 building fugitives				EU-3_MF	MELTFUG	EL/MP Metals	
EU-03 roof monitor				EU-3_RM	RMELT	EL/MP Metals	
EU-16ng	Natural Gas	Meltshop NG combustion	Baghouse	EU-16NG_BH01	BH01	NG Comb.	Inhalation
				EU-16NG_BH01A	BH01A		
				EU-16NG_RMELT	RMELT		
				EU-16NG_MF	MELTFUG		
EU-4	Natural Gas	Preheater NG Combustion		EU-4	VERTP		
Slag Handling							
EU-5	Slag	Slag Handling	Spray	EU-5 (daily)	SHF01V	EL/MP Metals	Inhalation, multipathway
				EU-5 (annual)	SHF01A	Dioxins/Furans	
						PCB	
						Fluorides	Inhalation
Billet Casting and Cutting							
EU-10ng	Natural Gas	Cutting		EU-10ng	BCUT	NG Comb.	Inhalation
EU-10	Molten Steel	Casting and Cutting		EU-10	BCUT	EL/MP Metals	
EU-12	Scrap Billets	Cutting	Baghouse	EU-12_BH01	BH01	EL/MP Metals	Inhalation, multipathway
				EU-12_BH01A	BH01A		
				EU-12_MELTFUG	MELTFUG		
EU-12ng	Natural Gas	NG Combustion	Baghouse	EU-12ng_BH01	BH01	NG Comb.	Inhalation
				EU-12ng_BH01A	BH01A		
				EU-12ng_MELTFUG	MELTFUG		
Rolling Mill Operations							
EU-7	Natural Gas	Reheat furnace		EU-7	RFS2	NG Comb.	Inhalation
EU-14	Natural Gas	Heat treatment Oven		EU-14	TTO	NG Comb.	Inhalation
Other Processes							
EU-15	Gasoline	Gas Dispensing		EU-15	GDF	Hydrocarbons	Inhalation
EU-11	Dust	Unpaved roads		EU-11	SCRAP1	EL/MP Metals	Inhalation, multipathway
				EU-11	SCRAP2		
EU-17	Weld wire	Welding		EU-17	MAINTFAB	Metals, Silica	
						Silica, Fluorides	Inhalation

3.0 Level 4 Risk Assessment

3.1 Methodology

Figure 3-1 shows the Level 4 Risk Assessment process. Using the CAO toxic air pollutant EI (e.g., AQ520 CAO spreadsheet), the 24-hr and annual average unit concentration files from AERMOD runs, the Risk Based Concentrations, and the land use designations at each receptor, the chronic cancer, chronic non-cancer and acute hazard index risk will be estimated at every receptor. A Level 4 risk assessment is identical to a Level 3 risk assessment except that a Level 4 Risk Assessment allows for site-specific adjustments to provide a more representative risk estimate. Under CAO, there are three types of Level 4 adjustments available: (1) changes in exposure time, frequencies, and durations, (2) the inclusion of relative bioavailability, and (3) site specific adjustments used in determining the multipathway factors (e.g. site specific deposition rates and uptakes rates).

Figure 3-1. Level 4 Refined Risk Assessment



3.2 Level 4 Adjustments

This Level 4 Risk Assessment will include an evaluation of the specific multipathway adjustment factors (MFAFs) detailed below. Table 3-1 shows the current multipathway adjustment Factors (MPAFs) used in CAO[1] for three TACs emitted by CSRM. These adjustment factors are from the South Coast Air Quality Management District (SCAQMD), Permit Application Package "M", March 2016, Table 8-1. South Coast Air Quality Management District, Facility Prioritization Procedures for AB 2588 Program, Nov. 2016, Table 3. For this analysis, the MPAFs of these three TACs will be evaluated.

Table 3-1. SCAQMD Multipathway Adjustment Factors

Toxic Air Contaminant	Cancer Resident MPA	Cancer Non-Resident MPA	Non-Cancer Resident MPA	Non-Cancer Non-Resident MPA
Arsenic	9.7	4.5	88	28
Chromium VI	1.6	1	2.4	1
Fluorides	--	--	5.7	2.9

SCAQMD generated these factors using the HARP2 Risk Assessment Standalone Tool (HARP2-RAST) based on exposures from inhalation, dermal contact, soil ingestion, consumption of home grown foods and consumption of breast milk and a deposition rate of 0.02 m/s. HARP2-RAST assumes 100% bioavailability for metals and accumulation of metals in the soils without losses.

In addition to the evaluation of MPAFs, CSRM asked ToxStrategies, a California-based scientific consulting firm specializing in toxicology and risk assessment, to evaluate the oral bioavailability of arsenic, and inhalation bioaccessibility of manganese, and ingestion rates for the homegrown produce and soil ingestion exposure pathways for the CSRM Level 4 risk assessment. ToxStrategies proposed site-specific alternatives to default assumptions for these factors are discussed below and described in more detail in their technical memorandum that ToxStrategies produced, which is provided in Appendix A.

Table 3-2 summarizes all the Level 4 adjustments to be incorporated into the CSRM risk assessment. In addition, the ODEQ's updated provisional acute TRV/RBC for manganese[2] will be incorporated (1.3 µg/m^3). These adjustments are described in more detail below.

[1] Cleaner Air Oregon Spreadsheet for Calculation of Toxicity Reference Values and Risk-Based Concentration, July 2020.

[2] DEQ Toxicity Reference Value (TRV) Proposal for 24-hour Acute Inhalation Exposure to Manganese, Memorandum to Ali Mirzakhalili, DEQ Air Quality Administrator from the Clean Air Oregon Toxicology Team, July 26, 2024

Table 3-2. Summary of Level 4 Adjustments

TEU ID	TEU Description	Level 4 Adjustment	Sampling*
EU-3_RM	Melt Shop Roof Monitor	Arsenic oral relative bioavailability	Yes – Roof Monitor D/R-01-042823
EU-3_MF	Melt Shop Fugitives	Arsenic oral relative bioavailability	Yes – Roof Monitor D/R-01-042823
EU-9sh_Main	Main Scrap Handling	Arsenic oral relative bioavailability	Yes – Truck Sweep Off-01-042823
EU-9sh_Sec	Secondary Scrap Handling	Arsenic oral relative bioavailability	Yes – Truck Sweep Off-01-042823
EU-5	Slag Handling	Mn inhalation bioaccessibility	Yes – EAF/LMF Slag-062123
EU-10	Caster Billet Cutting	Mn inhalation bioaccessibility	Yes – Billet Cut Vent D/R-A02-042823
EU-3_RM	Melt Shop Roof Monitor	Mn inhalation bioaccessibility	Yes – Roof Monitor D/R-02-042823
EU-3_MF	Melt Shop Fugitives	Mn inhalation bioaccessibility	Yes – Roof Monitor D/R-02-042823
EU-9sh_Main	Main Scrap Handling	Mn inhalation bioaccessibility	Yes – Truck Sweep Off-02-042823
EU-9sh_Sec	Secondary Scrap Handling	Mn inhalation bioaccessibility	Yes – Truck Sweep Off-02-042823
EU-18_ATDSiMn	SiMn Alloy Truck Dump	Mn inhalation bioaccessibility	Yes – SiMn Stockpile-062123
EU-18_AULDSiMn	SiMn Alloy Unload to Feeder	Mn inhalation bioaccessibility	Yes – SiMn Stockpile-062123
	All As, Cr6+, F- TEUs	Soil Ingestion Rates	N/A
	All As, Cr6+, F- TEUs	Produce Ingestion Rates	N/A

*See Appendix A, Tox Strategies' Technical Memorandum (including Attachment B) for more information regarding the sampling effort.

3.2.1 Intake rates

Soil Ingestion Rates (SIR)

The SCAQMD MPAF for soil ingestion uses the 95th percentile value. Use of a 95th percentile incidental soil ingestion rate in the MPAF, along with inhalation, dermal contact, and produce ingestion exposure pathways, creates an unrealistic estimate of upper-bound cumulative exposure by compounding upper-end exposure across multiple pathways.

In a previous Level 4 analysis for Owens-Brockway[3] and in the Oregon Health Authority (OHA) public health assessment of Bullseye Glass[4], ODEQ and OHA used different values for the soil ingestion rates, which result in an age-weighted soil ingestion rate that, with the exception of the 0 to 2 year old, fall between the OEHHA 95th percentile and the mean as shown in Table 3-3.

The OHA soil ingestion rate values used in those prior assessments are from Table 1 of Agency for Toxic Substances and Disease Registry's (ATSDR's) Exposure Dose Guidance for Soil and Sediment Ingestion, V2 (Oct. 26, 2016). These values represent the upper percentile (or the high end) of the exposure distribution. Table 3-4 shows the original ATSDR soil ingestion and body weights values and how they were averaged to determine the OHA age category values. CSRM proposes to use the age-category based values in their Level 4 risk assessment, for which more detailed calculations are provided in Table 3 in Appendix A.

[3] Oregon Department of Environmental Quality. 2022. Final Review of Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, OR.
[4] Oregon Health Authority, Public Health Division. 2023. Public Health Assessment Final Release. Bullseye Glass Co. (manufacturing site), 3722 SE 21st Avenue, Portland, OR 97202. Table I-3.

Table 3-3. HARP2-RAST Soil Ingestion Values and Proposed Values **(mg/kg-day)**

Age	Years	HARP2-RAST mean	HARP2-RAST 95th	Proposed OHA ATSDR 95th
0<2	2	20	40	15
2<16	14	3	10	6.8
16to 70	54	0.6	3	1.4
Age weighted SIR value	70	1.63	5.46	2.87

Table 3-4. ASTDR Age Specific IR and Body Weights

Group	Years	Ingestion Rate (mg/day)	Age Specific Body weight (kg)	Body weight Soil IR (mg/kg-day)	Bin Weighted SIR (mg/kg-day)
1	1	100	7.8	12.82	15
1-2	1	200	11.4	17.54	
2-6	4	200	17.4	11.49	6.8
6-11	5	200	31.8	6.29	
11-16	5	200	56.8	3.52	
16-21	5	200	71.6	2.79	1.4
21-70	49	100	80	1.25	

Home Grown Produce

The MPAF for TAC metals includes a contribution from potential ingestion of homegrown produce grown in soil affected by deposition from emissions. Similar to the discussion of soil ingestion rates, use of upper bound estimates of plant ingestion rates overestimates exposure when there are multiple exposure pathways. For this analysis, we propose the use of the 75th percentile for homegrown produce ingestion rates developed by OEHHA in their HARP2-RAST risk assessment documentation, which were determined as applicable by ODEQ/OHA for the previously identified Owens-Brockway Level 4 Risk Assessment. These values are shown in Table 3-5.

Table 3-5. Proposed 75th percentile homegrown produce ingestion rates (g/kg-day)

	0<2	2<16	16-70
Exposed	15.4	7.3	2.4
Leafy	5.3	2.3	1.5
Protected	7.5	4.9	2.1
Root	8.2	3.9	2.1

3.2.2. Arsenic Oral Bioavailability

The HARP2-RAST model assumes 100% arsenic bioavailability. In 2012, EPA compiled and reviewed data on the relative bioavailability (RBA) of arsenic in soils[5]. Based on that review, EPA set the default RBA for arsenic in soil at 60%. DEQ and OHA previously determined that use of EPA's default RBA was appropriate. Thus, for this Level 4 risk assessment, a maximum 60% default arsenic bioavailability is assumed except for those sources where site-specific evaluations were conducted.

Several emission sources of arsenic from CSRM were sampled to determine source-specific arsenic bioavailability values. These emission sources are the Melt Shop Roof Monitor and Melt Shop Fugitives and Main and Secondary Scrap Handling. For the Melt Shop Roof Monitor and Melt Shop Fugitives emission sources, material from around the roof monitor was collected to obtain a representative sample of these two sources. For the Main and Secondary Scrap Handling emission sources, representative samples of truck sweep-off material were collected. As described in the ODEQ approved CAO emissions inventory, the truck sweep-off material is the left-over residual left in the scrap trucks after the scrap material has been unloaded from the truck. Truck sweep-off material is representative of fine material that would be associated with emissions from scrap handling operations. For these sources, grab samples were taken around the source and then sieved to get the particle size fraction that was less than 150 microns in size following EPA Method 1340. For each source, a duplicate sample was taken.

Following EPA Method 1340[6] and EPA's *Release of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil* (SOP), in vitro bioaccessibility (IVBA) measurement were made for these two sources. From the IVBA measurements, the relative bioavailability (RBA) was calculated using the regression equation developed by USEPA specifically for arsenic. Details of the sampling and analysis method are described in Appendix A. Table 3-6 presents the values. Based on these data, we propose to use an oral RBA for arsenic of 47% for the Melt Shop Roof Monitor and Melt Shop Fugitives emission sources and 11% for the Scrap Handling (Main and Secondary) source emissions.

Table 3-6. Oral Arsenic IVBA and RBA values

Sample Description	IVBA	RBA	Applies to TEU
Roof Monitor	56%	47%	EU-3_RM
Roof Monitor – Dup.	51%	43%	& EU-3_MF
Truck Sweep-off Area	9.9%	11%	EU-9sh_Main
Truck Sweep-off Area – Dup.	9.5%	11%	& EU-9sh_Sec

[5] United States Environmental Protection Agency (U.S. EPA). 2012. Recommendations for Default Value for Relative Bioavailability of Arsenic in Soil. December. Compilation and Review of Data on Relative Bioavailability of Arsenic in Soil (epa.gov)

[6] U.S. EPA. 2017. Method 1340. *In Vitro* Bioaccessibility Assay for Lead in Soil. Revision 1. SW-846 Update VI. February. corrected July 6.
https://www.epa.gov/sites/default/files/2017-03/documents/method_1340_update_vi_final_3-22-17.pdf

3.2.3. Updated Acute Mn TRV value

Recently, ODEQ and OHA conducted a review of the Manganese (Mn) Acute Toxicity Reference Value (TRV) based on a petition to raise the TRV from 0.3 ug/m^3 to 5 ug/m^3. The process included convening a meeting with the Air Toxics Science Advisory Committee (ATSAC), which is a scientific body that ODEQ and OHA consult with on technical matters related to reviewing toxicological information for the purposes of updating our TRVs. Based on that review, ODEQ is proposing to change the acute Mn TRV to 1.3 ug/m^3 (rounded up from 1.25 ug/m^3).[7] Cascade will be using this provisional acute TRV value in its risk assessment.

3.2.4. Manganese Inhalation Biaccessibility

In reviewing the work on the Manganese (Mn) Acute Toxicity Reference Value (TRV), ToxStrategies identified that the manganese provisionally-approved acute TRV is based on freely soluble manganese sulfate. However, manganese emissions associated with steel production are expected to exist as less soluble oxides and/or bound in a mineral matrix (e.g., slag). These forms are known to be of lower inhalation bioavailability. Thus, similar to oral RBA, the inclusion of RBA for inhalation Mn exposures is also applicable in Level 4 risk assessments.

As discussed in Appendix A, no measures of inhalation RBA for manganese compounds were identified in publicly available literature; therefore, CSRM engaged ToxStrategies to measure the inhalation RBA for manganese from various facility emission sources. ToxStrategies completed measurements of the site-specific solubility of manganese from CSRM in lung biological fluids. Those measurements demonstrate that the manganese emissions from CSRM sources are less soluble than freely soluble manganese sulfate. Accordingly, those site-specific measures of bioaccessibility will be used as conservative estimate of RBA for the Level 4 risk assessment. The inhalation bioaccessibility values are provided and discussed further in Appendix A.

To simulate bioaccessibility by inhalation, solubility in two fluids that are relevant to dissolution in the lungs—interstitial fluid and lysosomal fluid—are assessed (Henderson et al. 2014).[8] The IVBA test and solutions (interstitial and lysosomal) simulate conditions in the lung that are relevant to the dissolution of metal ions and absorption of metals and systemic uptake. Additional detail for inhalation bioaccessibility testing is provided in Appendix A.

Sources sampled were the EAF/LMF slag, billet cutting vent, roof monitor, truck sweep off area, and silicon manganese (SiMn) stockpile. Details of the sampling approach are provided in Appendix A (Attachment B). Table 3-6 shows the inhalation bioavailability values for each

[7] ODEQ 2024. Memorandum: DEQ Toxicity Reference Value (TRV) Proposal for 24-hour Acute Inhalation Exposure to Manganese. July 26.

[8] Henderson, R.G., et al. 2014. Inter-laboratory validation of bioaccessibility testing for metals. Reg Tox and Pharm. 70: 170-181.

sample and the applicable TEU. CSRM proposes to use the lysosomal fluid IVBA values, which were more conservative (higher), as a measure of RBA.

Table 3-7. Source Specific Mn Inhalation IVBA

	Inhalation IVBA		Applies to TEU
Sample	**Lysosomal**	**Interstitial**	
EAF/LMF Slag	83%	6.0%	EU-5
Billet Cutting Vent	10%	0.6%	EU-10
Roof Monitor	54%	1.8%	EU-3_RM & EU-3_MF
Truck Sweep-off Area	61%	5.9%	EU-9sh_Main & EU-9sh_Sec
SiMn Stockpile	44%	0.6%	EU-18_ATDSiMn & EU-18_AULDSiMn

3.2.5. Application of the Level 4 adjustments

For all sources, the updated acute Mn TRV will be applied.

For the EAF/LMF slag, billet cutting vent, roof monitor, truck sweep off area, and silicon manganese (SiMn) stockpile sample concentrations were adjusted by the TEU specific Mn inhalation IVBA across all risk exposure classes. This approach was taken for each of the sources because Mn is not a multi-pathway chemical; i.e., inhalation exposures are what is evaluated for Mn.

For applying the soil ingestion and home grown produce intake rates, along with the arsenic oral RBA, a spreadsheet was prepared to show the original and revised MPAF values. In the sheet, the concentration was set to the chronic REL so that the inhalation HQ is 1. The sheet then calculates the doses for the various pathways (inhalation, dermal, soil ingestion, and food consumption). Two calculation sheets were included for each pollutant, one with the original MPAF calculation, and the other with the modified MPAF values. The revised MPAFs resulting from this Level 4 analysis are shown in Table 3-8.

Table 3-8. Revised Multipathway Adjustment Factors Considering Oral Bioavailability and Adjusted Produce and Soil Ingestion Rates

Toxic Air Contaminant	Cancer Resident MPA	Cancer Non-Resident MPA	Non-Cancer Resident MPA	Non-Cancer Non-Resident MPA
Arsenic (default)	9.7	4.5	88	28
Oral RBA 60%	4.58	2.67	39.8	19.86
Oral RBA 47%	4.25	2.49	37.2	17.09
Oral RBA 11%	3.32	1.98	29.9	14.92
Chromium VI (default)	1.6	1	2.4	1
Adjusted	1.38	1	1.37	1
Fluorides (default)			5.7	2.9
Adjusted			3.5	2.9

Application of the Level 4 adjustments to the RBCs is represented as:

$$RBC_{mod} = RBC_{org}\left[MPAF_{org}/MPAF_{mod}\right]$$

Table 3-9 shows the RBCs with adjustments applied. As part of the uncertainty analysis, CSRM will show how final risk estimates are impacted by the Level 4-adjusted value compared to the CAO default value based on the contribution of the variables described herein.

Table 3-9. RBCs used in the Level 4 Analysis

		Chronic Cancer Risk			Chronic Non-Cancer Risk			
CAS	**Compound**	**Res.**	**Child**	**Worker**	**Res.**	**Child**	**Worker**	**Acute**
75-07-0	Acetaldehyde	0.45	12	5.5	140	620	620	470
107-02-8	Acrolein	--	--	--	0.35	1.5	1.5	6.9
7429-90-5	Aluminum and compounds	--	--	--	5	22	22	--
7664-41-7	Ammonia	--	--	--	500	2200	2200	1200
7440-36-0	Antimony and compounds	--	--	--	0.3	1.3	1.3	1
7440-38-2*	Arsenic and compounds (60%)	0.000051	0.0022	0.0010	0.00038	0.0034	0.0034	0.2
	Arsenic and compounds (47%)	0.000055	0.0022	0.0010	0.00040	0.0034	0.0034	0.2
	Arsenic and compounds (11%)	0.000070	0.0022	0.0010	0.00050	0.0034	0.0034	0.2
71-43-2	Benzene	0.13	3.3	1.5	3	13	13	29
7440-41-7	Beryllium and compounds	0.00042	0.011	0.005	0.007	0.031	0.031	0.02
74-83-9	Bromomethane (Methyl bromide)	--	--	--	5	22	22	3900
7440-43-9	Cadmium and compounds	0.00056	0.014	0.0067	0.005	0.037	0.037	0.03
108-90-7	Chlorobenzene	--	--	--	50	220	220	--
74-87-3	Chloromethane (Methyl chloride)	--	--	--	90	400	400	1000
18540-29-9*	Chromium VI, chromate and dichromate particulate	0.000036	0.00052	0.001	0.15	0.88	0.88	0.3
7440-48-4	Cobalt and compounds	--	--	--	0.1	0.44	0.44	--
7440-50-8	Copper and compounds	--	--	--	--	--	--	100
110-82-7	Cyclohexane	--	--	--	6000	26000	26000	--
75-09-2	Dichloromethane (Methylene chloride)	59	620	1200	600	2600	2600	2100
100-41-4	Ethyl benzene	0.4	10	4.8	260	1100	1100	22000
C239*	Fluorides	--	--	--	3.6	20	20	240
50-00-0	Formaldehyde	0.17	4.3	2	9	40	40	49
118-74-1	Hexachlorobenzene	0.002	0.051	0.024	--	--	--	--
110-54-3	Hexane	--	--	--	700	3100	3100	--
7664-39-3	Hydrogen fluoride	--	--	--	2.1	19	19	16
7783-06-4	Hydrogen sulfide	--	--	--	2	8.8	8.8	98
98-82-8	Isopropylbenzene (Cumene)	--	--	--	400	1800	1800	--
7439-92-1	Lead and compounds	--	--	--	0.15	0.66	0.66	0.15
7439-96-5	Manganese and compounds	--	--	--	0.09	0.4	0.4	1.3
7439-97-6	Mercury and compounds	--	--	--	0.077	0.63	0.63	0.6
91-20-3	Naphthalene	0.029	0.76	0.35	3.7	16	16	200
C365	Nickel compounds, insoluble	0.0038	0.1	0.046	0.014	0.062	0.062	0.2
C447	Polybrominated diphenyl ethers (PBDEs)	--	--	--	--	--	--	6
1336-36-3	Polychlorinated biphenyls (PCBs)	0.00053	0.02	0.0092	--	--	--	--
C645	Polychlorinated biphenyls (PCBs) TEQ	0.000000001	0.00000009	0.000000042	0.00000013	0.000026	0.000026	--

CAS	Compound	Chronic Cancer Risk			Chronic Non-Cancer Risk			
		Res.	Child	Worker	Res.	Child	Worker	Acute
C646	Polychlorinated dibenzo-p-dioxins (PCDDs) & dibenzofurans (PCDFs) TEQ	0.000000001	0.00000009	0.000000042	0.00000013	0.000026	0.000026	--
C401	Polycyclic aromatic hydrocarbons (PAHs)	0.000043	0.0016	0.003	--	--	--	--
56-55-3	Benz[a]anthracene	0.00021	0.0078	0.015	--	--	--	--
50-32-8	Benzo[a]pyrene	0.000043	0.0016	0.003	0.002	0.0088	0.0088	0.002
205-99-2	Benzo[b]fluoranthene	0.000053	0.002	0.0038	--	--	--	--
191-24-2	Benzo[g,h,i]perylene	0.0047	0.17	0.34	--	--	--	--
207-08-9	Benzo[k]fluoranthene	0.0014	0.052	0.1	--	--	--	--
218-01-9	Chrysene	0.00043	0.016	0.03	--	--	--	--
53-70-3	Dibenz[a,h]anthracene	0.0000043	0.00016	0.0003	--	--	--	--
206-44-0	Fluoranthene	0.00053	0.02	0.038	--	--	--	--
193-39-5	Indeno[1,2,3-cd]pyrene	0.00061	0.022	0.043	--	--	--	--
7782-49-2	Selenium and compounds	--	--	--	--	--	--	2
7631-86-9	Silica, crystalline (respirable)	--	--	--	3	13	13	--
100-42-5	Styrene	--	--	--	1000	4400	4400	21000
79-34-5	1,1,2,2-Tetrachloroethane	0.017	0.45	0.21	--	--	--	--
108-88-3	Toluene	--	--	--	5000	22000	22000	7500
526-73-8	1,2,3-Trimethylbenzene	--	--	--	60	260	260	--
95-63-6	1,2,4-Trimethylbenzene	--	--	--	60	260	260	--
108-67-8	1,3,5-Trimethylbenzene	--	--	--	60	260	260	--
7440-62-2	Vanadium (fume or dust)	--	--	--	0.1	0.44	0.44	0.8
75-01-4	Vinyl chloride	0.11	0.22	2.7	100	440	440	1300
1330-20-7	Xylene (mixture), including m-xylene, o-xylene, p-xylene	--	--	--	220	970	970	8700

*RBC values for Arsenic, Chromium VI, and Fluorides have MPAF applied

3.3 Risk Calculation

Using the CAO toxic air contaminant emissions inventory (e.g., AQ520), the 24-hr and annual average concentration files from AERMOD runs, the RBCs with Level 4 adjustments applied, and the land use designations at each receptor, the chronic cancer, chronic non-cancer and acute hazard index risk will be estimated at every receptor. The risk at each receptor from source ($R_{r,s}$) is given by:

$$R_{r,s} = \chi_{r,s}\, C \sum_{p} \frac{Q_{s,p}\, TO_{p,o}}{RBC_{p,L(r)}}$$

where $\chi_{r,s}$ is the unit concentration for source s at receptor r, C is a constant to convert g/s to either lbs/day or lbs/year, $Q_{s,p}$ is the pollutant emission rate from the AQ520 form, $TO_{p,o}$ is the target organ factor (0 or 1) for pollutant p and organ o, A is the bioavailability factor, and $RBC_{p,L(r)}$ is the RBC for pollutant p and land use L at the receptor r. For manganese, an additional factor A is applied to its contribution to account for its inhalation bioaccessibility. For the TEUs with source-specific oral bioavailability, RBCs specific to those sources will be used.

For non-cancer risk, different pollutants impact different target organs, so the non-cancer risk is not additive. When applied, the target organ factor is set to 1 for pollutants that impact a particular target organ and zero otherwise. For cancer risk, TO is always 1 because carcinogens are considered cumulatively regardless of target organ.

Each receptor location has up to seven values for each source depending on and exposure scenarios (residential, child, and worker) and health endpoints: (acute non-cancer, chronic non-cancer, and cancer risk). For comparison purposes, the maximum cancer risk and chronic hazard values for any exposure scenario (residential, non-residential child, and worker) will be compared to the appropriate Existing Source Risk Action Levels (RALs) which are shown in Table 3-10.

Table 3-10. Existing Source Risk Action Levels

Level Description	Cancer # in a million	Non-Cancer Hazard Index
Source Permit Level	5	0.5
Community Engagement Level	25	1
TBACT Level	50	3/5 or RDR=1
Risk Reduction Level	200	6/10 or RDR=2
Permit Denial Level	500	12/20 or RDR=4

3.4 Uncertainty Analysis

CAO rules require that a quantitative or qualitative uncertainty evaluation be included in a Level 4 risk assessment.

AERMOD is designed to predict the overall maximum impact within the area modeled. However, it is well documented that the model cannot accurately predict the actual concentration at a specific location. Localized variations in winds, the influences of trees and terrain can influence when and where the worst-case impact may actually occur around a facility. For example, the downwash algorithm in AERMOD is a simplification of reality, treating all buildings as rectangular boxes. Wind tunnel studies have documented that for long buildings, modeled downwash is greatly overestimated downwind of the site. Downwash is also not well characterized when the winds are approaching a building from a diagonal direction (e.g. toward a corner). Thus, AERMOD has the potential to underpredict or overpredict concentrations at a particular location.

A chronic exposure location is defined in the CAO rules in terms of residential locations and non-residential locations. For residential locations, the rule indicates that the location is considered residential based on whether "… a person or persons may reasonably be present for most hours of each day over a period of many years" (340-245-0020 (21)(i)). For the chronic non-residential location, the rules state such a location is where "a person or persons may reasonably be present for a few hours several days per week, possibly over a period of several years" (340-245-0020 (21)(ii)). In practice, cancer risk estimates are based on a continuous exposure duration of 70 consecutive years, which is expected to overestimate chronic cancer exposures and, therefore, risk. For example, SCAQMD risk assessment guidance assumes residents are exposed for 30 years and workers are exposed for 25 years (SCAQMD, 2020).[9]

For acute exposures, the CAO regulation requires the use of the maximum 24-hour concentration that the computer model predicts using five years of meteorological data (1,825 days). Thus, the acute risk can be driven by the one "bad" meteorological day, regardless of whether such an impact would actually occur when the public is present or at the same time that the facility is emitting from all of its all TEU's at maximum capacity. Thus, using the 24-hr maximum provides a very conservative risk estimate as it assumes that someone will be present at a time when there is perfect alignment between worst-case meteorological conditions and maximum facility emissions.

Threshold risk values (TRVs) form the basis for the RBCs. Both the TRV and RBC values consider scientific uncertainty for safety, particularly in sensitive populations. Often the exact level of exposure that causes health effects in people is unknown because: 1) experiments are rarely conducted on people; 2) science experiments can only reflect the doses tested; and 3) different people have different sensitivities to the same dose. The greater the scientific uncertainty in determining potential harm, the larger the uncertainty factor applied to the TRV

[9] South Coast Air Quality Management District (SCAQMD). 2020. AB2588 and Rule 1402 Supplemental Guidelines (Supplemental Guidelines for Preparing risk Assessments for the Air Toxics "Hot Spots" Information and Assessment Act). October.

and RBC values. This results in risk and chronic hazard estimates that are well below levels at which adverse health effects have been observed.

ODEQ developed a new provisionally-accepted acute TRV for Mn, which incorporates a time adjustment factor that is particularly conservative considering that total exposure duration from the studies used as the point of departure was 90 hours (6 hours per day, 5 days per week for 3 weeks) and the TRV is for a single 24-hour period. As discussed in the recent publication by Perry et al., 2024[10], PBPK modeling demonstrates that a time adjustment factor for acute Mn exposure is not necessary because tissue concentrations in lung and brain tissue were essentially unchanged between the two exposure scenarios (6 hours per day, 5 days per week for 3 weeks compared to continuous 24-hour exposure). If the time-adjustment factor is not included, the TRV is 5 $\mu g/m^3$, thus the acute TRV is expected to overestimate the potential for acute effects associated with manganese exposure by a 4-fold.

As outlined above, one of the largest uncertainties is in the multipathway factors, especially for arsenic. The combined default non-cancer soil ingestion and home grown produce MPAFs for arsenic are over 80 times the inhalation risk, based on upper bound exposure assumptions and assumed 100% RBA. Applying the adjustments reduces the MPAF to approximately 40. In contrast, OHA found in the Bullseye Glass PHA that the arsenic soil ingestion and home-grown pathway risks were less than the inhalation pathway (e.g. MPAF < 1). Thus, MPAF values are highly uncertain and likely biased high.

[10] Perry et al. 2024. PBPK Modeling Demonstrates that Exposure Time Adjustment is Unnecessary for Setting an Acute Manganese Inhalation Exposure Guideline. *Reg Tox Pharm* (in press).

Appendix A. ToxStrategies Memo: Evaluation of Oral Bioavailability, Inhalation Bioavailability, Homegrown Produce Consumption, and Incidental Soil Ingestion Rates for Cascade Steel's Level 4 Cleaner Air Oregon Risk Assessment

ToxStrategies

Innovative solutions
Sound science

Memorandum

July 31, 2024

To:	Jim Spahr (Cascade Steel), and Kent Norville and John Browning (Bridgewater Group)
From:	Deborah Proctor and Ann Verwiel
Subject:	Evaluation of Oral Bioavailability, Inhalation Bioavailability, Homegrown Produce Consumption, and Incidental Soil Ingestion Rates for Cascade Steel's Level 4 Cleaner Air Oregon Risk Assessment

The Cascade Steel Rolling Mills (Cascade) facility at 3200 NE Highway 99W in McMinnville, OR is currently conducting a Level 4 Cleaner Air Oregon (CAO) risk assessment. In a Level 4 risk assessment, a facility can incorporate site-specific considerations to more accurately represent risk that may be over-estimated by default exposure assumptions. Cascade has retained ToxStrategies, a California based toxicology firm, to evaluate and propose changes to CAO multi-pathway adjustment factors (MPAFs) and other default assumptions. The proposed changes, as detailed in this memo, will produce a more site-specific and refined risk assessment (as compared to the default assumptions). The proposed changes in this memo include the following:

- Arsenic oral bioavailability adjustment
- Emission-unit-specific manganese inhalation bioavailability adjustment
- Soil ingestion rates
- Home grown produce ingestion rates

In support of this effort, we reviewed the public health assessments (PHAs) for Bullseye Glass Co. (manufacturing site) (2023)[1] and used information in the Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, OR approved on March 10, 2022.[2]

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[1] Oregon Health Authority, Public Health Division. 2023. Public Health Assessment Final Release. Bullseye Glass Co. (manufacturing site), 3722 SE 21st Avenue, Portland, OR 97202.

[2] Oregon Department of Environmental Quality. 2022. Final Review of Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, OR. March 10.

The authors of this memorandum each have been practicing in the field of human health risk assessment for more than 20 years. Ms. Verwiel and Ms. Proctor have both conducted multiple air toxic hot spot risk assessments for facilities in California and specifically with the South Coast Air Quality Management District (SCAQMD). The MPAFs that are used by Cleaner Air Oregon are based on the AB2588 air toxics program in California as implemented by SCAQMD. Ms. Verwiel and Ms. Proctor have also conducted relative bioavailability and bioaccessibility studies for metals in environmental media. Their resumes are included as Attachment A.

Oral Bioavailability of Arsenic in Particulate Emissions

DEQ applies default MPAF to account for potential exposures to CAO chemicals, including certain metals, which are emitted to ambient air but may deposit on soil and be incidentally ingested. The contribution of multi-pathway exposure for arsenic is significant in the Levels 1, 2, and 3 CAO risk assessments based on the default multi-pathway factors for arsenic applied by DEQ (9.71 for cancer effects and 88.03 for noncancer effects). The MPAF factor for CAO Level 1 to 3 risks assessments in Oregon incorporates a default assumption for oral relative bioavailability (RBA) of 100%. RBA is the bioavailability of a chemical in an environmental matrix, such as slag or baghouse dust, *relative to* the reference material used to develop the toxicity criteria for use in risk assessment.

The CAO rules specifically allow RBA measures to be included in Level 4 risk assessments. For example, oral RBA based on U.S. EPA's recommended default RBA of 60%[3] for arsenic was used rather than a default RBA of 100% in the approved Level 4 risk assessment for Owens-Brockway Glass.[4]

For Cascade, *in vitro* bioaccessibility (IVBA) measurements results for specific emissions sources containing arsenic are used to estimate oral RBA for use in the Level 4 risk assessment. Based on *in vivo* animal studies for arsenic, U.S. EPA has developed sufficient data to quantify the relationship of RBA to IVBA.[5] EPA Method 1340[6] and EPA's *Release of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and*

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[3] United States Environmental Protection Agency (U.S. EPA). 2012. Recommendations for Default Value for Relative Bioavailability of Arsenic in Soil. December. Compilation and Review of Data on Relative Bioavailability of Arsenic in Soil (epa.gov)

[4] Oregon Department of Environmental Quality. 2022. Final Review of Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, OR. March 10.

[5] U.S. EPA. 2017. Release of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil. STANDARD OPERATING PROCEDURE (corrected) FOR IN VITRO BIOACCESSIBILITY FOR LEAD AND ARSENIC IN SOIL (WITH MAY 5, 2017, TRANSMITTAL MEMO ATTACHED) (epa.gov)

[6] U.S. EPA. 2017. Method 1340. *In Vitro* Bioaccessibility Assay for Lead in Soil. Revision 1. SW-846 Update VI. February. corrected July 6. https://www.epa.gov/sites/default/files/2017-03/documents/method_1340_update_vi_final_3-22-17.pdf

Arsenic in Soil (SOP) provide calculations to predict RBA for arsenic for use in human health risk assessment based on an *in vitro* bioaccessibility (IVBA) measurements. The Level 4 risk assessment will use estimates of oral RBA for arsenic for certain specific emission sources based on EPA's methods, and the default RBA of 60% for other sources of arsenic where site-specific analysis applying EPA's methods was not completed.

To measure IVBA, samples of particulate were collected from around the roof monitor and from the truck sweep off area to represent air emissions from the Melt Shop Roof Monitor and Melt Shop Fugitive TEUs and the Main and Secondary Scrap Handling TEUs, respectively. Sampling was performed by Bridgewater LLC and the sampling methods are summarized in Attachment B. Prima Environmental, Inc. in El Dorado Hills, CA (Prima) performed the analyses, and the laboratory results for oral bioavailability are presented in Attachment C.[7] Prior to analysis and in accordance with EPA Method 1340[8], Prima sieved the samples; approximately 22% of the truck sweep off particulates and 27% of the roof monitor particulates were <150 microns. Prima analyzed the <150 micron fraction of the sample for total arsenic and for soluble arsenic using IVBA methods outlined in EPA Method 1340 and supplemental information provided in EPA guidance.[9]

As shown in Table 1, IVBA for arsenic in particulates from the roof monitor was 51% to 56%, corresponding to oral RBA of 43% to 47%, respectively, using EPA's equation to convert from arsenic IVBA to RBA (Table 1, footnote 1). For arsenic in truck sweep dust, the IVBA was estimated to be 9.5 to 9.9%, and the RBA was estimated to be 11%. As presented, duplicate sample results for each sample were very close, indicating good precision in the RBA data. Other quality assurance/quality control (QA/QC) measures were within limits except one laboratory control limit sample that was slightly low for arsenic as described in Attachment C.

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[7] The lab reports also include analysis for IVBA for lead at the roof monitors, but the results are not discussed further herein and were not used in the risk assessment work plan because lead was not a key chemical.

[8] U.S. EPA. 2017. Method 1340. *In Vitro* Bioaccessibility Assay for Lead in Soil. Revision 1. SW-846 Update VI. February. corrected July 6. https://www.epa.gov/sites/default/files/2017-03/documents/method_1340_update_vi_final_3-22-17.pdf

[9] U.S. EPA. 2017. Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil. STANDARD OPERATING PROCEDURE (corrected) FOR IN VITRO BIOACCESSIBILITY FOR LEAD AND ARSENIC IN SOIL (WITH MAY 5, 2017, TRANSMITTAL MEMO ATTACHED) (epa.gov)

Table 1. Oral IVBA results for arsenic proposed for use in the Level 4 Risk Assessment.

Toxic Emission Unit ID	Toxic Emission Unit Description	Representative Sample Description	Representative Sample ID	Sample Date	Arsenic	
					In Vitro Bioaccessibility (IVBA)	Relative Bioavailability (RBA) [1]
EU-3_RM	Melt Shop Roof Monitors	Roof Monitor	S1	6/1/23	56%	47%
EU-3_MF	Melt Shop Fugitives	Roof Monitor - Duplicate	S1-Dup	6/1/23	51%	43%
EU-9sh_Main	Main Scrap Handling	Truck Sweep Off	S2	6/1/23	9.9% J	11%
EU-9sh_Sec	Secondary Scrap Handling	Truck Sweep Off - Duplicate	S2-dup	6/1/23	9.5% J	11%

Notes
1. Relative bioavailability for arsenic is calculated as: arsenic RBA = (0.79 × IVBA) + 0.03 (U.S. EPA, 2017)
2. Relative bioavailability for lead is calculated as: lead RBA = (0.88 × IVBA) – 0.028 (U.S. EPA, 2017)
Oral RBA values to apply in Level 4 Risk Assessment.
Abbreviations
-- = not analyzed
IVBA = *in vitro* bioaccessibility
J = Value flagged as estimated by the laboratory
RBA = Relative bioavailability.
Reference
U.S. EPA, 2017. Standard Operating Procedure for an *In Vitro* Bioaccessibility Assessment for Lead and Arsenic in Soil. OLEM 9200.2-164. April 20.

Based on these data, we propose to use an oral RBA for arsenic of 47% for the Melt Shop Roof Monitor and Melt Shop Fugitive TEUs (EU-3_RM & EU-3_MF) and an oral RBA for arsenic of 11% for the Main and Secondary Scrap Handling TEUs (EU-9sh_Main & EU-9sh_Sec). In both cases, we elected to use the higher RBA value to be conservative. USEPA's default RBA for arsenic (60%) will be used for all other arsenic sources.

Inhalation Bioaccessibility of Manganese in Particulate Emissions

Similar to oral RBA, the inclusion of RBA for inhalation exposures is also applicable in Level 4 risk assessments. Quantitative measures of RBA are developed from research involving animals and rarely conducted for several reasons; rather inhalation RBA is estimated using measures of the solubility of metals in simulated lung fluids under extraction conditions designed to simulate lung condition (Henderson et al. 2014)[10].

For Cascade Steel, inhalation RBA values for manganese were estimated using IVBA for emission-specific sources, e.g., particulates suspended from slag handling. As discussed in the ATSDR Toxicological Profile for manganese, several animal studies have shown that soluble forms of manganese are better absorbed than forms of lesser solubility (ATSDR

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2012).[11] The provisionally approved TRV for acute exposures to Mn (1.3 µg/m^3)[12] is based on monkey bioassays wherein Mn was administered in the freely soluble manganese sulfate form. As discussed below, Mn in emissions from Cascade is less soluble in biological fluids and thus less available for absorption. Thus, to reduce uncertainty and conservatism in the Level 4 risk assessment, IVBA measures are used as estimates of inhalation RBA.

For inhalation exposures, there are two fluids that are relevant to absorption in the lungs: interstitial fluid and lysosomal fluid. Solubility in these fluids may lead to systemic absorption via inhalation. The IVBA test and solutions (interstitial and lysosomal) simulate conditions in the lung that are relevant to the dissolution of metal ions and absorption of metals and systemic uptake. Specifically, solubilized metal ions are important, because, for metal-containing particles, the free metal ion is usually considered to be responsible for the observed toxicity.[13] The solubility/bioaccessibility predicted by the IVBA tests are correlated with *in vivo* bioavailability, and IVBA values are reported to be more conservative (higher values) than the relative bioavailability values developed from *in vivo* testing.[14]

The test protocol to measure IVBA was consistent with that of Henderson et al. (2014)[15] (Attachment D) and involved two separate extractions, each analyzed at two timepoints. Our first extractions were conducted in simulated interstitial fluid for 24 and 72 hours. These experiments simulated the extracellular environment of the lungs (i.e., outside of the cells, conditions prior to absorption) to measure solubility of inhaled metals on the surface of lung cells. It is well-documented that inhaled particles can reside in the extracellular compartment of the lung, which is represented by the lung fluid of neutral pH (interstitial fluid).[16] Metals that dissolve in interstitial solution can be absorbed by simple diffusion, and those that do not dissolve are generally cleared from the lung by macrophages and mucociliary action. Any absorption of inhaled manganese from the oral pathway is considered negligible as the bioavailability of manganese from ingestion is only 3-5% (ATSDR 2012).

For the second set of extractions, IVBA extractions were conducted in the lysosomal fluid for 24 and 72 hours. Lysosomal fluid is representative of conditions inside of cells. Thus,

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[11] ATSDR 2012. Toxicological Profile for Manganese. US Department of Health and Human Services. Agency for Toxic Substances and Disease Registry (ATSDR). Page 225.

[12] ODEQ 2024. Memorandum: DEQ Toxicity Reference Value (TRV) Proposal for 24-hour Acute Inhalation Exposure to Manganese. July 26.

[13] Heim KE, Danzeisen R, Verougstraete V, Gaidou F, Brouwers, T, Oller AR. 2109. Bioaccessibility of nickel and cobalt in systemic gastric and lung fluids and in its potential use in alloy classification. Reg Toxciol Pharm.

[14] Ibid.

[15] Henderson, R.G., et al. 2014. Inter-laboratory validation of bioaccessibility testing for metals. Reg Tox and Pharm. 70: 170-181.

[16] Boisa N, Elom N, Dean JR, Deary ME, Bird G, Entwistle JA. 2014. Development and application of an inhalation bioaccessibility method (IBM) for lead in the PM10 size fraction of soil. Environ Int 70:131–142.

besides the extracellular environment of the lung, some chemical forms of metals can also be dissolved in the more acidic environment within the pulmonary macrophages and epithelial cells.[17] Hence, metals that are insoluble (i.e., do not dissolve) in interstitial fluid, may be absorbed via phagocytosis or other transport channels, and then dissolved inside the cells by lysosomal fluid. This fluid is more acidic, due to its citric acid content, and has greater capacity to dissolve water-insoluble metals, because it strongly binds metal ions and is thought to mimic the *in vivo* activity of proteins within macrophages.[18] (Heim et al. 2019).

Prima Environmental, Inc. in El Dorado Hills, California, measured IVBA in simulated lysosomal and interstitial fluids following procedures outlined in Henderson (2014) (Attachment D).[19] Emission source samples were collected as outlined in Attachment B. Samples were sieved to <75µm and extracted under simulated lung conditions after 24 hours and 72 hours for both fluids to measure any change in bioaccessibility with time. Five materials representing facility emission sources of particulates were analyzed (EAF-LMF slag, billet cutting vent, roof monitor, truck sweep off area, and SiMn stockpile). In addition, bioaccessibility was tested for two manganese standards ($MnSO_4$ and MnO) for quality control (QC) purposes. Duplicate samples were also run for all materials except EAF-LMF slag and the QC samples. Laboratory reports presenting the results are provided in Attachment E. Table 2 summarizes the results of the inhalation IVBA samples.

Lysosomal IVBA ranged from 9.1% to 83%, and interstitial IVBA ranged from 0.29% to 6%. For each emission source, the results were very similar between samples collected from the 24 hour and 72 hour extraction time periods. In all cases, interstitial IVBA was much lower than lysosomal IVBA. For this reason, the maximum IVBA in lysosomal fluids will conservatively be used to represent inhalation RBA in the Level 4 risk assessment. The values highlighted in blue for dissolution in lysosomal fluid in Table 2 will be used as the inhalation RBA for the respective emission sources. These values are considered conservative because insoluble particles must be taken into cells (e.g.,

macrophages) in the lung to be dissolved by the intracellular lysosomal fluid.

In addition to the high reproducibility in the duplicate samples, $MnSO_4*H_2O$ and MnO were 100% bioaccessible in the lysosomal fluid in contrast to the lower bioaccessibility for the site-specific and QC samples. $MnSO_4*H_2O$ is water soluble and MnO is water insoluble,[20] and although both were 100% bioaccessible in lysosomal fluid, solubility in interstitial fluid was greater for $MnSO_4*H_2O$ than MnO, which was <0.1% (Table 2). These

[17] Ibid.
[18] Heim KE, Danzeisen R, Verougstraete V, Gaidou F, Brouwers, T, Oller AR. 2109. Bioaccessibility of nickel and cobalt in systemic gastric and lung fluids and in its potential use in alloy classification. Reg Toxciol Pharm.
[19] Henderson, R.G., et al. 2014. Inter-laboratory validation of bioaccessibility testing for metals. Reg Tox and Pharm. 70: 170-181.
[20] ATSDR 2012.

results demonstrate the conservative nature of using lysosomal fluid solubility as a measure of RBA.

Table 2. Inhalation IVBA sample results for Mn proposed for use in the Level 4 Risk Assessment.

Toxic Emission Unit ID	Toxic Emission Unit Description	Sample ID	Duration of Test (hours)	Lysosomal			Interstitial		
				Lab ID	Extraction Date	Bioaccessibility	Lab ID	Extraction Date	Bioaccessibility
EU-5	Slag Handling	EAF/LMF Slag-062123	24	L24-1	7/12/23	83%	I24-1.2	7/24/23	3.8%
		EAF/LMF Slag-062123	72	L72-1	7/14/23	81%	I72-1	7/18/23	6.0%
EU-10	Caster Billet Cutting	Billet Cutting Vent D/R-A02-042823	24	L24-2	7/12/23	9.3%	I24-2 [2]	7/17/23	0.36%
		Billet Cutting Vent D/R-A02-042823	24	L24-2 dup	7/12/23	9.1%	I-24-2 dup [2]	7/17/23	0.29%
		Billet Cutting Vent D/R-A02-042823	72	L72-2	7/14/23	10%	I72-2 [2]	7/18/23	0.59%
		Billet Cutting Vent D/R-A02-042823	72	L72-2 dup	7/14/23	10%	I72-2 dup [2]	7/18/23	0.53%
EU_3 RM	Melt Shop Roof Monitor	Roof Monitor D/R-02-042823	24	L24-3	7/12/23	53%	I24-3.2	7/24/23	1.28%
		Roof Monitor D/R-02-042823	24	L24-3 dup	7/12/23	51%	I24-3.2 dup	7/24/23	1.08%
EU-3_MF	Melt Shop Fugitives	Roof Monitor D/R-02-042823	72	L72-3	7/14/23	54%	I72-3	7/18/23	1.76%
		Roof Monitor D/R-02-042823	72	L72-3 dup	7/14/23	51%	I72-3 dup	7/18/23	1.32%
EU-9sh_Main	Main Scrap Fugitives	Truck Sweep Off-02-042823	24	L24-4	7/12/23	59%	I24-4	7/17/23	2.85%
		Truck Sweep Off-02-042823	24	L24-4 dup	7/12/23	58%	I24-4 dup	7/17/23	2.24%
EU-9sh_Sec	Secondary Scrap Handling	Truck Sweep Off-02-042823	72	L72-4	7/14/23	61%	I72-4	7/17/23	5.86%
		Truck Sweep Off-02-042823	72	L72-4 dup	7/14/23	61%	I72-4 dup	7/17/23	5.23%
EU_18_ATDSiMn	SiMn Alloy Truck	SiMn Stockpile-062123	24	L24-6	7/12/23	43%	I24-6	7/17/23	0.39%
		SiMn Stockpile-062123	24	L24-6 dup	7/12/23	43%	I24-6 dup	7/17/23	0.39%
EU-18_AULDSiMn	SiMn Aloy Unload to Feeder	SiMn Stockpile-062123	72	L72-6	7/14/23	43%	I72-6	7/18/23	0.63%
		SiMn Stockpile-062123	72	L72-6 dup	7/14/23	44%	I72-6 dup	7/18/23	0.62%
QC Samples									
		$MnSO_4*H_2O$	24	L24-7	7/12/23	102%	I24-7.2 [1]	7/24/23	16%
		$MnSO_4*H_2O$	72	L72-7	7/14/23	103%	I72-7	7/18/23	6.0%
		MnO	24	L24-8	7/14/23	102%	I24-8 [1]	7/17/23	0.032%
		MnO	72	L72-8	7/14/23	102%	I72-8	7/18/23	0.062%

Notes:

1. The bioaccessibility of MnSO4•H2O decreased over time in the interstitial tests. The reason is presumably due to reaction of MnSO4•H2O with the extraction fluid. MnSO4•H2O is a pale pink solid that readily dissolves in deionized water. However, addition of MnSO4•H2O to interstitial extraction fluid turned the extraction fluid cloudy white. Settled solids were observed within 21 hours and a pinkish brown precipitate was noted within 7 days.
2. The results for these samples are estimated because the concentration in the extraction fluid was below the reporting limit but above the method detection limit.

Inhalation bioaccessibility values to represent bioavailability in Level 4 Risk Assessment.

Relative Bioavailability in Risk Calculations

Estimates of RBA for ingestion and inhalation exposures will be incorporated into the health risk calculations independently. Oral RBA was developed for arsenic for two emission sources. Inhalation RBA was developed for manganese for five emission sources. The subsequent sections discuss incorporating the RBAs into the soil ingestion and inhalation exposure equations to adjust the multi-pathway factors.

Incorporating Oral RBA into Soil Ingestion Exposures

The oral RBAs for arsenic (GRAF in the equation below) are applicable to the contribution from the melt shop and scrap pile TEUs to soil ingestion exposure. Dose from soil ingestion exposure is calculated as (OEHHA, 2015). [21]

$$Dose_{soil} = C_{soil} \times GRAF \times SIR \times \left(\frac{ET}{AT}\right) \times CF \times EF$$

Where:

$Dose_{soil}$ = Dose from soil (mg/kg-day)
C_{soil} = Concentration in soil (µg/kg)
GRAF = Gastrointestinal relative absorption fraction (RBA; unitless)
SIR = Soil ingestion rate (mg/kg-day)
ET = Exposure time (years)
AT = Averaging time (years)
CF = 10^{-9} kg/µg
EF = Exposure frequency (unitless; days/365 days)

The total soil concentration is equal to the contribute of arsenic from the melt shop, stockpile, and other sources of arsenic in soil as follows:

$$C_{soil} = C_{soil-MS} + C_{soil-SP} + C_{soil-other}$$

Where:
C_{soil} = total concentration in soil (µg/kg)
$C_{soil-MS}$ = total concentration in soil from the melt shop sources (µg/kg)
$C_{soil-SP}$ = total concentration in soil from the stockpile sources (µg/kg)
$C_{soil-other}$ = total concentration in soil from other sources (µg/kg)

This soil concentration (C_{soil}) is applicable to dermal absorption and to plant uptake and subsequent consumption. For incidental ingestion, the relative contribution of arsenic in soil from the melt shop and stockpiles should be adjusted for the oral RBA as follows:

[21] Office of Environmental Health Hazard Assessment (OEHHA). 2015. Risk Assessment Guidelines. Guidance Manual for Preparation of Health Risk Assessments. February. (Equation 5.4.3.1.1)

$$C_{soil-adj} = (C_{soil-MS} \times \text{RBA}_{MS}) + (C_{soil-SP} \times RBA_{SP}) + (C_{soil-other} \times RBA_D)$$

Where: $C_{soil-adj}$ = Adjusted total soil concentration for incidental ingestion (µg/kg)
RBA_{MS} = Relative bioavailability from melt shop sources
RBA_{SP} = Relative bioavailability from stockpile sources
RBA_D = Default relative bioavailability for arsenic (60%)
Other variables previously defined.

Specifically for arsenic, this would be:

$$C_{soil-adj(As)} = (C_{soil-MS} \times 47\%) + (C_{soil-SP} \times 11\%) + (C_{soil-other} \times 60\%)$$

Adjustments to the air emission sources (e.g., reduction of arsenic emissions in the air dispersion modeling for these two sources) or adjustments to the exposure calculations for these two sources could be made to incorporate oral RBA. This may require modeling air dispersion separately for these sources to distinguish the contribution to soil via incidental ingestion exposures from other exposure pathways that are not affected by oral RBA (e.g., inhalation, dermal contact, and plant uptake).

Incorporating RBA into Inhalation Exposures

The inhalation RBAs for manganese ("A" in the equation below) are applicable to the contribution from the EAF-LMF slag handling, caster billet cutting, melt shop, stockpiles, and SiMn alloy. Manganese is not a multi-pathway chemical so there is no additional exposure from non-inhalation exposure pathways. Dose from inhalation exposure is calculated as (OEHHA, 2015)[22]:

$$Dose_{air} = C_{air} \times \left\{\frac{BR}{BW}\right\} \times A \times EF \times CF$$

Where:

C_{air} = Concentration in air (µg/m^3)
$\left\{\frac{BR}{BW}\right\}$ = Daily breathing rate normalized to body weight (L/kg-day)
A = Inhalation absorption factor (RBA; unitless)
EF = Exposure frequency (unitless; days/365 days)
CF = 10^{-6} (mg-m^3/µg-L)

The inhalation RBA for manganese (A in the equation) was measured for materials representing five sources: EAF-LMF slag (slag handling), billet cutting vent (billet cutting), roof monitor (melt shop), truck sweep off (scrap piles), and SiMn stockpile. The

[22] Ibid. (Equation 5.4.1.1)

contribution of each of these sources of manganese must be adjusted to account for inhalation bioavailability before dose is calculated. The total air concentration is equal to the contribution from these five sources and any other sources of manganese in air at the facility as follows:

$$C_{air} = C_{air-EAF} + C_{air-BC} + C_{air-MS} + C_{air-SP} + C_{air-SiMnSP} + C_{air-other}$$

Where:
C_{air} = total concentration in air (µg/m^3)
$C_{air\text{-}EAF}$ = total concentration in air from the EAF-LMF slag (µg/m^3)
$C_{air\text{-}BC}$ = total concentration in air from the billet cutting (µg/m^3)
$C_{air\text{-}MS}$ = total concentration in air from the melt shop (µg/m^3)
$C_{air\text{-}SP}$ = total concentration in air from the scrap metals (µg/m^3)
$C_{air\text{-}SiMnSP}$ = total concentration in air from the SiMn stockpile (µg/m^3)
$C_{air\text{-}other}$ = total concentration in air from other sources (µg/m^3)

The relative contribution of manganese in air from the five sources to exposure to manganese via inhalation should be adjusted for the inhalation RBA as follows:

$$C_{air-adj} = (C_{air-EAF} \times \mathrm{RBA}_{EAF}) + (C_{air-BC} \times RBA_{BC}) + (C_{air-MS} \times \mathrm{RBA}_{MS}) + (C_{air-SP} \times RBA_{SP}) + \left(C_{air-SiMn\ stockpile} \times RBA_{SiMnSP}\right) + C_{air-other}$$

Where:
$C_{air\text{-}adj}$ = Adjusted total air concentration
RBA_{EAF} = Relative bioavailability from EAF-LMF slag
RBA_{BC} = Relative bioavailability from billet cutting
RBA_{MS} = Relative bioavailability from melt shop
RBA_{SP} = Relative bioavailability from scrap metals
$\mathrm{RBA}_{SiMn\ Sp}$ = Relative bioavailability from SiMn stockpile
Other variables previously defined.

Specifically for manganese in air, this would be:

$$C_{air-adj} = (C_{air-EAF} \times 83\%) + (C_{air-BC} \times 10\%) + (C_{air-MS} \times 54\%) + (C_{air-SP} \times 61\%) + (C_{air-SiMnSP} \times 44\%) + C_{air-other}$$

Manganese concentrations in air will be adjusted using the RBA for both acute and chronic inhalation exposures. Because manganese is only evaluated for inhalation exposures, the RBA can be applied to the emission rate from each of the relevant sources to predict the bioavailable concentration of manganese in air. There are no other exposure pathways that would be affected by these adjustment.

Soil Ingestion Rates

The OEHHA guidance used for the MPAF provides an option for using 95th percentile and mean incidental soil ingestion rates. Use of a 95th percentile incidental soil ingestion rate in the MPAF, along with inhalation, dermal contact, and produce ingestion exposure pathways, creates an unrealistic estimate of upper-bound cumulative exposure by compounding upper end exposure across multiple pathways.

Table 3 presents the soil ingestion data for the Public Health Assessment for Bullseye Glass Plant and soil ingestion rates used in the Cleaner Air Oregon Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, Oregon, which was approved by ODEQ in a letter dated March 10, 2022.[23] The Owens-Brockway soil ingestion rates are based on the data for Bullseye Glass Plant

Table 3. Incidental soil ingestion rates from Public Health Assessment for Bullseye Glass Company and Owens-Brockway Level 4 Risk Assessment

Age Group[1] (years)	Exposure Period by Age Group (years)	PHA Body Weight [1] (kg)	PHA Soil Ingestion Rate [1] (mg/day)	Age Group for HRA[2] (years)	Exposure Period by Age Group (years)	Time-weighted Average Soil Ingestion Rate[2,3] (mg/kg-day)
0 to <1	1	7.8	100	0 to <2	2	15
1 to <2	1	11.4	200			
2 to < 6	4	17.4	200	2 to <16	14	6.8
6 to <11	5	31.8	200			
11 to <16	5	56.8	200			
16 to <21	5	71.6	200	16 to 70	54	1.4
21 to 70	49	80	100			
Age-weighted Soil Ingestion Rate (SIR) (mg/kg-day)						2.87

Notes:

1. Oregon Health Authority, Public Health Division. 2023. Public Health Assessment Final Release. Bullseye Glass Co. (manufacturing site), 3722 SE 21st Avenue, Portland, OR 97202. Table I-3.
2. Bridgewater Group, Inc., 2022. Cleaner Air Oregon Modeling Protocol and Level-4 Risk Assessment Work Plan, Owens-Brockway Plant 21, Portland, Oregon.
3. Time-weighted Average Soil Ingestion Rate (mg/kg-day) =

$$\Sigma \frac{\text{PHA Ingestion Rate } \left(\frac{\text{mg}}{\text{day}}\right) \times \text{Exposure Period (years)}}{\text{Body Weight (kg)}} \Big/ \text{Age Group Exposure Period (years)}$$

□

Abbreviations:

mg — milligrams; kg — kilograms; PHA = Public Health Assessment

[23] Oregon Department of Environmental Quality. 2022. Final Review of Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, OR. March 10.

Homegrown Produce Ingestion Rates

The default screening MPAF for metals includes a contribution from potential ingestion of homegrown produce grown in soil affected by deposition from emissions. Similar to the discussion of soil ingestion rates, use of upperbound estimates of plant ingestion rates over estimates exposure across multiple exposure pathways. We propose to use assumptions for homegrown produce ingestion rates that are based on those approved for use in 2022 Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, Oregon.[24]

Homegrown produce is divided into four categories (exposed, leafy, protected, and root), each of which has unique consumption rates. For the Owens-Brockway Plant 21 Level 4 risk assessment, DEQ recommended using the 75th percentile produce consumption rates developed by California's Office of Environmental Health Hazard Assessment (OEHHA).[25] We propose to use a time-weighted average consumption rate using the 75th percentile for each age group (e.g., 0 to <2 years, 2 to <16 years, 16 to <70 years), which are provided by OEHHA.[26]

In Table 4, we present the 75th percentile produce consumption rates for each category of produce for three age groups: 0 to <2 years, 2 to <16 years and 16 to <70 years age groups. The corresponding exposure duration for each age group are 2 years, 14 years, and 54 years, respectively. For overall produce consumption by category, we calculate a time-weighted average consumption rate using the exposure duration for each of the three age groups as follows:

$$PC_{twa} = \frac{(PC_{0-<2} \times EP_{0-<2}) + (PC_{2-<16} \times EP_{2-<16}) + (PC_{16-<70} \times EP_{16-<70})}{Total\ Exposure\ Duration\ (70\ years)}$$

Where: PC_{twa} = Time-weighted average produce consumption by category (g/kg-day)
$PC_{0-<2}$ = Produce consumption rate for 0 to less than 2 years (g/kg-day)
$EP_{0-<2}$ = Exposure period for 0 to 2 years (2 years)
$PC_{2-<16}$ = Produce consumption rate for 2 to less than 16 years (g/kg-day)
$EP_{2-<16}$ = Exposure period 2 to <16 years (14 years)
$PC_{16-<70}$ = Produce consumption rate for 16 to 70 years (g/kg-day)
$EP_{16-<70}$ = Exposure period 16 to <70 years (54 years)

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[24] Oregon Department of Environmental Quality. 2022. Final Review of Level 4 Risk Assessment for Owens-Brockway Plant 21 in Portland, OR. March 10.

[25] Office of Environmental Health Hazard Assessment (OEHHA). 2012. Technical Support Document for Exposure Assessment and Stochastic Analysis. Final. August.

[26] Ibid

Table 4. OEHHA 75th percentile produce ingestion rates and time-weighted average ingestion rates proposed for use in the Level 4 Risk Assessment.[27]

Age Group	Exposure Period (years)	75th Percentile Exposed Produce Ingestion Rate [1] (g/kg-day)	75th Percentile Leafy Produce Ingestion Rate [1] (g/kg-day)	75th Percentile Protected Produce Ingestion Rate [1] (g/kg-day)	75th Percentile Root Produce Ingestion Rate [1] (g/kg-day)	Total 75th Percentile Produce Ingestion Rate (g/kg-day)
0 to <2 years	2	15.4	5.3	7.5	8.2	36.4
2 to <16 years	14	7.3	2.3	4.9	3.9	18.4
16 to < 70 years	54	2.4	1.5	2.1	2.1	8.1
Total Time- Weighted Average Produce Ingestion [2]		3.8	1.8	2.8	2.6	11

Notes:

1. Office of Environmental Health Hazard Assessment (OEHHA). 2012. Technical Support Document for Exposure Assessment and Stochastic Analysis. Final. August. Tables 7.9, 7-11, and 7-13.
2. Calculated using equations provided above.

Abbreviations:

g/kg-day — gram per kilogram-day

Table 4 presents a time-weighted average produce ingestion rate for each category of produce in units of g/kg-day. The total 70-year time-weighted average produce ingestion rate is 11 g/kg-day for all produce categories. Using the time-weighted average body weight from Table 3 (68.7 kg) and a total 11 g/kg-day total time-weighted average consumption rate, total consumption would be 1020 g/day of total produce or 2.2 pounds/day, which is an upperbound estimate of individual produce consumption on a daily basis.

[27] Ibid, Tables 7.9, 7.11, and 7.13

Conclusions

For the Level 4 risk assessment, we recommend using the following values:

- Oral RBA
    - 47% for arsenic for the Melt Shop Roof Monitor and Melt Shop Fugitive emissions (EU-3_RM and EU-3_MF)
    - 11% for arsenic for Scrap Handling emissions (EU-9sh_Main and EU-9sh_Sec)
    - 60% for arsenic for all other sources with arsenic emissions
- Inhalation RBA for Mn emissions
    - 83% EAF-LMF slag (EU-5)
    - 10% for caster billet cutting (EU-10)
    - 54% for the Melt Shop Roof Monitor and Melt Shop Fugitive emissions (EU-3_RM and EU-3_MF)
    - 61% for scrap handling (EU-9sh_Main and EU-9sh_Sec) represented by the truck sweep-off sample.
    - 44% for the SiMn Material Handling (EU-18_ATDSiMn and EU-18_AULDSiMn)
- A time-weighted average soil ingestion rate consistent with the Level 4 Risk Assessment for Owens-Brockway (Table 3).
- Time-weighted average produce ingestion rates by produce category based on 75th percentile ingestion rates published by OEHHA consistent with the Level 4 Risk Assessment for Owens-Brockway (Table 4).

Attachments

Attachment A Resumes
Attachment B Sample Collection for Oral and Inhalation IBVA Assessment
Attachment C Oral Bioaccessibility Analytical Results
Attachment D Inter-Laboratory Validation of Bioaccessibility Testing for Metals, (Henderson, et al, 2014)
Attachment E Inhalation Bioaccessibility Analytical Results

ATTACHMENT A

Resumes

Deborah Proctor

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CONTACT INFORMATION

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PROFESSIONAL PROFILE

Ms. Deborah Proctor has more than 30 years of experience in environmental and occupational health risk assessment, specializing in applied toxicology, mode-of-action evaluations for chemical carcinogens, air toxics and air pollution risk assessment, exposure reconstruction, and quantitative dose-response analysis for the purpose of developing toxicity criteria.

Ms. Proctor has technical expertise for assessing the potential human health risk associated with contaminated air, soil, sediments, groundwater, biota, and consumer products; evaluating failure-to-warn litigation claims pursuant to California Proposition 65, including determination of Safe Harbor Levels; designing risk-based site investigations; assessing the environmental fate and toxicity of metals in the environment; determining the bioavailability of metals in soil and solid media; and risk/hazard communications. Ms. Proctor has conducted studies of oral and inhalation bioaccessibility for metals in alloys, slags, and affected soil, dust, and baghouse dust, and has designed and conducted relative bioavailability studies for cobalt, nickel, and manganese. Ms. Proctor uses state-of-the-art scientific approaches to evaluate potential hazards and develop health-protective and science-driven remediation goals. She provides technical comments to regulatory agencies on policy and guidance documents, and technical support for public communication. Ms. Proctor has designed studies involving human volunteers and is experienced with the use of Internal Review Boards (IRBs) and the ethical requirements and considerations associated with research involving humans.

Ms. Proctor is a nationally recognized expert regarding the potential health risks associated with occupational and environmental exposure to chromium. She has published extensively in this field and managed research projects that have been used to develop federal and state regulatory health criteria. Additionally, she has extensive experience in metals risk assessment and specific expertise for evaluation of nickel, cobalt, titanium, manganese, lead, vanadium, beryllium, and arsenic. Ms. Proctor has experience using physiologically based pharmacokinetic (PBPK) modeling in risk assessment for chromium, lead, manganese, and perchlorate.

Ms. Proctor's research has been applied to support regulatory decisions and inform health-based criteria. Specific examples include the USEPA Inhalation Reference Concentration for hexavalent chromium using Malsh et al. (1994), the OSHA risk assessment for the 2006 Hexavalent Chromium Rule and revised Permissible Exposure Limit using Luippold et al. (2003); Crump et al. (2003), and Proctor et al. (2003; 2004), USEPA Office of Prevention, Pesticides and Toxic Substances 2008 Reregistration Eligibility Decision (RED) for Chromated Arsenicals using Technical Study Reports FPRL #012506 and FPRL #012406; and the New Jersey Department of Environmental Protection Soil Cleanup Criteria for dermal contact with hexavalent chromium using Fowler et al. (1999). She recently published an adverse outcome pathway (AOP) analysis for rodent forestomach tumors by nongenotoxic initiating events (Proctor et al., 2018).

Ms. Proctor is a regular science peer reviewer for the *Journal of Applied Toxicology*, *Toxicology*, *Regulatory Toxicology and Pharmacology*, *Chemico-Biological Interactions*, and *PLOS1*.

ACADEMIC CREDENTIALS

B.S., Environmental Toxicology, University of California, Davis, 1988
Graduate Studies, Epidemiology, University of Pittsburgh, 1996–1998

PROFESSIONAL AFFILIATIONS

Society for Risk Analysis (member)

Association for Environmental Health Sciences (Scientific Review Board member)

International Society of Exposure Assessment (member)

Society of Toxicology (Councilor, Risk Assessment Specialty Section)

PUBLICATION AND PRESENTATION AWARDS

Society of Toxicology (SOT) 2014
Awarded top 10 Risk Assessment Presentations at the Society of Toxicology conference (Proctor DM, Suh M, Tachovsky JA, Abraham L, Hixon JG, Brorby GP, Campleman SL) by the RASS.

SOT 2013
Awarded for Three of the Top Ten Risk Assessment Presentations at the Society of Toxicology conference (Kirman et al., Thompson et al., Kopec et al.) by the RASS.

SOT 2012
Awarded top nine published papers Advancing the Science of Risk Assessment by the Risk Assessment Specialty Section (Thompson CM, Haws LC, Harris MA, Gatto NM, Proctor DM) by the RASS.

SOT 2004
Awarded top five Risk Assessment Presentations at the Society of Toxicology conference (Leung H, Madl A, Proctor D, Hays S, Cohen E) by the RASS, Baltimore MD.

SOT 2002
Awarded top five Risk Assessment Presentations at the Society of Toxicology conference (Crump K and Proctor D) by the Risk Assessment Specialty Section (RASS), Nashville, TN.

MANUSCRIPTS

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CONFERENCE SYMPOSIA SESSION CHAIR

2018 ASSOCIATION OF ENVIRONMENTAL HEALTH SCIENCES: Session 5b: The Evolving Risk Assessment Landscape in California.

2017 AMERICAN INDUSTRIAL HYGIENE ASSOCIATION CONFERENCE: Challenges in Protecting Worker Health and Achieving Compliance in the World of Low Submicrogram Concentrations: A Case Study of Beryllium.

2016 SOCIETY OF TOXCIOLOGY: The Cancer Risk Assessment for Ingested Hexavalent Chromium: Challenges and Controversies

2015 SOCIETY OF TOXICOLOGY: Advanced Approaches for Quantitative Risk Assessment Using Human Data with Applications Across Disciplines

2014 TOXICOLOGY AND RISK ASSESSMENT: Using New Data and Methods to Improve the Risk Assessment of Environmental Perchlorate Exposure

2011 SOCIETY OF TOXICOLOGY: Using Mode of Action Data to Guide Quantitative Cancer Risk Assessment: A Case Study of Hexavalent Chromium in Drinking Water

2003 SOCIETY OF TOXICOLOGY: Health Risk Assessment of Hexavalent Chromium in Drinking Water: Carcinogenicity, Research and Regulation.

1996 ASSOCIATION FOR THE ENVIROMENTAL HEALTH OF SOIL: Chromium in Soil: Perspectives in Chemistry, Health and Environmental Regulation.

ABSTRACTS AND PRESENTATIONS

Proctor DM, Vivanco S, Blanchette A. Relative oral bioavailability of manganese in electric arc furnace steel slag is influenced by high iron content and low bioaccessibility. Poster presented at Society of Toxicology Annual Meeting, Nashville, TN, March 2023.

Thompson CM, Wikoff DS, **Proctor DM**, Harris MA. An evaluation of risk assessments on hexavalent chromium [Cr(VI)]: The past, present, and future of mode of action research. Poster presented at Society of Toxicology Annual Meeting, Nashville, TN, March 2023.

Perry C, **Proctor D**. Short-term environmental inhalation toxicity criteria for airborne manganese protective of neurological and respiratory effects for use in air toxics risk assessment. Presentation 5-15.t-04 to Society of Environmental Toxicology and Chemistry, Pittsburgh PA, November 2022.

Proctor D, Mittal L, Vivanco S, Perry C, Blanchette A. Probabilistic health risk assessment for residential exposures to metals in electric arc furnace (EAF) steel slag. Presentation 5.15.P-Th123 to Society of Environmental Toxicology and Chemistry, Pittsburgh PA, November 2022.

Proctor DM, Mittal L, Vivanco S, Antonijevic T. Probabilistic health risk assessment for residential exposures to metals in electric arc furnace (EAF) steel slag. Poster at Society of Environmental Toxicology and Chemistry (SETAC), Philadelphia, PA, November 2022.

Proctor DM, Antonijevic T. Refined health risk assessment for residential exposures to manganese in EAF steel slag. Poster presented at Society of Toxicology Annual Meeting, San Diego, CA, March 2022.

Thompson CM, Chappell GA, Mittal L, Gorman B, **Proctor DM**, Haws LC, Harris MA. Use of targeted mode-of-action research to inform human health risk assessment of hexavalent chromium. Poster presented at Society of Toxicology Annual Meeting, San Diego, CA, March 2022.

Suh M, Verwiel A, **Proctor D**. Oral and inhalation bioaccessibility of cobalt and nickel in metal alloys: A critical consideration for site-specific human health risk assessments and read across. Poster for Society of Toxicology, Virtual Annual Meeting, 2020, https://eventpilotadmin.com/web/page.php?page=Session&project=SOT20&id=P3190.

Proctor D. Use of the latest science in cancer risk assessment for hexavalent chromium: Is it time to step away from the default regulatory approaches? Invited presentation to the International Union of Toxicology (IUTOX) / International Congress of Toxicology (ICT) meeting, Honolulu, HI, June 17, 2019.

Ring CL, Suh M, Casteel S, Dunsmore M, Verwiel A, **Proctor D**. Relative oral bioavailability of cobalt and nickel in residential soil and dust affected by metal grinding operations. Presented at Joint Annual Meeting of International Society of Exposure Science and International Society for Environmental Epidemiology (ISES-ISEE 2018), Ottawa, Canada, August 2018.

Suh M, Wikoff D, Harvey S, Mittal L, Lipworth L, Goodman M, Goodmanson A, Ring C, Rohr A, **Proctor D**. Hexavalent chromium and stomach cancer: A systematic review and meta-analysis. Presented at Joint Annual Meeting of International Society of Exposure Science and International Society for Environmental Epidemiology (ISES-ISEE 2018), Ottawa, Canada, August 2018.

Proctor, DM. Hexavalent chromium in drinking water: When is the science sufficient to deviate from defaults? Invited Speaker, Genetic and Environmental Toxicology Association (GETA). Thresholds in Toxicology and Risk Assessment Fall Symposium. Oakland, CA, November 14, 2018.

Proctor, DM. Updating the regulatory risk assessment for hexavalent chromium in California: Implications for regulatory standards. Association of Environmental Health Sciences San Diego, CA, March 20, 2018.

Thompson CM, Suh M, **Proctor DM**, Harris MA. Ten factors for considering the mode of action of Cr(VI)-induced intestinal tumors in rodents. Society of Toxicology Annual Meeting, San Antonio, TX, March 11-15.

Thompson CM, Wolf JC, Suh M, **Proctor DM**, HJaws LC, Harris MA. Toxicity and recovery in the duodenum of B6C3F1mice following treatment with intestinal carcinogens; captan, folpet, and hexavalent chromium: Evidence for an adverse outcome pathway. Society of Toxicology Annual Meeting, San Antonio, TX, March 11-15.

Proctor DM, Corbett ME. The world of low submicrogram beryllium concentrations. Session F5, American Industrial Hygiene Conference and Exhibition (AIHce), Seattle, WA, June 6, 2017.

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Verwiel A, **Proctor D**, Tachovsky A. Principal component analysis of metals in soil and dust to distinguish background and anthropogenic sources in an urban area. Association for Environmental Health and Sciences Foundation Annual Meeting. San Diego, CA. March 14, 2016.

Verwiel A, **Proctor DM**. Oral bioaccessibility of nickel and cobalt from metal alloy emissions in soil and dust. Society for Risk Analysis Annual Meeting. Arlington, VA, December 7, 2015.

Proctor, DM. Overview of hexavalent chromium mode of action (MOA) and implications for determining safe drinking water concentrations. Naturally occurring compounds of regulatory concern. Groundwater Resources Association Symposium. Garden Grove, CA, November 18, 2015.

Brorby G, Suh M, Bichteler A, **Proctor D**. Use of cluster analysis and homogeneity testing to characterize distributions of exposures among beryllium workers: Tools for developing occupational exposure limits from quantitative risk assessment. 2015 International Society For Exposure Science Annual Meeting. Henderson, NV, October 22, 2015.

Kirman CR, **Proctor DM**, Suh M, Hays S. Reduction of hexavalent chromium by gastric fluids from fed and fasted individuals with applications to toxicokinetic modeling. Presented at the Society of Toxicology's 54th Annual Meeting. San Diego, CA, March 22-26, 2015.

Suh M, Mittal L, Hirsch S, Valdes R, Bartlett C, Rohr A, **Proctor D**. Lung cancer risk in chromate production workers exposed to hexavalent chromium. Presented at the Society of Toxicology's 54th Annual Meeting. San Diego, CA, March 22-26, 2015.

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Thompson CM, Young RR, Suh M, Dinesdurage H, Elbekai R, Harris, MA, Rohr AC, **Proctor DM**. Hexavalent Chromium Does Not Induce Mutations in the Oral Mucosa of Transgenic Big Blue® Rats following Drinking Water Exposures at a Carcinogenic Dose. Presented at the Society of Toxicology's 54th Annual Meeting. San Diego, CA, March 22-26, 2015.

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Proctor DM, Suh M, Tachovsky JA, Abraham L, Hixon JG, Brorby GP, Campleman SL. Cumulative Risk Assessment of Urban Air Toxics: A Pilot Study in San Antonio, TX. Presented at the Society of Toxicology's 53rd Annual Meeting. Phoenix, AZ, March 23-27, 2014.

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Hays SM, Kirman CR, Suh M, **Proctor DM**. Gastric Reduction of Hexavalent Chromium [Cr(VI)] in Fed and Fasted Human Stomach Samples. Presented at the Society of Toxicology's 53rd Annual Meeting. Phoenix, AZ, March 23-27, 2014.

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Kirman C, Thompson C, **Proctor D**, Suh M, Haws L, Harris MA, Hays S. Using PBPK modeling to address diurnal variation and age differences in hexavalent chromium toxicokinetics in humans. Presented at the Society of Toxicology's 52st Annual Meeting. San Antonio, TX March 10-14, 2013.

Thompson C, Kirman C, **Proctor D**, Suh M, Hays S, Haws L, Harris MA. A chronic oral reference dose for hexavalent chromium. Presented at the Society of Toxicology's 52nd Annual Meeting. San Antonio, TX, March 10-14, 2013.

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O'Brien TJ, Hao D, Suh M, **Proctor D**, Thompson CM, Harris MA, Parsons BL, Meyers MB. K-ras codon 12 GGT to GAT mutation is not elevated in the duodenum of mice subchronically exposed to hexavalent chromium in drinking water. Presented at the Society of Toxicology's 51st Annual Meeting. San Francisco, CA, March 11-15, 2012.

Proctor DM, Thompson CM, Suh M, Haws LC, Harris MA. Mode of action for intestinal carcinogenesis of ingested hexavalent chromium in mice. Presented at the Society of Toxicology's 51st Annual Meeting. San Francisco, CA. March 11-15, 2012.

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Haws L, **Proctor D**, Thompson C, Harris M. Research plan to fill data gaps in the mode of action for cancer risk assessment of hexavalent chromium in drinking water. Presented at the Society of Toxicology's 50th Annual Meeting. Washington, DC, March 6-10, 2011.

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Proctor D, Haws L, Tachovsky A, Harris M. Critical Evaluation of the data underlying the USA Today rankings of air quality at schools. Toxicologist. Abstract 1909. Presented at the Society of Toxicology Conference. Salt Lake City, UT, March 2010.

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Hong S, **Proctor D**, Finley B. Assessment of LA sewage spills on Santa Monica Bay beaches. Society of Toxicology 45th Annual Meeting. San Diego, CA, March 2006.

Hong SJ, **Proctor DM**, Finley BL. Exposure to sewage spill-related pathogens at Santa Monica Bay beaches. 4th Society of Environmental Toxicology and Chemistry World Congress and 25th Annual Meeting. Portland, OR, November 2004.

Proctor D. Exposure assessment for perchlorate in milk. Abstract 421. Society of Toxicology 45th Annual Meeting. New Orleans, LA, 2005.

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Proctor D, Cohen E, Leung H, Hays S, Barraj L, Madl A. Exposure assessment for perchlorate in drinking water. Abstract 1754. Society of Toxicology 44th Annual Meeting. Baltimore, MD, 2004.

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Proctor D, Ohanian E. Health risk assessment of hexavalent chromium in drinking water: Carcinogenicity, research and regulation. Symposium Chairman. Abstract 277. Society of Toxicology 42nd Annual Meeting, Salt Lake City, UT, 2003.

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Proctor D, Kelsh M, Lau E, Exuzides A, Cahill J. Analysis beyond publication: Further evaluation of an occupational study of chromium workers. Abstract 318. Society of Epidemiological Research. 2003.

Proctor DM, Su S, Finley BL. Multi-media exposure scenario survey for defining the conceptual site model of a human health risk assessment for a highly urbanized area. Society of Risk Analysis Conference. December 8, 2002.

Shay E, **Proctor D**, Long T. Community response and health risk assessment of a PCB release from a natural gas pipeline rupture. Association for the Environmental Health of Soils. San Diego, CA, March 2000.

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Ann Holbrow Verwiel, M.P.P.

SENIOR MANAGING SCIENTIST

CONTACT INFORMATION

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PROFESSIONAL PROFILE

Ms. Verwiel has more than 20 years of experience in environmental consulting in the areas of human health risk assessment, site assessment, and environmental regulation. Over her career, she has focused on integrating risk assessment into an overall risk management approach to problem definition, investigation, and mitigation. She has successfully applied this approach in negotiations with regulatory agencies and public groups to develop cost-effective investigations, assessments, and mitigation strategies. She has published and presented papers on a wide variety of topics, including probabilistic risk assessment (Monte Carlo analysis), environmental fate and transport of contaminants, and environmental auditing.

Ms. Verwiel has managed and conducted numerous human health risk assessments that addressed a wide variety of chemicals in soil, soil vapor, air, and groundwater. Petroleum, aerospace, electronics, mining, and MGP sites are among some of the most common sites for which she has performed these risk assessments. She has evaluated the chemical signatures, transport mechanisms and ultimate fate, and likely current and future human exposures as key first steps in the health risk evaluation. She has worked to develop investigation strategies and assess exposure to indoor and ambient air, which included evaluating air emission sources, modeling, soil vapor measurements, and indoor/ambient air measurements. At sites where volatile organic compounds (VOCs) are present in the subsurface, she has addressed the potential for a vapor intrusion exposure pathway through modeling and measurement.

Ms. Verwiel has a detailed understanding of a broad cross section of environmental regulations, which she has applied to regulatory impact analyses, environmental compliance, and training programs. She has evaluated potential impacts of new regulations on operating facilities and new developments, assessed compliance at operating facilities with a wide variety of environmental regulations, and developed training materials to help regulatory agencies establish their requirements clearly and help regulated entities comply. She has conducted air toxics analysis to meet the requirements of California Proposition 65, the AB2588 Toxic “Hot Spots” Act, and the California Environmental Quality Act (CEQA).

Ms. Verwiel also has communicated risk to formal public groups, such as Restoration Advisory Boards, as well as the general public, in open meetings and direct written communications. She has worked with regulatory public participation specialists, public affairs officers, and others to develop written summaries and presentation materials to convey complex technical issues to the public. She has provided litigation support for several projects involving disputes between owners and operators, alleged air emissions exposures, and Proposition 65 litigation.

EDUCATION AND DEGREES EARNED

1996 Master of Public Policy, Georgetown University, Washington, DC

1987 B.S., Chemistry, University of California, Irvine

CERTIFICATIONS

OSHA 40-hour training (updated annually since 1987)

OSHA Supervisor training

PROFESSIONAL AFFILIATIONS

American Chemical Society (ACS; member)

Society of Environmental Toxicology and Chemistry (SETAC; member)

Society of Risk Analysis (SRA; member)

SELECTED PROJECT EXPERIENCE

Air Toxics Health Risk Assessments

Prepared a modeling and risk assessment protocol and completed an air toxics human health risk assessment for a metals recycling facility in Ohio to support a RCRA Part B permit. As part of their permit conditions, Ohio EPA required that the operator complete a risk assessment. Results of community air monitoring had previously resulted in evaluation of the facilities' air emission sources. A protocol was developed to achieve concurrence on the plan for emission estimates, modeling, and risk assessment. Going forward, comments on the protocol from Ohio EPA will be incorporated, and then emissions from various handling, storage, and treatment operations will be characterized and used in an air dispersion model (AERMOD) to estimate off-site concentrations in air and potential risk.

Managed a California AB2588 health risk assessment (AB2588 HRA) for a metal forge operation in southern California. This facility was the focus of public interest related to odors being observed in the neighborhood, and air emission sources were discussed in a series of public meetings. A community air monitoring program was also in place in the neighborhood. Mitigation strategies were developed, and as a result of source controls and operating procedure changes, the potential exposures from air emissions were below significance levels.

Managed a California AB2588 health risk assessment (AB2588 HRA) to evaluate emissions from a metal-finishing facility in the South Coast Air Quality Management District (SCAQMD). The SCAQMD also instituted a community air monitoring program to assess off-site impacts from this facility and others in the area that identified localized increases in air concentrations of some metals. An air toxics risk assessment was performed that required generating emission estimates for unique sources, characterizing source operations for a facility that operated

24 hours per day, conducting air dispersion modeling, and completing risk evaluation and comparisons to local monitoring data. All work was performed on an expedited schedule to meet agency enforcement deadlines, and the results were reported during a community meeting.

Developed a risk assessment protocol and emission estimates for an explosives manufacturing facility to support a risk assessment prior to renewal of the RCRA Part B permit application for storage and open burning of explosive wastes. Evaluated various waste materials and combustion by-products to identify emission estimates and toxicity criteria. Used air dispersion modeling to estimate off-site concentrations and estimated potential human health risks for off-site residents, ranchers, and recreators.

Managed evaluation of source material testing for metals (including hexavalent chromium) at various emission sources at a cement manufacturing plant in northern California.

Performed a California AB2588 HRA for a manufacturing facility in northern California, and obtained regulatory approval from the Bay Area Air Quality Management District (BAAQMD), receiving only minimal comments.

Prepared a California AB2588 HRA for a film-processing facility with emissions of PCE and other solvents used in film developing and cleaning processes.

Evaluated chemical emissions from multiple air emission sources at an urban medical center, in support of an Environmental Impact Report (EIR) under CEQA.

Evaluated chemical emissions from multiple emission sources at the University of California – Riverside campus, to support preparation of an EIR for the long-range development plan for the university.

Project manager responsible for evaluating potential worker exposure to vehicle emissions in a proposed subterranean parking garage for a convention center that managed large volumes of material transport requiring diesel-emitting trucks. Findings were used to revise the building design to mitigate potential exposures incurred by workers in the garage.

Led a study to evaluate emissions from neighboring industrial sources and a highway prior to construction of a child-care facility at a food production facility, for the convenience of their employees. Conducted air monitoring to understand concentrations and looked at industrial sources in the vicinity of the food production facility.

Project manager responsible for evaluating potential health effects associated with emissions from an oil drilling operation in a highly urban area of Los Angeles.

Prepared an HHRA for remedial action activities, including dust generation and diesel exhaust, in support of a permit application for a remedial action at a former burn dump and shooting range. Managed development and implementation of an air monitoring plan to document concentrations of particulates and lead during remediation activities for comparison to acceptable levels established in the monitoring plan. Monitoring data were made available to the public electronically, which required rapid assessment of the results and adjustments to remedial activities as necessary.

Vapor Intrusion Risk Assessments

Evaluated PCE in groundwater for potential vapor intrusion to off-site residents. Considered the unique geologic setting of a thick, competent clay layer between groundwater and the surface, which likely serves to mitigate vapor intrusion from groundwater to off-site residences. However, soil gas measurements near a sanitary sewer line detected concentrations of VOCs that complicated the interpretation and required additional evaluation.

Evaluated potential vapor intrusion of petroleum hydrocarbons into a building adjacent to a former gas station planned for use as a daycare center. Soil at the gas station was remediated, but a groundwater plume appeared to extend beneath the building, and because excavation would have affected the integrity of the building, residual

petroleum hydrocarbons remained in soil near and potentially under the building. Multiple rounds of indoor air samples were collected to demonstrate that vapor intrusion was not an issue for this building.

Evaluated vapor intrusion of TCE at two industrial buildings adjacent to a shallow soil vapor source. The buildings were monitored over a period of 2 years, and results demonstrated minimal impacts, with indoor air concentrations below health-based screening levels.

Evaluated potential vapor intrusion of TCE and six other VOCs at 100+ homes in the vicinity of a shallow groundwater plume. Developed an indoor air sampling protocol, health-based screening levels, and letters reporting results to residents. We developed presentations for the lead agency and other regulators to define the scope of the evaluation, results, and conclusions. Continued monitoring at fewer than 10 homes after 5 years, when the extent of TCE in groundwater was formally assessed.

Provided third-party review for a vapor intrusion assessment at a future residential development. Worked with land owner to design a development plan that minimizes potential impacts to new homes. Open spaces and parks were used for areas where vapor intrusion may have been an issue. Homes were located at least 100 feet from these areas.

Lead risk assessor for a vapor intrusion HHRA at a former manufacturing facility redeveloped as a business park in southern California. VOCs, primarily trichloroethylene (TCE), were detected in subsurface soil, groundwater, and soil vapor. Developed an indoor air sampling program, calculated site-specific screening levels, and evaluated off-site migration using soil vapor measurements under regulatory oversight.

Lead risk assessor for a vapor intrusion evaluation at an operating hazardous waste treatment facility with chlorinated solvents present in soil and groundwater both on and off site. Evaluated potential human health risks at nearby residences for on-site workers.

Conducted an indoor air evaluation using multiple lines of evidence to evaluate conditions at a surgical hospital prior to a property transaction. Soil gas, sub-slab soil gas, and indoor air samples were collected simultaneously to provide information for decision making within the time frame of the property transaction.

Conducted an indoor air evaluation at a public building to address potential vapor intrusion issues related to a tetrachloroethene (PCE) plume from a former dry cleaning operation at the site.

Lead risk assessor responsible for evaluating potential human health risks associated with free product on the groundwater table approximately 200 feet below ground surface at a former refinery, and for assessing potential impacts to off-site residents.

Lead risk assessor for an HHRA for a former (UST) site where potential indoor air impacts were the key issue following soil remediation because of residual concentrations of petroleum constituents and 1,2-dichloroethane in groundwater at the site and off-site.

California Proposition 65 Evaluations

Evaluated concentrations of chemical ingredients in lubricant products such as gear oils, greases, and other oils and lubricants, that would require a warning label pursuant to California's Safe Drinking Water and Toxic Enforcement Act of 1986 (commonly referred to as Proposition 65). Developed exposure scenarios relevant to each product group, such as chemical-specific dermal absorption factors, potential incidental ingestion, product-specific density, and product-specific exposure frequencies. Using these exposure parameters, estimated potential exposures to the listed chemicals in the product, to assess whether Proposition 65 notifications were required.

Provided support to legal counsel and their client in the evaluation of potential off-site exposure to diesel exhaust from ski resort operations.

Performed a Proposition 65 evaluation for a metal forge operation in southern California; results demonstrated that notification was not required for off-site residents.

Evaluated potential exposures to lead in a dietary supplement and in a skin product, based on daily use suggested by the product label. Recommended additional analysis to assess bioavailability to more accurately assess exposure.

Sixty-day notices were sent by plaintiffs' attorneys to numerous industrial facilities in California based on the simple listing of a Proposition 65 chemical in their emission inventory reported to local air districts and made publicly available. Assisted several clients by conducting simple evaluations of their emissions, which showed that, under conservative assumptions, specific regulatory levels for the Proposition 65-listed chemicals had not been exceeded.

Evaluated requirement to notify off-site persons potentially exposed to emissions from an industrial facility in southern California. Developed specific regulatory levels when such levels had not been published by the state.

Provided technical support in negotiations with the California Attorney General's office on behalf of a manufacturing facility that was issued a 60-day notice based on erroneous interpretations of a public air toxics risk assessment report.

Developed a Proposition 65 emission calculator for diesel exhaust from construction activities for a client that conducts numerous construction projects every year, to assess whether notification may be required,

Evaluated building materials, furniture, and chemical products at a large child-care facility, to identify Proposition 65-listed chemicals and assess whether Proposition 65 notification may be required.

Estimated potential exposure to cadmium and lead in a food product, including evaluating laboratory data and researching typical consumption patterns.

Multi-Media Environmental Human Health Risk Assessments

Managed the risk assessment planning process for the soil operating unit of a former airport, aircraft maintenance facility, and military manufacturing site. Worked with EPA to attain concurrence on a scoping document for the risk assessment that addressed the major questions regarding the approach to the risk assessment. This allowed the risk assessment to proceed quickly and streamlined EPA's review.

Managed a site-wide HHRA for an active chemical manufacturing facility subject to RCRA under EPA oversight. Chemicals at the site included VOCs, semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), pesticides, dioxin/furans, and inorganics. Key factors included an upgradient contribution of VOCs from an adjacent Superfund site, shallow groundwater (~5 feet below ground surface), redevelopment of a portion of the former site as a regional park, off-site residences 350 feet from the site boundary, a nearby creek, and a variety of source areas.

Managed human health risk assessment activities at a confidential Superfund mining site. Over the last 10 years, participated in the project management team that developed work plans, performed site characterization activities, evaluated nature and extent of affected areas, and developed a baseline human health risk assessment work plan. Unique features of this project included:

- Developed a site conceptual model that incorporated unique receptors, including Native American tribal members and foragers
- Evaluated incremental sampling methods for mine-waste piles
- Conducted bioaccessibility testing for key metals

- Prepared a work plan and collected data to develop site-specific plant uptake factors
- Collected site-specific background data sets for multiple media and calculated statistically based benchmarks for comparison to site data.

Provided technical expertise to Nevada Department of Environmental Protection (NDEP) to review documents related to redevelopment of a former manganese mine. ToxStrategies' responsibility was to review documents related to the assessment of potential human health risk, such as the baseline human health risk assessment work plan, the baseline human health risk assessment, the Remedial Investigation report, and other documents. ToxStrategies provided comments to NDEP, which were forwarded to the responsible party and incorporated into their documents.

Developed cleanup goals for future redevelopment of a former Department of Energy facility that was being decommissioned. ToxStrategies was hired by the developer to assist in evaluating the implications of hundreds of due diligence samples collected in support of the property transaction. Developed site-specific cleanup goals for more than 50 chemicals in soil, soil gas, and/or groundwater and evaluated these data with respect to the cleanup goals. The cleanup goals were also used by the developer to estimate remediation costs and strategies. The project team worked with regulators—including Missouri's Department of Natural Resources and Department of Health and Senior Services—to achieve regulatory concurrence on the cleanup goals and enable the project to move forward.

Managed a human health and ecological risk assessment for an operating lumber mill for on-site impacts in operational areas and off-site impacts in a slough of dioxins and other chemicals. Developed a baseline human health risk assessment (HHRA) and cleanup levels for upland soil and performed the scoping ecological and off-site human health risk assessment to evaluate ecological and human health risks associated with chemicals present in the slough, both of which received regulatory approval. Developed a sediment management strategy to document that conditions in the slough remained protective of aquatic organisms.

Developed a risk assessment approach for the investigation of former ponds believed to have been affected by mine drainage from a nearby mine. Developed a risk-based investigation and risk assessment work plan to evaluate the residual material and assess the effort necessary to mitigate the impacts at the site.

Project Manager responsible for evaluating environmental issues associated with an approximately 1100-acre ranch where wastewater from a nearby pulp and paper mill was used to irrigate specific agricultural fields, resulting in dioxin in the soil. Developed presentation materials for a public meeting and supported various parts of the California Environmental Quality Act (CEQA) process related to future use of the site as a gravel mine, including preparing public information sheets on dioxins.

Performed an HHRA in support of a Remedial Action Workplan (RAW) for two parcels that were formerly part of a larger manufactured gas plant where PAHs and benzene were key chemicals of potential concern (COPCs) in soil, groundwater, and/or indoor air. The HHRA was approved by the California Department of Toxic Substances Control (DTSC), and the RAW was implemented.

Performed an HHRA and developed risk-based remediation goals for future residential or commercial/industrial land use at a former manufacturing site with metals in soil and VOCs in soil vapor, which were approved by DTSC.

Managed a multi-disciplinary project to provide consulting services to the operators of a former fuel storage terminal (the terminal) in the Port of Los Angeles. Performed the HHRA, obtained regulatory concurrence, developed remediation goals, negotiated with the regulatory agency, and provided support to the client's negotiations with the landowner.

Used a risk-based approach to evaluate off-site risk resulting from a groundwater plume that had migrated from a bulk petroleum storage facility beneath an adjacent residential neighborhood. Worked with members of the public

in a formal Restoration Advisory Board (RAB) to refine the existing HHRA Work Plan, perform the risk assessment, and achieve regulatory concurrence.

Managed a multi-phase investigation of petroleum hydrocarbons in soil at a residential development that was discovered after redevelopment. Worked with the City, developer, and numerous regulatory agencies to prioritize investigation needs, conduct a comprehensive investigation, and perform a screening risk assessment. Work was completed in an expedited time frame, and the development was able to move forward.

Managed preparation of an HHRA Work Plan for a jet-fuel plume at a major U.S. airport that focused on current and potential future receptors. Negotiated acceptance of the work plan with property owner, and completed the risk assessment.

Lead risk assessor for a residential development planned adjacent to a former agricultural chemical manufacturing facility (the site) where groundwater had been affected by agricultural chemicals and VOCs. Completed the risk assessment, which was approved by the regulators, within strict time constraints required to obtain approval of development financing by lending agencies.

Lead risk assessor for site characterization activities and subsequent remediation measures related to VOCs in soil gas, VOCs, and hexavalent and total chromium in soil and groundwater at a former metal-plating facility pursuant to a Cleanup and Abatement Order with the Los Angeles Regional Water Quality Control Board (RWQCB).

Managed the health risk assessment components of the evaluation of waste piles at a former mine site. Performed a background comparison and a risk assessment to evaluate site conditions.

PUBLICATIONS

Verwiel A, Racz L, Mittal L, Rish W. 2022. CDC's national report on human exposure to environmental chemicals. SETAC Globe 23(6), https://globe.setac.org/cdc_report_human_exposure_to_chemicals/.

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Verwiel A, Proctor D, Suh M. Glyphosate risk assessment to assess Proposition 65 requirements for pesticide applicators and construction workers: Risk communication case study. Poster for Society of Toxicology, Virtual Annual Meeting, 2020. https://eventpilotadmin.com/web/page.php?page=IntHtml&project=SOT20&id=2097.

Johnson D, Thompson C, **Verwiel A**, Brorby B. Derivation of California Proposition 65 safe harbor levels for nine chemicals. Poster for Society of Toxicology, Virtual Annual Meeting, 2020. https://eventpilotadmin.com/web/page.php?page=IntHtml&project=SOT20&id=2633.

Suh M, **Verwiel A**, Proctor D. Oral and inhalation bioaccessibility of cobalt and nickel in metal alloys: A critical consideration for site-specific human health risk assessments and read across. Poster for Society of Toxicology, Virtual Annual Meeting, 2020, https://eventpilotadmin.com/web/page.php?page=Session&project=SOT20&id=P3190.

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Verwiel, A.H. Assessment of the Risk of Bias in the Evidence Base for Gestational Exposure to TCE and Development of Congenital Heart Defects. Air & Waste Management Association Meeting, Vapor Intrusion, Remediation, and Site Closure. Phoenix, AZ, December. 2018.

Proctor DM, Suh M, Dunsmore D, **Verwiel A**, Casteel S. Bioaccessibility and relative oral bioavailability of cobalt and nickel from metal alloys in soil and dust. Poster presented at Society of Toxicology Annual Meeting. March 15, 2017. Baltimore, MD.

Holbrow Verwiel A. Development of a long-term monitoring solution for dioxins/furans in sediment. Poster Presentation at Dioxin 2010: 30th International Symposium on Halogen Persistent Organic Pollutants, San Antonio, TX. September 12-17, 2010.

Croteau D, Bernhardt T, **Holbrow A**, Conti E, and Ellery B. Site characterization using a dioxin screening method: Former sawmill, California, United States of America. Proceedings of the Dioxin 2008: 28th International Symposium on Halogenated Persistent Organic Pollutants, Birmingham, England. Aug 17-22, 2008. Also published in Organohalogen Compounds, v. 70. 2008.

Connor K, Kelly C, Cheung R, and **Holbrow A**. Risk-based screening values for vapor intrusion pathway and flux chamber data. Society for Risk Analysis 2005 Annual Meeting, Orlando, FL. December 6, 2005.

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Holbrow AM, Nazmi N, Smith JS and Brorby GP. Implementing a risk assessment work plan developed by stakeholder consensus. Presented at the Society for Risk Analysis Conference, Atlanta, GA. December 1999.

Spencer AL, **Holbrow AM** and Graf T. The 'free product' dilemma: Is free-product removal required to achieve site closure? International Petroleum Institute Conference, Albuquerque, NM. Oct. 20–23, 1998.

Marquis SA, Copeland TL and **Holbrow AM**. A site-specific health-based approach for determining groundwater cleanup concentrations - Part I: Advective transport modeling. Presented at Hazmacon '93, San Jose, CA. April 1993.

Copeland TL, **Holbrow AM** and Marquis SA. A site-specific health-based approach for determining groundwater cleanup concentrations - Part II: Vapor emission modeling and risk characterization. Presented at Hazmacon '93, San Jose, CA. April 1993.

Holbrow AM, Copeland TL and Sullivan MJ. Data characterization methods for contaminated soil and the effects on exposure estimates calculated using a Monte Carlo simulation. Presented at Society for Risk Analysis, San Diego, CA. December 1992.

McCullough ML, Dagdigian JV and **Holbrow AM**. Developing air compliance plans. Presented at the Eighth Annual EA Environmental Compliance Conference, San Diego, CA. August 1992.

Connor K, **Holbrow AM** and Copeland TL, and Paustenbach D. Use of quantitative uncertainty analysis in air toxics risk assessment. Presented at the 85th Annual Meeting of the Air and Waste Management Association, Kansas City, MO. June 21–26, 1992.

Connor K, **Holbrow AM** and Copeland TL. Monte Carlo analysis applied to multipathway risk assessment of chlorinated dibenzo-p-dioxins and dibenzofurans. Poster presentation at the Society of Toxicology 1992 Annual Meetings, Seattle.

Continuing Education

1989 UC Irvine, Hazardous Waste Certification Program

ATTACHMENT B

Sample Collection for Oral and Inhalation IVBA Assessment

1.0 Background

Bridgewater mobilized to the Cascade Steel Rolling Mill (CSRM) facility on April 28, 2023 and June 21, 2023 to collect dust samples for oral and inhalation *in vitro* bioaccessibility (IVBA) measurements as part of a Cleaner Air Oregon (CAO) Toxic Air Contaminant Level 4 Health Risk Assessment. Samples were taken from the Toxic Emission Unit (TEU) emission sources or from surrogate representative materials. For example, as part of the CAO emission inventory process DEQ approved the use of a compositional analysis of dust collected at the truck sweep off area to determine TAC emission speciation for scrap handling. Table 1 summarize the TEUs, IVBA evaluations, and samples taken.

Table 1. Summary of samples collected and related emission unit

TEU ID	TEU Description	IVBA	Sample ID
EU-3_RM	Melt Shop Roof Monitor	Arsenic oral	Roof Monitor D/R-01-042823
EU-3_MF	Melt Shop Fugitives	Arsenic oral	
EU-9sh_Main	Main Scrap Handling	Arsenic oral	Truck Sweep Off-01-042823
EU-9sh_Sec	Secondary Scrap Handling	Arsenic oral	
EU-5	Slag Handling	Manganese Inhalation	EAF/LMF Slag-062123
EU-10	Caster Billet Cutting	Manganese Inhalation	Billet Cut Vent D/R-A02-042823
EU-3_RM	Melt Shop Roof Monitor	Manganese Inhalation	Roof Monitor D/R-02-042823
EU-3_MF	Melt Shop Fugitives	Manganese Inhalation	
EU-9sh_Main	Main Scrap Handling	Manganese Inhalation	Truck Sweep Off-02-042823
EU-9sh_Sec	Secondary Scrap Handling	Manganese Inhalation	
EU-18_ATDSiMn	SiMn Alloy Truck Dump	Manganese Inhalation	SiMn Stockpile-062123
EU-18_AULDSiMn	SiMn Alloy Unload to Feeder	Manganese Inhalation	

Sample collection of Billet Cutting Vent, Roof Monitor, and Truck Sweep off locations occurred on April 28, 2023 with EAF/LMF Slag Pile and SiMn Stockpile samples collected on June 21, 2023.

2.0 Sample Collection Methods

At each sampling location, the following procedures were followed:

- Record a general physical description of the material.
- Remove large gross organic materials or rocks from the sample (by sieve or hand).
- Homogenize the remaining sample material in a large sample jar
- Collect a subsample sample of material by filling a 1.0-oz sample jar from the homogenized sample material.
- Place the 1.0-oz of subsample sample material into the primary sample

container, an 8-oz or 16-oz laboratory supplied glass jar. The sample containers were stored in a chilled cooler during and after the subsamples were added. There was one sampling container of aggregated subsamples representing one composite sample at each location.

- Repeat to collect a total of 12 subsamples for Truck Sweep location and 6 subsamples for EAF/LMF Stockpile, SiMn Stockpile, Billet Cutting Vent, and Roof Monitor location.
- For Truck Sweep and Roof Monitor locations, two sample containers (one for inhalation and one for oral bioaccessibility) were filled with the subsamples.
- Transport the sample containers to the laboratory under chain-of-custody (COC) for processing and analysis.

Prior to sample collection, five lab supplied 8-oz wide-mouth glass jars, were labeled on the lid and side with sample IDs (Figure 5).

3.0 Sample Locations

Samples were collected from five locations for oral and/or inhalation bioaccessibility analysis. Some samples were sieved to be sure enough fine particles were available for analysis. Other samples were not sieved as part of sample collection, but all samples were sieved by the laboratory prior to analysis. Photologs of each sample location are attached.

Un-sieved Samples Collection Method:

Billet Cutting Vent – Fugitive emissions from billet cutting are emitted through a vent opening at the west end of the melt shop building. Billet cutting vent dust was collected from six equally spaced locations on the roof. Approximately 1.0-oz of material was collected from each of six roof locations and placed directly into the 8-oz lab-supplied sampling container.

Roof Monitor – Melt shop emissions that are not captured by the baghouses and are emitted through the melt shop roof monitor or other melt shop openings (i.e., Melt Shop Fugitives). Roof monitor dust was collected from six equally spaced locations along the roof monitor. Approximately 1.0-oz of material was collected from each of six sampling locations and placed directly into the 8-oz lab-supplied sampling container.

Truck Sweep Off - Material from the front of the truck sweep off area was collected with a broom and dustpan. Approximately 1.0-oz of fine material from the truck sweep off area was collected from 12 equally spaced locations. Collected material was placed directly into the 16-oz lab-supplied sampling container.

Sieved Sample Collection Method for Stockpiles:
The two stockpile samples were collected in six increments or locations. Two increments equally spaced on the top, middle, and bottom of stockpile. Samples were collected with shovel and placed into 5-gallon buckets. After samples were dry, the sample material was sieved in the field through #4 mesh stainless steel screen and then through a #40 mesh screen into a 5-gallon bucket. Subsamples were collected from sieved material and placed in the composite sample container. Sieves and buckets were decontaminated between samples. Stockpile sample collection followed AP-42, Appendix C-1 Section C.1.3 – Samples from Storage Pile.

EAF/LMF Slag- The EAF/LMF slag is moved out of the melt shop and placed in a pile to cool before being loaded into trucks for offsite processing. Fugitive emissions of EAF/LMF slag occurs during handling of material into or out of the slag pile. EAF/LMF slag was collected in six increments (approximately one gallon of material at each location or subsample) and sieved through two pans. Approximately 1.0-oz from each subsample was directly placed into the 8-oz lab supplied sampling container.

SiMn Stockpile- SiMn stockpile samples were collected in six increments (approximately one gallon of material at each location or subsample) and sieved through two pans. Approximately 1.0-oz from each subsample was directly placed into the 8-oz lab supplied sampling container.

3.1 Sample Quantities and Nomenclature

The sampling approach resulted in seven samples being submitted to the laboratory for further processing.

The samples were named as follows:
Location Dust Residue Indicator-Sample Container Number - Date

Where;
Location = Location of sample collected (example=EAF/LMF Slag)
D/R = Dust residue (if appropriate for sample)
Sample Container Number (as necessary) = 01 or 02. (01 was designated for oral and 02 for inhalation for materials analyzed for both).
Date = Date of sample collection (example=062123 for June 21, 2023)

For example, the sample for the roof monitor was labeled:
Roof Monitor D/R-01-042823

3.2 Sample Container Decontamination

Sampling equipment was decontaminated between sampling locations. Given that all the subsamples were composited into lab supplied containers, there was no need for

extensive decontamination of sieves and buckets between subsamples for the same sampling location. Residual particles were removed from sampling equipment with a clean cloth or brush in between subsamples.

4.0 Laboratory and Sample Analysis

Sample were packaged and shipped under chain of custody procedure to Prima Environmental, Inc., 5070 Robert J. Mathews Parkway, Suite 300, El Dorado Hills, CA, 95762. Five samples were submitted for inhalation bioaccessibility analysis for manganese (see Table 2). Two samples were submitted for oral bioaccessibility analysis for arsenic (Table 2).

Table 2. Summary of samples submitted to the laboratory

Sample ID	Date Sampled	Sample Method	Volume of Sample	Bioaccessibility Analysis	Sample Container
Billet Cutting Vent D/R-A02-042823	04/28/2023	6 Point Composite	6 ounces	Inhalation (Mn)	Glass
Roof Monitor D/R-01-042823	04/28/2023	6 Point Composite	6 ounces	Oral (As)	Glass
Roof Monitor D/R-02-042823	04/28/2023	6 Point Composite	6 ounces	Inhalation (Mn)	Glass
Truck Sweep Off-01-042823	04/28/2023	12 Point Composite	12 ounces	Oral (As)	Glass
Truck Sweep Off-02-042823	04/28/2023	12 Point Composite	12 ounces	Inhalation (Mn)	Glass
EAF/LMF Slag-062123	06/21/2023	6 Point Composite	6 ounces	Inhalation (Mn)	Glass
SiMn Stockpile-062123	06/21/2023	6 Point Composite	6 ounces	Inhalation (Mn)	Glass

Photologs

DUST SAMPLING PHOTOGRAPHS

Sample ID: EAF/LMF Slag-062123
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 1

Description
Looking at EAF/LMF Slag Pile Sampling Location and steam from adding cooling water to hot slag

Photo No. 2

Description
Looking at EAF/LMF Slag Pile Sampling Location and steam from adding cooling water to hot slag

DUST SAMPLING PHOTOGRAPHS

Sample ID: EAF/LMF Slag-062123
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 3

Description
Close up of one of the subsample locations.

Photo No. 4

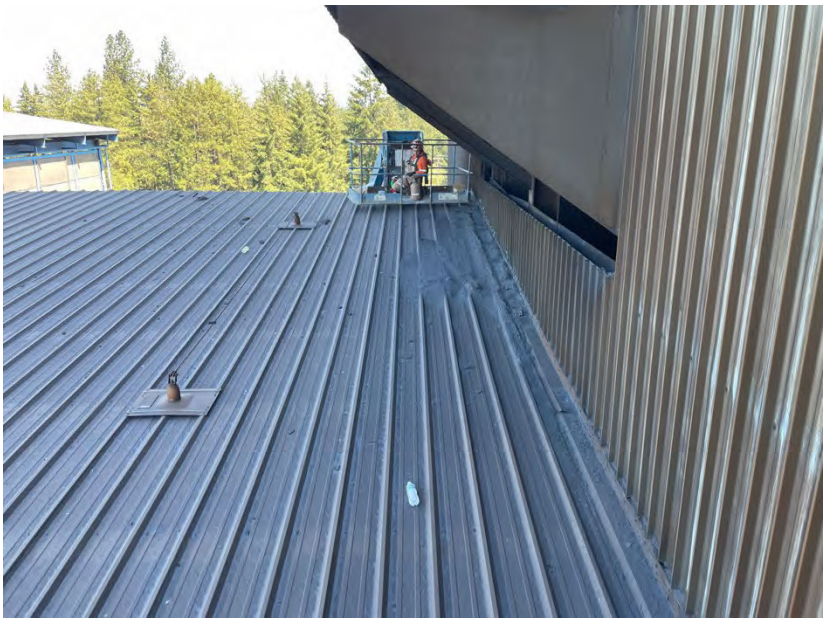
Description
Photo of dried subsample before sieving

DUST SAMPLING PHOTOGRAPHS

Sample ID: Billet Cutting Vent D/R-A02-042823
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 01

Description
Close up of one of the subsample locations.



DUST SAMPLING PHOTOGRAPHS

Sample ID: SiMn Stockpile-062123
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 1

Description
Looking west at SiMn Stockpile Area

Photo No. 02

Description
Closeup of SiMn Stockpile Material.

DUST SAMPLING PHOTOGRAPHS

Sample ID: Roof Monitor D/R-A02-042823
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 01

Description
Looking at Roof Monitor.

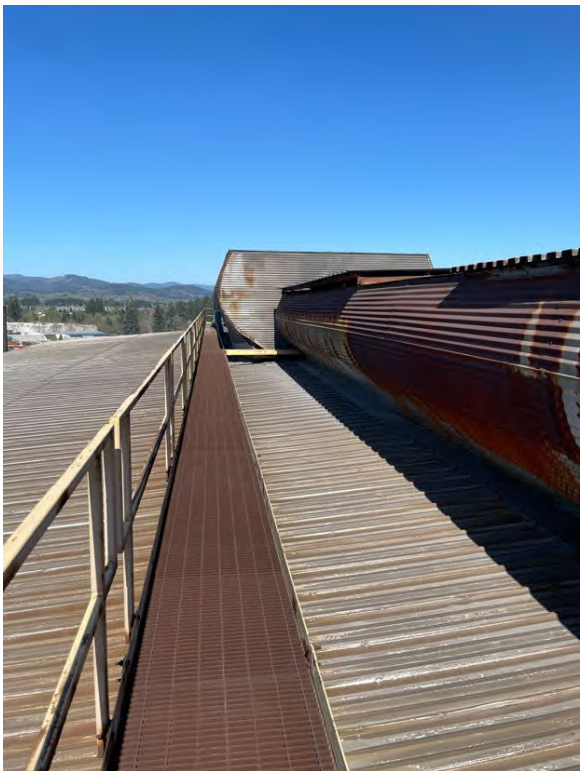
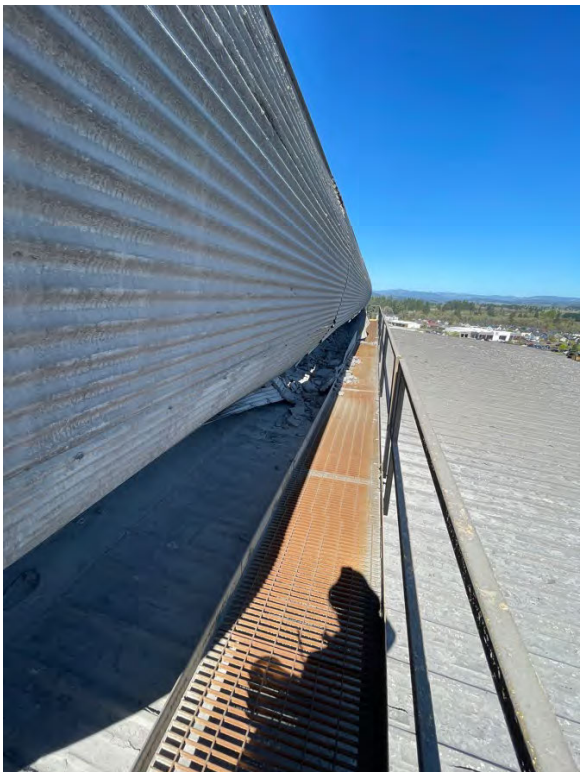


Photo No. 02

Description
Subsample Location 01



DUST SAMPLING PHOTOGRAPHS

Sample ID: Roof Monitor D/R-A02-042823
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 3

Description
Subsample Location 02

Photo No. 4

Description
Subsample Location 03

DUST SAMPLING PHOTOGRAPHS

Sample ID: Roof Monitor D/R-A02-042823
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 5

Description
Subsample Location 04

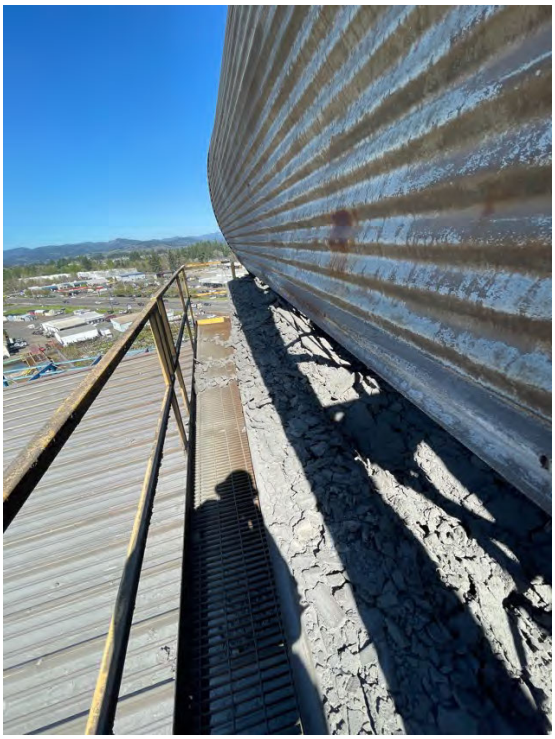


Photo No. 6

Description
Subsample Location 05

DUST SAMPLING PHOTOGRAPHS

Sample ID: Truck Sweep Off D/R-A02-042823
Project Name: Cascade Steel Rolling Mills
Date: 06/21/23

Photo No. 1

Description
Truck Sweep Off Area
Looking South.

Photo No. 02

Description
Truck Sweep Off Area
Looking West.

Field Sampling Sheets

SAMPLING DATA FOR STORAGE PILES

Date Collected 6/21/23 Recorded by J. Pounds

Type of material sampled EAF/LMF Slag Pile

Sampling location* EAF/LMF Slag Pile

METHOD:

1. Sampling device: pointed shovel (hollow sampling tube if inactive pile is to be sampled) Shovel
2. Sampling depth: 0-6 inches
   For material handling of active piles: 10-15 cm (4-6 in.)
   For material handling of inactive piles: 1 m (3 ft)
   For wind erosion samples: 2.5 cm (1 in.) or depth of the largest particle (whichever is less)
3. Sample container: bucket with sealable liner
4. Gross sample specifications: 6 increments Top, middle, bottom of pile
   For material handling of active or inactive piles: minimum of 6 increments with total sample weight of 5 kg (10 lb) [10 increments totalling 23 kg (50 lb) are recommended]
   For wind erosion samples: minimum of 6 increments with total sample weight of 5 kg (10 lb)

Refer to AP-42 Appendix C.1 for more detailed instructions.

Indicate any deviations from the above: No, 6 subsamples or increments combined into 1 composite

SAMPLING DATA COLLECTED:

Sample No.	Time	Location* of Sample Collection	Device Used S/T **	Depth	Mass of Sample
EAF/LMF Slag 06-21-23		Slag Pile	Shovel	0-6"	6- 5gal buckets → 8oz jar

* Use code given of plant or area map for pile/sample identification. Indicate each sampling location on map.

** Indicate whether shovel or tube.

Figure C.1-5. Example data form for storage piles.

SAMPLING DATA FOR STORAGE PILES

Date Collected 6/21/2023 Recorded by J. Pounds

Type of material sampled Si MN stockpile

Sampling location* Si MN stockpile - CSRM

METHOD:

1. Sampling device: pointed shovel (hollow sampling tube if inactive pile is to be sampled)
2. Sampling depth:
   For material handling of active piles: 10-15 cm (4-6 in.)
   For material handling of inactive piles: 1 m (3 ft)
   For wind erosion samples: 2.5 cm (1 in.) or depth of the largest particle (whichever is less)
3. Sample container: bucket with sealable liner
4. Gross sample specifications:
   For material handling of active or inactive piles: minimum of 6 increments with total sample weight of 5 kg (10 lb) [10 increments totalling 23 kg (50 lb) are recommended]
   For wind erosion samples: minimum of 6 increments with total sample weight of 5 kg (10 lb)

Refer to AP-42 Appendix C.1 for more detailed instructions.

Indicate any deviations from the above: Co subsamples or increments collected. Subsample locations distributed from top, middle, and bottom of stockpile (0-6 inches in depth). Material placed in bucket, then sieved

SAMPLING DATA COLLECTED:

Sample No.	Time	Location* of Sample Collection	Device Used S/T **	Depth	Mass of Sample
1-6		Si MN stockpile	Shovel	0-6"	10 lbs per increment

* Use code given of plant or area map for pile/sample identification. Indicate each sampling location on map.

** Indicate whether shovel or tube.

Figure C.1-5. Example data form for storage piles.

SAMPLING DATA FOR PAVED ROADS

Date Collected April 28, 2023 Recorded by JP

Sampling location* Truck Sweep No. of Lanes 0

Surface type (e.g., asphalt, concrete, etc.) concrete

Surface condition (e.g., good, rutted, etc.) rutted

* Use code given on plant or road map for segment identification. Indication sampling location on map.

METHOD:

1. Sampling device: portable vacuum cleaner (whisk broom and dustpan if heavy loading present) broom / dustpan
2. Sampling depth: loose surface material (do not sample curb areas or other untravelled portions of the road) 0-1 inches
3. Sample container: tared and numbered vacuum cleaner bags (bucket with sealable liner if heavy loading present)
4. Gross sample specifications: Vacuum swept samples should be at least 200 g (0.5 lb), with the exposed filter bag weight should be at least 3 to 5 times greater than the empty bag tare weight.

Refer to AP-42 Appendix C.1 for more detailed instructions.

Indicate any deviations from the above: Swept across front of truck sweep area and collect dust

SAMPLING DATA COLLECTED:

Sample No.	Vacuum Bag ID	Vacuum Bag Tare Wgt (g)	Sampling Surface Dimensions (l x w)	Time	Mass of Broom-Swept Sample +
Truck sweep off 02-042823			1' x 80'		1 Loz

\+ Enter "0" if no broom sweeping is performed.

Figure C.1-4. Example data form for paved roads.

ATTACHMENT C

Oral Bioaccessibility Analytical Results

June 16, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: EPA 1340 IVBA for Arsenic and Lead
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the bioaccessibility of arsenic and lead in submitted materials received May 2, 2023.

Procedure. IVBA tests were performed using EPA Method 1340 revision February 2017. Additional information relating to arsenic was found in USEPA Memorandum "Release of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil and Validation Assessment of the In Vitro Arsenic Bioaccessiblity Assay for Predicting Relative Bioavailablity of Arsenic in Soils and Soil-like Materials" dated May 5, 2017. Each sample was air-dried, then sieved to obtain the < 150 micron (< 100 mesh) size fraction required for the IVBA extraction. The entire sample was sieved by hand and the percent < 150 micron (μm) was calculated (**Table 1**). The IVBA tests used 100 ± 0.5 mL of extraction fluid and 1.00 ± 0.05 g sample. The soil was extracted at 37°C for 60 minutes using an end-over-end extraction apparatus. The initial pH of the 0.4 M glycine extraction fluid was 1.5 ± 0.05. The final pH of each extract was within ± 0.5 pH units of the initial pH.

Results. The arsenic and lead concentrations in the extraction solution and in the unextracted sieved soil (< 150 μm fraction) are shown in **Table 2**. The *Bioaccessibility* is given in terms of percent (Eqn. 1), and as the mass of soluble metal per mass of soil (Eqn. 2). Note that the mass of soil is the mass of the *sieved* fraction used in the test, not the mass of bulk soil. The QC results are given in **Table 3**. All QC parameters were within limits, except the LCS NIST 2710a, which was slightly lower than expected (29.8% versus 32.9% for arsenic and 59.9% versus 60.7% for lead); LCS NIST 2711a was within control limits.

$$\text{Bioaccessibility (\%)} = 100 \times \frac{\text{(concentration in extract, mg/L) * (0.1L)}}{\text{(concentration in soil, mg/kg) * (0.001kg)}} \qquad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{\text{(concentration in extract, mg/L) * (volume extract, L)}}{\text{(mass of soil, kg)}} \qquad \text{Eqn. 2}$$

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Percent of Soil Less Than 150 μm (100 mesh).

Sample ID	Mass > 150 μm	Mass < 150 μm	Percent > 150 μm	Percent < 150 μm
	g	g		
Roof Monitor D/R-01-042823	152	54.9	73	27

Note: Samples were sieved by hand, not via mechanical shaker.

Table 2. Results of IVBA Tests.

				Arsenic				Lead Concentration			
PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility		Concentration		Bioaccessibility	
				Sieved Sample	Extraction Fluid			Sieved Sample	Extraction Fluid		
			g	mg/kg	mg/L	%	mg As/kg sample	mg/kg	mg/L	%	mg Pb/kg sample
S1	1-Jun-2023	Roof Monitor D/R-01-042823	0.9972	13	0.073	56	7.3	650	4.5	9.5	61
S1-dup	1-Jun-2023	Roof Monitor D/R-01-042823 dup	0.9831	13	0.065	51	6.6	650	4.5	10	68

^ "Dup" is a duplicate extraction. The soil was extracted twice, but metals in the soil were measured once.

Table 3. QC Data for IVBA Tests.

			Arsenic						Lead					
PRIMA ID	Date	Sample Description	Conc. mg/L	Spike, mg/L	% Recovery	IVBA, %	RPD	Limits	Conc. mg/L	Spike, mg/L	% Recovery	IVBA, %	RPD	Limits
RB	1-Jun-2023	Reagent Blank	< 0.00037	--	--	--	--	< 0.02 mg/L	0.00082 J	--	--	--	--	< 0.050 mg/L
MB	1-Jun-2023	Method Blank	0.00084 J	--	--	--	--	< 0.02 mg/L	0.00061 J	--	--	--	--	< 0.050 mg/L
NIST2710a	1-Jun-2023	LCS (NIST 2710a)^	4.1	--	--	29.8	--	32.9-49.1%	4.1	--	--	59.9	--	60.7-74.2%
NIST2711a	1-Jun-2023	LCS (NIST 2711a)^	0.50			56.9		NE	11.0	--	--	85.8		75.2-96.2%
SPK	1-Jun-2023	Blank - Spike	0.93	1.0	93	--	--	85-115%	0.92	1.0	92	--	--	85-115%
S1	1-Jun-2023	Roof Monitor D/R-01-042823	--	--	--	56	10	+/- 20%	--	--	--	9.5	10	--
S1-dup		Roof Monitor D/R-01-042823 dup				51						10		

^ IVBA limits from EPA Method 1340 and/or US EPA Memorandum " elease of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil and Validation Assessment of the In Vitro Arsenic Bioaccessiblity Assay for Predicting Relative Bioavailablity of Arsenic in Soils and Soil-like Materials" dated May 5, 2017.

RPD = relative percent difference

J = estimated value. The analyte was positively detected; the quantitation is an estimation.

NE = not established

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 5/2/23 10:30

Client/Company: Tox Strat

Project: Bridge IVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐
If no, list number of samples and Sample ID			
Ice present?	☒	☐	☐
If no, what is temperature? ____			
Samples in good condition?	☒	☐	☐
If no, explain:			
Do sample IDs on containers match IDs on COC?	☒	☐	☐
If no, explain:			

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As, Pb	Inhal RBA - Cr6+, Mn	EPA 3050//6020 - Total Pb, As, Cr, and Mn	EPA 3060A/7199 - CrVI					Comments
Roof Monitor D/R-01-042823	4/28/23	1030	S	1	X								

Special Instructions	Relinquished by:		Received by:
[redacted]	Company: Bridgewater Group	Date 5/1/23	Company Prima Env. 5/2/23
	Printed Name: Justin Pounds	Time 10:30	Printed Name Maria Fakhri
	Signature		Signature
	Relinquished by:		Received by:
	Company	Date	Company
	Printed Name	Time	Printed Name
	Signature		Signature

Matrix key: S - soil/sediment; W - water; OT - other

June 16, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: EPA 1340 IVBA for Arsenic and Lead
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the bioaccessibility of arsenic in submitted materials received May 2, 2023.

Procedure. IVBA tests were performed using EPA Method 1340 revision February 2017. Additional information relating to arsenic was found in USEPA Memorandum "Release of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil and Validation Assessment of the In Vitro Arsenic Bioaccessiblity Assay for Predicting Relative Bioavailablity of Arsenic in Soils and Soil-like Materials" dated May 5, 2017. Each sample was air-dried, then sieved to obtain the < 150 micron (< 100 mesh) size fraction required for the IVBA extraction. The entire sample was sieved by hand and the percent < 150 micron (μm) was calculated (**Table 1**). The IVBA tests used 100 ± 0.5 mL of extraction fluid and 1.00 ± 0.05 g sample. The soil was extracted at 37°C for 60 minutes using an end-over-end extraction apparatus. The initial pH of the 0.4 M glycine extraction fluid was 1.5 ± 0.05. The final pH of each extract was within ± 0.5 pH units of the initial pH.

Results. The arsenic and lead concentrations in the extraction solution and in the unextracted sieved soil (< 150 μm fraction) are shown in **Table 2**. The *Bioaccessibility* is given in terms of percent (Eqn. 1), and as the mass of soluble metal per mass of soil (Eqn. 2). Note that the mass of soil is the mass of the *sieved* fraction used in the test, not the mass of bulk soil. The QC results are given in **Table 3**. All QC parameters were within limits, except the LCS NIST 2710a, which was slightly lower than expected (29.8% versus 32.9% for arsenic and 59.9% versus 60.7% for lead); LCS NIST 2711a was within control limits.

$$\text{Bioaccessibility (\%)} = 100 \times \frac{\text{(concentration in extract, mg/L) * (0.1L)}}{\text{(concentration in soil, mg/kg) * (0.001kg)}} \qquad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{\text{(concentration in extract, mg/L) * (volume extract, L)}}{\text{(mass of soil, kg)}} \qquad \text{Eqn. 2}$$

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Percent of Sample Less Than 150 μm (100 mesh).

Sample ID	Mass > 150 μm	Mass < 150 μm	Percent > 150 μm	Percent < 150 μm
	g	g		
Truck Sweep Off-01-042823	196	55.9	78	22

Note: Samples were sieved by hand, not via mechanical shaker.

Table 2. IVBA Results.

				Arsenic			
PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample	Extraction Fluid		
			g	mg/kg	mg/L	%	mg As/kg Sample
S2	1-Jun-2023	Truck Sweep Off-01-042823	1.0092	16	0.016 J	9.9 J	1.6 J
S2-dup	1-Jun-2023	Truck Sweep Off-01-042823 dup	0.9901	16	0.015 J	9.5 J	1.5 J

^ "Dup" is a duplicate extraction. The sample was extracted twice, but metals in the sample were measured once.

J = estimated value

Table 3. QC Data for IVBA Tests.

			Arsenic						Lead					
PRIMA ID	Date	Sample Description	Conc. mg/L	Spike, mg/L	% Recovery	IVBA, %	RPD	Limits	Conc. mg/L	Spike, mg/L	% Recovery	IVBA, %	RPD	Limits
RB	1-Jun-2023	Reagent Blank	< 0.00037	--	--	--	--	< 0.02 mg/L	0.00082 J	--	--	--	--	< 0.050 mg/L
MB	1-Jun-2023	Method Blank	0.00084 J	--	--	--	--	< 0.02 mg/L	0.00061 J	--	--	--	--	< 0.050 mg/L
NIST2710a	1-Jun-2023	LCS (NIST 2710a)^	4.1	--	--	29.8	--	32.9-49.1%	4.1	--	--	59.9	--	60.7-74.2%
NIST2711a	1-Jun-2023	LCS (NIST 2711a)^	0.50			56.9		NE	11.0	--	--	85.8		75.2-96.2%
SPK	1-Jun-2023	Blank - Spike	0.93	1.0	93	--	--	85-115%	0.92	1.0	92	--	--	85-115%
S2	1-Jun-2023	Truck Sweep Off-01-042823	--	--	--	10	4.5	+/- 20%	--	--	--	--	--	--
S2-dup		Truck Sweep Off-01-042823 dup				9.5						--		

^ IVBA limits from EPA Method 1340 and/or US EPA Memorandum " elease of Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil and Validation Assessment of the In Vitro Arsenic Bioaccessiblity Assay for Predicting Relative Bioavailablity of Arsenic in Soils and Soil-like Materials" dated May 5, 2017.

RPD = relative percent difference

J = estimated value. The analyte was positively detected; the quantitation is an estimation.

NE = not established

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 5/2/23 10:30

Client/Company: Tox Strat

Project: Bridge IVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐
If no, list number of samples and Sample ID			
Ice present?	☒	☐	☐
If no, what is temperature? ____			
Samples in good condition?	☒	☐	☐
If no, explain:			
Do sample IDs on containers match IDs on COC?	☒	☐	☐
If no, explain:			

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As	Inhal RBA - Cr6+, Mn	EPA 3050//6020 - Total Pb, As, Cr, and Mn	EPA 3060A/7199 - CrVI						Comments
Truck Sweep Off-01-042823	4/28/23	1258	S	1	X									

Special Instructions

[redacted]

Relinquished by:		Received by:	
Company: Bridgewater Group	Date 5.1.23	Company Prima Env.	5/2/23
Printed Name: Justin Pounds	Time	Printed Name Maria Fakhri	10:30
Signature		Signature Maria Fakhri	
Relinquished by:		**Received by:**	
Company	Date	Company	
Printed Name	Time	Printed Name	
Signature		Signature	

Matrix key: S - soil/sediment; W - water; OT - other

PRIMA ENVIRONMENTAL Inc. Questions Answered.

ATTACHMENT D

Inter-Laboratory Validation of Bioaccessibility Testing for Metals

(Henderson, et al, 2014)

Regulatory Toxicology and Pharmacology 70 (2014) 170–181

Contents lists available at ScienceDirect

Regulatory Toxicology and Pharmacology

journal homepage: www.elsevier.com/locate/yrtph

Inter-laboratory validation of bioaccessibility testing for metals

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ARTICLE INFO

Article history:
Received 9 May 2014
Available online 28 June 2014

Keywords:
Metals
Alloys
UVCBs
Classification
Bioelution
Bioaccessibility
Read-across
Inter-laboratory validation

ABSTRACT

Bioelution assays are fast, simple alternatives to *in vivo* testing. In this study, the intra- and inter-laboratory variability in bioaccessibility data generated by bioelution tests were evaluated in synthetic fluids relevant to oral, inhalation, and dermal exposure. Using one defined protocol, five laboratories measured metal release from cobalt oxide, cobalt powder, copper concentrate, Inconel alloy, leaded brass alloy, and nickel sulfate hexahydrate. Standard deviations of repeatability (s_r) and reproducibility (s_R) were used to evaluate the intra- and inter-laboratory variability, respectively. Examination of the s_R:s_r ratios demonstrated that, while gastric and lysosomal fluids had reasonably good reproducibility, other fluids did not show as good concordance between laboratories. Relative standard deviation (RSD) analysis showed more favorable reproducibility outcomes for some data sets; overall results varied more between- than within-laboratories. RSD analysis of s_r showed good within-laboratory variability for all conditions except some metals in interstitial fluid. In general, these findings indicate that absolute bioaccessibility results in some biological fluids may vary between different laboratories. However, for most applications, measures of relative bioaccessibility are needed, diminishing the requirement for high inter-laboratory reproducibility in absolute metal releases. The inter-laboratory exercise suggests that the degrees of freedom within the protocol need to be addressed.

1. Introduction

As the demand for understanding the potential hazard and risk of chemicals to human health continues to grow, the data required for elucidating these concerns continues to expand as well. Meeting the new and evolving demands of regulatory programs such as the Registration, Evaluation, and Authorization of Chemicals (REACH) regulation in Europe (Regulation (EC) No 1907/2006, 2006) necessitates the generation of new and scientifically robust data on chemical substances, including metals. The *in vivo* testing that would be required to fill these needs is often cost-prohibitive and time-consuming, and also raises concerns with regards to

Abbreviations: CEN, European Committee for Standardization; CLP, classification, labeling and packaging of substances and mixtures regulation; RBA, relative bioavailability; ECHA, European Chemicals Agency; RBALP, relative bioaccessibility leaching procedure; REACH, Registration Evaluation and Authorization of Chemicals; RSD, relative standard deviation; s_r, repeatability standard deviation; s_R, reproducibility standard deviation; UBM, unified BARGE method.

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http://dx.doi.org/10.1016/j.yrtph.2014.06.021
0273-2300/

animal welfare due to the extent of testing potentially required. As such, alternative approaches such as read-across (extrapolation of known data from one substance to another substance) based on structure activity relationships or bioavailability are often encouraged to perform hazard and risk assessment while reducing animal testing (ECHA, 2008, 2013). For most routes of exposure and health endpoints, it is indeed the bioavailability of the metal at the target site in an organism that is the most important factor determining its potential toxicity. Bioaccessibility, referring in this context to the amount of metals released from a given material in fluids designed to mimic those of the human body that may become available for uptake (e.g., synthetic gastric fluid to simulate oral exposure) (Ruby et al., 1999; Henderson et al., 2012), provides a conservative estimate of bioavailability. Bioaccessibility is measured in *in vitro* bioelution assays, whose application to hazard and risk assessment has been increasingly used as an alternative to *in vivo* testing in recent years. Bioaccessibility is a conservative concept because not all metals available will be absorbed or induce damage (effects will depend on dose and metal speciation). Bioaccessibility data are particularly informative, as the presence of a metal does not always impart its biological properties on a given material, for example when the release of the metals and their absorption may be limited due to surface and material properties (e.g., for alloys).

The comparison of bioaccessibility data for two or more forms of the same metal (e.g., a pure metal and an alloy with the same metal constituent) enables an estimate of their relative *in vivo* bioavailability. This type of information can be used in a variety of ways for metals assessment, including: as a tool in determining hazard classification (e.g., using relative bioavailability to determine classification or justifying a derogation because of a lack of bioavailability; ECHA, 2013), to aid in establishing categories of metal substances (grouping; ECHA, 2008), as part of the weight of evidence approach applied in performing read-across (e.g., Henderson et al., 2012), and for risk assessments for exposure to metals required by some consumer product safety regulations (Brock and Stopford, 2003). In addition, relative bioaccessibility can be used to estimate the effective concentration (defined as the bioaccessible concentration of a constituent substance in a complex material) of a metal in a complex material where matrix effects may occur (e.g., alloys) and enable read-across between these materials (Stockmann-Juvala et al., 2013; Hedberg et al., 2013).

The bioaccessibility concept is already incorporated in some standard bioelution test methods and regulatory frameworks, such as the European standard for release of nickel in artificial sweat (BS EN 1811, 2011), ASTM D5517 (2007) for metals in art materials, and BS EN 71-3 (2013) that specifies safety requirements for metals in toys. Bioaccessibility has been listed as a possible approach for complying with information requirements of REACH as part of the chapter on grouping of chemicals (ECHA, 2008).

Method development for – and utilization of – bioelution testing by independent and government research groups have increased. The bioaccessibility approach to estimate metal bioavailability has been applied in recent years to human exposures to metals and minerals in soils, consumer products, and to the evaluation of metal substances (Hillwalker and Anderson, 2014; Henderson et al., 2012; Stopford et al., 2003; Herting et al., 2008; Hedberg et al., 2010; Mazinanian et al., 2013; Oller et al., 2009; Hamel et al., 1998; Vasiluk et al., 2011; Drexler and Brattin, 2007; Wragg et al., 2011; Ellickson et al., 2001; Turner, 2011; Gray et al., 2010; Twining et al., 2005; Hedberg et al., 2013, 2012; Hedberg and Odnevall Wallinder, 2013; Jiang et al., 2012; Guney and Zagury, 2014). In addition, some groups have developed research programs to perform inter-laboratory validation of bioelution methods for specific systems and metals. For example, Drexler and Brattin (2007) reported the outcome of a validation exercise for a method to estimate *in vivo* bioavailability of lead (Pb) from soils. Additionally, a separate group also performed a round-robin study for a different physiologically-based method for estimating the bioaccessibility of Pb, as well as cadmium (Cd) and As, from soils (Wragg et al., 2011). Cordeiro et al. (2012) reported the results of an inter-laboratory comparison of 8 metal releases in comminuted flakes from alkyd resin paints simulating a toy coating using BS EN 71-3 (2013).

Although some groups have sought to standardize specific methods (Drexler and Brattin, 2007; Wragg et al., 2011; Ashley et al., 2012; Cordeiro et al., 2012), generally standardized fluid compositions and testing protocols for the basic bioelution method are lacking. In addition, there are no reference standards to ensure the accuracy of these bioaccessibility results and existing studies have demonstrated that sample characteristics and methodological differences (e.g., temperature, pH, sample loading) can affect the amount of metals released (Stopford et al., 2003; Midander et al., 2006; Hedberg et al., 2013).

The aim of the current study, therefore, was to perform a cross-laboratory testing of different metal-containing materials in select simulated biological fluids that are relevant to characterizing key routes of human exposure, using a defined protocol. To do so, five laboratories measured the release of metal from six different metals and metal-containing materials in synthetic gastric, lysosomal/interstitial, and perspiration fluids (representing oral, inhalation, and dermal routes of exposure, respectively). The results of these bioelution analyses were evaluated by characterizing within-laboratory repeatability and between-laboratory reproducibility measures.

2. Materials and methods

2.1. General study design

The five laboratories participating in the inter-laboratory validation study were Center of Ecotoxicology and Chemistry of Metals, Universidad Adolfo Ibañez (Santiago, Chile), ECTX-Consult (Hasselt, Belgium) with analytical work conducted at Labtium Oy (Finland), Kirby Memorial Health Center (Wilkes-Barre, PA, USA), Oregon State University (Corvallis, Oregon, USA) and KTH Royal Institute of Technology (Stockholm, Sweden). Each laboratory was assigned an identification code of A–E in no specific order and is referred to by its respective coding throughout this manuscript. All labs performed bioaccessibility testing in the following four simulated biological fluids: gastric, lysosomal, interstitial, and perspiration. Labs were asked to follow a Standard Operating Procedure (SOP; dated November 2010) provided and discussed prior to study initiation. In brief, test materials were added to simulated fluids and extracted for a set period of time under standard conditions (e.g., pH, temperature). Following a filtration step, extracts were analyzed and the amounts of metals released into solution were reported. Laboratories measured the release of seven different metals (Cr, Co, Cu, Fe, Ni, Pb and Zn) depending on the composition of the test materials.

2.2. Test materials

The six materials tested are listed in Table 1 with their respective chemical formula, CAS number, metal content, mean particle size, surface area, and supplier. The materials were Co oxide, Co powder, Cu concentrate, Inconel alloy, leaded brass alloy, and Ni sulfate hexahydrate. All test materials were powders with a median particle size <60 μm in diameter representing a size range relevant for oral and dermal exposures. However, although the SOP

Table 1
Description of test materials used in this study: sample ID, CAS number, chemical formula, primary metal content, mean particle size, surface area and supplier.

Test Material	Sample ID	CAS No	Formula	Metal Content (%)[a]	$D_{0.5}$ (m)[b]	SA (m^2/g)[c]	Supplier
Cobalt oxide	C32.10-PTL	1308-06-1	Co_3O_4	Co (73.43)	2.7	0.92	Umicore (Belgium)
Cobalt metal	C23.8-PTL	7440-48-4	Co	Co (99.98)	3.4	2.30	Umicore (USA)
Copper concentrate	908753	N/A	N/A	Cu (23.58)	59.2	0.40	Rio Tinto (Canada)
Inconel alloy	N130.6-PTL	N/A	N/A	Cr (18.3), Fe (14.6), Ni (67.1)	6.1[d]	0.16[d]	Powder Alloy Corporation (USA)
Leaded brass alloy	Wieland Z32-profil	N/A	N/A	Cu (58.45), Pb (3.22), Zn (37.75)	56.2	0.15	Wieland – Werke AG (Germany)
Nickel sulfate hexahydrate	N131.6-PTL	10101-97-0	$NiSO_4{\cdot}6H_20$	Ni (23.07)	12.4[d]	0.91[d]	Sigma-Aldrich (USA)

N/A, not applicable.
[a] Composition information from Certificate of Analysis as provided by supplier. Each metal constituent within a given test material is referred to within the manuscript as "X metal ion in Y test material", e.g., Cu in Cu concentrate.
[b] Particle size measured with laser diffraction as reported by supplier unless otherwise noted; $d_{0.5}$ corresponds to the median particle diameter from the volume (mass) distribution.
[c] Surface area measured by BET gas absorption methodology as reported by supplier unless otherwise noted.
[d] Analysis conducted by Particle Technology Labs, Ltd.

required particles sized <10 μm for testing in interstitial and lysosomal fluids, which are considered to be representative of the respirable fraction, only three samples met this criterion. As Ni sulfate hexahydrate is hygroscopic, the salt agglomerated to a mean particle size of 12.4 μm. However, its particle size is not relevant as it is readily soluble in aqueous solutions. The copper concentrate was ground during the concentration process and the smallest attainable particles were sent to the labs for testing (mean diameter of 59.2 μm). As lead in the leaded brass alloy sample has lubricating properties, additional milling would have likely smeared the particles together. Therefore, a sieve was used to separate the smallest fraction for testing with a mean particle size of 56.2 μm. Laboratories were supplied with 100 g of each test material from the same original batch and samples were tested as received without further grinding or other manipulation to alter particle size.

2.3. Laboratory equipment

In general, laboratories used similar equipment and any major deviations are listed in the Supplemental Online Material. All chemicals used to prepare the test fluids were of analytical grade reagent quality or better unless otherwise stated. Test vessels were inert, chemical resistant, covered Erlenmeyer flasks of 250 mL. All glassware was cleaned by acid soaking for 24 h (10% HNO_3) then rinsed four times in ultrapure water (18.2 MΩ cm) and dried (by air or oven). A thermostated linear shaker (150 rpm; stroke length = 2.54 cm) or a thermostated orbital shaker (171 rpm stroke length = 2.54 cm) was used for agitation. Controlled thermometers with a readability of 0.1 °C and calibrated pH meters with a readability of 0.01 units were utilized. A calibrated micro balance with a readability of 0.01 mg or 0.001 mg was used. For filtration, 0.2 μm membrane filters (e.g., Whatman UNIFLO syringe filters, Pall Acrodisc syringe filters or equivalent filter system), latex- and oil-free syringes, and polypropylene tubes were used.

2.4. Bioaccessibility assays

All fluids and experimental set ups were prepared by each individual laboratory. The compositions and general testing conditions of each of the simulated fluids, including pH, temperature, loading, and extraction duration, are described in Table 2. The use of synthetic gastric fluid (pH 1.5) to represent oral exposure has been used extensively, starting with the Comité Européen de Normalisation standard, Safety of Toys (BS EN 71-3, 2013), which has been adopted in the United States as ASTM D5517 (2007; Standard Method for Determining the Solubility of Metals in Art Materials). Interstitial and lysosomal fluids are used as surrogates for inhalation. Interstitial fluid (pH 7.4), comprised primarily of Gamble's solution, represents fluid deep within the lung and has been used for many years to evaluate a range of materials. In this study, 5% CO_2 in air was used to keep the interstitial fluid test solutions at pH 7.4 ± 0.2. The approach used by each laboratory to maintain this pH varied and is described in the Supplemental Online Material. Simulated lysosomal fluid, which mimics intracellular conditions with a pH of 4.5 similar to that found in lysosomes of alveolar macrophages, was also used (de Meringo et al., 1994; Stopford et al., 2003). Finally, synthetic perspiration (pH 6.5) was used to represent release from test materials on the skin and was prepared according to BS EN 1811 (2011).

Ultrapure water was added to the fluid compositions listed in Table 2 up to a final volume of 1 L. Temperature and pH were measured at the start of each test and fluids were adjusted with HCl or NaOH as necessary to achieve the desired pH. Temperature and pH were also measured in the remaining blank control for each test solution after sampling. All bioaccessibility tests were conducted at 37°C except for tests in synthetic perspiration where a temperature of 30°C was used (BS EN 1811, 2011). Sample loadings were 0.2 and 2.0 g/L for gastric and all other fluids, respectively (Midander et al., 2006; Henderson et al., 2012; Stopford et al., 2003; Turner, 2011).

Extractions in gastric fluid were conducted for 2 h based on an average half time for gastric emptying of 17.7 min and complete emptying of 91 min in human volunteers (Tomlin et al., 1993; Wang et al., 2001). In addition, this duration has been shown to be correlated with acute oral toxicity of nickel compounds in a recent study by Henderson et al. (2012). All other extractions were carried out for 24 h or 168 h to be representative of longer-term exposures. All extractions were prepared and analyzed in triplicate.

Filtered extracts from blank controls and test vessels were analyzed for metal concentrations using ICP-OES, ICP-MS, or AAS (flame or graphite furnace, depending on concentration) as noted in the Supplemental Online Material. Bioaccessibility measurements underwent a Quality Assurance (QA) check and were reported as released μg metal/g sample.

2.5. Quality assurance

Each laboratory generated a comprehensive report, which underwent a QA exercise. A detailed review and comparison between the SOP and the 5 laboratory reports was performed. As part of this review, individual exchanges were held with the labs to address information gaps and confirm data when necessary. Some differences in methodology between labs were noted. As a result of this exercise, some datasets were excluded from statistical analysis.

Table 2
General description of bioaccessibility fluids and protocols.

	Gastric		Lysosomal		Interstitial		Perspiration	
Composition of fluid	Reagent	g/L	Reagent	g/L	Reagent	g/L	Reagent	g/L
	Hydrochloric acid	2.55	Sodium chloride	3.21	Magnesium chloride hexahydrate	0.203	Sodium chloride	5.0
			Sodium hydroxide	6.00	Sodium chloride	6.02	Urea	1.0
			Citric acid	20.8	Potassium chloride	0.298	Lactic acid	1.06
			Calcium chloride dihydrate	0.097	Sodium phosphate	0.142		
			Sodium phosphate heptahydrate	0.179	Sodium sulfate	0.071		
			Sodium sulfate	0.039	Calcium chloride dihydrate	0.368		
			Magnesium chloride hexahydrate	0.106	Sodium acetate trihydrate	0.953		
			Glycine	0.059	Sodium bicarbonate	2.60		
			Sodium citrate dihydrate	0.077	Sodium citrate dihydrate	0.097		
			Sodium tartrate dihydrate	0.090				
			Sodium lactate	0.085				
			Sodium pyruvate	0.086				
			Formaldehyde	1.0 mL				
pH	1.5 ± 0.1		4.7 ± 0.2		7.4 ± 0.2		6.5 ± 0.1	
Temp (°C)	37 ± 1		37 ± 1		37 ± 1		30 ± 1	
Loading (g/L)	0.2		2		2		2	
Time (hours)	2		24, 168		24, 168		24, 168	
Protocol overview	Ten (10.0 ± 0.5) mg of test material was weighed in triplicate into three separate 250 mL Erlenmeyer flasks. Subsequently, 50 mL of extraction fluid was added to each test vessel flask and to one blank control flask. After adjusting for pH, the flasks were covered with a stopper or parafilm, placed into shaker bath, and agitated for 1 h. Flasks were allowed to sit without agitation for one additional hour before sampling.		One hundred (100.0 ± 5.0) mg of test material was weighed in triplicate into three separate 250 mL Erlenmeyer flasks. Subsequently, 50 mL of extraction fluid was added to each test vessel flask and to two blank control flasks. After adjusting for pH, the flasks were covered with a stopper or parafilm, placed into shaker bath, and agitated for 24 or 168 h. After the appropriate extraction time, the test vessels were left to settle for 3–5 min.		One hundred (100.0 ± 5.0) mg of test material was weighed in triplicate into three separate 250 mL Erlenmeyer flasks. Subsequently, 50 mL of extraction fluid was added to each test vessel flask and two blank control flasks. After adjusting for pH, flasks were covered with a stopper or parafilm, placed into a shaker bath, and agitated for 24 or 168 h. To maintain the pH during the extraction at 7.4 ± 0.2, 5% CO_2 was introduced in the test vessel during the test. After the defined extraction time, test vessels were left to settle for 3–5 min.		One hundred (100.0 ± 5.0) mg of test material was weighed in triplicate into three separate 250 mL Erlenmeyer flasks. Subsequently, 50 mL of extraction fluid was added to each test vessel flask and to two blank control flasks. After adjusting for pH, the flasks were covered with a stopper or parafilm, placed into shaker bath without agitation for 24 or 168 h. After the appropriate extraction time, the test vessels were left to settle for 3–5 min.	
	A syringe was used to remove a 10 mL aliquot from each test vessel at a depth of two third of the supernatant. The samples were filtered through a 0.2 μm syringe filter and transferred to tubes for storage of less than one month.							

2.6. Statistical approach

Amounts of released metals that were not reported by the laboratories or were below the respective limit of detection were excluded from any analysis. In addition, any fluid/time point/lab dataset with 2 or more labs reporting results below the limit of detection (<LOD) were excluded from the inter-laboratory validation.

The statistical analysis of the measurement results was based on ISO 5725-2 (1994). According to this method, measurement results obtained in an inter-laboratory study are inspected for consistency by plotting Mandel's h and k statistics and for outliers by application of the Grubbs tests and the Cochran test. A laboratory mean or a within-laboratory standard deviation was marked as a straggler if the outlier test result was significant at the 5% level, and marked as an outlier if the outlier test result was significant at the 1% significance level. Following ISO 5725-2 recommendations, outliers were discarded and stragglers retained unless no other explanations for the outlying observations were found.

Repeatability standard deviation (s_r; within-lab) and reproducibility standard deviation (s_R; between-labs) were used as measurements of precision. The ratio of the repeatability standard deviation and the reproducibility standard deviation (s_R:s_r) of the log-concentration was determined and used as an indicator of the (dis)agreement between the mean results of the laboratories. Ratios up to 3 were considered to represent good agreement, ratios between 3 and 6 to represent fair agreement, and >6 were considered to mean that agreement between the laboratories needed to be improved.

Relative standard deviation (RSD) was used to assess the fluctuations in the data relative to the data mean. Expressed in percentage terms, the formula for RSD is: (sd/mean log concentration) * 100. RSD values and associated thresholds represent an attempt to define absolute levels of acceptable sample-to-sample result variability (repeatability, r) and lab-to-lab result variability (reproducibility, R). Standards for RSD have been developed in the literature in an attempt to define absolute levels of acceptable variability in sample-to-sample measurements. Criteria for the analysis were based on Wragg et al. (2011) and Ashley et al. (2012) who suggest that the RSD for reproducibility should be less than 20%, and Wragg et al. who further suggest that RSD for repeatability should be less than 10%.

3. Results

The five laboratories performed bioaccessibility testing on the same six distinct metal-containing materials in four simulated biological fluids. A total of 70 datasets were generated: seven time

points with up to ten metal/test substance extractions each. However, some datasets were excluded from analyses as described in Section 3.1.

3.1. Data exclusion

3.1.1. Quality control of protocol implementation

Differences in protocol implementation between labs identified as part of the QA exercise (see Section 2.5) are summarized in detail in the Supplemental Online Material. The outcome of this exercise led to exclusion of several fluid/time point/lab datasets from statistical analyses when the identified deviations from the SOP had potential to impact the experimental procedures, as discussed below.

- For synthetic perspiration, both datasets (24 and 168 h) for Lab D were excluded from analyses of perspiration data as this lab reported using a different temperature during extraction (37 °C instead of 30 °C).
- Four of the five labs demonstrated lower Pb values for the 168 h time point in perspiration compared to 24 h. The reported lower values could be due to Pb ion complexation and subsequent precipitation. Indeed two labs reported seeing precipitation with a naked eye. This phenomenon is likely to be associated with pH changes. Labs A and E reported a drift in pH up to 7.7–7.9 after 168 h (no information on pH was provided by Lab D; Lab B reported pH around 6.5). While these effects are related to the underlying chemistry of metal ion dominated by complexation with fluid constituents and subsequent precipitation effects, they introduce a greater source of variability to the assays. The results from multiple labs suggest that this combination of fluid composition, time point, and loading is less suitable to assess the repeatability and reproducibility of bio-elution tests for Pb. Thus Pb from leaded brass alloy at 168 h was not included in this evaluation.
- Lab E reported significant evaporation in many of the test vessels containing interstitial fluid at both time points, with some data points not reported at all due to 100% evaporation. Therefore, Lab E data were not included in analyses of interstitial fluid.
- Release of Ni from Ni compound in interstitial fluid at 168 h was less than that at 24 h for Labs B, C, and D; while Lab E only had one of the triplicate samples reported due to evaporation (data already excluded). Labs A and B reported observations of precipitation with Ni compounds in this fluid at this time point and Lab B reported a pH shift upwards of ~1 unit in some cases. While related to the underlying chemistry of metal ion interactions (as described above for Pb) in this particular fluid, these effects introduce a greater source of variability to the assays. The results from multiple labs suggest that this combination of fluid composition, time point, and loading is not suitable to assess the repeatability and reproducibility of bio-elution tests for Ni from Ni compound, therefore data from 168 h were not included in this evaluation.

3.1.2. Limitations imposed by limits of detection

The LODs varied depending upon the metal, fluid, loading and analytical methodology used (e.g., AAS-flame or AAS-GF) and are provided in the Supplemental Online Material. Since one of the goals of this study was to determine reproducibility of measurements between labs, the variable LODs precluded the possibility of using the measurements that were below the LOD (only the case for the Inconel alloy), either by substituting them with the LOD or replacing them by a fraction of the LOD. Therefore, all measurements <LOD were noted as such and excluded from any statistical analyses.

Table 3
Results of outlier analysis.

Treatment	Laboratory	Metal – test substance	Outlier test
Gastric fluid (2 h)	D	Fe – Inconel alloy 718	Cochran test
Perspiration fluid (24 h)	E	Pb – leaded brass alloy	Single high Grubbs test
	E	Zn – leaded brass alloy	Cochran test
Perspiration fluid (168 h)	A	Cu – copper concentrate	Cochran test
	B	Cr – Inconel alloy 718	Cochran test
	E	Co – cobalt compound	Single high Grubbs test
	E	Zn – leaded brass alloy	Cochran test
Lysosomal fluid (24 h)	C	Ni – Inconel alloy 718	Single low Grubbs test
	C	Zn – leaded brass alloy	Cochran test
Lysosomal fluid (168 h)	C	Cr – Inconel alloy 718	Single low Grubbs test
Interstitial fluid (24 h)	B	Cu – copper concentrate	Cochran test
Interstitial fluid (168 h)	No outliers were detected in this treatment dataset		

Datasets with 2 or more labs reporting results <LOD and therefore excluded from the inter-laboratory validation were only an issue for the release of Fe and Cr from the Inconel alloy: Cr in gastric fluid, Cr and Fe in 24 h perspiration, Fe in 168 h perspiration, Cr and Fe in 24 and 168 h interstitial fluid, and Cr in 24 h lysosomal fluid.

3.1.3. Precision measures and outliers

As illustrated in Table 3, there were a total of 11 outliers identified among all treatments, with at least one outlier present within each treatment except the 168 h extraction of interstitial fluid. Per ISO 5725-2 recommendations, all outliers were discarded from the database prior to subsequent analyses. Retained datasets (number of labs and number of measurements) are summarized in Table 4.

3.2. Results from statistical analyses

3.2.1. Repeatability and reproducibility results

For the retained test substances and treatment fluid conditions, the means and measures of repeatability (s_r) and reproducibility (s_R) of the logarithms of the measurements were calculated and presented under each treatment fluid condition in Table 4. General observations based on intra-laboratory and inter-laboratory measurement variability for each treatment conditions are presented below according to their respective s_r and s_R calculations.

3.2.1.1. Gastric 2 h. Laboratory data for bioaccessibility after 2 h in synthetic gastric fluid were available for all but the Cr from Inconel alloy (Table 4). In this treatment condition, Ni from Ni compound measurements were the least variable within and across labs, with Pb from leaded brass alloy and Co from Co compound also demonstrating relatively low variability for both measures. Iron from the Inconel alloy, a dataset with the fewest bioaccessibility measures for the gastric fluid treatment, demonstrated some of the highest variability for both measures.

Table 4
Results of repeatability and reproducibility analyses.

Metal – test substance	Number of labs	Total measures	Log mean concentration in fluid (μg/g)	S_r	S_R
Gastric (2 h)					
Co – cobalt compound	5	15	2.58	0.033	0.065
Co – cobalt powder	5	15	5.73	0.044	0.272
Cu – copper concentrate	5	15	3.39	0.052	0.174
Cu – leaded brass alloy	4	12	3.42	0.059	0.252
Fe – Inconel alloy 718	3	9	2.21	0.083	0.255
Ni – Inconel alloy 718	5	15	2.14	0.06	0.1
Ni – nickel compound	5	15	5.35	0.009	0.024
Pb – leaded brass alloy	5	15	4.45	0.019	0.068
Zn – leaded brass alloy	5	15	4.73	0.068	0.237
		Average	3.78	0.047	0.161
Perspiration (24 h)					
Co – cobalt compound	4	11	2.18	0.027	0.647
Co – cobalt powder	4	12	4.13	0.043	0.547
Cu – copper concentrate	4	12	2.43	0.024	0.096
Cu – leaded brass alloy	4	12	3.81	0.026	0.518
Ni – Inconel alloy 718	4	12	1.60	0.02	0.107
Ni – nickel compound	4	12	5.34	0.011	0.044
Pb – leaded brass alloy	3	9	3.15	0.02	0.131
Zn – leaded brass alloy	3	9	4.09	0.021	0.399
		Average	3.34	0.024	0.311
Perspiration (168 h)					
Co – cobalt compound	3	9	3.95	0.016	0.166
Co – cobalt powder	4	12	4.38	0.065	0.359
Cr – Inconel alloy 718	3	9	0.55	0.013	0.546
Cu – copper concentrate	3	9	3.27	0.017	0.041
Cu – leaded brass alloy	4	12	3.78	0.059	0.242
Ni – Inconel alloy 718	4	12	1.92	0.021	0.099
Ni – nickel compound	4	12	5.38	0.025	0.057
Zn – leaded brass alloy	3	9	4.35	0.011	0.045
		Average	3.45	0.028	0.194
Lysosomal (24 h)					
Co – Cobalt compound	5	15	4.23	0.013	0.027
Co – cobalt powder	5	15	5.81	0.037	0.369
Cu – copper concentrate	5	15	3.58	0.014	0.077
Cu – leaded brass alloy	5	15	4.72	0.481	0.751
Fe – Inconel alloy 718	5	15	2.12	0.016	0.086
Ni – Inconel alloy 718	4	12	2.14	0.012	0.023
Ni – nickel compound	5	15	5.34	0.013	0.03
Pb – leaded brass alloy	5	15	4.50	0.012	0.034
Zn – leaded brass alloy	4	12	5.29	0.028	0.16
		Average	4.19	0.07	0.173
Lysosomal (168 h)					
Co – cobalt compound	5	15	4.32	0.008	0.051
Co – cobalt powder	5	15	6.01	0.007	0.033
Cr – Inconel alloy 718	4	12	1.77	0.012	0.025
Cu – copper concentrate	5	15	3.95	0.032	0.113
Cu – leaded brass alloy	5	15	5.77	0.018	0.059
Fe – Inconel alloy 718	5	15	2.15	0.019	0.218
Ni – Inconel alloy 718	5	15	2.46	0.009	0.095
Ni – nickel compound	5	15	5.34	0.006	0.024
Pb – leaded brass alloy	5	15	4.54	0.014	0.053
Zn – leaded brass alloy	5	15	5.58	0.016	0.067
		Average	4.19	0.014	0.074
Interstitial (24 h)					
Co – cobalt compound	3	9	3.15	0.029	0.206
Co – cobalt powder	4	12	4.11	0.033	0.434
Cu – copper concentrate	3	9	2.97	0.06	0.388
Cu – leaded brass alloy	4	12	2.77	0.295	0.514
Ni – Inconel alloy 718	3	9	1.36	0.1	0.386
Ni – Nickel compound	4	12	5.07	0.032	0.097
Pb – leaded brass alloy	2	6	1.47	0.422	0.470
Zn – leaded brass alloy	3	9	1.58	0.569	0.920
		Average	2.81	0.193	0.427
Interstitial (168 h)					
Co – cobalt compound	4	12	3.77	0.092	0.266
Co – cobalt powder	4	12	4.43	0.03	0.12
Cu – copper concentrate	4	12	3.21	0.05	0.417
Cu – leaded brass alloy	4	12	3.40	0.068	0.216
Ni – Inconel alloy 718	3	9	1.64	0.036	0.088
Zn – leaded brass alloy	4	12	2.06	0.223	0.424
		Average	3.09	0.083	0.191

3.2.1.2. Perspiration – 24 h. For the bioaccessibility dataset after 24 h in synthetic perspiration fluid, data were retained for all but the Cr and Fe from the Inconel alloy (Table 4). Under these conditions, both Ni-containing test substances and the Cu from Cu concentrate demonstrated a combination of low variability for both the repeatability and reproducibility measures. On the other hand, both Co-containing test substances demonstrated some of the highest variability for both measures under these conditions.

3.2.1.3. Perspiration – 168 h. For the extended 168 h exposure to perspiration fluid, the bioaccessibility data were retained for all but the Fe from Inconel alloy and Pb from leaded brass alloy (Table 4). Again, Ni from the Ni compound demonstrated relatively little variability within and between labs, along with Zn from leaded brass and Cu from Cu concentrate. Similar to the 24 h perspiration treatment, Co from Co powder had a relatively high variability for both measures.

3.2.1.4. Lysosomal – 24 h. With the exception of Cr from the Inconel alloy, bioaccessibility measurement data were retained for all metal/test substance analyses in lysosomal fluid for 24 h (Table 4). The measurement variability within and between labs was relatively low for both Ni-containing test substances, Pb from leaded brass alloy, and the Co from Co compound. In contrast, Co from Co powder and Cu from leaded brass alloy had relatively large s_r and s_R values.

3.2.1.5. Lysosomal – 168 h. Bioaccessibility measurement data were retained for all metal/test substance analyses conducted over the extended 168 h period in lysosomal fluid (Table 4). Under these conditions, the variability in measurements both within and between labs was relatively low for Ni from Ni compound, Cr from Inconel alloy, and Co from Co powder. On the other hand, Fe from the Inconel alloy and Cu from Cu concentrate measurements demonstrated relatively high variability for both measures under these conditions.

3.2.1.6. Interstitial – 24 h. For the bioaccessibility dataset after 24 h in interstitial fluids, data that passed QA check and outlier evaluations were available for all but the Cr and Fe measurements from the Inconel alloy (Table 4). In general, the dataset for this treatment condition was the most variable as it relates to both repeatability and reproducibility. Only Ni from the Ni compound had relatively low variability for both parameters, whereas the three metals measured from the leaded brass alloy sample (Cu, Pb, and Zn) demonstrated some of the highest variability in the overall dataset.

3.2.1.7. Interstitial – 168 h. For the extended 168 h exposure to interstitial fluid, the bioaccessibility data were not retained for four of the 10 metal/test substance analyses, including Cr and Fe measurements from Inconel alloy, as well as Pb from leaded brass alloy and Ni from Ni compound (Table 4). The measurement variability within and between labs was relatively low for Ni from Inconel alloy and the Co from Co powder. In contrast, Zn from leaded brass alloy had relatively large s_r and s_R values.

3.3. s_R:s_r ratio results

As demonstrated in Table 4, the average repeatability standard deviation (s_r) of the log-concentration among all treatment conditions varied slightly (between 0.014 and 0.083), with the exception of interstitial fluid at the 24 h extraction time period. These findings demonstrate good within-lab agreement. However, the between-lab agreement relative to the within-lab agreement was not as satisfactory. This can be illustrated for many of the treatment condition datasets by calculating the ratio of the reproducibility standard deviation (s_R) and the repeatability standard deviation (s_r) of the log-concentration, which was used as an indicator of the agreement/disagreement between the mean results of the laboratories (Table 5). Even after exclusion of measurements obtained outside the SOP (Section 3.1.1) or datasets with more than 2 values below the LOD (Section 3.1.2), the reproducibility standard deviations of log-concentrations for perspiration fluid (24 h and 168 h extraction time) and lysosomal fluid (168 h extraction time) remain very large as compared with the repeatability standard deviations. This is reflected in the high s_R:s_r ratios in several of the metals measurements for these treatment conditions. Based on the criteria used to interpret the s_R:s_r ratio the perspiration treatment, conditions were poorly reproduced between labs. This is especially true at 24 h for Co from Co compound (24.0) and Co powder (12.7), and all three metals (Cu, Pb, and Zn) measured from leaded brass alloy (19.9, 6.6, and 19.0, respectively). There was fair agreement in variability between repeatability and reproducibility measurements under the gastric and long-term lysosomal treatments (average s_R:s_r for all 10 metal/test substance analyses equal to 3.4 and 5.3, respectively), while the average s_R:s_r ratios for interstitial fluids (24 h and 168 h) and the short-term lysosomal treatment indicated good agreement in variability within and between labs (average s_R:s_r for all 10 metal/test substance analyses equal to 2.2, 2.3, and 2.5, respectively).

From the perspective of the metal/test substance analyses, both Ni-containing substances, the three metals from the leaded brass sample (Cu, Zn, Pb), and Cu from the Cu concentrate all displayed fair inter-laboratory agreement (relative to intra-laboratory agreement) across treatment conditions. The remaining metal/test

Table 5
Results of s_R:s_r analysis.

	Gastric - 2 h	Perspiration - 24 h	Perspiration - 168 h	Lysosomal - 24 h	Lysosomal - 168 h	Interstitial - 24 h	Interstitial - 168 h
Metal - Test Substance	s_R:s_r ratio						
Co - Cobalt compound	2.0	24.0	10.4	2.1	6.4	7.1	2.9
Co - Cobalt powder	6.2	12.7	5.5	10.0	4.7	13.2	4.0
Cr - Inconel alloy 718	-	-	42.0	-	2.1	-	-
Cu - Copper concentrate	3.3	4.0	2.4	5.5	3.5	6.5	8.3
Cu - Leaded brass	4.3	19.9	4.1	1.6	3.3	1.7	3.2
Fe - Inconel alloy 718	3.1	-	-	5.4	11.5	-	-
Ni - Inconel alloy 718	1.7	5.4	4.7	1.9	10.6	3.9	2.4
Ni - Nickel compound	2.7	4.0	2.3	2.3	4.0	3.0	-
Pb - Leaded brass alloy	3.6	6.6	-	2.8	3.8	1.1	-
Zn - Leaded brass alloy	3.5	19.0	4.1	5.7	4.2	1.6	1.9
Treatment Averages	3.4	13.0	6.9	2.5	5.3	2.2	2.3

s_R:s_r = between-laboratory variability relative to within-laboratory variability; shaded cells = s_R:s_r ratio exceeds 6, indicating poor agreement between the variability in repeatability and reproducibility.

substance (Fe, Cr) analyses showed poor agreement between repeatability and reproducibility, indicating that the agreement between the laboratories needs to be improved.

3.4. RSD results

Relative standard deviation (RSD) analysis of the log concentration is another way to consider intra- and inter-laboratory measurement variability. This approach examines s_r and s_R measures individually, assessing the fluctuations in the data relative to the log mean. In our study there were only five instances where the standard for repeatability (e.g., 10%) was exceeded (all with metals from the leaded brass sample treated with lysosomal or interstitial fluids) out of a potential 70 treatment + metal/test substance analyses combinations. Fig. 1 demonstrates that with the exception of interstitial, all other fluids have fairly low within-lab variability for the time point shown (<4%). This suggests that measurements were satisfactory based on within-lab variability for all treatment conditions (Table 6).

According to the RSD analysis, the inter-laboratory variability appears to be unacceptable (e.g., >20%) in the interstitial fluid treatment (24 h) for Pb and Zn from the leaded brass alloy, and Ni from the Inconel alloy. Additionally, the RSD analysis indicates very large reproducibility RSD values for Co from Co compound (perspiration, 24 h), Cr from Inconel alloy (perspiration, 168 h), and Zn in leaded brass alloy (interstitial, 168 h). Fig. 2 demonstrates the variability observed between laboratories.

4. Discussion

Bioelution methods have been used extensively as an alternative to *in vivo* testing for evaluation of metals and metal-containing materials over the last 15 years. Existing publications include those evaluating the bioaccessibility of various metals (Co, Ni, Cr, Pb, Zn, Cu, Cd, arsenic, beryllium, manganese, tin, and uranium) from metal compounds, alloys, soils, household dust, welding fumes, and mine waste in various synthetic fluids (Stopford et al., 2003; Stefaniak et al., 2014; Hillwalker and Anderson, 2014; Oller et al., 2009; Hamel et al., 1998; Vasiluk et al., 2011; Drexler and Brattin, 2007; Wragg et al., 2011; Ellickson et al., 2001; Turner, 2011; Gray et al., 2010; Twining et al., 2005; Mazinanian et al., 2013; Hedberg et al., 2013). A series of studies published by the KTH laboratory primarily reported on the bioaccessibility of

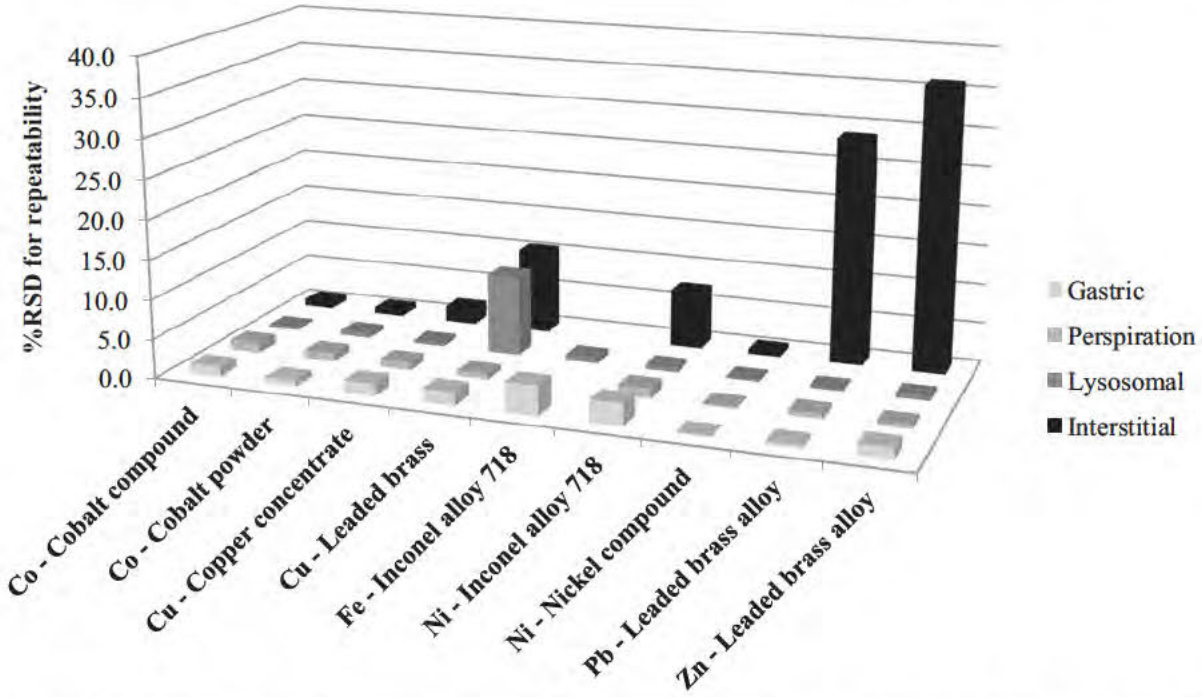

Fig. 1. Within-laboratory variability. All fluids except interstitial fluid have fairly low within-lab variability (<4%) for the time point shown (2 h, gastric; 24 h, all others). %RSD = percent relative standard deviation.

Table 6
Results of relative standard deviation analysis.

	Gastric - 2 h		Perspiration - 24 h		Perspiration - 168 h		Lysosomal - 24 h		Lysosomal - 168 h		Interstitial - 24 h		Interstitial - 168 h	
Metal - Test Substance	%RSD r	%RSD R	%RSD r	%RSD R	%RSD r	%RSD R	%RSD r	%RSD R	%RSD r	%RSD R	%RSD r	%RSD R	%RSD r	%RSD R
Co - Cobalt compound	1.3	2.5	1.2	29.6	0.4	4.2	0.3	0.6	0.2	1.2	0.9	6.5	2.4	7.1
Co - Cobalt powder	0.8	4.7	1.0	13.2	1.5	8.2	0.6	6.4	0.1	0.5	0.8	10.5	0.7	2.7
Cr - Inconel alloy 718	-	-	-	-	2.4	99.6	-	-	0.7	1.4	-	-	-	-
Cu - Copper concentrate	1.5	5.1	1.0	3.9	0.5	1.3	0.4	2.2	0.8	2.9	2.0	13.1	1.6	13.0
Cu - Leaded brass	1.7	7.4	0.7	13.6	1.6	6.4	10.2	15.9	0.3	1.0	10.7	18.6	2.0	6.3
Fe - Inconel alloy 718	3.8	11.6	-	-	-	-	0.8	4.1	0.9	10.1	-	-	-	-
Ni - Inconel alloy 718	2.8	4.7	1.3	6.7	1.1	5.2	0.6	1.1	0.4	3.9	7.4	28.5	2.2	5.4
Ni - Nickel compound	0.2	0.4	0.2	0.8	0.5	1.1	0.2	0.6	0.1	0.4	0.6	1.9	-	-
Pb - Leaded brass alloy	0.4	1.5	0.6	4.2	-	-	0.3	0.8	0.3	1.2	28.7	32.0	-	-
Zn - Leaded brass alloy	1.4	5.0	0.5	9.8	0.3	1.0	0.5	3.0	0.3	1.2	36.1	58.3	10.8	20.6
Treatment Averages	1.2	4.3	0.7	9.3	0.8	5.6	1.7	4.1	0.3	1.8	6.9	15.2	2.7	6.2

%RSD_r = relative standard deviation for repeatability; %RSD_R = relative standard deviation for reproducibility; shaded cells = %RSD exceeds criteria (10% for repeatability and 20% for reproducibility).

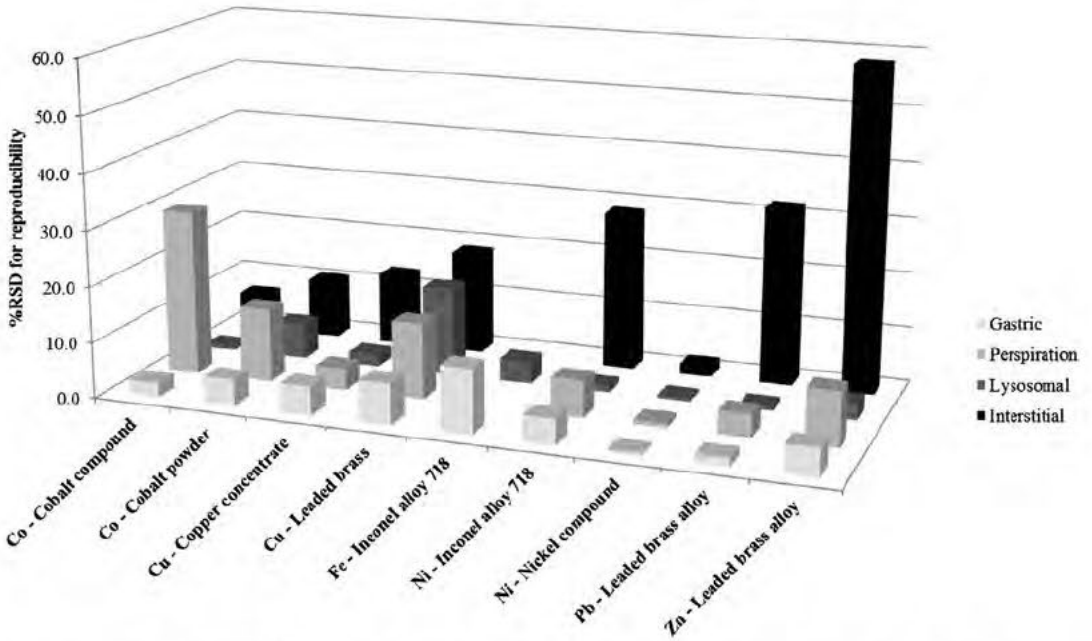

Fig. 2. Between-laboratory variability. Results varied between laboratories depending on the metal and fluid tested. As shown here, gastric and lysosomal fluids had larger reproducibility than other fluids at the time point shown (2 h, gastric; 24 h, all others). %RSD = percent relative standard deviation.

Fe, Cr, and Ni from various alloys and metals (Herting et al., 2008; Hedberg et al., 2010, 2013, 2011; Mazinanian et al., 2013; Midander et al., 2010; Hedberg and Odnevall Wallinder, 2013; Jiang et al., 2012; Stockmann-Juvala et al., 2013).

In recent years, various metals associations have also used bioaccessibility methods to meet regulatory requirements imposed under REACH. Prior to REACH, precedents for the use of bioaccessibility in regulatory frameworks already existed. For example, the European standard for release of nickel in artificial perspiration (BS EN 1811, 2011) has also been incorporated into Europe's classification, labeling and packaging of substances and mixtures regulation (CLP). This regulation stipulates that Ni-containing alloys are classified as dermal sensitizers according to the amount of nickel released (EC, 2008). Another example is the restriction of 19 metals in consumer articles that can be mouthed by children based on the use of EN71.3 (EC, 2013). In the United States, the soluble (bioaccessible) fraction of cadmium in surface coatings of children's jewelry is also restricted (US CPSC, 2008; ASTM F963, 2011).

As evidenced by the number of recent publications on this topic, a variety of fluid compositions and protocols for performing bioaccessibility testing exist. While these are generally similar in nature, it was important in this inter-laboratory study to establish one SOP that could be followed by each of the participating laboratories. The methods and simulated fluids were selected based on their relevance to oral, inhalation and dermal exposure; those previously published by Stopford et al. (2003) served as the basis of developing the SOP.

With regards to gastric fluid, the protocol of ASTM D5517 (2007) was employed for the estimation of metal solubility in the stomach. Synthetic gastric fluid extractions such as this one have been compared with the *in vivo* solubility of lead silicates in the stomach of rats (Ruby et al., 1999) and more recently with the acute oral toxicity in rats exposed to nickel compounds (Henderson et al., 2012). While additional compartments such as saliva and intestinal fluids can be informative in assessing the bioavailability of some metals, these fluids were not included in the present validation program. The ASTM D5517 (2007) protocol was also followed for extractions with simulated interstitial and lysosomal fluids; with the interstitial fluid closely matching Gamble's solution. The interstitial fluid represents lung fluid and uses citrate in place of proteins while acetate is used to represent organic acids. The interstitial fluid has been used to compare the pulmonary durability of inhaled man-made fibers (Zoitos et al., 1997; Lehuede et al., 1997). The solubility of substances that have been phagocytized and subsequently released into the intracellular environment has been estimated using lysosomal fluid (de Meringo et al., 1994; Thélohan et al., 1994). This fluid includes glycine, a variety of salts of organic acids, and citric acid. Citric acid and other organic acids in lysosomal fluid are known to form complexes with metals, resulting in increased release of metals (Hedberg et al., 2010, 2011; Hillwalker and Anderson, 2014). Finally, the synthetic perspiration fluid cited in standard EN 1811 (2011) and approved by the European Committee for Standardization (CEN) in 1998 was used here to simulate the release of soluble metal onto skin. Other compositions for artificial perspiration have also been tested (e.g., Stefaniak et al., 2014). Hillwalker and Anderson (2014) compared the bioaccessibility results from a variety of alloys (stainless steels AISI 304 and 316, Inconel, and Monel (a nickel- and copper-based alloy)) in fluids with slightly different compositions and concluded that Ni and Cr absolute releases from alloys are especially sensitive to fluid composition and extraction time.

In the current study, analyses of repeatability measures using two different approaches (s_R:s_r ratios and RSD) show that the within-laboratory variability was generally satisfactory for all treatment conditions with the exception of some metals in interstitial fluid (Tables 5 and 6). However, variability between laboratories was found to exceed accepted criteria, the extent of which depended on whether the s_R:s_r ratios or the RSD approaches were used. Using the ratio of s_R:s_r, the inter-laboratory concordance for synthetic perspiration was found to be poor overall (ratios > 6; see Table 5). Testing in gastric and 168 h lysosomal fluids resulted in fair agreement between labs (ratios = 3–6), while testing in interstitial and 24 h lysosomal fluids resulted in good agreement in variability within labs (ratios < 3). Similarly, while RSD analysis showed better agreement between laboratories overall, higher inter-laboratory than within-laboratory variability was observed.

A study aimed at evaluating analytical procedures among labs was conducted prior to initiating the present round robin

bioaccessibility study. Samples of interstitial fluid spiked with known metal concentrations (blank, Co, Cu, Ni, Pb, and Zn) were provided (in blind fashion) to each of the laboratories to determine the analytical concentrations. After eliminating outliers, the statistical analysis resulted in an s_R:s_r ratio of about 6, indicating a lack of harmonization among laboratories (data not shown). As a result of this analytical exercise, several recommendations for improving reproducibility were subsequently implemented in the SOP utilized in the bioaccessibility inter-laboratory exercise.

Still, careful comparison of each of the laboratory reports for the round robin revealed that the SOP might not have been precise enough for some parameters (e.g., buffering method). A systematic comparison between the SOP and the reports from the 5 labs also identified a number of methodological differences. For interstitial fluid, the method of CO_2 buffering varied widely among all 5 labs including equipment, location (headspace, fluid, or chamber), and moisturizing gas, etc. Although this is a potential major source of variation, and even though all labs performed this step differently, no clear association between the results for this fluid and any specific lab was identified. Another difference observed between labs was the incidence of evaporation in some fluids. Lab E reported evaporation at 24 h in interstitial fluid while Labs A, B, C, and E reported evaporation over time and difficulty in measuring/maintaining pH in this fluid. Also in interstitial fluid, Lab A noted precipitation with Ni compound and Pb from leaded brass alloy and Lab B reported precipitation with Ni compound. This precipitation may have been due in part to the evaporation taking place in the vessels. Control of pH, particularly in the lysosomal fluid, also presented challenges. This issue was also noted in the unified BARGE method (UBM) study, which concluded that tighter control of pH was critical in gastric fluid (Wragg et al., 2011). Finally, when measurements approach the limit of determination (e.g., <25 μg/g; but even <100 μg/g), the reproducibility outcomes worsened.

Several lessons can be learned from this exercise. The SOP used in this study had too many degrees of freedom as written, and as such, additional details should be incorporated into future drafts. Substances that are being compared (e.g., Cu metal and Cu alloy) should always be tested side-by-side or at least in the same lab. The choice of particle loading is crucial to minimize effects such as agglomeration and abrasion (Hedberg et al., 2010; Henderson et al., 2012; Stopford et al., 2003; Turner, 2011). On the other hand, it is possible that higher sample loadings could overcome the variability associated with low metal releases close to the LOD. In all cases, realistic conditions need to be considered. It might also be useful to measure metal releases over time (e.g., μg/g/h) that can better define the kinetics of metal release (Herting et al., 2008; Hedberg et al., 2010, 2013; Hillwalker and Anderson, 2014; Stefaniak et al., 2014).

Limiting longer exposure times when complicating factors such as CO_2 buffering are introduced may reduce inter-laboratory variability. For example, metal complexation and precipitation and difficulties in maintaining the pH may provide an explanation for the change in repeatability observed between 24 and 168 h in some fluids. In particular, this is an example of why longer time points (168 h) may be pushing the limitations of experimental methods where pH, precipitation, changes in volume, buffering, etc. can all introduce variation. Improvements to the SOP are clearly needed to obtain better within- and between-laboratory agreements. Recommendations for refining the SOP include better defining pH control measures, CO_2 buffering technique, and agitation methods, and ways to minimize evaporation. This is especially true for the interstitial fluid, which stands out as a fluid that requires the most improvement.

It is useful to compare the results of the current study to those of similar inter-laboratory validation studies of specific bioelution methods. In the study of Drexler and Brattin (2007) an *in vitro* relative bioaccessibility leaching procedure (RBALP) designed to mimic oral Pb exposure conditions was performed by three laboratories on 19 different test materials. The results of each lab were subsequently compared to *in vivo* relative bioavailability (RBA) measures. The authors reported that the intra- and inter-laboratory *in vitro* results were "highly reproducible" with a coefficient of variation (e.g., RSD) equal to 6% and 4%, respectively, and concluded that the RBALP method could reliably estimate Pb RBA *in vivo*. Another round-robin study looked at a different physiologically-based method for estimating the bioaccessibility of Pb, as well as Cd and As, from soils (Wragg et al., 2011). The UBM method, which includes synthetic saliva, gastric and intestinal fluids, was used to assess metal release from As, Cd, and Pb samples. Measurements from seven laboratories were compared to *in vivo* RBA data and the overall outcomes were evaluated based on a set of four benchmark criteria. Results of the UBM method were reported to have met the inter-laboratory criteria for As (RSD = 7.43% for stomach phase and 15.72% for stomach + intestine phase). However, compliances for the stomach phase only for Pb (RSD = 22.78%) and stomach plus intestine phases for Cd and Pb (RSD = 35.35% and 81.39%, respectively) were above the benchmark criteria (i.e., RSD $\leqslant$ 20%). The authors suggested that tighter control of gastric pH may be helpful and noted that a follow up inter-laboratory study would be needed.

Using the same RSD criteria the results of the current study appear to be in line with those of Wragg et al. (2011), with the possible exception of interstitial fluid at 24 h (Table 6). In the context of some other studies of similar characteristics it is possible that the criteria used here (RSD $\leqslant$ 10% and $\leqslant$ 20% for intra- and inter-laboratory variability, respectively) may be too stringent. An RSD of 30% or even 40% may be a more realistic cut-off for determining acceptable variation between laboratories. For example, in one study using a saliva migration test for organic plasticizers, where 15 labs performed validation of the SOP, an RSD of 30% was found to be the best obtainable reproducibility (EUR 19826 EN, 2001). Similarly, in a study to validate a method for environmental assessment of metals, Skeaff et al. (2011) reported that the inter-laboratory variability ranged according to analysis by % Coefficient of Variance (%CV; similar to %RSD). In this study, 12/37 measurements had %CV values between 25–56% and 10/37 had values $\geqslant$ 57%. If an RSD of 30% or 40% had been used as the standard for the current study, all between laboratory reproducibility would have been deemed acceptable for all metals and treatment conditions, with the exception of Cr from Inconel alloy in 168 h perspiration fluid and Zn from leaded brass alloy in 24 h interstitial fluid.

The above discussion applies exclusively to estimates of absolute metal release. However, for most applications, only measures of relative metal release from two or more forms (e.g., metal and alloy) of the same metal are needed, diminishing the requirement for high inter-laboratory reproducibility in absolute metal releases. The high within-laboratory repeatability supports the use of these methods for the assessment of relative metal release and calculation of effective concentration of metals in complex materials where a matrix effects can be present.

In the current exercise we included two alloy samples (Inconel and leaded brass alloys) but we did not include the pure metal components of these alloys (e.g., Cr, Fe, Ni in case of Inconel) as reference materials. Thus effective concentrations of metals in these alloys cannot be calculated based on the data from the present round robin. However, two laboratories that participated in this study previously tested the same sample of a Ni metal powder in lysosomal fluid (Mazinanian et al., 2013; KMHC, 2010). Based on the Ni releases from Ni metal and Inconel alloy in 24 h lysosomal

fluid, the effective concentration of Ni in Inconel alloy can be calculated as 0.05 and 0.2%, for Mazinanian et al. (2013) and KMHC (2010), respectively (calculations not shown). Using different Ni metal and Inconel samples, an effective concentration of Ni in Inconel of 0.4% was calculated, based on bioaccessibility data in lysosomal fluid at 72 h reported by Hillwalker and Anderson (2014). In summary, three different laboratories calculated similar effective concentrations of Ni metal in Inconel alloy (relevant to the inhalation route of exposure) even when using different alloys and nickel metal samples and with slightly different absolute releases. The effective concentration of Ni in a SS316 alloy has been recently shown to be a better predictor of *in vivo* inhalation toxicity than its content (Stockmann-Juvala et al., 2013).

In general, this approach could be applied for the classification of alloys based on classifications of their constituent metals. The relative bioaccessibility in gastric, perspiration and lysosomal fluids could allow the calculation of effective concentration of classified metals in alloys and permit more toxicologically relevant classifications when effective concentrations are compared to classification cut-off limits for mixtures. A similar approach could be applied to other complex materials, such as ores and concentrates, where matrix effects are suspected.

5. Conclusion

In conclusion, the outcome of this inter-laboratory validation exercise for bioelution testing of metals demonstrates overall satisfactory within-laboratory variability in bioaccessibility data for synthetic gastric fluid, lysosomal fluid, interstitial fluid, and perspiration fluid for all treatment conditions. With regards to between-laboratory agreement, a higher inter-laboratory than within-laboratory variability in bioaccessibility results was observed for most metals and treatment conditions suggesting that, for the methods tested, the absolute bioaccessibility results in some biological fluids may not always be in line among different laboratories. There are a number of potential sources of variation that may have contributed to this outcome. The most reproducible results were typically observed with shorter extraction times. The inter-laboratory exercise suggests that the degrees of freedom within the SOP need to be addressed to achieve better concordance in absolute metal releases. However, for hazard and risk assessment applications, the use of these methods to generate relative release data for read-across purposes or to calculate effective concentration of metals in alloys and other complex materials appears to be acceptable.

Acknowledgments

We would like to thank the following individuals for their help with data analysis and/or manuscript preparation: Dr. Wendy Hillwalker (OSU) and Dr. Jon Urban (ToxStrategies).

The project was supported by the following: the Nickel Producers Environmental Research Association, Inc. (Durham, NC, USA), the Cobalt Development Institute (Guildford, Surrey UK), the International Zinc Association (Brussels, Belgium), the European Copper Institute (Brussels, Belgium), Eurometaux (Brussels, Belgium) and the Food Safety and Environmental Stewardship Program at OSU.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.yrtph.2014.06.021.

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ATTACHMENT E

Inhalation Bioaccessibility Analytical Results

August 11, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: Inhalation Bioaccessibility, Manganese
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the inhalation bioaccessibility of manganese (Mn) in submitted materials. Inhalation bioaccessibility simulated lysosomal and interstitial conditions following procedures based on those described in Henderson, R.G. et al "Inter-laboratory validation of bioaccessibility testing for metals", *Regulatory Toxicology and Pharmacology*, **70** (2014) 170-181. Procedures and results are described in this letter report.

Sample Receipt and Preparation. One sample – EAF/LMF Slag-062123 – was received on June 27, 2023. It was sieved by hand through a 200 mesh screen to obtain the less than 75 micron (μm) fraction (**Table 1**), which was used for the inhalation bioaccessibility tests.

Materials. All reagents were reagent grade or better quality. Lysosomal and Interstitial extraction fluids were prepared using the recipes presented in Table 2 of Henderson et al. 5% Carbon dioxide/95% Air was obtained from Magnegas.

Procedures. Inhalation bioaccessibility tests using simulated lysosomal and interstitial conditions were run based on methods described in Henderson et al. In this method, the < 75μm fraction of test material (200 mg for lysosomal, 350 mg for interstitial) was extracted with 100 mL lysosomal fluid or 175 mL interstitial fluid for approximately 24 hours or 72 hours at 37° C, after which the extraction fluid was filtered through 0.2 μm filter then submitted to Enthalpy Analytical (Orange, CA) for analysis of Mn. The primary modifications to the Henderson et al method were use of closed HDPE bottles rather than stoppered flasks, end-over-end mixing rather than orbital shaking, and use of large headspace containing 5% CO_2 in air in order to maintain pH in the interstitial tests rather than constant bubbling of CO_2 into each reactor. The pH was monitored periodically and adjusted as needed using hydrochloric acid or sodium hydroxide.

Results. The concentrations of metals in the extraction solution and in the concentration in unextracted sieved test material (< 75 μm fraction) are shown in **Tables 2** and **3 f**or the lysosomal and interstitial tests, respectively. The *Bioaccessibility* is given in terms of percent (**Eqn. 1**), and as the mass of soluble metal per mass of sample (**Eqn. 2**). Note that the mass of test material is the mass of the *sieved* fraction used in the test, not the mass of bulk material. The final pH values of all extracts were within the target range (4.7±0.2 for Lysosomal fluid and 7.4±0.2 for Interstitial fluid).

$$\text{Bioaccessibility (\%)} = \frac{100 \times (\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{concentration in test material, mg/kg}) * (\text{mass of test material, kg})} \quad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{(\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{mass of test material, kg})} \quad \text{Eqn. 2}$$

Quality Control (QC). The QC results are given in **Tables 4 and 5** for lysosomal and interstitial tests, respectively. QC limits have not been established for these tests, but the QC is reasonable and indicates good quality data. Mn, if detected in the reagent blank or method blank, was present at or below 0.04 mg/L, while spike recoveries 88% to 97%.

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Analytes in Test Material.

Sample ID	Mass > 75 µm	Mass < 75 µm	Percent > 75 µm	Percent < 75 µm
	g	g	%	%
EAF/LMF Slag-062123	148	68	69	31

Table 2. Results of Lysosomal Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
L24-1	7/12/2023	EAF/LMF Slag-062123	0.2078	11,000	19	83	9,100
		72 hours					
L72-1	7/14/2023	EAF/LMF Slag-062123	0.2031	11,000	18	81	8,900

* Less than 75 µm fraction.

Table 3. Results of Interstitial Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
I24-1.2	7/24/2023	EAF/LMF Slag-062123	0.3482	11,000	0.83	3.8	420
		72 hours					
I72-1	7/18/2023	EAF/LMF Slag-062123	0.3457	11,000	1.3	6.0	660

* Less than 75 µm fraction.

Table 4. Quality Control - Lysosomal Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
L24-RB	12-Jul-2023	Reagent Blank	0.04 J	--	--	NE
L24-MB	12-Jul-2023	Method Blank	< 0.015	--	--	NE
L24-SPK	12-Jul-2023	Spike	0.88	1.0	88	NE
L72-RB	14-Jul-2023	Reagent Blank	< 0.015	--	--	NE
L72-MB	14-Jul-2023	Method Blank	< 0.015	--	--	NE
L72-SPk	14-Jul-2023	Spike	0.95	1.0	95	NE

NE = not established

"L24" = QC samples associated with Lysosomal 24hr tests.

"L72" = QC samples associated with Lysosomal 72hr test.

J = Estimated value

Table 5. Quality Control - Interstitial Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
I24-RB	17-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I24-MB	17-Jul-2023	Method Blank	0.018 J	--	--	NE
I24-SPK	17-Jul-2023	Spike	0.93	1.0	93	NE
I24-RB2	24-Jul-2023	Reagent Blank	< 0.0081	--	--	NE
I24-MB2	24-Jul-2023	Method Blank	< 0.0081	--	--	NE
I24-SPK2	24-Jul-2023	Spike	0.97	1.0	97	NE
I72-RB	18-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I72-MB	18-Jul-2023	Method Blank	< 0.014	--	--	NE
I72-SPk	18-Jul-2023	Spike	0.90	1.0	90	NE

NE = not established

"I24" = QC samples associated with the Interstitial 24hr tests.

"I72" = QC samples associated with the Interstitial 72hr test.

J = Estimated value

PRIMA ENVIRONMENTAL INC.
Questions Answered.

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 6/27/23 9:40

Client/Company: Tox Strat

Project: Bridge IVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐

If no, list number of samples and Sample ID

Ice present?	☒	☐	☐

If no, what is temperature? _____

Samples in good condition?	☒	☐	☐

If no, explain:

Do sample IDs on containers match IDs on COC?	☒	☐	☐

If no, explain:

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature J. Pounds

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As, Pb	Inhal RBA - Mn	EPA 3050//6020 - Total Pb, As, Cr, and Mn	EPA 3060A/7199 - CrVI						Comments
EAF/LMF Slag-062123	6/21/23	1201	S	1		x								

Special Instructions	Relinquished by:		Received by:
[redacted]	Company: Bridgewater Group	Date 6/25	Company Prima Env. 6/27/23
	Printed Name: Justin Pounds	Time 1000	Printed Name Maria Fakhri 9:40
	Signature		Signature Maria Fakhri
	Relinquished by:		Received by:
	Company	Date	Company
	Printed Name	Time	Printed Name
	Signature		Signature

Matrix key: S - soil/sediment; W - water; OT - other

PRIMA ENVIRONMENTAL Inc.

August 11, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: Inhalation Bioaccessibility, Manganese
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the inhalation bioaccessibility of manganese (Mn) in submitted materials. Inhalation bioaccessibility simulated lysosomal and interstitial conditions following procedures based on those described in Henderson, R.G. et al "Inter-laboratory validation of bioaccessibility testing for metals", *Regulatory Toxicology and Pharmacology*, **70** (2014) 170-181. Procedures and results are described in this letter report.

Sample Receipt and Preparation. One sample – Billet Cutting Vent D/R-A02-042823 – was received on May 2, 2023. It was sieved by hand through a 200 mesh screen to obtain the less than 75 micron (μm) fraction (**Table 1**), which was used for the inhalation bioaccessibility tests.

Materials. All reagents were reagent grade or better quality. Lysosomal and Interstitial extraction fluids were prepared using the recipes presented in Table 2 of Henderson et al. 5% Carbon dioxide/95% Air was obtained from Magnegas.

Procedures. Inhalation bioaccessibility tests using simulated lysosomal and interstitial conditions were run based on methods described in Henderson et al. In this method, the less than 75μm fraction of test material (200 mg for lysosomal, 350 mg for interstitial) was extracted with 100 mL lysosomal fluid or 175 mL interstitial fluid for approximately 24 hours or 72 hours at 37° C, after which the extraction fluid was filtered through 0.2 μm filter then submitted to Enthalpy Analytical (Orange, CA) for analysis of Mn. The primary modifications to the Henderson et al method were use of closed HDPE bottles rather than stoppered flasks, end-over-end mixing rather than orbital shaking, and use of large headspace containing 5% CO_2 in air in order to maintain pH in the interstitial tests rather

than constant bubbling of CO_2 into each reactor. The pH was monitored periodically and adjusted as needed using hydrochloric acid or sodium hydroxide.

Results. The concentrations of metals in the extraction solution and in the concentration in unextracted sieved test material (< 75 μm fraction) are shown in **Tables 2** and **3 f**or the lysosomal and interstitial tests, respectively. The *Bioaccessibility* is given in terms of percent (**Eqn. 1**), and as the mass of soluble metal per mass of sample (**Eqn. 2**). Note that the mass of test material is the mass of the *sieved* fraction used in the test, not the mass of bulk material. The final pH values of all extracts were within the target range (4.7±0.2 for Lysosomal fluid and 7.4±0.2 for Interstitial fluid).

$$\text{Bioaccessibility (\%)} = \frac{100 \times (\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{concentration in test material, mg/kg}) * (\text{mass of test material, kg})} \quad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{(\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{mass of test material, kg})} \quad \text{Eqn. 2}$$

Quality Control (QC). The QC results are given in **Tables 4 and 5** for lysosomal and interstitial tests, respectively. QC limits have not been established for these tests, but the QC is reasonable and indicates good quality data. Mn, if detected in the reagent blank or method blank, was present at or below 0.04 mg/L, while spike recoveries 88% to 97%.

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Analytes in Test Material.

Sample ID	Mass > 75 µm	Mass < 75 µm	Percent > 75 µm	Percent < 75 µm
	g	g	%	%
Billet Cutting Vent D/R A02-042823	261	240	52	48

Samples were sieved by hand.

Table 2. Results of Lysosomal Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
L24-2	7/12/2023	Billet Cutting Vent D/R A02-042823	0.2149	5,500	1.1	9.3	510
L24-2 dup	7/12/2023	Billet Cutting Vent D/R A02-042823	0.2201	5,500	1.1	9.1	500
		72 hours					
L72-2	7/14/2023	Billet Cutting Vent D/R A02-042823	0.1958	5,500	1.1	10	560
L72-2 dup	7/14/2023	Billet Cutting Vent D/R A02-042823	0.1977	5,500	1.1	10	560

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 3. Results of Interstitial Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
I24-2	7/17/2023	Billet Cutting Vent D/R A02-042823	0.3579	5,500	0.040 J	0.36 J	20 J
I24-2 dup	7/17/2023	Billet Cutting Vent D/R A02-042823	0.3568	5,500	0.032 J	0.29 J	16 J
		72 hours					
I72-2	7/18/2023	Billet Cutting Vent D/R A02-042823	0.3505	5,500	0.065 J	0.59 J	32 J
I72-2 dup	7/18/2023	Billet Cutting Vent D/R A02-042823	0.3497	5,500	0.058 J	0.53 J	29 J

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 4. Quality Control - Lysosomal Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
L24-RB	12-Jul-2023	Reagent Blank	0.04 J	--	--	NE
L24-MB	12-Jul-2023	Method Blank	< 0.015	--	--	NE
L24-SPK	12-Jul-2023	Spike	0.88	1.0	88	NE
L72-RB	14-Jul-2023	Reagent Blank	< 0.015	--	--	NE
L72-MB	14-Jul-2023	Method Blank	< 0.015	--	--	NE
L72-SPk	14-Jul-2023	Spike	0.95	1.0	95	NE

NE = not established

"L24" = QC samples associated with Lysosomal 24hr tests.

"L72" = QC samples associated with Lysosomal 72hr test.

J = Estimated value

Table 5. Quality Control - Interstitial Tests.

PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
I24-RB	17-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I24-MB	17-Jul-2023	Method Blank	0.018 J	--	--	NE
I24-SPK	17-Jul-2023	Spike	0.93	1.0	93	NE
I72-RB	18-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I72-MB	18-Jul-2023	Method Blank	< 0.014	--	--	NE
I72-SPk	18-Jul-2023	Spike	0.90	1.0	90	NE

NE = not established

"I24" = QC samples associated with the Interstitial 24hr tests.

"I72" = QC samples associated with the Interstitial 72hr test.

J = Estimated value

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 5/2/23 10:30

Client/Company: Tox Strat

Project: Bridge IVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐
If no, list number of samples and Sample ID			
Ice present?	☒	☐	☐
If no, what is temperature? ____			
Samples in good condition?	☒	☐	☐
If no, explain:			
Do sample IDs on containers match IDs on COC?	☒	☐	☐
If no, explain:			

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As, Pb	Inhal RBA - Cr6+, Mn	EPA 3050//6020 - Total Pb, As, Cr, and Mn	EPA 3060A/7199 - CrVI						Comments
Billet Cutting Vent D/R-A02-042823	4/28/23	1201	S	1		x								

Special Instructions	Relinquished by:		Received by:
[redacted]	Company: Bridgewater Group	Date 5.1.2023	Company Prima Env. 5/2/23
	Printed Name: Justin Pounds	Time 1000	Printed Name Maria Fakhri 10:30
	Signature		Signature
	Relinquished by:		Received by:
	Company	Date	Company
	Printed Name	Time	Printed Name
	Signature		Signature

Matrix key: S - soil/sediment; W - water; OT - other

August 11, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: Inhalation Bioaccessibility, Manganese
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the inhalation bioaccessibility of manganese (Mn) in submitted materials. Inhalation bioaccessibility simulated lysosomal and interstitial conditions following procedures based on those described in Henderson, R.G. et al "Inter-laboratory validation of bioaccessibility testing for metals", *Regulatory Toxicology and Pharmacology*, **70** (2014) 170-181. Procedures and results are described in this letter report.

Sample Receipt and Preparation. One sample – Roof Monitor D/R-02-042823 – was received on May 2, 2023. It was sieved by hand through a 200 mesh screen to obtain the less than 75 micron (μm) fraction (**Table 1**), which was used for the inhalation bioaccessibility tests.

Materials. All reagents were reagent grade or better quality. Lysosomal and Interstitial extraction fluids were prepared using the recipes presented in Table 2 of Henderson et al. 5% Carbon dioxide/95% Air was obtained from Magnegas.

Procedures. Inhalation bioaccessibility tests using simulated lysosomal and interstitial conditions were run based on methods described in Henderson et al. In this method, the < 75μm fraction of test material (200 mg for lysosomal, 350 mg for interstitial) was extracted with 100 mL lysosomal fluid or 175 mL interstitial fluid for approximately 24 hours or 72 hours at 37° C, after which the extraction fluid was filtered through 0.2 μm filter then submitted to Enthalpy Analytical (Orange, CA) for analysis of Mn. The primary modifications to the Henderson et al method were use of closed HDPE bottles rather than stoppered flasks, end-over-end mixing rather than orbital shaking, and use of large headspace containing 5% CO_2 in air in order to maintain pH in the interstitial tests rather

than constant bubbling of CO_2 into each reactor. The pH was monitored periodically and adjusted as needed using hydrochloric acid or sodium hydroxide.

Results. The concentrations of metals in the extraction solution and in the concentration in unextracted sieved test material (< 75 μm fraction) are shown in **Tables 2** and **3 f**or the lysosomal and interstitial tests, respectively. The *Bioaccessibility* is given in terms of percent (**Eqn. 1**), and as the mass of soluble metal per mass of sample (**Eqn. 2**). Note that the mass of test material is the mass of the *sieved* fraction used in the test, not the mass of bulk material. The final pH values of all extracts were within the target range (4.7±0.2 for Lysosomal fluid and 7.4±0.2 for Interstitial fluid).

$$\text{Bioaccessibility (\%)} = \frac{100 \times (\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{concentration in test material, mg/kg}) * (\text{mass of test material, kg})} \quad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{(\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{mass of test material, kg})} \quad \text{Eqn. 2}$$

Quality Control (QC). The QC results are given in **Tables 4 and 5** for lysosomal and interstitial tests, respectively. QC limits have not been established for these tests, but the QC is reasonable and indicates good quality data. Mn, if detected in the reagent blank or method blank, was present at or below 0.04 mg/L, while spike recoveries 88% to 97%.

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Analytes in Test Material.

Sample ID	Mass >75 µm	Mass <75 µm	Percent >75 µm	Percent <75 µm
	g	g	%	%
Roof Monitor D/R-02-042823	146	41	78	22

Samples were sieved by hand.

Table 2. Results of Lysosomal Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
L24-3	7/12/2023	Roof Monitor D/R-02-042823	0.205	12,000	13	53	6,300
L24-3 dup	7/12/2023	Roof Monitor D/R-02-042823	0.2133	12,000	13	51	6,100
		72 hours					
L72-3	7/14/2023	Roof Monitor D/R-02-042823	0.2	12,000	13	54	6,500
L72-3 dup	7/14/2023	Roof Monitor D/R-02-042823	0.1967	12,000	12	51	6,100

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 3. Results of Interstitial Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
I24-3.2	7/24/2023	Roof Monitor D/R-02-042823	0.3526	12,000	0.31	1.28	150
I24-3.2 dup	7/24/2023	Roof Monitor D/R-02-042823	0.3505	12,000	0.26	1.08	130
		72 hours					
I72-3	7/18/2023	Roof Monitor D/R-02-042823	0.3489	12,000	0.42	1.76	210
I72-3 dup	7/18/2023	Roof Monitor D/R-02-042823	0.3542	12,000	0.32	1.32	160

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 4. Quality Control - Lysosomal Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
L24-RB	12-Jul-2023	Reagent Blank	0.04 J	--	--	NE
L24-MB	12-Jul-2023	Method Blank	< 0.015	--	--	NE
L24-SPK	12-Jul-2023	Spike	0.88	1.0	88	NE
L72-RB	14-Jul-2023	Reagent Blank	< 0.015	--	--	NE
L72-MB	14-Jul-2023	Method Blank	< 0.015	--	--	NE
L72-SPk	14-Jul-2023	Spike	0.95	1.0	95	NE

NE = not established

"L24" = QC samples associated with Lysosomal 24hr tests.

"L72" = QC samples associated with Lysosomal 72hr test.

J = Estimated value

Table 5. Quality Control - Interstitial Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
I24-RB	17-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I24-MB	17-Jul-2023	Method Blank	0.018 J	--	--	NE
I24-SPK	17-Jul-2023	Spike	0.93	1.0	93	NE
I24-RB2	24-Jul-2023	Reagent Blank	< 0.0081	--	--	NE
I24-MB2	24-Jul-2023	Method Blank	< 0.0081	--	--	NE
I24-SPK2	24-Jul-2023	Spike	0.97	1.0	97	NE
I72-RB	18-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I72-MB	18-Jul-2023	Method Blank	< 0.014	--	--	NE
I72-SPk	18-Jul-2023	Spike	0.90	1.0	90	NE

NE = not established

"I24" = QC samples associated with the Interstitial 24hr tests.

"I72" = QC samples associated with the Interstitial 72hr test.

J = Estimated value

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 5/2/23 10:30

Client/Company: Tox Strat

Project: Bridge IVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐
If no, list number of samples and Sample ID			
Ice present?	☒	☐	☐
If no, what is temperature? ____			
Samples in good condition?	☒	☐	☐
If no, explain:			
Do sample IDs on containers match IDs on COC?	☒	☐	☐
If no, explain:			

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As, Pb	Inhal RBA - Cr6+, Mn	EPA 3050//6020 - Total Pb, As, Cr, and Mn	EPA 3060A/7199 - CrVI						Comments
Roof Monitor D/R-02-042823	4/28/23	1035	S	1		x								

Special Instructions	Relinquished by:		Received by:
[redacted]	Company: Bridgewater Group	Date 5/1/23	Company Prima Env 5/10 5/2/23
	Printed Name: Justin Pounds	Time 1000	Printed Name Maria Fakhri 10:30
	Signature		Signature
	Relinquished by:		**Received by:**
	Company	Date	Company
	Printed Name	Time	Printed Name
	Signature		Signature

Matrix key: S - soil/sediment; W - water; OT - other

August 11, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: Inhalation Bioaccessibility, Manganese
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the inhalation bioaccessibility of manganese (Mn) in submitted materials. Inhalation bioaccessibility simulated lysosomal and interstitial conditions following procedures based on those described in Henderson, R.G. et al "Inter-laboratory validation of bioaccessibility testing for metals", *Regulatory Toxicology and Pharmacology*, **70** (2014) 170-181. Procedures and results are described in this letter report.

Sample Receipt and Preparation. One sample – Truck Sweep Off-02-042823 – was received on May 2, 2023. It was sieved by hand through a 200 mesh screen to obtain the less than 75 micron (μm) fraction (**Table 1**), which was used for the inhalation bioaccessibility tests.

Materials. All reagents were reagent grade or better quality. Lysosomal and Interstitial extraction fluids were prepared using the recipes presented in Table 2 of Henderson et al. 5% Carbon dioxide/95% Air was obtained from Magnegas.

Procedures. Inhalation bioaccessibility tests using simulated lysosomal and interstitial conditions were run based on methods described in Henderson et al. In this method, the < 75μm fraction of test material (200 mg for lysosomal, 350 mg for interstitial) was extracted with 100 mL lysosomal fluid or 175 mL interstitial fluid for approximately 24 hours or 72 hours at 37° C, after which the extraction fluid was filtered through 0.2 μm filter then submitted to Enthalpy Analytical (Orange, CA) for analysis of Mn. The primary modifications to the Henderson et al method were use of closed HDPE bottles rather than stoppered flasks, end-over-end mixing rather than orbital shaking, and use of large headspace containing 5% CO_2 in air in order to maintain pH in the interstitial tests rather

than constant bubbling of CO_2 into each reactor. The pH was monitored periodically and adjusted as needed using hydrochloric acid or sodium hydroxide.

Results. The concentrations of metals in the extraction solution and in the concentration in unextracted sieved test material (< 75 μm fraction) are shown in **Tables 2** and **3 f**or the lysosomal and interstitial tests, respectively. The *Bioaccessibility* is given in terms of percent (**Eqn. 1**), and as the mass of soluble metal per mass of sample (**Eqn. 2**). Note that the mass of test material is the mass of the *sieved* fraction used in the test, not the mass of bulk material. The final pH values of all extracts were within the target range (4.7±0.2 for Lysosomal fluid and 7.4±0.2 for Interstitial fluid).

$$\text{Bioaccessibility (\%)} = \frac{100 \times (\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{concentration in test material, mg/kg}) * (\text{mass of test material, kg})} \quad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{(\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{mass of test material, kg})} \quad \text{Eqn. 2}$$

Quality Control (QC). The QC results are given in **Tables 4 and 5** for lysosomal and interstitial tests, respectively. QC limits have not been established for these tests, but the QC is reasonable and indicates good quality data. Mn, if detected in the reagent blank or method blank, was present at or below 0.04 mg/L, while spike recoveries 88% to 97%.

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Analytes in Test Material.

Sample ID	Mass > 75 µm	Mass < 75 µm	Percent > 75 µm	Percent < 75 µm
	g	g	%	%
Truck Sweep Off-02-042823	225	31	88	12

Samples were sieved by hand.

Table 2. Results of Lysosomal Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
L24-4	7/12/2023	Truck Sweep Off-02-042823	0.2108	4,900	6.1	59	2,900
L24-4 dup	7/12/2023	Truck Sweep Off-02-042823	0.2142	4,900	6.1	58	2,800
		72 hours					
L72-4	7/14/2023	Truck Sweep Off-02-042823	0.2041	4,900	6.1	61	3,000
L72-4 dup	7/14/2023	Truck Sweep Off-02-042823	0.2042	4,900	6.1	61	3,000

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 3. Results of Interstitial Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
I24-4	7/17/2023	Truck Sweep Off-02-042823	0.3513	4,900	0.28	2.85	140
I24-4 dup	7/17/2023	Truck Sweep Off-02-042823	0.3509	4,900	0.22	2.24	110
		72 hours					
I72-4	7/17/2023	Truck Sweep Off-02-042823	0.3476	4,900	0.57	5.86	290
I72-4 dup	7/17/2023	Truck Sweep Off-02-042823	0.3484	4,900	0.51	5.23	260

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 4. Quality Control - Lysosomal Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
L24-RB	12-Jul-2023	Reagent Blank	0.04 J	--	--	NE
L24-MB	12-Jul-2023	Method Blank	< 0.015	--	--	NE
L24-SPK	12-Jul-2023	Spike	0.88	1.0	88	NE
L72-RB	14-Jul-2023	Reagent Blank	< 0.015	--	--	NE
L72-MB	14-Jul-2023	Method Blank	< 0.015	--	--	NE
L72-SPk	14-Jul-2023	Spike	0.95	1.0	95	NE

NE = not established

"L24" = QC samples associated with Lysosomal 24hr tests.

"L72" = QC samples associated with Lysosomal 72hr test.

J = Estimated value

Table 5. Quality Control - Interstitial Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
I24-RB	17-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I24-MB	17-Jul-2023	Method Blank	0.018 J	--	--	NE
I24-SPK	17-Jul-2023	Spike	0.93	1.0	93	NE
I24-RB2	24-Jul-2023	Reagent Blank	< 0.0081	--	--	NE
I24-MB2	24-Jul-2023	Method Blank	< 0.0081	--	--	NE
I24-SPK2	24-Jul-2023	Spike	0.97	1.0	97	NE
I72-RB	18-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I72-MB	18-Jul-2023	Method Blank	< 0.014	--	--	NE
I72-SPk	18-Jul-2023	Spike	0.90	1.0	90	NE

NE = not established

"I24" = QC samples associated with the Interstitial 24hr tests.

"I72" = QC samples associated with the Interstitial 72hr test.

J = Estimated value

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 5/2/23 10:30

Client/Company: Tox Strat

Project: Bridge IVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐
If no, list number of samples and Sample ID			
Ice present?	☒	☐	☐
If no, what is temperature? ____			
Samples in good condition?	☒	☐	☐
If no, explain:			
Do sample IDs on containers match IDs on COC?	☒	☐	☐
If no, explain:			

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As, Pb	Inhal RBA - Cr6+, and, Mn	EPA 3050//6020 - Total As, Cr, and Mn	EPA 3060A/7199 - CrVI						Comments
Truck Sweep Off-02-042823	4/28/23	1259	S	1		x								

Special Instructions	Relinquished by:		Received by:
[redacted]	Company: Bridgewater Group	Date 5.1.23	Company Prima Env. 5/2/23
	Printed Name: Justin Pounds	Time 1000	Printed Name Maria Fakhri 10:30
	Signature		Signature Maria Fakhri
	Relinquished by:		**Received by:**
	Company	Date	Company
	Printed Name	Time	Printed Name
	Signature		Signature

Matrix key: S - soil/sediment; W - water; OT - other

August 11, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: Inhalation Bioaccessibility, Manganese
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the inhalation bioaccessibility of manganese (Mn) in submitted materials. Inhalation bioaccessibility simulated lysosomal and interstitial conditions following procedures based on those described in Henderson, R.G. et al "Inter-laboratory validation of bioaccessibility testing for metals", *Regulatory Toxicology and Pharmacology*, **70** (2014) 170-181. Procedures and results are described in this letter report.

Sample Receipt and Preparation. One sample – SiMn Stockpile-062123 – was received on June 27, 2023. It was sieved by hand through a 200 mesh screen to obtain the less than 75 micron (μm) fraction (**Table 1**), which was used for the inhalation bioaccessibility tests.

Materials. All reagents were reagent grade or better quality. Lysosomal and Interstitial extraction fluids were prepared using the recipes presented in Table 2 of Henderson et al. 5% Carbon dioxide/95% Air was obtained from Magnegas.

Procedures. Inhalation bioaccessibility tests using simulated lysosomal and interstitial conditions were run based on methods described in Henderson et al. In this method, the < 75μm fraction of test material (200 mg for lysosomal, 350 mg for interstitial) was extracted with 100 mL lysosomal fluid or 175 mL interstitial fluid for approximately 24 hours or 72 hours at 37° C, after which the extraction fluid was filtered through 0.2 μm filter then submitted to Enthalpy Analytical (Orange, CA) for analysis of Mn. The primary modifications to the Henderson et al method were use of closed HDPE bottles rather than stoppered flasks, end-over-end mixing rather than orbital shaking, and use of large headspace containing 5% CO_2 in air in order to maintain pH in the interstitial tests rather than constant bubbling of CO_2 into each reactor. The pH was monitored periodically and adjusted as needed using hydrochloric acid or sodium hydroxide.

Results. The concentrations of metals in the extraction solution and in the concentration in unextracted sieved test material (< 75 μm fraction) are shown in **Tables 2** and **3 f**or the lysosomal and interstitial tests, respectively. The *Bioaccessibility* is given in terms of percent (**Eqn. 1**), and as the mass of soluble metal per mass of sample (**Eqn. 2**). Note that the mass of test material is the mass of the *sieved* fraction used in the test, not the mass of bulk material. The final pH values of all extracts were within the target range (4.7±0.2 for Lysosomal fluid and 7.4±0.2 for Interstitial fluid).

$$\text{Bioaccessibility (\%)} = \frac{100 \times (\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{concentration in test material, mg/kg}) * (\text{mass of test material, kg})} \quad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{(\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{mass of test material, kg})} \quad \text{Eqn. 2}$$

Quality Control (QC). The QC results are given in **Tables 4 and 5** for lysosomal and interstitial tests, respectively. QC limits have not been established for these tests, but the QC is reasonable and indicates good quality data. Mn, if detected in the reagent blank or method blank, was present at or below 0.04 mg/L, while spike recoveries 88% to 97%.

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Analytes in Test Material.

Sample ID	Mass > 75 µm	Mass < 75 µm	Percent > 75 µm	Percent < 75 µm
	g	g	%	%
SiMn Stockpile-062123	606	160	79	21

Samples were sieved by hand.

Table 2. Results of Lysosomal Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
L24-6	7/12/2023	SiMn Stockpile-062123	0.2056	560,000	500	43	240,000
L24-6 dup	7/12/2023	SiMn Stockpile-062123	0.2001	560,000	480	43	240,000
		72 hours					
L72-6	7/14/2023	SiMn Stockpile-062123	0.2066	560,000	500	43	240,000
L72-6 dup	7/14/2023	SiMn Stockpile-062123	0.2066	560,000	510	44	250,000

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 3. Results of Interstitial Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
I24-6	7/17/2023	SiMn Stockpile-062123	0.3552	560,000	4.4	0.39	2,200
I24-6 dup	7/17/2023	SiMn Stockpile-062123	0.3484	560,000	4.4	0.39	2,200
		72 hours					
I72-6	7/18/2023	SiMn Stockpile-062123	0.3533	560,000	7.1	0.63	3,500
I72-6 dup	7/18/2023	SiMn Stockpile-062123	0.349	560,000	6.9	0.62	3,500

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 4. Quality Control - Lysosomal Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
L24-RB	12-Jul-2023	Reagent Blank	0.04 J	--	--	NE
L24-MB	12-Jul-2023	Method Blank	< 0.015	--	--	NE
L24-SPK	12-Jul-2023	Spike	0.88	1.0	88	NE
L72-RB	14-Jul-2023	Reagent Blank	< 0.015	--	--	NE
L72-MB	14-Jul-2023	Method Blank	< 0.015	--	--	NE
L72-SPk	14-Jul-2023	Spike	0.95	1.0	95	NE

NE = not established

"L24" = QC samples associated with Lysosomal 24hr tests.

"L72" = QC samples associated with Lysosomal 72hr test.

J = Estimated value

Table 5. Quality Control - Interstitial Tests.

PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
I24-RB	17-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I24-MB	17-Jul-2023	Method Blank	0.018 J	--	--	NE
I24-SPK	17-Jul-2023	Spike	0.93	1.0	93	NE
I72-RB	18-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I72-MB	18-Jul-2023	Method Blank	< 0.014	--	--	NE
I72-SPk	18-Jul-2023	Spike	0.90	1.0	90	NE

NE = not established

"I24" = QC samples associated with the Interstitial 24hr test.

"I72" = QC samples associated with the Interstitial 72hr test.

J = Estimated value

PRIMA ENVIRONMENTAL INC.
Questions Answered.

5070 Robert J Mathews Parkway, Suite 300
El Dorado Hills, CA 95762
916-939-7300
www.primaenvironmental.com

Sample Receipt Summary

Date/Time: 6/27/23 9:40

Client/Company: Tox Strat

Project: Bridge TVBA

	Yes	No	N/A
Custody seals intact?	☒	☐	☐
Chain of custody Present?	☒	☐	☐

If no, list number of samples and Sample ID

Ice present?	☒	☐	☐

If no, what is temperature? ____

Samples in good condition?	☒	☒	☐

If no, explain:

Glass jar for sample SiMn Stockpile-062123 was broken.

Do sample IDs on containers match IDs on COC?	☒	☐	☐

If no, explain:

Other Comments:

5070 Robert J. Mathews Parkway, Suite 300 • El Dorado Hills, CA 95762
(916) 939-7300 • www.primaenvironmental.com

Project Manager: Geoff Tichenor

Company: Stoel Rives LLP, 760 SW Ninth Ave, Suite 3000 Portland, Oregon

Phone: 503-294-9389

Email: geoffrey.tichenor@stoel.com

CSRM Dust Sampling

Project Number: CSRM-007

TAT : Normal

Sampler Signature: J. Pounds

SAMPLE ID	Date	Time	Matrix	# Containers	EPA 1340 - As, Pb	Inhal RBA - Mn	EPA 3050//6020 - Total Pb, As, Cr, and Mn	EPA 3060A/7199 - CrVI						Comments
SiMn Stockpile-062123	6/21/23	10:25	S	1		x								

Special Instructions	Relinquished by:		Received by:
[redacted]	Company: Bridgewater Group	Date 6/26	Company PRIMA ENV 6/27/23
	Printed Name: Justin Pounds	Time 1000	Printed Name Mousa Fakhuri 7:40
	Signature		Signature
	Relinquished by:		Received by:
	Company	Date	Company
	Printed Name	Time	Printed Name
	Signature		Signature

Matrix key: S - soil/sediment; W - water; OT - other

August 11, 2023

Geoff Tichenor
Stoel Rives LLP
760 SW Ninth Ave., Ste 3000
Portland, OR 97205-2587

RE: Inhalation Bioaccessibility, Manganese
Client Project No.: CSRM-007
Client Project ID: CSRM Dust Sampling
PRIMA Project ID: ToxStrat-Bridge IVBA

Dear Mr. Tichenor:

PRIMA Environmental, Inc. performed in vitro bioaccessibility test (IVBA) to measure the manganese inhalation bioaccessibility of manganese sulfate heptahydrate ($MnSO_4 \bullet H_2O$), and manganese oxide (MnO). Inhalation bioaccessibility simulated lysosomal and interstitial conditions following procedures based on those described in Henderson, R.G. et al "Inter-laboratory validation of bioaccessibility testing for metals", *Regulatory Toxicology and Pharmacology*, **70** (2014) 170-181. Procedures and results are described in this letter report.

Materials. All reagents were reagent grade or better quality. Lysosomal and Interstitial extraction fluids were prepared using the recipes presented in Table 2 of Henderson et al. 5% Carbon dioxide/95% Air was obtained from Magnegas. $MnSO_4 \bullet H_2O$ and MnO were obtained from Bean Town Chemical. MnO was a powder, greater than 200 mesh; it was used as received. $MnSO_4 \bullet H_2O$ was a powder; it was used as received.

Procedures. Inhalation bioaccessibility tests using simulated lysosomal and interstitial conditions were run based on methods described in Henderson et al. In this method, the < 75μm fraction of test material (200 mg for lysosomal, 350 mg for interstitial) was extracted with 100 mL lysosomal fluid or 175 mL interstitial fluid for approximately 24 hours or 72 hours at 37° C, after which the extraction fluid was filtered through 0.2 μm filter then submitted to Enthalpy Analytical (Orange, CA) for analysis of Mn. The primary modifications to the Henderson et al method were use of closed HDPE bottles rather than stoppered flasks, end-over-end mixing rather than orbital shaking, and use of large headspace containing 5% CO_2 in air in order to maintain pH in the interstitial tests rather than constant bubbling of CO_2 into each reactor. The pH was monitored periodically and adjusted as needed using hydrochloric acid or sodium hydroxide.

Results. The concentrations of metals in the extraction solution and in the concentration in unextracted sieved test material (< 75 μm fraction) are shown in **Tables 1** and **2 f**or the lysosomal and interstitial tests, respectively. The *Bioaccessibility* is given in terms of percent (**Eqn. 1**), and as the mass of soluble metal per mass of sample (**Eqn. 2**). Note that the mass of test material is the mass of the *sieved* fraction used in the test, not the mass of bulk material. The final pH values of all extracts were within the target range (4.7±0.2 for Lysosomal fluid and 7.4±0.2 for Interstitial fluid).

The bioavailability of $MnSO_4 \bullet H_2O$ decreased over time in the interstitial tests. The reason is presumably due to reaction of $MnSO_4 \bullet H_2O$ with the extraction fluid. $MnSO_4 \bullet H_2O$ is a pale pink solid that readily dissolves in deionized water. However, addition of $MnSO_4 \bullet H_2O$ to interstitial extraction fluid turned the extraction fluid cloudy white. Settled solids were observed within 21 hours and a pinkish brown precipitate was noted within 7 days.

$$\text{Bioaccessibility (\%)} = \frac{100 \times (\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{concentration in test material, mg/kg}) * (\text{mass of test material, kg})} \quad \text{Eqn. 1}$$

$$\text{Bioaccessibility (mg As/kg soil)} = \frac{(\text{concentration in extract, mg/L}) * (\text{volume extraction fluid, L})}{(\text{mass of test material, kg})} \quad \text{Eqn. 2}$$

Quality Control (QC). The QC results are given in **Tables 4 and 5** for lysosomal and interstitial tests, respectively. QC limits have not been established for these tests, but the QC is reasonable and indicates good quality data. Mn, if detected in the reagent blank or method blank, was present at or below 0.04 mg/L, while spike recoveries 88% to 97%.

If you have any questions, please give me a call at 916-939-7300. Thank you for the opportunity to be of service.

Sincerely,
PRIMA Environmental, Inc.

Cindy G. Schreier, Ph.D.
President & Chief Scientist

Attachments

Table 1. Results of Lysosomal Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
L24-7	7/12/2023	MnSO4*H2O	0.2059	310,000	650	102	320,000
L24-8	7/12/2023	MnO	0.2074	760,000	1600	102	770,000
		72 hours					
L72-7	7/14/2023	MnSO4*H2O	0.1936	310,000	620	103	320,000
L72-8	7/14/2023	MnO	0.1943	760,000	1500	102	770,000

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 2. Results of Interstitial Bioaccessibility Tests - Manganese.

PRIMA ID	Extraction Date	Sample	Mass Extracted	Concentration		Bioaccessibility	
				Sieved Sample*	Extraction Fluid		
			g	mg/kg	mg/L	%	mg /kg sample
		24 hours					
I24-7.2	7/24/2023	MnSO4*H2O	0.3523	310,000	100	16	50,000
I24-8	7/17/2023	MnO	0.3544	760,000	0.49	0.032	240
		72 hours					
I72-7	7/18/2023	MnSO4*H2O	0.3464	310,000	37	6.0	19,000
I72-8	7/18/2023	MnO	0.3498	760,000	0.94	0.062	470

* Less than 75 µm fraction.

^ "Dup" is a duplicate extraction - the sample was extracted twice, but Mn in the sample was measured once.

Table 3. Quality Control - Lysosomal Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
L24-RB	12-Jul-2023	Reagent Blank	0.04 J	--	--	NE
L24-MB	12-Jul-2023	Method Blank	< 0.015	--	--	NE
L24-SPK	12-Jul-2023	Spike	0.88	1.0	88	NE
L72-RB	14-Jul-2023	Reagent Blank	< 0.015	--	--	NE
L72-MB	14-Jul-2023	Method Blank	< 0.015	--	--	NE
L72-SPk	14-Jul-2023	Spike	0.95	1.0	95	NE

NE = not established

"L24" = QC samples associated with Lysosomal 24hr tests.

"L72" = QC samples associated with Lysosomal 72hr test.

J = Estimated value

Table 4. Quality Control - Interstitial Tests.

			Manganese			
PRIMA ID	Extraction Start Date	Sample ID	Conc. mg/L	Spike, mg/L	% Rec	Limits
I24-RB	17-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I24-MB	17-Jul-2023	Method Blank	0.018 J	--	--	NE
I24-SPK	17-Jul-2023	Spike	0.93	1.0	93	NE
I24-RB2	24-Jul-2023	Reagent Blank	< 0.0081	--	--	NE
I24-MB2	24-Jul-2023	Method Blank	< 0.0081	--	--	NE
I24-SPK2	24-Jul-2023	Spike	0.97	1.0	97	NE
I72-RB	18-Jul-2023	Reagent Blank	< 0.014	--	--	NE
I72-MB	18-Jul-2023	Method Blank	< 0.014	--	--	NE
I72-SPk	18-Jul-2023	Spike	0.90	1.0	90	NE

NE = not established

"I24" = QC samples associated with the Interstitial 24hr tests.

"I72" = QC samples associated with the Interstitial 72hr test.

J = Estimated value