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July 7, 2017

Mr. Tom Brodell
Georgia Environmental Protection Division Land Protection Branch
2 Martin Luther King, Jr. Drive SE
Suite 1456 East
Atlanta, Georgia 30334

Subject: July 2017 Semi-Annual Voluntary Remediation Program Progress Report
North Berkeley Lake Road Site (HSI No. 10844)
Duluth, Gwinnett County, Georgia

Dear Mr. Brodell:

This Progress Report documents the activities completed for the North Berkeley Lake Road Site, also referred to as Fire Station 19, in Duluth, Georgia from January 2017 through June 2017. This report constitutes the fifth semi-annual progress report and follows the schedule outlined in the Georgia Environmental Protection Division's (EPD's) letter dated January 15, 2015.

This Progress Report includes the following:

- Work Performed This Period;
- Work Anticipated for the Next Period;
- Schedule; and
- Professional Certification.

Work Performed This Period

The U.S. Environmental Protection Agency (EPA) has now approved the new model and test method for evaluating the bioavailability of arsenic. As previously discussed, this method and our site-specific data are proposed for use in finalizing the Risk Reduction Standards (RRSs) for Fire Station 19.

EPA reported a validation assessment (**Attachment A**) of arsenic *in vitro* bioaccessibility (IVBA) assay for predicting relative bioavailability of arsenic in soils at Superfund sites in April 2017. Following the validation, the laboratory method was published by EPA. A copy of the approved method was provided to CDM Smith by EPA and is attached (**Attachment B**). The approved method is based on the work of the publication provided to EPD in our July 2016 Semi-Annual Progress Report (Predicting oral relative bioavailability of arsenic in soil from IVBA, Diamond et al., 2016). As shown in Appendix B of the method, the replicate data used to calculate the method reference values were derived from two laboratories: EPA's National Exposure Research Laboratory and the University of Colorado laboratory operated by Dr. John Drexler.

As you are aware, Dr. Drexler also completed the arsenic *in vitro* bioaccessibility analyses for Fire Station 19 in his University of Colorado laboratory and the EPA approved method is unchanged from Dr. Drexler's method. However, the formula used to calculate the relative bioavailability (RBA) using the laboratory data has been updated to the Diamond 2016 formula, as provided below.

$$RBA_{arsenic} (\%) = 0.79 * IVBA (\%) + 3.0$$

The soil IVBA previously derived for Fire Station 19 was developed using the approved method and ranged from 0.6 percent to 13.3 percent. This produces an RBA range from 3.5 percent to 13.5 percent using the approved formula. To be conservative, CDM Smith has assumed a 13.5 percent RBA.

The RBA Risk Reduction Standard (RRS) Technical Memorandum (CDM Smith, March 17, 2015) that was attached to the first Semi-Annual Status Report, proposed Type 4 and Type 5 (construction worker) soil arsenic RRSs. Using the same calculation approach that EPD reviewed with the recently approved RBA formula, the new Type 4 arsenic RRS is 280 mg/kg and the new Type 5 arsenic RRS is 990 mg/kg. The updated Type 5 arsenic RRS also includes the increase in the soil ingestion rate previously recommended by EPD. The supporting calculations are shown in **Attachment C**.

The arsenic sampling locations and tabulated data from the Corrective Action Plan are shown included in **Attachment D**. These data show that all locations are below the currently proposed RRSs.

Work Anticipated for the Next Period

Gwinnett County and CDM Smith propose that the next actions for Fire Station 19 be the implementation of the Environmental Covenant and submittal of the Compliance Status Report (CSR).

Schedule

Gwinnett County and CDM Smith propose to initiate the Environmental Covenant process within 30 days and expect it to be fully executed by the end of September. The draft CSR is expected to be completed prior to the finalization of the Environmental Covenant and the CSR will be submitted to EPD immediately upon Environmental Covenant finalization.

Professional Certification

Attachment E contains the professional certification and summary of incurred professional engineer and geologist hours for the period from January 1, 2017 through June 30, 2017.

If you have any questions regarding this Progress Report, please do not hesitate to contact me at (678) 376-6953 or richard.schoeck@gwinnettcounty.com.

Sincerely,
GWINNETT COUNTY DEPARTMENT OF WATER RESOURCES

Richard Schoeck, P.E., PMP
Division Director of Project Controls
Attachments

cc: Tom Duffey, CDM Smith
Forrest Fields, Gwinnett County
J.C. Lan, Gwinnett County
John Reichling, CDM Smith

Attachment A
Arsenic IVBA Validation Assessment

Validation Assessment of *In Vitro* Arsenic Bioaccessibility Assay for Predicting Relative Bioavailability of Arsenic in Soils and Soil-like Materials at Superfund Sites

1. Introduction

This report summarizes the basis for the Agency's determination that the IVBA method for arsenic has satisfied the validation and regulatory acceptance criteria for application of the method in an appropriate regulatory context. Validation and regulatory acceptance criteria developed by the U.S. Environmental Protection Agency (U.S. EPA, 2007a), as adapted from the Interagency Coordinating Committee on the Validation of Alternative Methods (ICCVAM, 1997), have been applied to an *in vitro* arsenic bioaccessibility (IVBA) assay described in detail by Brattin et al. (2013). The arsenic IVBA method estimates site-specific relative bioavailability (RBA) of arsenic in soils quickly and inexpensively relative to *in vivo* methods. The arsenic IVBA assay is well suited for regulatory use in arsenic risk assessment for several reasons: (1) the assay does not sacrifice animals; (2) the reduced cost and analysis time from use of the IVBA assay in place of *in vivo* RBA assays will facilitate greater numbers of soil samples analyzed at each site to improve representativeness; (3) regulatory acceptance of the arsenic IVBA assay would lower bioavailability assessment costs by enabling simultaneous assessments of RBA for both arsenic and lead using the existing Standard Operating Procedure (SOP) for the IVBA extraction protocol, which has been previously validated for assessment of RBA of lead in soil (U.S. EPA 2009, 2012a); and (4) some of the U.S. EPA Regional laboratories and commercial laboratories have analytical and quality control experience with the SOP gained from use of the identical assay for lead.

2. Validation Assessment of the *In Vitro* Arsenic Bioaccessibility Assay

This section discusses the validation criteria established in the Agency soil bioavailability guidance (U.S. EPA, 2007a). Criteria for method validation and regulatory acceptance were consolidated because many of the criteria overlap.

2.1. Scientific and regulatory rationale for the test method, including a clear statement of its proposed use, should be available.

The scientific and regulatory rationale for the arsenic IVBA method is presented in the following:

U.S. EPA. (2007a) Guidance for Evaluating the Bioavailability of Metals in Soils for Use in Human Health Risk Assessment. OSWER 9285.7-80. May 2007. Available online at https://semspub.epa.gov/work/11/175333.pdf

U.S. EPA. (2012b) Recommendations for Default Value for Relative Bioavailability of Arsenic in Soil. OSWER 9200.1-113. December 2012. Available online at https://semspub.epa.gov/work/11/175338.pdf

Regulatory and scientific rationale: The *Guidance for Evaluating the Bioavailability of Metals in Soils for Use in Human Health Risk Assessment* (U.S. EPA, 2007a) articulates the regulatory rationale for determining the bioavailability of metals from soils when assessing human health risks at hazardous waste sites:

> *Accounting for potential differences in oral bioavailability of metals in different exposure media can be important to site risk assessment (U.S. EPA, 1989). This is true for all chemicals, but is of special importance for ingested metals. This is because metals can exist in a variety of chemical and physical forms, and not all forms of a given metal are absorbed to the same extent. For example, a metal in contaminated soil may be absorbed to a lesser extent than when ingested in drinking water or food. Thus, if the oral RfD or CSF for a metal is based on studies using the metal administered in water or food, risks from ingestion of the metal in soil might be overestimated. Even a relatively small adjustment in oral bioavailability can have significant impacts on estimated risks and cleanup goals.* (U.S. EPA, 2007a)

The *Recommendations for Default Value for Relative Bioavailability of Arsenic in Soil* (U.S. EPA, 2012b) document articulates the regulatory rationale for site-specific assessment of arsenic bioavailability in soils:

> *The current default assumption for assessing risk from arsenic in soil is that the bioavailability of arsenic in soil is the same as the bioavailability of arsenic in water (relative bioavailability [RBA] soil/water = 100%). However, recent bioavailability studies conducted in animal models show that bioavailability of arsenic in soil is typically less than that of highly water soluble forms of arsenic (e.g., sodium arsenate dissolved in water). This suggests that bioavailability of arsenic in soil will typically be less than that of arsenic dissolved in drinking water (i.e., RBA<100%). At sites where this applies, the default assumption of RBA=100% will result in an overestimation of risk.* (U.S. EPA, 2012b)
>
> *In general, the Agency (U.S. EPA, 2007a) recommends that efforts be made to collect data that support site-specific estimates, rather than relying on the default value recommended in this memorandum which may not accurately represent arsenic RBA at any specific site. Use of the national default in place of site specific estimates may underestimate or overestimate risk. Where development of site-specific RBA estimates is not feasible (e.g., screening-level assessments), the default value of 60% can be used, recognizing that the default value is an estimate that is not likely to be exceeded at most sites and is preferable to the assumption of an RBA equal to 100%.* (U.S. EPA, 2012b)

2.2. Relationship of the test method endpoint(s) to the endpoint of interest must be described.

The endpoint of interest for risk assessment is a prediction of the oral RBA of arsenic in soil (ratio of oral bioavailability of arsenic in soil to that of water-soluble arsenic) based on a measurement of IVBA of arsenic in soil (solubility of arsenic in soil at gastric pH). The test soil sample is assayed for IVBA, and the corresponding RBA is predicted from a regression model relating IVBA and RBA. This same approach has been validated by EPA for predicting RBA of lead in soil from IVBA (U.S. EPA, 2009).

The IVBA assay for predicting RBA of arsenic in soil is the same extraction procedure validated for predicting the RBA of lead in soil (U.S. EPA, 2009, 2012a). In brief, the IVBA assay consists of incubating a 1 g soil sample with end-over-end mixing in 100 mL of 0.4 M glycine buffer (pH 1.5) for 1 hour at 37°C (body temperature).

The regression model for predicting RBA of arsenic in soil from IVBA is based on a meta-analysis of concordant data from studies in mice and swine (Bradham et al., 2011, 2013; Brattin et al., 2013; Juhasz et al., 2009, 2014a). Data were combined into a validation dataset consisting of paired IVBA and RBA measurements made on 83 soils collected from different sites and mineral types, including mining, smelting, and pesticide or herbicide application (see Section 2.3 for mineral types). Paired measurements of IVBA and RBA for each of the 83 soil samples were included in a weighted linear regression model (Equation 1) in which IVBA and RBA were based on their respective variances (1/variance). The estimated slope is 0.79 ± 0.01 (SE) and intercept is 3.0 ± 0.1 (SE). The equation of the model is:

$$RBA(\%) = 0.79 \cdot IVBA(\%) + 3.0 \qquad \text{Eq. (1)}$$

This model explains approximately 87% of the variance in RBA (weight-adjusted $R^2 = 0.87$). The 95% prediction limit for a single RBA measurement was ±19% RBA. A detailed description of the derivation of the regression model is provided in Diamond et al. (2016). This regression model could be updated periodically by incorporating more data sets as they become available.

2.3. A detailed protocol for the test method must be available and should include a description of the materials needed, a description of what is measured and how it is measured, acceptable test performance criteria (e.g., positive and negative control responses), a description of how data will be analyzed, a list of the materials for which the test results are applicable, and a description of the known limitations of the test, including a description of the classes of materials that the test can and cannot accurately assess.

Standard Operating Procedure: The arsenic IVBA assay extraction protocol is the same as SOP 92000.2-86 for the IVBA assay for lead in soil (U.S. EPA, 2012a, 2017). EPA has developed an SOP specifically for arsenic that includes the SOP 09000.2-86 extraction protocol along with the corresponding analytical procedures for measuring arsenic in the soil and soil-like materials and extracts. The IVBA method is included under the validated methods tab on the SW-846 website as Method 1340 for lead, which will be updated to include arsenic.

Aside from the standard laboratory glassware, reagents, supplies, and equipment, the materials needed for the IVBA assay include 0.4 M glycine (free base, reagent-grade glycine in deionized water, adjusted to a pH of 1.50 ± 0.05 at 37°C using trace metal-grade concentrated hydrochloric acid), and either a water bath or an incubated air chamber with sample rotator is necessary for the extraction of the samples at 37°C. In addition, reference standards NIST 2710a SRM or Flat Creek SRM need to be purchased for use as the control soils in the QA/QC samples. These materials and equipment do not require a large investment from laboratories interested in performing the IVBA assay.

The IVBA assay is meant to measure the fraction of the amount of ingested arsenic that would be solubilized at the low pH of the stomach. The samples are sieved at 150 μm to mimic the fraction of soil that is likely to stick to human hands and thereby be ingested (U.S. EPA, 2016). The samples are then extracted in a 0.4 M glycine solution, pH 1.5 at 37°C for 1 hour with rotation to mimic gastric conditions. Following the extraction by IVBA assay, the concentration of arsenic in the extraction solution is measured by ICP-MS or ICP-AES. The total concentration of arsenic in the sample is measured by SW-846 Method 3051A.

As part of the quality control/quality assurance for the IVBA assay, the method requires that a set of quality control samples be run in a batch of samples. Quality control samples are reagent blank (extraction fluid that is not run through the extraction procedure), method blank (extraction fluid that has been run through the extraction procedure), laboratory control sample (LCS; extraction fluid spiked with arsenic that is run through the extraction procedure), matrix spike (spiked matrix, e.g., soil, that is run through the extraction procedure), duplicate sample, and control soil. Control limits and frequency for each quality control sample for arsenic are shown in Table 1.

Table 1. Recommended Control Limits for Quality Control Samples for Arsenic

Quality Control Samples	Frequency	Control Limits for Arsenic
Reagent blank	once per batch (minimum 1 in 20 samples)	<25 µg/L arsenic
Method blank	once per batch (minimum 1 in 20 samples)	<50 µg/L arsenic
LCS (10 mg/L)	once per batch (minimum 1 in 20 samples)	85–115% recovery
Matrix spike (10 mg/L)	once per batch (minimum 1 in 10 samples)	75–125% recovery
Duplicate sample	once per batch (minimum 1 in 10 samples)	±20% RPD
NIST 2710a[a]	once per batch (minimum 1 in 20 samples)	32.9–49.1%

RPD = Relative percent difference
[a]Appendix A

The % IVBA for a sample is determined from the analytical results by Equation 2.

$$IVBA(\%) = [(As_{ext} \times V_{ext})/(As_{soil} \times Soil_{mass}) \times 100 \qquad \text{Eq. (2)}$$

where:

As_{ext} = mass concentration of arsenic in the IVBA extract (mg/L)
V_{ext} = IVBA extract solution volume (L)
As_{soil} = total arsenic concentration (as determined by SW-846 Method 3051A or equivalent) (mg/kg)
$Soil_{mass}$ = mass of soil extracted by IVBA (kg)

Equation 1 is applied to the % IVBA results to determine the % RBA (see section 2.2).

Applicable test materials: Application of the IVBA method SOP is expected to yield predictions of RBA for individual soil samples that fall within the prediction interval of the assay (±19 RBA%). The prediction interval was based on results from various sources, including mining, smelting, or pesticide applications. Although arsenic mineralogy has not been evaluated for all soils in the data set, the following arsenic mineral phases were identified: sorbed As^{V} and As^{III}, arsenic trioxide, arsenopyrite, lollingite, realgar, scorodite, and a variety of arsenic-metal oxides (Bradham et al., 2011, 2013, 2015; Brattin et al., 2013; Juhasz et al., 2007). It is possible that some soils may fall outside of the established prediction interval as a result of an unusual arsenic mineralogy or soil composition not represented in the validation dataset. Therefore, whenever a sample is suspected of containing an unusual and/or untested source material or arsenic mineralogy, this should be identified as a potential data gap and source of uncertainty in the resulting prediction of RBA. As additional samples with a variety of new and different arsenic forms are tested by both *in vivo* and *in vitro* methods, the range of applicability of the method should be refined and expanded.

Assay limitations: The following uncertainties may apply to applications of the IVBA assay.

i. **Sample arsenic concentration limits**: The arsenic concentrations of soils tested in the development of the regression model relating IVBA and RBA and its associated prediction interval for the IVBA assay ranged from 40 to 13,000 ppm. This validation range should be sufficient for most applications of the methodology. Although there is no basis for predicting what errors would necessarily be introduced into the predictions of RBA if sample concentrations outside this range were used in the IVBA assay, use of such samples without validating comparisons with results of an *in vivo* assay will introduce additional uncertainty into estimates of RBA. However, applications of the IVBA assay to such high arsenic concentrations (e.g., >7,000 ppm) are unlikely to change risk management decisions; thus, this limitation is not a serious constraint for the utility of the method to support cleanup decisions. If additional data suggests modification of the limits, then the Agency will issue additional guidance. In addition, the minimum soil concentration in the sample is determined by that which is measurable in the assay using the SOP.

ii. **Particle size**: Soil samples in the validation dataset were sieved for particles less than 250 μm. Particle size can be expected to affect dissolution of arsenic embedded in soil particles (Karna et al., 2017). Therefore, additional uncertainty will be associated with RBA estimates from IVBA assays of soil samples having particle sizes excluded from the validation dataset (i.e., >250 μm) U.S. EPA recommends a sieving size of <150 μm to represent the particle fraction having the highest likelihood of incidental ingestion (Ruby and Lowney, 2012; U.S. EPA, 2016). Arsenic IVBA in soils sieved to <250 μm were not different from IVBA measured in soils sieved to <150 μm (Karna et al., 2017).

iii. **Uncertainty in predicted RBA value**: The IVBA assay for arsenic measures IVBA for a test soil and converts this to an estimate of RBA using a regression equation estimated from a meta-analysis of 83 samples. The predicted RBA is the most likely (highest probability) estimate corresponding to the IVBA, but the actual RBA (if measured *in vivo*) might be either higher or lower than the predicted value. The 95% prediction limit for the arsenic IVBA-RBA regression model is relatively narrow in the context of its application to risk assessment, ±19 RBA%. This means that there will be a 95% probability that individual RBA measurements will be ±19 of the RBA% predicted from IVBA. In general, the most likely estimate of RBA is the most appropriate value for use in risk assessments because there is an equal probability of the true RBA being above or below the predicted value; however, other values from within the RBA prediction interval could also be evaluated as part of an uncertainty analysis.

iv. **Predicting RBA in humans**: The IVBA assay was developed to predict arsenic RBA in humans, although there are no data in humans to provide a direct validation of RBA predictions in humans. Therefore, the arsenic IVBA assay was evaluated with estimates of RBA made from studies conducted in two different juvenile swine bioassays and a mouse bioassay. The use of animals for establishing arsenic RBA values to be used in regulatory contexts has several precedents: (1) a national default soil arsenic RBA, to be used when site-specific estimates are not available (it is always better to collect and analyze site-specific data than to rely on a default value), was derived based on a large sample of soil RBA measurements made in mice, monkeys, and swine (U.S. EPA, 2012a,c); (2) an IVBA assay was validated for predicting lead RBA based on soil RBA measurements made in a swine assay (U.S. EPA, 2009); and (3) animal bioassays (e.g., mice, monkeys, swine) remain valid for establishing site-specific soil arsenic and lead RBA, but are not recommended because it is better to run IVBA analyses on many samples (e.g., a statistical sample) than to rely on a smaller number of samples analyzed in animal bioassays (U.S. EPA, 2007b, 2010). Significantly greater costs and time to complete will limit the number of animal bioassays.

Although there is no quantitative support for discerning which animal bioassay provides a more accurate prediction of arsenic RBA in humans, RBA estimates obtained from the mouse and swine assays are in close agreement (Bradham et al., 2013; Juhasz et al., 2014b).

2.4. The extent of within-test variability and the reproducibility of the test within and among laboratories must have been demonstrated. The degree to which sample variability affects this test reproducibility should be addressed.

Within-test variability: Precision of the IVBA protocol was assessed with analyses of soils included in the validation dataset, which included contributions from three laboratories. Each laboratory achieved consistent and relatively low coefficients of variation (CV=standard deviation/mean): 2.1, 4.0, and <5% (Brattin et al., 2013; Diamond et al., 2016).

Inter-laboratory reproducibility: An inter-laboratory comparison of the IVBA was conducted with four participating laboratories: ACZ Laboratories Inc.; EPA Region 7 laboratory; EPA Region 8 laboratory; and University of Colorado at Boulder (Brattin et al., 2013). Each laboratory applied the IVBA method to analyses (in triplicate) of 12 test soils. Average within-laboratory variability (coefficient of variation, CV) ranged from 1.3 to 11.0%. The inter-laboratory coefficient ranged from 2.2 to 15% (mean: 5.4%).

Effects of sample variability: The prediction interval for the IVBA assay was derived based on analysis of 83 soil samples from a variety of site types: mining, smelting, or pesticide application. The IVBA range for the soil samples was 0–80% (mean: 27.2 ± 20 SD). The within-laboratory coefficient of variation for IVBA was <0.05 (Diamond et al., 2016).

2.5. The test method performance must have been demonstrated using reference materials or test materials representative of the types of substances to which the test method will be applied, and should include both known positive and known negative agents.

Performance with reference materials: Precision of the IVBA protocol was assessed with replicate arsenic analyses of standard reference materials (SRMs; National Institute of Standards and Technology [NIST] SRM 2710A) conducted by the EPA Office of Research and

Development National Exposure Research Laboratory [ORD NERL]) over several years (Appendix B). The mean relative percent difference ranged from -10.2 to 9.6% (mean: -0.14 ± 5.3% SD).

Performance with representative materials: The prediction interval for the IVBA assay was derived based on analysis of samples having a variety of arsenic mineral phases from a variety of different types of sites: mining, smelting, and pesticide application.

2.6. Sufficient data should be provided to permit a comparison of the performance of a proposed substitute test with that of the test it is designed to replace.

The IVBA assay is a cost-effective and time-saving alternative to *in vivo* RBA assays that can improve data quality by increasing the number of samples analyzed while reducing costs and turn-around time. For the dataset used to derive the regression model, the model accounted for approximately 87% of the observed variance in RBA. The 95% prediction interval for the model is ±19 RBA%, based on 83 soil samples from a variety of site types that are expected to be typical applications of the assay for site risk assessment (mining, smelting, and or pesticide application). The standard errors for the RBA estimates for this sample of 83 soils ranged from 0.2 to 20% (median 2%), and the ratios of the SE to the mean RBA (SE/mean) ranged from 0.02 to 0.48 (median 0.09).

2.7. Data supporting the validity of a test method should be obtained and reported in accordance with Good Laboratory Practices (GLPs).

Data supporting validity of the IVBA assay are reported in detail in a published report (Diamond et al., 2016). Data used in the analysis is provided in Appendix C.

2.8. Data supporting the assessment of the validity of the test method must be available for review.

Data supporting the assessment of the validity of the IVBA assay are available online at http://www.tandfonline.com/doi/full/10.1080/15287394.2015.1134038.

2.9. The methodology and results should have been subjected to independent scientific review.

The arsenic IVBA methodology was reviewed by EPA scientists and evaluated in several peer-reviewed publications (Bradham et al., 2011, 2013, 2015; Brattin et al., 2013; Juhasz et al., 2009, 2014a,b). The report describing derivation of the prediction regression model was reviewed by the EPA Office of Superfund Remediation and Technology Innovation (OSRTI) Technical Review Workgroup Bioavailability Committee, EPA ORD peer-review for release of publication, and editorial peer-review for publication (Diamond et al., 2016).

2.10. The method should be time and cost effective.

Costs of assessment of a soil sample using the IVBA assay are expected to range from approximately 10-fold to 100-fold less than the costs of a bioassay. Time requirements for the IVBA assay are expected to range from approximately 10-fold to 50-fold less than that required to conduct an *in vivo* bioassay (i.e., days compared to several weeks). Additional cost and time efficiencies are expected for applications at sites where arsenic and lead are chemicals of interest because the same IVBA extraction protocol can be used to predict arsenic and lead RBA. These efficiencies can be used to analyze a greater number of samples.

2.11. The method should be one that can be harmonized with similar testing requirements of other agencies and international groups.

Other international efforts (e.g., Australia, Canada, European Union, United Kingdom) are pursuing the development of methods for *in vitro* assessment of RBA of arsenic and of other metals and inorganic contaminants in soil. The IVBA assay is directly applicable to these national and international programs. It satisfies the Bioaccessibility Research Canada (BARC) acceptance criteria for use in risk assessment (BARC, 2016; Koch and Reimer, 2012) and the IVBA assay has been used widely to characterize soil arsenic bioaccessibility; recent examples of international use include reports from Africa, Australia, Canada, China, and Great Britain (Dodd et al., 2013; Ettler et al., 2012; Juhasz et al., 2015; Koch and Reimer 2012; Kribek et al., 2014; Li et al., 2015a,b; Meunier et al., 2010; Morales et al., 2015; Silvetti et al., 2014; Wang et al., 2012; Yang et al., 2015). The meta-analysis that forms the basis for the predictive regression model for RBA included contributors from the United States and Australia (Diamond et al., 2016). Various EPA and non-government laboratories provided data to support the validation.

2.12. The method should be suitable for international acceptance.

The IVBA assay is suitable for international acceptance (see section 2.11 for further discussion).

2.13. The method must provide adequate consideration for the reduction, refinement, and replacement of animal use.

The IVBA assay replaces bioassays and will decrease the use of animals for assessing RBA of arsenic in soil.

3. Summary

The IVBA assay for arsenic has been evaluated against validation criteria established by EPA (U.S. EPA, 2007a) for validation of test methods to be used in a regulatory context. All validation criteria have been satisfied. SOPs have been established and tested for intra-laboratory precision and inter-laboratory reproducibility. The quantitative relationship between the IVBA assay output and output from *in vivo* animal bioassays, which the IVBA assay is meant to replace, has been reliably established. The description in the method SOP is expected to yield predictions of RBA that fall within acceptable prediction limits for applications in arsenic site risk assessment. The prediction interval is based on assays of samples collected from a variety of arsenic mineral phases from a variety of different sites and, as a result, the method is expected to be widely applicable to soil typically encountered at arsenic waste sites. Based on this assessment, EPA concludes that the IVBA method is valid for predicting RBA of arsenic in soils in support of site-specific risk assessments. The following regression model is recommended for applications to risk assessment (Equation 1):

$$RBA(\%)=IVBA(\%)\cdot 0.79+3.0(\%) \qquad \text{Eq. (1)}$$

The Agency strongly encourages use of this methodology when implemented in context with the decision framework described in its soil bioavailability guidance (U.S. EPA, 2007a).

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APPENDIX A

Provisional Reference Values for Arsenic IVBA of NIST 2710A Standard Reference Material

Consensus values for In Vitro Bioaccessibility (IVBA) of arsenic in soil reference materials (RM) are needed to support the Standard Operating Procedures (SOP) for determination of arsenic IVBA in soil. EPA intends to conduct multi-laboratory evaluations of arsenic IVBA for NIST 2710A and USGS Flat Creek RMs. and has conducted similar evaluations of lead IVBA for these RMs. Until the arsenic IVBA evaluations are completed, EPA recommends using the provisional reference values for NIST 2710A in Table A-1. Although, the provisional reference values are based on data from only two laboratories, the estimated prediction interval (±20%) is in the range observed for lead IVBA reference values (Table A-2). The data on which the arsenic IVBA reference values are based are provided in Tables A-3 (summary) and A-4 (individual replicates).

Table A-1. Recommended Provisional Reference Value for Arsenic IVBA% of NIST 2710A

Laboratory	Reference Material	Laboratory Analysis	Total Soil Arsenic Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
All Labs[a]	NIST2710A	Arsenic IVBA	NIST Certificate[b]	%	131	32.9	41.0	49.1	± 19.8

[a]Data provided by Karen Bradham (EPA PRD NERL) and John Drexler (University of Colorado)
[b]NIST certificate median soil arsenic concentration: 1400 mg/kg

Table A-2. Reference Values for Lead IVBA% of Standard Reference Materials

Laboratory	Reference Material	Laboratory Analysis	Total Soil Lead Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
QATS Round Robin	NIST2710A	Lead IVBA	NIST Certificate	%	35	60.7	67.5	74.2	±10
QATS Round Robin	NIST2711A	Lead IVBA	NIST Certificate	%	35	75.2	85.7	96.2	±12.3
QATS Round Robin	Flat Creek	Lead IVBA	EPA 3051A	%	30, 35[a]	56.0	71.0	86.0	±21.1

[a]Based on n=35 estimates of total Pb (mg/kg) and 30 estimates of IVBA Pb (mg/kg)

Table A-3. Values for Arsenic IVBA% of NIST 2710A Based Data from Individual Laboratories and Combined Data

Laboratory[a]	Reference Material	Laboratory Analysis	Total Soil Arsenic Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
EPA NERL	NIST2710A	Arsenic IVBA	NIST Certificate	%	117	33.1	40.8	48.4	± 18.8
U Colorado	NIST2710A	Arsenic IVBA	NIST Certificate	%	14	30.7	43.0	55.2	± 28.5
All Labs	NIST2710A	Arsenic IVBA	NIST Certificate	%	131	32.9	41.0	49.1	± 19.8

[a]Data provided by Karen Bradham (EPA PRD NERL) and John Drexler (University of Colorado)

Table A-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
1	EPA NERL	1.00	5.59	1400	39.9
2	EPA NERL	1.00	5.56	1400	39.6
3	EPA NERL	1.00	5.33	1400	38.0
4	EPA NERL	1.00	5.14	1400	36.7
5	EPA NERL	1.00	6.40	1400	45.6
6	EPA NERL	1.00	6.40	1400	45.6
7	EPA NERL	1.00	5.98	1400	42.7
8	EPA NERL	1.00	6.15	1400	43.9
9	EPA NERL	1.00	5.46	1400	38.9
10	EPA NERL	1.00	5.82	1400	41.4
11	EPA NERL	1.00	6.39	1400	45.5
12	EPA NERL	1.00	5.25	1400	37.5
13	EPA NERL	1.00	5.26	1400	37.6
14	EPA NERL	1.00	5.19	1400	37.1
15	EPA NERL	1.00	5.54	1400	39.5
16	EPA NERL	1.00	5.43	1400	38.8
17	EPA NERL	1.00	5.52	1400	39.3
18	EPA NERL	1.00	5.20	1400	37.0
19	EPA NERL	1.00	5.08	1400	36.3
20	EPA NERL	1.00	5.19	1400	37.0
21	EPA NERL	1.00	5.24	1400	37.4
22	EPA NERL	1.00	6.01	1400	42.9
23	EPA NERL	1.00	5.57	1400	39.7
24	EPA NERL	1.00	5.58	1400	39.6
25	EPA NERL	1.00	5.66	1400	40.4
26	EPA NERL	1.00	5.25	1400	37.4
27	EPA NERL	1.00	5.25	1400	37.5
28	EPA NERL	1.00	5.51	1400	39.4
29	EPA NERL	1.00	4.89	1400	35.0
30	EPA NERL	1.00	5.61	1400	40.0
31	EPA NERL	1.00	5.36	1400	38.2
32	EPA NERL	1.01	5.94	1400	42.1
33	EPA NERL	1.00	5.86	1400	41.8
34	EPA NERL	1.00	5.84	1400	41.6
35	EPA NERL	1.00	4.83	1400	34.4
36	EPA NERL	1.00	5.12	1400	36.5
37	EPA NERL	1.00	5.29	1400	37.7
38	EPA NERL	1.00	5.88	1400	41.9

Table A-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
39	EPA NERL	1.00	5.69	1400	40.6
40	EPA NERL	1.00	5.88	1400	41.8
41	EPA NERL	1.00	5.70	1400	40.6
42	EPA NERL	1.00	5.44	1400	38.8
43	EPA NERL	1.00	5.35	1400	38.2
44	EPA NERL	1.00	5.38	1400	38.3
45	EPA NERL	1.00	5.37	1400	38.3
46	EPA NERL	1.00	5.42	1400	38.7
47	EPA NERL	1.00	5.30	1400	37.9
48	EPA NERL	1.00	5.10	1400	36.3
49	EPA NERL	1.00	6.00	1400	42.7
50	EPA NERL	1.00	5.21	1400	37.1
51	EPA NERL	1.00	5.19	1400	37.0
52	EPA NERL	1.00	6.29	1400	44.8
53	EPA NERL	1.00	5.92	1400	42.1
54	EPA NERL	1.00	5.64	1400	40.1
55	EPA NERL	1.00	5.60	1400	39.9
56	EPA NERL	1.00	5.73	1400	40.8
57	EPA NERL	1.00	5.90	1400	42.0
58	EPA NERL	1.00	5.59	1400	39.9
59	EPA NERL	1.00	5.55	1400	39.5
60	EPA NERL	1.00	5.73	1400	40.7
61	EPA NERL	1.00	5.95	1400	42.4
62	EPA NERL	1.00	5.83	1400	41.6
63	EPA NERL	1.00	5.63	1400	40.2
64	EPA NERL	1.00	5.64	1400	40.2
65	EPA NERL	1.00	6.18	1400	44.1
66	EPA NERL	1.00	5.70	1400	40.6
67	EPA NERL	1.00	5.39	1400	38.3
68	EPA NERL	1.00	5.85	1400	41.6
69	EPA NERL	1.00	6.14	1400	43.7
70	EPA NERL	1.00	6.05	1400	43.1
71	EPA NERL	1.00	6.53	1400	46.6
72	EPA NERL	1.00	6.13	1400	43.7
73	EPA NERL	1.00	6.35	1400	45.3
74	EPA NERL	1.00	6.21	1400	44.2
75	EPA NERL	1.00	5.24	1400	37.3
76	EPA NERL	1.00	5.60	1400	40.0

Table A-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
77	EPA NERL	1.00	6.05	1400	43.1
78	EPA NERL	1.00	5.99	1400	42.6
79	EPA NERL	1.00	5.45	1400	38.9
80	EPA NERL	1.00	5.73	1400	40.8
81	EPA NERL	1.00	5.79	1400	41.2
82	EPA NERL	1.00	5.55	1400	39.5
83	EPA NERL	1.01	6.09	1400	43.1
84	EPA NERL	1.00	5.68	1400	40.4
85	EPA NERL	1.00	5.28	1400	37.6
86	EPA NERL	1.00	5.26	1400	37.5
87	EPA NERL	1.00	5.50	1400	39.2
88	EPA NERL	1.01	5.67	1400	40.2
89	EPA NERL	1.00	5.36	1400	38.2
90	EPA NERL	1.01	5.70	1400	40.5
91	EPA NERL	1.00	5.68	1400	40.4
92	EPA NERL	1.01	5.48	1400	38.8
93	EPA NERL	1.01	5.35	1400	37.9
94	EPA NERL	1.00	5.62	1400	40.0
95	EPA NERL	1.00	5.63	1400	40.1
96	EPA NERL	1.01	5.94	1400	42.0
97	EPA NERL	1.00	6.57	1400	46.9
98	EPA NERL	1.00	5.77	1400	41.2
99	EPA NERL	1.00	6.14	1400	43.8
100	EPA NERL	1.00	6.50	1400	46.5
101	EPA NERL	1.01	6.36	1400	44.9
102	EPA NERL	1.01	6.14	1400	43.5
103	EPA NERL	1.01	6.62	1400	46.7
104	EPA NERL	1.01	6.21	1400	44.0
105	EPA NERL	1.01	6.70	1400	47.5
106	EPA NERL	1.00	6.45	1400	46.1
107	EPA NERL	1.00	5.73	1400	40.8
108	EPA NERL	1.01	5.87	1400	41.7
109	EPA NERL	1.01	5.98	1400	42.5
110	EPA NERL	1.00	6.04	1400	43.0
111	EPA NERL	1.00	5.42	1400	38.6
112	EPA NERL	1.00	5.49	1400	39.1
113	EPA NERL	1.01	6.15	1400	43.6
114	EPA NERL	1.01	6.63	1400	46.9

Table A-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
115	EPA NERL	1.01	5.93	1400	42.0
116	EPA NERL	1.01	6.14	1400	43.5
117	EPA NERL	1.00	6.44	1400	45.9
118	U. Colorado	1.00	5.10	1400	36.3
119	U. Colorado	1.02	5.22	1400	36.7
120	U. Colorado	1.01	5.69	1400	40.3
121	U. Colorado	1.01	6.55	1400	46.5
122	U. Colorado	1.00	6.69	1400	47.7
123	U. Colorado	1.00	6.34	1400	45.1
124	U. Colorado	1.00	6.75	1400	48.2
125	U. Colorado	1.00	6.45	1400	46.1
126	U. Colorado	1.00	6.34	1400	45.2
127	U. Colorado	1.01	6.46	1400	45.8
128	U. Colorado	1.02	5.79	1400	40.4
129	U. Colorado	1.01	5.69	1400	40.3
130	U. Colorado	1.00	5.68	1400	40.4
131	U. Colorado	1.01	6.02	1400	42.4

[a]Data provided by Karen Bradham *(EPA ORD NERL) and John Drexler, University of Colorado
[b]NIST certificate median soil arsenic concentration

APPENDIX B

Replicate IVBA results for NIST2710A (March 2010 – January 2015)
EPA Office of Research and Development National Exposure Research Laboratory

Replicate	IVBA (%)	RPD
1	42.4	3.9
2	40.0	-1.9
3	38.5	-5.7
4	37.2	-9.2
5	40.9	0.3
6	37.6	-8.1
7	39.5	-3.2
8	43.7	6.9
9	42.5	4.1
10	42.8	4.8
11	40.9	0.3
12	39.6	-2.9
13	38.8	-5.0
14	40.9	0.3
15	41.6	2.0
16	39.0	-4.4
17	42.5	4.1
18	36.8	-10.2
19	43.4	6.2
20	43.3	6.0
21	42.5	4.1
22	42.8	4.8
23	40.9	0.3
24	39.9	-2.2
25	39.6	-2.9
26	44.9	9.6
27	38.4	-6.0
Mean	40.8.	-0.14
SD	2.2	5.32
Min	36.8	-10.25
Maximum	44.9	9.63

APPENDIX C

Data Used for Meta-analysis of IVBA Assay for Predicting Oral RBA of Arsenic

ID	As Source	Soil As (ppm)	IVBA (%)	IVBA SD (%)	RBA (%)	RBA SE (%)	RBA Assay
1	Mining/smelting	676	13.0	0.7	38.1	1.6	Swine UEF
2	Mining/smelting	313	32.5	1.6	52.4	2.0	Swine UEF
3	Pesticide (orchard)	290	21.0	1.1	31.0	4.0	Swine UEF
4	Pesticide (orchard)	388	18.6	0.9	40.8	1.8	Swine UEF
5	Pesticide (orchard)	382	19.4	0.4	48.7	4.7	Swine UEF
6	Pesticide (orchard)	364	30.6	1.5	52.8	2.3	Swine UEF
7	Mining/smelting	234	8.8	0.3	17.8	3.2	Swine UEF
8	Mining/smelting	367	6.0	0.3	23.6	2.4	Swine UEF
9	Mining/smelting	181	50.4	2.5	50.7	5.9	Swine UEF
10	Mining	200	78.0	3.9	60.2	2.7	Swine UEF
11	Mining	3957	11.0	0.6	18.6	0.9	Swine UEF
12	Mining/smelting	590	55.1	2.8	44.1	2.3	Swine UEF
13	Mining/smelting	1400	42.2	0.6	41.8	1.4	Swine UEF
14	Mining/smelting	312	41.8	2.1	40.3	3.6	Swine UEF
15	Mining/smelting	983	33.2	1.7	42.2	3.8	Swine UEF
16	Mining/smelting	390	40.3	0.7	36.7	3.3	Swine UEF
17	Mining/smelting	813	22.0	1.1	23.8	2.4	Swine UEF
18	Mining/smelting	368	18.7	0.9	21.2	2.1	Swine UEF
19	Mining/smelting	516	18.6	0.9	23.5	2.6	Swine UEF
20	Herbicide (railway corridor)	267	57.3	2.2	72.2	19.9	Swine AUC
21	Herbicide (railway corridor)	42	42.7	0.8	41.6	6.6	Swine AUC
22	Herbicide (railway corridor)	1114	17.2	0.4	20.0	9.5	Swine AUC
23	Herbicide (railway corridor)	257	10.5	0.1	10.1	2.5	Swine AUC
24	Herbicide (railway corridor)	751	22.2	0.0	22.5	2.2	Swine AUC
25	Herbicide (railway corridor)	91	80.0	0.3	80.5	6.9	Swine AUC
26	Pesticide (dip site)	713	17.8	0.1	29.3	8.7	Swine AUC
27	Pesticide (dip site)	228	55.4	0.6	43.8	5.6	Swine AUC
28	Mining	807	40.0	0.1	41.7	4.4	Swine AUC
29	Mining	577	3.8	0.0	7.0	2.9	Swine AUC
30	Gossan	190	19.0	0.2	16.4	5.2	Swine AUC
31	Gossan	88	14.0	0.2	12.1	4.9	Swine AUC
32	Pesticide	275	5.7	0.2	10.8	0.7	Swine AUC
33	Pesticide	210	7.7	0.4	12.9	1.2	Swine AUC
34	Pesticide	81	41.7	1.1	6.8	1.2	Swine AUC
35	Pesticide	358	6.5	0.1	10.1	3.5	Swine AUC
36	Pesticide	200	13.1	0.3	10.9	3.9	Swine AUC
37	Pesticide	215	7.2	0.2	18.2	3.8	Swine AUC

Data Used for Meta-analysis of IVBA Assay for Predicting Oral RBA of Arsenic

ID	As Source	Soil As (ppm)	IVBA (%)	IVBA SD (%)	RBA (%)	RBA SE (%)	RBA Assay
38	Pesticide	981	9.7	0.2	16.4	3.6	Swine AUC
39	Pesticide	1221	15.1	0.6	15.7	1.9	Swine AUC
40	Mining	949	52.9	0.1	45.8	2.6	Swine AUC
41	Mining	1126	36.9	1.1	30.7	4.1	Swine AUC
42	Mining	1695	38.1	1.3	27.5	0.7	Swine AUC
43	Mining	1306	78.4	0.4	70.5	6.8	Swine AUC
44	Mining	2270	43.5	3.4	36.2	1.5	Swine AUC
45	Mining	244	18.1	0.40	15.5	1.3	Mouse UEF
46	Mining	173	6.8	0.80	14.1	1.2	Mouse UEF
47	Mining	6899	17.5	0.60	14.7	1.0	Mouse UEF
48	Mining	280	53.6	0.20	39.9	1.7	Mouse UEF
49	Mining	4495	8.8	0.10	14.5	1.6	Mouse UEF
50	Mining	448	22.8	0.6	17.2	0.5	Mouse UEF
51	Mining	195	25.7	3.4	18.8	2.7	Mouse UEF
52	Mining/smelting	837	18.2	2.70	11.2	0.3	Mouse UEF
53	Mining/smelting	182	32.9	0.20	26.7	1.8	Mouse UEF
54	Mining/smelting	990	73.1	0.60	48.7	2.4	Mouse UEF
55	Mining/smelting	829	74.3	1.30	49.7	2.1	Mouse UEF
56	Mining/smelting	379	53.2	0.50	51.6	2.1	Mouse UEF
57	Pesticide (orchard)	322	18.8	0.30	26.3	1.4	Mouse UEF
58	Pesticide (orchard)	462	16.1	0.40	35.2	2.0	Mouse UEF
59	Pesticide (orchard)	401	18.0	0.20	20.9	2.2	Mouse UEF
60	Pesticide (orchard)	422	27.9	0.80	35.0	1.8	Mouse UEF
61	Pesticide (orchard)	340	35.4	1.90	33.2	2.4	Mouse UEF
62	Pesticide (orchard)	396	48.1	0.80	46.4	1.4	Mouse UEF
63	Pesticide (dip site)	965	9.0	0.40	21.7	1.5	Mouse UEF
64	Pesticide (dip site)	313	36.4	1.30	29.1	1.7	Mouse UEF
65	Herbicide (railway corridor)	246	47.0	2.10	45.1	2.7	Mouse UEF
66	Herbicide (railway corridor)	108	27.0	0.80	23.8	1.9	Mouse UEF
67	Herbicide (railway corridor)	184	11.9	0.20	23.0	1.8	Mouse UEF
68	Herbicide (railway corridor)	981	54.3	2.50	36.3	1.3	Mouse UEF
69	Mining	573	3.5	0.30	6.4	0.3	Mouse UEF
70	Mining	583	21.2	0.20	14.2	0.3	Mouse UEF
71	Gossan	239	12.3	0.70	20.4	1.9	Mouse UEF
72	Mining	197	21.9	0.20	29.0	2.7	Mouse UEF
73	Mining	884	16.9	0.40	23.2	3.3	Mouse UEF
74	Mining	293	12.3	0.30	17.9	0.7	Mouse UEF
75	Mining	223	17.3	0.10	19.8	1.9	Mouse UEF
76	Mining	494	15.5	0.10	18.0	1.8	Mouse UEF

Data Used for Meta-analysis of IVBA Assay for Predicting Oral RBA of Arsenic

ID	As Source	Soil As (ppm)	IVBA (%)	IVBA SD (%)	RBA (%)	RBA SE (%)	RBA Assay
77	Mining	738	13.4	3.50	11.2	0.9	Mouse UEF
78	Mining	777	0.0	0.00	4.3	0.7	Mouse UEF
79	Mining	943	0.1	0.00	3.0	0.2	Mouse UEF
80	Mining	898	0.1	0.00	1.9	0.2	Mouse UEF
81	Mining	668	0.0	0.00	3.6	0.3	Mouse UEF
82	Mining/smelting (SRM)	601	54.0	4.10	42.9	1.2	Mouse UEF
83	Mining/smelting (SRM)	1513	41.8	1.70	42.1	1.1	Mouse UEF
84	Mining/smelting (SRM)	879	14.5	0.20	14.6	0.8	Mouse UEF

As, arsenic; AUC, area under the curve; ID, sample identification number; IVBA, *in vitro* bioaccessibility; RBA, relative bioavailability; SD, standard deviation; SE, standard error; SRM, standard reference material; UEF, urinary excretion fraction

Attachment B
EPA Method 1340
Standard Operating Procedure for an In Vitro Assay for Lead and Arsenic in Soil

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

MAY - 5 2017

OFFICE OF
SOLID WASTE AND
EMERGENCY RESPONSE

NOW THE
OFFICE OF LAND AND
EMERGENCY MANAGEMENT

MEMORANDUM

SUBJECT: 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 Bioaccessibility Assay for Predicting Relative Bioavailability of Arsenic in Soils and Soil-like Materials at Superfund Sites

FROM: Schatzi Fitz-James, Acting Director
Assessment and Remediation Division
Office of Superfund Remediation and Technology Innovation (OSRTI)

TO: Superfund National Program Managers, Regions 1-10

The purpose of this memorandum is to transmit the Technical Review Workgroup (TRW) for Metals and Asbestos technical documents entitled "Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil" and "Validation Assessment of In Vitro Arsenic Bioaccessibility Assay for Predicting Relative Bioavailability of Arsenic in Soils and Soil-like Materials at Superfund Sites." The Standard Operating Procedure provides an update to EPA Method 1340 (Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil, April 2012, EPA 9200.2-86) by including an assessment of arsenic bioaccessibility. The Validation Assessment Report presents the basis for the Agency's determination that the In Vitro Bioaccessibility Assay (IVBA) method has satisfied the validation and regulatory acceptance criteria for application of the method for arsenic.

EPA Method 1340 was first published as an SW-846 Method by EPA Office of Resource Conservation and Recovery in 2013 for the assessment of lead bioaccessibility as a method to calculate Relative Bioavailability (RBA) and is now regularly used at Superfund sites. Since then, the TRW has worked to incorporate the assessment of arsenic bioaccessibility into this same method. Arsenic and lead are commonly found together at Superfund sites and accurately measuring their RBA has a significant impact on the risk assessment and on the selection of soil cleanup levels. The addition of arsenic to this method allows the arsenic RBA to be measured rapidly and inexpensively. The method does not require the use or sacrifice of animals, and the reduced cost per sample allows risk assessors to obtain a more representative number of soil samples per exposure unit. Additionally, the incorporation of arsenic into the already existing method for lead means that laboratories already have experience performing the assay.

These two documents can be accessed on the US EPA Superfund Website: https://www.epa.gov/superfund/soil-bioavailability-superfund-sites-guidance#arsenic. Please contact Matt Lambert at lambert.matthew@epa.gov or 703-603-7174 if you have any questions or concerns.

Attachments:

1. "Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead and Arsenic in Soil"
2. "Validation Assessment of In Vitro Arsenic Bioaccessibility Assay for Predicting Relative Bioavailability of Arsenic in Soils and Soil-like Materials at Superfund Sites."

cc:
James Woolford, OLEM/OSRTI
Barbara Hostage, OLEM/OPM
Reggie Cheatham, OLEM/OEM
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Sally Dalzell, OECA/FFEO
Karen Melvin and Jill Lowe, Region 3 – Lead Region
TRW Committee Members
NARPM Co-Chairs
OHHRRAF Members

OLEM 9200.2-164
April 20, 2017

Standard Operating Procedure for an *In Vitro* Bioaccessibility Assay for Lead and Arsenic in Soil

1.0 SCOPE AND APPLICATION

1.1 The purpose of this standard operating procedure (SOP) is to define the proper analytical procedure for the validated in vitro bioaccessibility (IVBA) assay for lead and arsenic in soil (U.S. EPA, 2007b, 2017), to describe the typical working range and limits of the assay, quality assurance (QA), and to indicate potential interferences. The method described herein has been validated only for lead and arsenic in soil, not other contaminants or matrices (e.g., water, air, amended soils, dust, food, etc.) (U.S. EPA, 2007b, 2017).

1.2 The SOP described herein is typically applicable for the characterization of lead and arsenic bioaccessibility in contaminated soil. Users are cautioned that deviations in the method from the assay as described may impact the results and the validity of the method. Users are strongly encouraged to document and report any deviations, as well as any comparisons, with other methods and associated Quality Assurance (QA) requirements.

1.3 This document is intended to be used as a reference for developing site-specific Quality Assurance Project Plans (QAPPs) and Sampling and Analysis Plans (SAPs), but not intended to be used as a substitute for a site-specific QAPP or a detailed SAP or laboratory Standard Operating Procedure. The information contained in this method is provided by EPA as guidance for the analyst and the regulatory community to use in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.4 Mention of trade names or commercial products does not constitute endorsement or recommended use by U.S. EPA.

1.5 For additional information on method development, see method EPA SW-846-1340 (https://www.epa.gov/sites/production/files/2015-12/documents/1340.pdf) and general information on quality assurance and hazardous waste materials test methods (https://www.epa.gov/hw-sw846/quality-assurance-and-hazardous-waste-test-methods).

2.0 DEFINITIONS

2.1 Bioavailability (BA): The fraction of an ingested dose (i.e., *in vivo*) that crosses the gastrointestinal epithelium and becomes available for distribution to internal target tissues and organs.

2.2 Absolute bioavailability: Bioavailability expressed as a fraction (or percentage) of a dose.

2.3 Relative bioavailability (RBA): The ratio of the bioavailability of a metal in one exposure context (i.e., physical chemical matrix or physical chemical form of the metal) to that in another exposure context. For example, for this method, RBA is defined as the ratio of bioavailability of lead in soil to lead in water.

2.4 Bioaccessibility: An *in vitro* measure of the physiological solubility of the metal that may be available for absorption into the body.

2.5 Batch: A group of analytical and control/QC samples that are extracted simultaneously and is limited to 20 environmental samples in addition to the batch QC samples.

2.6 Phosphate-amended soil: Phosphate rich materials (e.g., fertilizers) applied to lead-contaminated soils.

2.7 Amended soil: *In-situ* remediation approach to sequester a soil contaminant for the purpose of reducing its bioavailability and transport.

2.8 *In vitro*: Outside the living body and in an artificial environment.

2.9 *In vivo*: In the living body of an animal.

3.0 BACKGROUND AND METHOD SUMMARY

3.1 Background

Reliable analysis of the potential health hazards from ingestion of lead and arsenic in the environment depends on accurate information on a number of key parameters, including (1) concentration of metal in environmental media (soil, dust, water, food, air, etc.), (2) intake rates of each medium, and (3) the rate and extent of absorption of lead or arsenic (i.e., "bioavailability") from each medium. Knowledge of bioavailability is important because the amount of lead or arsenic that actually enters the blood and body tissues from an ingested medium depends on the physical-chemical properties of both the contaminants and the medium. For example, lead in soil may exist, at least in part, as poorly water-soluble minerals, and may also exist inside particles of inert matrices such as rock or slag of variable size, shape, and association. These chemical and physical properties may tend to influence (usually decrease) the bioavailability of lead when ingested. Thus, equal ingested amounts of different forms of lead in different media may not be of equal health concern.

Since solubilization is usually required for absorption across membranes, poorly soluble forms of metals, with low bioaccessibility, may also have low bioavailability. In certain circumstances, if solubility is the major determinant of absorption at the portal of entry, bioaccessibility may be a predictor of bioavailability. Lead is an example of this, as is further discussed in U.S. EPA (2007b).

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$$In\ vitro\ bioaccessibility\ (IVBA\%) = \frac{C_{ext} \cdot V_{ext} \cdot 100}{Soil_{conc} \cdot Soil_{mass}}$$

where:

C_{ext} = *in vitro* extractable contaminant (i.e., lead or arsenic) in the in vitro extract (mg/L)

V_{ext} = extraction solution volume (L)

$Soil_{conc}$ = contaminant concentration (i.e., lead or arsenic) in the soil sample being assayed (mg/kg)

$Soil_{mass}$ = mass of soil sample being assayed (kg)

The *in vitro* bioaccessibility assay described in this SOP provides a rapid and relatively inexpensive alternative to *in vivo* assays for predicting RBA of lead and arsenic in soils and soil-like materials (i.e., sediments, mining materials). The method, which measures the extent of metal solubilization in an extraction solvent that resembles gastric fluid, is based on the concept that solubilization of metals in gastrointestinal fluid is likely to be an important determinant of bioavailability *in vivo*. The IVBA is used to estimate the *in vivo* RBA. Measurements of IVBA using this assay have been shown to be a reliable predictor of *in vivo* RBA of lead and arsenic in a wide range of soil types and phases from a variety of different sites (U.S. EPA, 2007b, 2017).

3.2 Rationale for Method

Most previous *in vitro* test systems have employed more complex fluid intended to simulate gastric fluid. For example, Medlin (1997) used a fluid that contained pepsin and a mixture of citric, malic, lactic, acetic, and hydrochloric acids. When the bioaccessibility of a series of test substances were compared using 0.4M glycine buffer (pH 1.5) with and without the inclusion of the enzyme and metabolic acids, no significant difference was observed. This indicates that the simplified buffer employed in the procedure is appropriate, even though it lacks some constituents known to be present in gastric fluid.

The dissolution of a contaminant from a test material into the extraction fluid depends on a number of variables including extraction fluid composition, temperature, pH, time, agitation, and solid/fluid ratio. Additional discussion of these procedures is available in U.S. EPA (2007b) and Drexler and Brattin (2007). The following is a discussion of the reasons why the particular variables were established as they were for this IVBA method along with a few caveats:

Temperature. A temperature of 37°C is used because this is approximately the temperature of gastric fluid *in vivo* in humans.

pH. The human gastric pH values tend to range from 1 to 4 during fasting (see U.S. EPA, 2007b, Appendix A). A pH of 1.5 was selected because the highest amounts of lead and arsenic are extracted at pH 1.5, compared with higher pHs (Brattin et al., 2013; U.S. EPA, 2007b).

Extraction Time. The time that ingested material is present in the stomach (i.e., stomach-emptying time) is about 1 hour for a child, particularly when a fasted state is assumed (see U.S. EPA, 2007a, Appendix A). Thus, an extraction time of 1 hour should be used. It was found that allowing the bottles to stand at room temperature for up to 4 hours after rotation at 37°C caused no significant variation (<10%) in lead concentration (U.S. EPA, 2007b).

Agitation. If the test material is allowed to accumulate at the bottom of the extraction apparatus, the effective surface area of contact between extraction fluid and the test material may be reduced, which may influence the extent of contaminant solubilization. Depending on which theory of dissolution is relevant (Nernst and Brunner, 1904 or Dankwerts, 1951), agitation will greatly affect either the diffusion layer thickness or the rate of production of fresh surface. Previous workers have noted problems associated with both stirring and argon bubbling methods (Medlin, 1997). Although no systematic comparison of agitation methods was performed, an end-over-end method of agitation is recommended.

Soil/Fluid Ratio and Mass of Test Material. A solid-to-fluid ratio of 1/100 (mass per unit volume) should be used to reduce the effects of metal dissolution when lower ratios (1/5 and 1/25) are used. Tests using NIST Standard Reference Material (SRM) 2710 showed no significant variation (within ±1% of control means) in the fraction of lead extracted with soil masses as low as 0.2 g per 100 mL (U.S. EPA, 2007b). However, use of low masses of test material could introduce variability due to small scale heterogeneity in the sample and/or to weighing errors. Therefore, the final method employs 1.0 g of test material in 100 mL of extraction fluid.

In special cases, the mass of test material may need to be <1.0 g to avoid the potential for saturation of the extraction solution. Tests performed using lead acetate, lead oxide, and lead carbonate indicate that if the bulk concentration of a test material containing these relatively soluble forms of lead exceed approximately 50,000 ppm, the extraction fluid becomes saturated at 37°C and, upon cooling to room temperature and below, lead chloride crystals will precipitate. To prevent precipitation this from occurring, the concentration of lead in the test material should not exceed 50,000 ppm, or the mass of the test material should be reduced to 0.50 ± 0.01 g (U.S. EPA, 2007b). The IVBA extraction has been conducted on soils with arsenic concentrations up to 13,000 ppm (Juhasz et al., 2007). However, studies to determine if the extraction fluid becomes saturated at soil arsenic concentrations >13,000 ppm have not been conducted.

3.3 Summary of Method

After drying and sieving to 150 μm, 1 g of soil sample is rotated with 100 mL (0.1 L) of 0.4 M glycine buffered extraction fluid (pH 1.50) at 37°C for 1 hour. The supernatant is separated from the sample by filtration and analyzed for lead and/or arsenic by an appropriate analytical method (e.g., Method 6010 and Method 6020).

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4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences during sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents may be necessary.

4.2 While the predictive relationship between IVBA and RBA for lead and arsenic has been shown to be applicable to the variety of soil types, anthropogenic sources (e.g., mining operations, orchards), and elemental forms (U.S. EPA, 2007b, 2017), the bioavailability of contaminated soil is influenced by a variety of site-specific considerations and there are limitations when applying both the *in vivo* and *in vitro* assays (U.S. EPA, 2007b). As such, it is essential to identify IVBA samples containing unusual and/or untested forms of either lead or arsenic as potential sources of uncertainty. These samples will help to inform future research to better understand limits on applicability of the methods outlined in this SOP.

4.3 Excess phosphate in the sample medium may result in interference for the measurement of lead. IVBA results for phosphate-treated soils have not been shown to correlate with extraction results from juvenile swine *in vivo* assays (Scheckel et al., 2013). As a result, the methodology discussed in this SOP is not suited for lead in phosphate-amended soils. The role of phosphate on arsenic IVBA and RBA is not clear; however, phosphate amendments should be avoided in arsenic contaminated soils to avoid unintended transport. The impact of other soil amendments (i.e., iron-based or organic [compost] amendments) have not been fully examined to determine if they influence IVBA results relative to *in vivo* data.

4.4 It is not recommended to analyze lead IVBA for soils exceeding a total lead concentration of 50,000 ppm in order to avoid saturation of the extraction fluid, and because risk management decisions are not likely to be improved by analyzing IVBA for soil with concentrations of lead above this level.

4.5 The IVBA extraction for arsenic has been conducted on soils with arsenic concentrations up to 13,000 ppm (Juhasz et al., 2007). However, users should be cautioned that studies to determine if the extraction fluid becomes saturated at soil arsenic concentrations >13,000 ppm have not been conducted.

4.6 Additional information on interferences and potential problems are discussed further in Section 11.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

This method recommends the use of a water bath (Section 6.1) or an incubated air chamber (Section 6.2).

A statistical comparison (t-test) was made between the NIST SRM data for lead derived from IVBA extractions that were performed by laboratories employing air (incubator type) as the temperature controlling (37 ± 2°C) medium, versus water (aquarium type water bath). The comparison showed that, for this set of results, there was no statistical difference between the two (2) techniques of controlling the temperature of sample bottles during the extraction.

Additional testing to confirm these results was conducted by EPA's NERL and included four *in vitro* scenarios using NIST SRM 2710a (n = 27 for each scenario):

1. Water bath + preheated gastric solution
2. Water bath + room temperature gastric solution
3. Air incubator + preheated gastric solution
4. Air incubator + room temperature gastric solution

Results of the t-tests indicate that there was no statistically significant difference in observed mean Pb IVBA values for NIST 2710a SRM between scenarios 1 and 2; 1 and 3; and 2 and 3. The mean Pb IVBA value from scenario 4 (air temperature controlled, gastric solution not- preheated) was slightly lower. Therefore, the mean Pb IVBA value for scenario 4 was statistically different from the other three scenarios (Nelson et al., 2013).

6.1 Water Bath

If the water bath option is used, the specific extraction device is an electric motor (the same motor as is used in the toxicity characteristic leaching procedure (TCLP, Method 1311) driven flywheel, which drives a rotating block situated inside a temperature-controlled water bath (See Figure 1). The extraction device must be capable of holding a capped 125-mL wide-mouth high density polyethylene (HDPE) bottle. The water bath should be filled such that the extraction bottles are completely immersed. Temperature in the water bath should be maintained at 37 ± 2°C using an immersion circulator heater, and the water bath temperature should be monitored and recorded. The electric motor must be capable of 30 ± 2 rpm.

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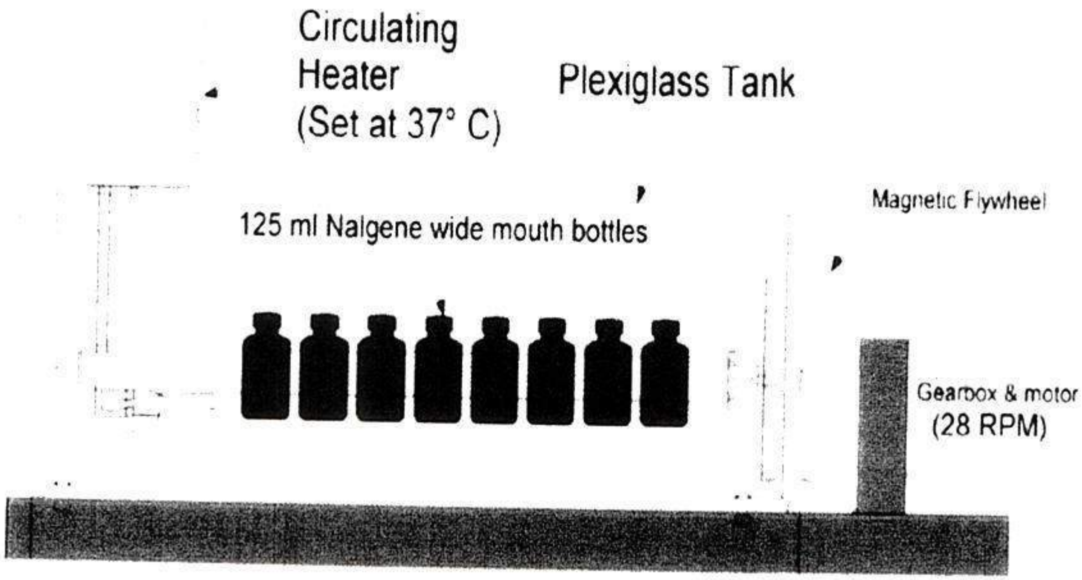
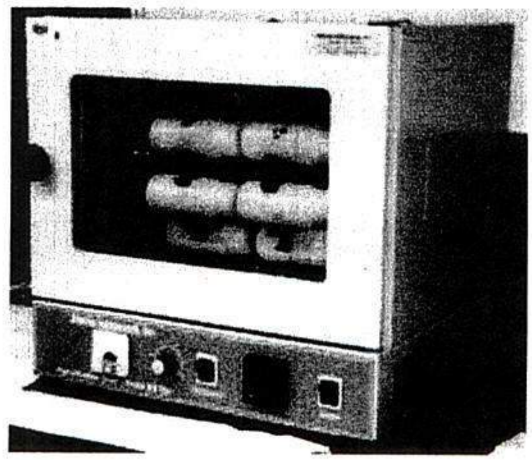
Figure 1. Example of *In Vitro* Bioaccessibility Extraction Apparatus with Water Bath.



6.2 Incubated Air Chamber
If the air incubator option is used, the specific extraction device will rotate the extraction bottles within an incubated air chamber. It must be capable of rotating at 30 ± 2 rpm and designed to hold capped 125-mL wide-mouth HDPE bottles (see Figure 2 for an example of an extraction device in an incubated air chamber). The incubator must be capable of maintaining 37 ± 2°C. The temperature inside the incubator should be monitored and recorded.

Figure 2. Example of *In Vitro* Bioaccessibility Extraction Apparatus with Air Incubator.



6.3 HDPE bottles, 125 mL in size, equipped with airtight screw-cap seals should be used. Care should be taken to ensure that the bottles do not leak and to minimize contamination during the extraction procedure.

6.4 Automated temperature compensation (ATC) pH electrode – used for measuring the pH of the extraction fluid both prior to and after the experiment

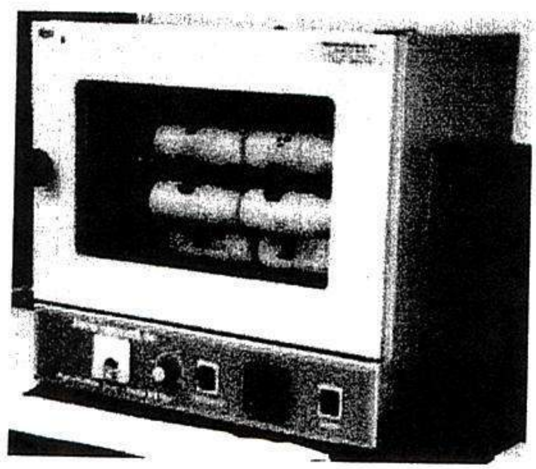
7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals, at a minimum, should be used in all tests. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 All reagents shall be free of lead and arsenic. For lead, the final extraction fluid shall be tested to confirm that lead concentrations are <¼ (<one-fourth) of the project-required detection limit (PRDL) of 100 µg/L (i.e., less than 25 µg/L lead in the unprocessed reagent blank). For arsenic, the final extraction fluid shall be tested to confirm that arsenic

6.2 *Incubated Air Chamber*
If the air incubator option is used, the specific extraction device will rotate the extraction bottles within an incubated air chamber. It must be capable of rotating at 30 ± 2 rpm and designed to hold capped 125-mL wide-mouth HDPE bottles (see Figure 2 for an example of an extraction device in an incubated air chamber). The incubator must be capable of maintaining 37 ± 2°C. The temperature inside the incubator should be monitored and recorded.

Figure 2. Example of *In Vitro* Bioaccessibility Extraction Apparatus with Air Incubator.



6.3 HDPE bottles, 125 mL in size, equipped with airtight screw-cap seals should be used. Care should be taken to ensure that the bottles do not leak and to minimize contamination during the extraction procedure.

6.4 Automated temperature compensation (ATC) pH electrode – used for measuring the pH of the extraction fluid both prior to and after the experiment

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals, at a minimum, should be used in all tests. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 All reagents shall be free of lead and arsenic. For lead, the final extraction fluid shall be tested to confirm that lead concentrations are <¼ (<one-fourth) of the project-required detection limit (PRDL) of 100 µg/L (i.e., less than 25 µg/L lead in the unprocessed reagent blank). For arsenic, the final extraction fluid shall be tested to confirm that arsenic

concentrations are <¼ (<one-fourth) of the project-required detection limit (PRDL) of 100 µg/L (i.e., less than 25 µg/L arsenic in the unprocessed reagent blank).

7.3 Reagent water must be interference free. All references to water in this method refer to reagent water, unless otherwise specified.

7.4 Cleanliness of all materials used to prepare and/or store the extraction fluid and buffer is essential. All glassware and equipment used to prepare standards and reagents shall be properly cleaned, acid washed, and triple-rinsed with deionized water prior to use.

7.5 Extraction fluid – 0.4 M glycine (free base, reagent-grade glycine in deionized water), adjusted to a pH of 1.50 ± 0.05 at 37°C using trace metal-grade concentrated hydrochloric acid (HCl).

7.5.1 Prepare 2 liters (L) of extraction fluid in a volumetric flask (Class A) using American Society for Testing and Materials (ASTM) Type II deionized (DI) water. Record within two significant digits the weight of glycine using an analytical balance and measure 1.9 L of deionized water ± 1 mL in a pre-acid washed flask. Add 60.06 ± 0.05 grams of glycine (free base) to a flask containing 1.9 L of deionized water. Glycine should be weighed using an analytical balance calibrated daily according to the manufacturer's instructions. Solution can be transferred to a wide-mouth HDPE bottle for ease of handling. Place the HDPE bottle containing the extraction fluid in a water bath at 37°C and heat until the extraction fluid reaches 37°C. Standardize the pH meter using an ATC pH electrode at 37°C or pH buffers maintained at 37°C in the water bath. Add trace metal-grade concentrated HCl (12.1 N) until the solution pH reaches 1.50 ± 0.05. Bring the solution to a final volume of 2 L (0.4 M glycine).

7.5.2 If the extraction fluid is prepared in advance of the extraction, the extraction fluid must be heated to 37°C and the pH shall be adjusted to 1.5 using trace metal grade concentrated HCl prior to conducting the extraction batch.

8.0 SOIL SAMPLE PREPARATION, PRESERVATION, AND STORAGE

8.1 All test soils should be prepared by drying (<40°C) and sieving to <150 µm. The <150 µm size fraction was used because this particle size is representative of that which adheres to children's hands (U.S. EPA, 2016). Stainless steel sieves are recommended. Samples should be thoroughly mixed prior to use to ensure homogenization. Mixing and aliquoting of samples using a riffle splitter is recommended. Clean HDPE storage bottles are recommended.

8.2 To perform this assay, soil standards and test soils should be weighed using an analytical balance calibrated daily according to the manufacturer's instructions. Soil samples should be weighed to ***four significant digits*** (i.e., the nearest 0.0001 gram).

8.3 All samples should be archived after analysis and retained for further analysis for a period of six (6) months. No preservatives or special storage conditions are required.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulatory community in making judgments necessary to generate results that meet the DQOs for the intended application.

9.1 Initial demonstration of proficiency (IDP)
Each laboratory must demonstrate initial proficiency by generating data of acceptable precision and bias for target analytes in a clean matrix. It is recommended that the laboratory repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation and/or procedures are made.

9.2 Quality assurance for the extraction procedure are as follows (summarized in Table 1 for lead and Table 2 for arsenic):

9.2.1 Reagent blank: Unprocessed (not run through the extraction procedure) extraction fluid should be analyzed for each new batch of extraction fluid. The reagent blank is considered within control limits if its result is less than the lower limit of quantitation (LLOQ). The corrective action for a blank hit above LLOQ should include preparing a new batch of extraction fluid and reprocessing any samples that were prepared with the failing reagent fluid. The reagent blank should be run at a frequency of 1 in 20 samples (minimum of one per batch).

9.2.2 Method blank: Extraction fluid only (i.e., no test soil) is carried through all steps of the method at a frequency of 1 in 20 samples (minimum of 1 per batch). The method blank is considered within control limits if its result is less than the LLOQ. The corrective action for a recovery above the LLOQ should include making a new extraction fluid and reprocessing any samples that were prepared with the failing method blank.

9.2.3 Laboratory Control Sample (LCS): A LCS consisting of a spiked blank should be run once per batch (minimum 1 in 20 samples). The LCS may be spiked with the same source as the calibration standards and needs to be carried through all steps of the rotation procedure. The extraction fluid should be spiked at either 10 mg/L lead or 10 mg/L arsenic. The control limits are 85–115% recovery. The corrective action for outliers should include an analyst review that all dilutions and spike concentrations were performed correctly. If no error is found, either re-extract the samples or flag and narrate the defect and possible bias in the data.

8.3 All samples should be archived after analysis and retained for further analysis for a period of six (6) months. No preservatives or special storage conditions are required.

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Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulatory community in making judgments necessary to generate results that meet the DQOs for the intended application.

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9.2 Quality assurance for the extraction procedure are as follows (summarized in Table 1 for lead and Table 2 for arsenic):

9.2.1 Reagent blank: Unprocessed (not run through the extraction procedure) extraction fluid should be analyzed for each new batch of extraction fluid. The reagent blank is considered within control limits if its result is less than the lower limit of quantitation (LLOQ). The corrective action for a blank hit above LLOQ should include preparing a new batch of extraction fluid and reprocessing any samples that were prepared with the failing reagent fluid. The reagent blank should be run at a frequency of 1 in 20 samples (minimum of one per batch).

9.2.2 Method blank: Extraction fluid only (i.e., no test soil) is carried through all steps of the method at a frequency of 1 in 20 samples (minimum of 1 per batch). The method blank is considered within control limits if its result is less than the LLOQ. The corrective action for a recovery above the LLOQ should include making a new extraction fluid and reprocessing any samples that were prepared with the failing method blank.

9.2.3 Laboratory Control Sample (LCS): A LCS consisting of a spiked blank should be run once per batch (minimum 1 in 20 samples). The LCS may be spiked with the same source as the calibration standards and needs to be carried through all steps of the rotation procedure. The extraction fluid should be spiked at either 10 mg/L lead or 10 mg/L arsenic. The control limits are 85–115% recovery. The corrective action for outliers should include an analyst review that all dilutions and spike concentrations were performed correctly. If no error is found, either re-extract the samples or flag and narrate the defect and possible bias in the data.

9.2.4 Matrix Spike (MS): A MS should be run once per batch (minimum 1 in 20 samples). The MS should be prepared after extraction and filtration of the supernatant. The matrix spike should be prepared at either 10 mg/L lead and/or 10 mg/L arsenic. The control limits are 75–125% recovery. The corrective action for outliers should include an analyst review that all dilutions and spike concentrations were performed correctly. If no error is found, either re-extract the samples or flag and narrate the defect and possible bias in the data.

9.2.5 Duplicate sample: A duplicate sample should be run once per batch (minimum 1 in 20 samples) and carried through all steps of the method. The relative percent difference (RPD) should be less than 20%. The corrective action for outliers should include either re-extraction of the samples or flagging the data.

9.2.6 Control soils for Lead: The National Institute of Standards and Testing (NIST) standard reference materials (SRMs) 2710a or 2711a (Montana Soil) can be used as control soils. The reference material shall be carried through all steps of the method and analyzed at a frequency of 1 in 20 samples (minimum of 1 per batch). The IVBA is calculated using the equation shown in Section 12.1.

9.2.6.1 NIST SRM 2710a: Analysis of the NIST SRM 2710a for lead should yield a mean IVBA result of 67.5%, with an acceptable range of 60.7–74.2%. The IVBA result in terms of mg/kg should be 3,440 mg/kg, with a range of 3,096–3,785 mg/kg (Shaw Environmental, Inc., 2011). For the lead concentration (Pb_{soil}) in the SRM, the median lead concentration presented in the Addendum to the NIST certificate for leachable concentrations determined using Method 3050 (5,100 mg/kg) should be used (NIST, 2009a).

9.2.6.2 NIST SRM 2711a: Analysis of the NIST SRM 2711a for lead should yield a mean IVBA result of 85.7%, with an acceptable IVBA range of 75.2–96.2%. The IVBA result in terms of mg/kg should be 1,114 mg/kg, with a range of 980–1,249 mg/kg (Shaw Environmental, Inc., 2011). For the lead concentration (Pb_{soil}) in the SRM, the median lead concentration presented in the Addendum to the NIST certificate for leachable concentrations determined using Method 3050 (1,300 mg/kg) should be used (NIST, 2009b).

9.2.7 Control soils for Arsenic

Note: NIST SRM 2711a is not an appropriate control soil for the IVBA assay for arsenic due to the low arsenic concentration.

9.2.7.1 NIST SRM 2710a: Analysis of the NIST SRM 2710a for arsenic should yield a mean IVBA result of 41.0%, with an acceptable IVBA range 32.9–49.1% (Appendix B). For the arsenic concentration (As_{soil}) in NIST 2710a, the median lead concentration presented in the Addendum to the

NIST certificate for leachable concentrations determined using Method 3050 (1,400 mg/kg) should be used (NIST, 2009a).

9.3 Lower limit of quantitation check standard

9.3.1 The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. The LLOQ should be verified by the analysis of at least seven (7) replicate samples, which are spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases, the mean recovery should be ±35% of the true value and the RSD should be ≤20%. In-house limits may be calculated when sufficient data points exist. The monitoring of recovery data for the LLOQ check standard over time is useful for assessing precision and bias. Refer to a scientifically valid and published method (such as Chapter 9 of *Quality Assurance of Chemical Measurements* [Taylor, 1987] or the Report of the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs [http://water.epa.gov/scitech/methods/cwa/det/index.cfm]) for calculating precision and bias for LLOQ.

9.3.2 Ongoing LLOQ verification, at a minimum, is carried out on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than or equal to the desired regulatory action levels based on the stated project-specific requirements.

NIST certificate for leachable concentrations determined using Method 3050 (1,400 mg/kg) should be used (NIST, 2009a).

9.3 Lower limit of quantitation check standard

9.3.1 The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. The LLOQ should be verified by the analysis of at least seven (7) replicate samples, which are spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases, the mean recovery should be ±35% of the true value and the RSD should be ≤20%. In-house limits may be calculated when sufficient data points exist. The monitoring of recovery data for the LLOQ check standard over time is useful for assessing precision and bias. Refer to a scientifically valid and published method (such as Chapter 9 of *Quality Assurance of Chemical Measurements* [Taylor, 1987] or the Report of the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs [http://water.epa.gov/scitech/methods/cwa/det/index.cfm]) for calculating precision and bias for LLOQ.

9.3.2 Ongoing LLOQ verification, at a minimum, is carried out on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than or equal to the desired regulatory action levels based on the stated project-specific requirements.

Table 1. Recommended Control Limits for Quality Control Samples: Lead

Analysis	Frequency	Control Limits	Corrective Action
Reagent blank	once per batch (minimum 1 in 20 samples)	<25 µg/L lead	Make new extraction fluid and rerun all analyses.
Method blank	once per batch (minimum 1 in 20 samples)	<50 µg/L lead	Make new extraction fluid and rerun all analyses.
LCS (10 mg/L)	once per batch (minimum 1 in 20 samples)	85–115% recovery	Ensure dilutions and spike concentrations are correct. If no error is found, re-extract the samples or flag the data.
Matrix spike (10 mg/L)	once per batch (minimum 1 in 10 samples)	75–125% recovery	Ensure dilutions and spike concentrations are correct. If no error is found, re-extract the samples or flag the data.
Duplicate sample	once per batch (minimum 1 in 10 samples)	±20% RPD	Re-extract the samples or flag the data.
Control soil (NIST SRMs 2710a and 2711a)	once per batch (minimum 1 in 20 samples)	NIST 2710a mean 67.5% (acceptable range: 60.7–74.2%) NIST 2711a mean 85.7% (acceptable range: 75.2–96.2%)	Re-extract the samples or flag the data.

RPD, Relative percent difference

Table 2. Recommended Control Limits for Quality Control Samples: Arsenic

Analysis	Frequency	Control Limits	Corrective Action
Reagent blank	once per batch (minimum 1 in 20 samples)	<25 μg/L arsenic	Make new extraction fluid and rerun all analyses.
Method blank	once per batch (minimum 1 in 20 samples)	<50 μg/L arsenic	Make new extraction fluid and rerun all analyses.
LCS (10 mg/L)	once per batch (minimum 1 in 20 samples)	85–115 % recovery	Ensure dilutions and spike concentrations are correct. If no error is found, re-extract the samples or flag the data.
Matrix spike (10 mg/L)	once per batch (minimum 1 in 10 samples)	75–125% recovery	Ensure dilutions and spike concentrations are correct. If no error is found, re-extract the samples or flag the data.
Duplicate sample	once per batch (minimum 1 in 10 samples)	±20% RPD	Re-extract the samples or flag the data.
Control soil (NIST 2710a)	once per batch (minimum 1 in 20 samples)	NIST 2710a mean 41.0% (acceptable range: 32.9–49.1%)	Re-extract the samples or flag the data.

RPD, Relative percent difference

10.0 CALIBRATION AND STANDARDIZATION

10.1 An automated temperature compensation (ATC) pH electrode shall be used for measuring the pH of the extraction fluid prior and post experiment. Each instrument/ electrode system must be calibrated at a minimum of two points that bracket the expected pH (1.5) of the samples and are approximately two pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value as indicated in SW-846 method 9045D for Soil and Waste pH (http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9045d.pdf). The pH meter should be calibrated and checked with standard solutions within the calibration range (e.g., pH = 1 and 2) according to the manufacturer's instructions. After calibration, the meter is ready to analyze samples.

10.2 Thermometers capable of measuring 37 ± 2°C are needed.

10.3 The analytical balance should be calibrated daily in accordance with the manufacturer's instructions.

10.4 Pipettes should be calibrated in accordance with the manufacturer's instructions and the laboratory QA plan.

Table 2. Recommended Control Limits for Quality Control Samples: Arsenic

Analysis	Frequency	Control Limits	Corrective Action
Reagent blank	once per batch (minimum 1 in 20 samples)	<25 μg/L arsenic	Make new extraction fluid and rerun all analyses.
Method blank	once per batch (minimum 1 in 20 samples)	<50 μg/L arsenic	Make new extraction fluid and rerun all analyses.
LCS (10 mg/L)	once per batch (minimum 1 in 20 samples)	85–115 % recovery	Ensure dilutions and spike concentrations are correct. If no error is found, re-extract the samples or flag the data.
Matrix spike (10 mg/L)	once per batch (minimum 1 in 10 samples)	75–125% recovery	Ensure dilutions and spike concentrations are correct. If no error is found, re-extract the samples or flag the data.
Duplicate sample	once per batch (minimum 1 in 10 samples)	±20% RPD	Re-extract the samples or flag the data.
Control soil (NIST 2710a)	once per batch (minimum 1 in 20 samples)	NIST 2710a mean 41.0% (acceptable range: 32.9–49.1%)	Re-extract the samples or flag the data.

RPD, Relative percent difference

10.0 CALIBRATION AND STANDARDIZATION

10.1 An automated temperature compensation (ATC) pH electrode shall be used for measuring the pH of the extraction fluid prior and post experiment. Each instrument/ electrode system must be calibrated at a minimum of two points that bracket the expected pH (1.5) of the samples and are approximately two pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value as indicated in SW-846 method 9045D for Soil and Waste pH (http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9045d.pdf). The pH meter should be calibrated and checked with standard solutions within the calibration range (e.g., pH = 1 and 2) according to the manufacturer's instructions. After calibration, the meter is ready to analyze samples.

10.2 Thermometers capable of measuring 37 ± 2°C are needed.

10.3 The analytical balance should be calibrated daily in accordance with the manufacturer's instructions.

10.4 Pipettes should be calibrated in accordance with the manufacturer's instructions and the laboratory QA plan.

11.0 PROCEDURE

11.1 The extraction fluid for this procedure is 0.4 M glycine (free base, reagent grade glycine in deionized water), adjusted to a pH of 1.50 ± 0.05 at 37 ± 2°C using trace metal grade concentrated hydrochloric acid (HCl). See Section 7.5 for extraction fluid preparation details.

11.2 Pre-heat the TCLP extractor water bath OR incubator (See Section 6.0) to 37°C. Record the temperature at the beginning and end of each extraction batch (an example of an extraction data recording form is provided in Appendix A).

11.3 Soil samples should be thoroughly mixed immediately prior to removing aliquots for extraction to ensure homogenization (i.e., rotate sample bottles using X, Y, Z motion).

11.4 The extraction procedure is begun by placing 1.00 ± 0.05 g of sieved test material (<150 µm; U.S. EPA, 2016) into a 125-mL wide-mouth HDPE bottle. **Record weight of soil to *four significant digits*** (i.e., the nearest 0.0001 gram). Care should be taken to ensure that static electricity does not cause soil particles to adhere to the lip or outside threads of the bottle; if necessary, an antistatic brush should be used to eliminate static electricity prior to adding the test substrate.

11.5 Measure 100 ± 0.5 mL of the 37 ± 2°C buffered extraction fluid (0.4 M glycine, pH 1.5), using a graduated cylinder or automated dispenser, and transfer extraction fluid to the 125-mL wide-mouth HDPE bottle.

11.6 The bottle should be tightly sealed and then shaken or inverted to ensure that there is no leakage and that no soil is caked on the bottom of the bottle.

11.7 Fill the extractor (TCLP extractor OR rotating extractor inside of a pre-heated incubator, see Section 6.0 for details) with 125-mL bottles containing test materials or Quality Control samples (see Section 7.0). Record start time of rotation.

NOTE: Care should be taken to prevent contamination of the samples during rotation (e.g., getting bath water in the threads around the cap and possibly into the sample when the cap is removed). Precautions that laboratories may consider include but are not limited to: the type of bottle that is used, sealing the samples in plastic freezer bags with air expelled before installing in the water bath extractor, and/or sealing the bottles with tape or Parafilm®.

11.8 Samples are extracted by rotating the samples at 30 ± 2 rpm for 1 hour.

11.9 After 1 hour, the bottles should be removed from the rotator, dried, and placed upright on the bench top to allow the soil to settle to the bottom.

11.10 A 40-mL sample of supernatant fluid is then removed directly from the extraction bottle into a disposable syringe. After withdrawal of the sample into the syringe, a Luer-Lok attachment fitted with a 0.45-µm cellulose acetate disk filter (25 mm diameter) is attached, and the 15 mL aliquot of fluid is filtered through the attachment to remove any particulate matter into a pre-acid washed polypropylene centrifuge tube or other appropriate sample vial for analysis.

11.11 Record the time that the extract is filtered (i.e., extraction is stopped). If the total time elapsed for the extraction and filtration process exceeds 90 minutes, the test must be repeated (i.e., Steps 11.1–11.10).

11.12 Measure and record the pH of fluid remaining in the extraction bottle. If the fluid pH is not within ±0.5 pH units of the starting pH, the test must be discarded and the sample re-analyzed. In some cases (mainly slag soils), the test material can increase the pH of the extraction buffer, and this could influence the results of the bioaccessibility measurement. To guard against this, the pH of the fluid should be measured at the end of the extraction step (just after a sample was withdrawn for filtration and analysis). If the pH is not within 0.5 pH units of the starting pH (1.5), the sample should be re-analyzed. If the second test also results in an increase in pH of >0.5 units, it is reasonable to conclude that the test material is buffering the solution. In these cases, the test should be repeated using manual pH adjustment during the extraction process, stopping the extraction at 5, 10, 15, and 30 minutes and manually adjusting the pH down to pH 1.5 at each interval by drop-wise addition of HCl.

11.13 Store filtered sample(s) in a refrigerator at 4 ± 2°C until they are analyzed. This filtered sample of extraction fluid is then analyzed for lead and/or arsenic by ICP-AES or ICP-MS (U.S. EPA Method 6010C or Method 6020A). For lead, the method detection limit (MDL) in extraction fluid should be approximately 20 µg/L for Method 6010 and 0.1–0.3 µg/L for Method 6020 (U.S. EPA, 2012a, b). For arsenic, the MDL in extraction fluid should be approximately 20–40 µg/L for Method 6010 and 1–5 µg/L for Method 6020.

NOTE: In some cases, high dissolved solids (e.g., Fe oxides) in the extracts may cause nebulizer performance issues by inductively coupled plasma-optical emission spectrometry (ICP-OES) or inductively coupled plasma-mass spectrometry (ICP-MS). If this is encountered, dilution of the extracts tenfold is recommended before analysis. Correct for any dilutions in the calculations. Alternately, a high solids nebulizer may be useful. Graphite furnace atomic absorption spectrophotometry (GFAA) should be avoided due to the high levels of HCl in the extracts.

NOTE: In some cases, the amount of lead present in the sample will begin to saturate the extraction fluid, and the extraction response will cease to be linear. If the concentration of lead in the extract exceeds approximately 500 mg/L (depending on the sample matrix and mineralogy), this upper limit may have been reached. It is not recommended to analyze IVBA for soils exceeding a total lead concentration of 50,000 ppm in order to avoid saturation of the extraction fluid, and because risk management decisions are not likely to be improved by analyzing IVBA for soil with concentrations of lead above this level.

11.14. Examples of an extraction record, gastric extraction fluid preparation record, and an example batch format and IVBA calculation are provided in Appendix A (Tables A1–A3).

attached, and the 15 mL aliquot of fluid is filtered through the attachment to remove any particulate matter into a pre-acid washed polypropylene centrifuge tube or other appropriate sample vial for analysis.

11.11 Record the time that the extract is filtered (i.e., extraction is stopped). If the total time elapsed for the extraction and filtration process exceeds 90 minutes, the test must be repeated (i.e., Steps 11.1–11.10).

11.12 Measure and record the pH of fluid remaining in the extraction bottle. If the fluid pH is not within ±0.5 pH units of the starting pH, the test must be discarded and the sample re-analyzed. In some cases (mainly slag soils), the test material can increase the pH of the extraction buffer, and this could influence the results of the bioaccessibility measurement. To guard against this, the pH of the fluid should be measured at the end of the extraction step (just after a sample was withdrawn for filtration and analysis). If the pH is not within 0.5 pH units of the starting pH (1.5), the sample should be re-analyzed. If the second test also results in an increase in pH of >0.5 units, it is reasonable to conclude that the test material is buffering the solution. In these cases, the test should be repeated using manual pH adjustment during the extraction process, stopping the extraction at 5, 10, 15, and 30 minutes and manually adjusting the pH down to pH 1.5 at each interval by drop-wise addition of HCl.

11.13 Store filtered sample(s) in a refrigerator at 4 ± 2°C until they are analyzed. This filtered sample of extraction fluid is then analyzed for lead and/or arsenic by ICP-AES or ICP-MS (U.S. EPA Method 6010C or Method 6020A). For lead, the method detection limit (MDL) in extraction fluid should be approximately 20 µg/L for Method 6010 and 0.1–0.3 µg/L for Method 6020 (U.S. EPA, 2012a, b). For arsenic, the MDL in extraction fluid should be approximately 20–40 µg/L for Method 6010 and 1–5 µg/L for Method 6020.

NOTE: In some cases, high dissolved solids (e.g., Fe oxides) in the extracts may cause nebulizer performance issues by inductively coupled plasma-optical emission spectrometry (ICP-OES) or inductively coupled plasma-mass spectrometry (ICP-MS). If this is encountered, dilution of the extracts tenfold is recommended before analysis. Correct for any dilutions in the calculations. Alternately, a high solids nebulizer may be useful. Graphite furnace atomic absorption spectrophotometry (GFAA) should be avoided due to the high levels of HCl in the extracts.

NOTE: In some cases, the amount of lead present in the sample will begin to saturate the extraction fluid, and the extraction response will cease to be linear. If the concentration of lead in the extract exceeds approximately 500 mg/L (depending on the sample matrix and mineralogy), this upper limit may have been reached. It is not recommended to analyze IVBA for soils exceeding a total lead concentration of 50,000 ppm in order to avoid saturation of the extraction fluid, and because risk management decisions are not likely to be improved by analyzing IVBA for soil with concentrations of lead above this level.

11.14. Examples of an extraction record, gastric extraction fluid preparation record, and an example batch format and IVBA calculation are provided in Appendix A (Tables A1–A3).

11.15. Once received by the laboratory, all samples and extracts should be checked-in, verified, and maintained under standard chain-of-custody (e.g., U.S. EPA, 2012c).

12.0 DATA ANALYSIS AND CALCULATIONS

A split of each solid material (sieved to <150 μm) that has been subjected to this extraction procedure should be analyzed for total lead and/or total arsenic concentration using analytical procedures taken from the U.S. EPA SW-846 (U.S. EPA, 2012d) or a non-destructive method such as Instrumental Neutron Activation Analysis. If SW-846 methods are used, the solid material should be acid digested according to SW-846 Method 3050B (December 1996 revision) or 3051A (microwave-assisted digestion, February 2007 revision), and the digestate analyzed for lead and/or arsenic concentrations determined by ICP-AES analysis (Method 6010C, February 2007 revision) or ICP-MS (Method 6020A, February 2007 revision). Note that although SW-846 Method 3050B states a hot plate is acceptable as a heating source, a hot plate should not be used; the heating source should be a block digestor.

12.1 *In vitro* bioaccessibility (IVBA) is calculated and expressed on a percentage basis using the following equation:

$$In\ vitro\ bioaccessibility = \frac{C_{ext} \cdot V_{ext} \cdot 100}{Soil_{conc} \cdot Soil_{mass}}$$

where:

C_{ext} = *in vitro* extractable contaminant (i.e., lead/arsenic) in the in vitro extract (mg/L)

V_{ext} = extraction solution volume (L)

$Soil_{conc}$ = contaminant concentration (i.e., lead/arsenic) in the soil sample being assayed (mg/kg)

$Soil_{mass}$ = mass of soil sample being assayed (kg)

12.2 In order for an *in vitro* bioaccessibility test system to be useful in predicting the *in vivo* RBA of a test material, it is necessary to empirically establish that a strong correlation exists between the *in vivo* and the *in vitro* results across many different samples. The currently preferred models for predicting RBA from IVBA for lead (U.S. EPA, 2007b) and arsenic (Diamond et al., 2016; U.S. EPA, 2017) are:

$$RBA_{lead}(\%) = 0.88 \bullet IVBA(\%) - 0.028\ (R^2 = 0.92)$$

$$RBA_{arsenic}(\%) = 0.79 \bullet IVBA(\%) + 3.0\ (R^2 = 0.87)$$

where RBA and IVBA are expressed as percentages (not fractions). It is important to recognize that use of this equation to calculate RBA from a given IVBA measurement will yield the "typical" RBA value expected for a test material with that IVBA, and the true RBA may be somewhat different (either higher or lower).

12.3 If dilutions were performed, apply the appropriate corrections to the sample values.

13.0 METHOD PERFORMANCE

13.1 Method Performance for Lead. NIST SRMs 2710a and 2711a should be used as control soils for lead. The soil standard will be analyzed at a frequency of 1 in 20 samples (minimum 1 per batch). The NIST SRMs 2710a and 2711a are available from the National Institute of Standards and Technology, Standard Reference Materials Program (http://www.nist.gov/srm/). Acceptable performances of soil standards for lead are shown in Table 3. The calculations for percent Pb IVBA is shown in Section 12.1.

Table 3. Method Performance for Lead

Soil Standard	Mean mg/kg Result	Acceptable mg/kg Range	Mean IVBA Result (%)	Acceptable IVBA Range (%)
NIST 2710a	3,440	3,096–3,785	67.5	60.7–74.2
NIST 2711a	1,114	980–1,249	85.7	75.2–96.2

13.2 Method Performance for Arsenic. NIST SRM 2710a should be used as a control soil for arsenic. The soil standard will be analyzed at a frequency of 1 in 20 samples (minimum 1 per batch). The NIST SRM 2710a is available from the National Institute of Standards and Technology, Standard Reference Materials Program (http://www.nist.gov/srm/). Acceptable performances of soil standards for arsenic are shown in Table 4. The calculation for percent As IVBA is shown in Section 12.1.

Table 4. Method Performance for Arsenic

Soil Standard	Mean mg/kg Result	Acceptable mg/kg Range	Mean IVBA Result (%)	Acceptable IVBA Range (%)
NIST 2710a	1400	1300–1600	41.0	32.9–49.1

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice (SW-846). Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

12.3 *If dilutions were performed*, apply the appropriate corrections to the sample values.

13.0 METHOD PERFORMANCE

13.1 Method Performance for Lead. NIST SRMs 2710a and 2711a should be used as control soils for lead. The soil standard will be analyzed at a frequency of 1 in 20 samples (minimum 1 per batch). The NIST SRMs 2710a and 2711a are available from the National Institute of Standards and Technology, Standard Reference Materials Program (http://www.nist.gov/srm/). Acceptable performances of soil standards for lead are shown in Table 3. The calculations for percent Pb IVBA is shown in Section 12.1.

Table 3. Method Performance for Lead

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Table 4. Method Performance for Arsenic

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NIST 2710a	1400	1300–1600	41.0	32.9–49.1

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice (SW-846). Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, DC 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices are consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult The Waste Management Manual for Laboratory Personnel, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, DC 20036, (202) 872-4477.

16.0 REFERENCES

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APPENDIX A

IVBA Extraction Forms and Calculation

Table A-1. Example Extraction Record

Date:
Sample ID:
BATCH No:
Extraction Fluid ID: Glycine & HCl, pH 1.5; SRM ID:
Spike solution concentration: 10 mg/L Pb or 10 mg/L As
Lead and/or As Spiking Solution Vendor, Lot No. (X mL of standard added to X mL extraction solutions (100 mL total volume) labeled as "spikes")

	Sample Preparation			Extraction					
Sample ID	**Bottle No.**	**Volume (mL)**	**Sample mass (g)**	**Agitation Time (min)**	**Initial pH**	**Final pH**	**Start Temp (C)**	**End Temp (C)**	**Total Time[a] (min)**
Acceptable Range		**100 ± 0.5**	**1.00 ± 0.01**	**60 ± 5**	**1.50 ± 0.5**	**1.50 ± 0.5**	**37 ± 2**	**37 ± 2**	**≤90**
Method Blank	1								
LCS	2								
Control Soil	3								
Sample ID	4								
Sample ID	5								
Sample ID	6								
Sample ID	7								
Sample ID	8								
Sample ID	9								
Sample ID	10								
Sample ID	11								
Sample ID	12								
Regent blank	13								

Reagent blank is not extracted through the *in vitro* process.
[a]Time between start of agitation and filtration

APPENDIX A

IVBA Extraction Forms and Calculation

Table A-1. Example Extraction Record

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Sample ID:
BATCH No:
Extraction Fluid ID: Glycine & HCl, pH 1.5; SRM ID:
Spike solution concentration: 10 mg/L Pb or 10 mg/L As
Lead and/or As Spiking Solution Vendor, Lot No. (X mL of standard added to X mL extraction solutions (100 mL total volume) labeled as "spikes")

	Sample Preparation			Extraction					
Sample ID	**Bottle No.**	**Volume (mL)**	**Sample mass (g)**	**Agitation Time (min)**	**Initial pH**	**Final pH**	**Start Temp (C)**	**End Temp (C)**	**Total Time[a] (min)**
Acceptable Range		**100 ± 0.5**	**1.00 ± 0.01**	**60 ± 5**	**1.50 ± 0.5**	**1.50 ± 0.5**	**37 ± 2**	**37 ± 2**	**≤90**
Method Blank	1								
LCS	2								
Control Soil	3								
Sample ID	4								
Sample ID	5								
Sample ID	6								
Sample ID	7								
Sample ID	8								
Sample ID	9								
Sample ID	10								
Sample ID	11								
Sample ID	12								
Regent blank	13								

Reagent blank is not extracted through the *in vitro* process.
[a]Time between start of agitation and filtration

Table A-2. Gastric Extraction Fluid Preparation

Sample Batch No:
Date Prepared:

Component	**Lot ID**	**Fluid Preparation**		**Actual Quantity**	**Comments**
		1L	**2L**		
Deionized water	ASTM Type II	0.95 L (approximate)	1.90 L (approximate)		
Glycine	Sigma Lot No.	30.04 ± 0.05g	60.08 ± 0.05g		
HCl (12.1N; Tr. Metal)	Fisher Optima	(approximate)	(approximate)		
Final Volume	—	1.0 L (class A)	2.0 L (class A)		
pH at 37°C	—	1.50 ± 0.05	1.50 ± 0.05		

Table A3. Example Batch Format and IVBA Calculation

Date: Sample ID:
Batch No.
Extraction Fluid ID: Glycine & HCl, pH 1.5
SRM ID:
Spike solution concentration: 10 mg/L Pb
Lead Spiking Solution Vendor, Lot No. (X mL of standard added to X mL extraction solutions (100 mL total volume) labeled as "spikes")

Batch #	Bottle No.	Type	Sample ID	Soil weight (grams)	Soil weight (kg)	Volume (mL)	Volume (L)	ICP (Pb) (mg/L)	Soil [Pb] (mg/kg)	% IVBA	Avg % IVBA	SD of IVBA%
Insert No.	1	Method Blank	Method blank	n/a	n/a	100	0.1			n/a		
	2	LCS	LCS	n/a	n/a	100	0.1			n/a		
	3	Control soil	SRM 2710a	1.0019	0.00100	100	0.1	34.24	5100	67		
	4	Sample	Sample1 a	1.0016	0.00100	100	0.1	32.24	5100	63		
	5	Sample	Sample1 b	1.0006	0.00100	100	0.1	33.24	5100	65	64.1	1.4
	6	Matrix spike	Sample + spike	0.9985	0.00100	100	0.1					
	7	Sample	Sample2 a	1.0029	0.00100	100	0.1				Avg of Dups	SD
	8	Sample	Sample2 b	1.0022	0.00100	100	0.1					
	9	Matrix spike	Sample + spike	1.0028	0.00100	100	0.1					
	10	Sample	Sample3 a	1.0004	0.00100	100	0.1				Avg of Dups	SD
	11	Sample	Sample3 b	1.0029	0.00100	100	0.1					
	12	Matrix spike	Sample + spike	0.9972	0.00100	101	0.1		n/a	n/a		
	13	Reagent blank	unprocessed sample	n/a	n/a	100	0.1		n/a	n/a		

$$\% IVBA = \frac{(\text{Concentration in IVBA extract mg/L})\ (0.1\ \text{L})}{(\text{Concentration in solid mg/kg})\ (\text{weight of sample kg})} \bullet 100$$

Table A3. Example Batch Format and IVBA Calculation

Date: Sample ID:
Batch No.
Extraction Fluid ID: Glycine & HCl, pH 1.5
SRM ID:
Spike solution concentration: 10 mg/L Pb
Lead Spiking Solution Vendor, Lot No. (X mL of standard added to X mL extraction solutions (100 mL total volume) labeled as "spikes")

Batch #	Bottle No.	Type	Sample ID	Soil weight (grams)	Soil weight (kg)	Volume (mL)	Volume (L)	ICP (Pb) (mg/L)	Soil [Pb] (mg/kg)	% IVBA	Avg % IVBA	SD of IVBA%
Insert No.	1	Method Blank	Method blank	n/a	n/a	100	0.1					
	2	LCS	LCS	n/a	n/a	100	0.1			n/a		
	3	Control soil	SRM 2710a	1.0019	0.00100	100	0.1			n/a		
	4	Sample	Sample1 a	1.0016	0.00100	100	0.1	34.24	5100	67		
	5	Sample	Sample1 b	1.0006	0.00100	100	0.1	32.24	5100	63		
	6	Matrix spike	Sample + spike	0.9985	0.00100	100	0.1	33.24	5100	65	64.1	1.4
	7	Sample	Sample2 a	1.0029	0.00100	100	0.1					
	8	Sample	Sample2 b	1.0022	0.00100	100	0.1				Avg of Dups	SD
	9	Matrix spike	Sample + spike	1.0028	0.00100	100	0.1					
	10	Sample	Sample3 a	1.0004	0.00100	100	0.1					
	11	Sample	Sample3 b	1.0029	0.00100	100	0.1				Avg of Dups	SD
	12	Matrix spike	Sample + spike	0.9972	0.00100	101	0.1		n/a	n/a		
	13	Reagent blank	unprocessed sample	n/a	n/a	100	0.1		n/a	n/a		

$$\% IVBA = \frac{(\text{Concentration in IVBA extract mg/L})\ (0.1\ L)}{(\text{Concentration in solid mg/kg})\ (\text{weight of sample kg})} \bullet 100$$

APPENDIX B

Provisional Reference Values for Arsenic IVBA of NIST 2710A Standard Reference Material

Consensus values for In Vitro Bioaccessibility (IVBA) of arsenic in soil reference materials (RM) are needed to support the Standard Operating Procedures (SOP) for determination of arsenic IVBA in soil. EPA is currently conducting multi-laboratory evaluations of arsenic IVBA for NIST 2710A and USGS Flat Creek RMs and has conducted similar evaluations of lead IVBA for these RMs. Until the arsenic IVBA evaluations are completed, EPA recommends using the provisional reference values for NIST 2710A in Table B-1. Although the provisional reference values are based on data from only two laboratories, the estimated prediction interval (±20%) is in the range observed for lead IVBA reference values (Table B-2). The data on which the arsenic IVBA reference values are based are provided in Tables B-3 (summary) and B-4 (individual replicates).

Table B-1. Recommended Provisional Reference Value for Arsenic IVBA% of NIST 2710A

Laboratory	Reference Material	Laboratory Analysis	Total Soil Arsenic Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
All Labs[a]	NIST2710A	Arsenic IVBA	NIST Certificate[b]	%	131	32.9	41.0	49.1	±19.8

[a]Data provided by Karen Bradham (EPA PRD NERL) and John Drexler (University of Colorado).
[b]NIST certificate median soil arsenic concentration: 1400 mg/kg.

Table B-2. Reference Values for Lead IVBA% of Standard Reference Materials

Laboratory	Reference Material	Laboratory Analysis	Total Soil Lead Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
QATS Round Robin	NIST2710A	Lead IVBA	NIST Certificate	%	35	60.7	67.5	74.2	±10
QATS Round Robin	NIST2711A	Lead IVBA	NIST Certificate	%	35	75.2	85.7	96.2	±12.3
QATS Round Robin	Flat Creek	Lead IVBA	EPA 3051A	%	30, 35[a]	56.0	71.0	86.0	±21.1

[a]Based on n=35 estimates of total Pb (mg/kg) and 30 estimates of IVBA Pb (mg/kg).

Table B-1. Recommended Provisional Reference Value for Arsenic IVBA% of NIST 2710A

Laboratory	Reference Material	Laboratory Analysis	Total Soil Arsenic Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
All Labs[a]	NIST2710A	Arsenic IVBA	NIST Certificate[b]	%	131	32.9	41.0	49.1	±19.8

[a]Data provided by Karen Bradham (EPA PRD NERL) and John Drexler (University of Colorado).
[b]NIST certificate median soil arsenic concentration: 1400 mg/kg.

Table B-2. Reference Values for Lead IVBA% of Standard Reference Materials

Laboratory	Reference Material	Laboratory Analysis	Total Soil Lead Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
QATS Round Robin	NIST2710A	Lead IVBA	NIST Certificate	%	35	60.7	67.5	74.2	±10
QATS Round Robin	NIST2711A	Lead IVBA	NIST Certificate	%	35	75.2	85.7	96.2	±12.3
QATS Round Robin	Flat Creek	Lead IVBA	EPA 3051A	%	30, 35[a]	56.0	71.0	86.0	±21.1

[a]Based on n=35 estimates of total Pb (mg/kg) and 30 estimates of IVBA Pb (mg/kg).

Table B-3. Values for Arsenic IVBA% of NIST 2710A Based Data from Individual Laboratories and Combined Data

Laboratory[a]	Reference Material	Laboratory Analysis	Total Soil Arsenic Method	Units	Number of Replicates	Lower 99% Prediction Limit	Mean	Upper 99% Prediction Limit	PI as Percent of Mean
EPA NERL	NIST2710A	Arsenic IVBA	NIST Certificate	%	117	33.1	40.8	48.4	±18.8
U Colorado	NIST2710A	Arsenic IVBA	NIST Certificate	%	14	30.7	43.0	55.2	±28.5
All Labs	NIST2710A	Arsenic IVBA	NIST Certificate	%	131	32.9	41.0	49.1	±19.8

[a]Data provided by Karen Bradham (EPA PRD NERL) and John Drexler (University of Colorado).

Table B-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
1	EPA NERL	1.00	5.59	1400	39.9
2	EPA NERL	1.00	5.56	1400	39.6
3	EPA NERL	1.00	5.33	1400	38.0
4	EPA NERL	1.00	5.14	1400	36.7
5	EPA NERL	1.00	6.40	1400	45.6
6	EPA NERL	1.00	6.40	1400	45.6
7	EPA NERL	1.00	5.98	1400	42.7
8	EPA NERL	1.00	6.15	1400	43.9
9	EPA NERL	1.00	5.46	1400	38.9
10	EPA NERL	1.00	5.82	1400	41.4
11	EPA NERL	1.00	6.39	1400	45.5
12	EPA NERL	1.00	5.25	1400	37.5
13	EPA NERL	1.00	5.26	1400	37.6
14	EPA NERL	1.00	5.19	1400	37.1
15	EPA NERL	1.00	5.54	1400	39.5
16	EPA NERL	1.00	5.43	1400	38.8
17	EPA NERL	1.00	5.52	1400	39.3
18	EPA NERL	1.00	5.20	1400	37.0
19	EPA NERL	1.00	5.08	1400	36.3
20	EPA NERL	1.00	5.19	1400	37.0
21	EPA NERL	1.00	5.24	1400	37.4
22	EPA NERL	1.00	6.01	1400	42.9
23	EPA NERL	1.00	5.57	1400	39.7
24	EPA NERL	1.00	5.58	1400	39.6
25	EPA NERL	1.00	5.66	1400	40.4
26	EPA NERL	1.00	5.25	1400	37.4
27	EPA NERL	1.00	5.25	1400	37.5
28	EPA NERL	1.00	5.51	1400	39.4
29	EPA NERL	1.00	4.89	1400	35.0
30	EPA NERL	1.00	5.61	1400	40.0
31	EPA NERL	1.00	5.36	1400	38.2
32	EPA NERL	1.01	5.94	1400	42.1
33	EPA NERL	1.00	5.86	1400	41.8
34	EPA NERL	1.00	5.84	1400	41.6
35	EPA NERL	1.00	4.83	1400	34.4
36	EPA NERL	1.00	5.12	1400	36.5

Table B-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
1	EPA NERL	1.00	5.59	1400	39.9
2	EPA NERL	1.00	5.56	1400	39.6
3	EPA NERL	1.00	5.33	1400	38.0
4	EPA NERL	1.00	5.14	1400	36.7
5	EPA NERL	1.00	6.40	1400	45.6
6	EPA NERL	1.00	6.40	1400	45.6
7	EPA NERL	1.00	5.98	1400	42.7
8	EPA NERL	1.00	6.15	1400	43.9
9	EPA NERL	1.00	5.46	1400	38.9
10	EPA NERL	1.00	5.82	1400	41.4
11	EPA NERL	1.00	6.39	1400	45.5
12	EPA NERL	1.00	5.25	1400	37.5
13	EPA NERL	1.00	5.26	1400	37.6
14	EPA NERL	1.00	5.19	1400	37.1
15	EPA NERL	1.00	5.54	1400	39.5
16	EPA NERL	1.00	5.43	1400	38.8
17	EPA NERL	1.00	5.52	1400	39.3
18	EPA NERL	1.00	5.20	1400	37.0
19	EPA NERL	1.00	5.08	1400	36.3
20	EPA NERL	1.00	5.19	1400	37.0
21	EPA NERL	1.00	5.24	1400	37.4
22	EPA NERL	1.00	6.01	1400	42.9
23	EPA NERL	1.00	5.57	1400	39.7
24	EPA NERL	1.00	5.58	1400	39.6
25	EPA NERL	1.00	5.66	1400	40.4
26	EPA NERL	1.00	5.25	1400	37.4
27	EPA NERL	1.00	5.25	1400	37.5
28	EPA NERL	1.00	5.51	1400	39.4
29	EPA NERL	1.00	4.89	1400	35.0
30	EPA NERL	1.00	5.61	1400	40.0
31	EPA NERL	1.00	5.36	1400	38.2
32	EPA NERL	1.01	5.94	1400	42.1
33	EPA NERL	1.00	5.86	1400	41.8
34	EPA NERL	1.00	5.84	1400	41.6
35	EPA NERL	1.00	4.83	1400	34.4
36	EPA NERL	1.00	5.12	1400	36.5

Table B-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
37	EPA NERL	1.00	5.29	1400	37.7
38	EPA NERL	1.00	5.88	1400	41.9
39	EPA NERL	1.00	5.69	1400	40.6
40	EPA NERL	1.00	5.88	1400	41.8
41	EPA NERL	1.00	5.70	1400	40.6
42	EPA NERL	1.00	5.44	1400	38.8
43	EPA NERL	1.00	5.35	1400	38.2
44	EPA NERL	1.00	5.38	1400	38.3
45	EPA NERL	1.00	5.37	1400	38.3
46	EPA NERL	1.00	5.42	1400	38.7
47	EPA NERL	1.00	5.30	1400	37.9
48	EPA NERL	1.00	5.10	1400	36.3
49	EPA NERL	1.00	6.00	1400	42.7
50	EPA NERL	1.00	5.21	1400	37.1
51	EPA NERL	1.00	5.19	1400	37.0
52	EPA NERL	1.00	6.29	1400	44.8
53	EPA NERL	1.00	5.92	1400	42.1
54	EPA NERL	1.00	5.64	1400	40.1
55	EPA NERL	1.00	5.60	1400	39.9
56	EPA NERL	1.00	5.73	1400	40.8
57	EPA NERL	1.00	5.90	1400	42.0
58	EPA NERL	1.00	5.59	1400	39.9
59	EPA NERL	1.00	5.55	1400	39.5
60	EPA NERL	1.00	5.73	1400	40.7
61	EPA NERL	1.00	5.95	1400	42.4
62	EPA NERL	1.00	5.83	1400	41.6
63	EPA NERL	1.00	5.63	1400	40.2
64	EPA NERL	1.00	5.64	1400	40.2
65	EPA NERL	1.00	6.18	1400	44.1
66	EPA NERL	1.00	5.70	1400	40.6
67	EPA NERL	1.00	5.39	1400	38.3
68	EPA NERL	1.00	5.85	1400	41.6
69	EPA NERL	1.00	6.14	1400	43.7
70	EPA NERL	1.00	6.05	1400	43.1
71	EPA NERL	1.00	6.53	1400	46.6
72	EPA NERL	1.00	6.13	1400	43.7

Table B-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
73	EPA NERL	1.00	6.35	1400	45.3
74	EPA NERL	1.00	6.21	1400	44.2
75	EPA NERL	1.00	5.24	1400	37.3
76	EPA NERL	1.00	5.60	1400	40.0
77	EPA NERL	1.00	6.05	1400	43.1
78	EPA NERL	1.00	5.99	1400	42.6
79	EPA NERL	1.00	5.45	1400	38.9
80	EPA NERL	1.00	5.73	1400	40.8
81	EPA NERL	1.00	5.79	1400	41.2
82	EPA NERL	1.00	5.55	1400	39.5
83	EPA NERL	1.01	6.09	1400	43.1
84	EPA NERL	1.00	5.68	1400	40.4
85	EPA NERL	1.00	5.28	1400	37.6
86	EPA NERL	1.00	5.26	1400	37.5
87	EPA NERL	1.00	5.50	1400	39.2
88	EPA NERL	1.01	5.67	1400	40.2
89	EPA NERL	1.00	5.36	1400	38.2
90	EPA NERL	1.01	5.70	1400	40.5
91	EPA NERL	1.00	5.68	1400	40.4
92	EPA NERL	1.01	5.48	1400	38.8
93	EPA NERL	1.01	5.35	1400	37.9
94	EPA NERL	1.00	5.62	1400	40.0
95	EPA NERL	1.00	5.63	1400	40.1
96	EPA NERL	1.01	5.94	1400	42.0
97	EPA NERL	1.00	6.57	1400	46.9
98	EPA NERL	1.00	5.77	1400	41.2
99	EPA NERL	1.00	6.14	1400	43.8
100	EPA NERL	1.00	6.50	1400	46.5
101	EPA NERL	1.01	6.36	1400	44.9
102	EPA NERL	1.01	6.14	1400	43.5
103	EPA NERL	1.01	6.62	1400	46.7
104	EPA NERL	1.01	6.21	1400	44.0
105	EPA NERL	1.01	6.70	1400	47.5
106	EPA NERL	1.00	6.45	1400	46.1
107	EPA NERL	1.00	5.73	1400	40.8
108	EPA NERL	1.01	5.87	1400	41.7

Table B-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
73	EPA NERL	1.00	6.35	1400	45.3
74	EPA NERL	1.00	6.21	1400	44.2
75	EPA NERL	1.00	5.24	1400	37.3
76	EPA NERL	1.00	5.60	1400	40.0
77	EPA NERL	1.00	6.05	1400	43.1
78	EPA NERL	1.00	5.99	1400	42.6
79	EPA NERL	1.00	5.45	1400	38.9
80	EPA NERL	1.00	5.73	1400	40.8
81	EPA NERL	1.00	5.79	1400	41.2
82	EPA NERL	1.00	5.55	1400	39.5
83	EPA NERL	1.01	6.09	1400	43.1
84	EPA NERL	1.00	5.68	1400	40.4
85	EPA NERL	1.00	5.28	1400	37.6
86	EPA NERL	1.00	5.26	1400	37.5
87	EPA NERL	1.00	5.50	1400	39.2
88	EPA NERL	1.01	5.67	1400	40.2
89	EPA NERL	1.00	5.36	1400	38.2
90	EPA NERL	1.01	5.70	1400	40.5
91	EPA NERL	1.00	5.68	1400	40.4
92	EPA NERL	1.01	5.48	1400	38.8
93	EPA NERL	1.01	5.35	1400	37.9
94	EPA NERL	1.00	5.62	1400	40.0
95	EPA NERL	1.00	5.63	1400	40.1
96	EPA NERL	1.01	5.94	1400	42.0
97	EPA NERL	1.00	6.57	1400	46.9
98	EPA NERL	1.00	5.77	1400	41.2
99	EPA NERL	1.00	6.14	1400	43.8
100	EPA NERL	1.00	6.50	1400	46.5
101	EPA NERL	1.01	6.36	1400	44.9
102	EPA NERL	1.01	6.14	1400	43.5
103	EPA NERL	1.01	6.62	1400	46.7
104	EPA NERL	1.01	6.21	1400	44.0
105	EPA NERL	1.01	6.70	1400	47.5
106	EPA NERL	1.00	6.45	1400	46.1
107	EPA NERL	1.00	5.73	1400	40.8
108	EPA NERL	1.01	5.87	1400	41.7

Table B-4. NIST 2710A Arsenic IVBA Replicate Data Used in Calculation of Provisional Reference Values

Replicate	Laboratory[a]	Soil Mass (g)	Extracted As (mg/L)	Total Soil As[b] (mg/kg)	As IVBA (%)
109	EPA NERL	1.01	5.98	1400	42.5
110	EPA NERL	1.00	6.04	1400	43.0
111	EPA NERL	1.00	5.42	1400	38.6
112	EPA NERL	1.00	5.49	1400	39.1
113	EPA NERL	1.01	6.15	1400	43.6
114	EPA NERL	1.01	6.63	1400	46.9
115	EPA NERL	1.01	5.93	1400	42.0
116	EPA NERL	1.01	6.14	1400	43.5
117	EPA NERL	1.00	6.44	1400	45.9
118	U. Colorado	1.00	5.10	1400	36.3
119	U. Colorado	1.02	5.22	1400	36.7
120	U. Colorado	1.01	5.69	1400	40.3
121	U. Colorado	1.01	6.55	1400	46.5
122	U. Colorado	1.00	6.69	1400	47.7
123	U. Colorado	1.00	6.34	1400	45.1
124	U. Colorado	1.00	6.75	1400	48.2
125	U. Colorado	1.00	6.45	1400	46.1
126	U. Colorado	1.00	6.34	1400	45.2
127	U. Colorado	1.01	6.46	1400	45.8
128	U. Colorado	1.02	5.79	1400	40.4
129	U. Colorado	1.01	5.69	1400	40.3
130	U. Colorado	1.00	5.68	1400	40.4
131	U. Colorado	1.01	6.02	1400	42.4

[a]Data provided by Karen Bradham *(EPA ORD NERL) and John Drexler, University of Colorado.
[b]NIST certificate median soil arsenic concentration.

Attachment C
Risk Reduction Standard Calculations

Type 4 Risk Reduction Standards (RRSs) pose no significant risk on the basis of SITE-SPECIFIC EXPOSURE ASSUMPTIONS for NON-RESIDENTIAL PROPERTIES.

RAGS Equation 6
Non-Residential Soil - Carcinogenic Effects

$$C_{car}\ \text{mg/kg} = \frac{TR \times BW \times AT_{car} \times 365\ \text{days/year}}{EF \times ED \times [(SFo \times 10^{-6}\ \text{kg/mg} \times IR_{soil}) + (SF_i \times IR_{air} \times \{1/VF + 1/PEF\})]}$$

Parameter	Definition (units)	Default Value	Source
C_{car} =	Concentration in soil (mg/kg)	Calculated	Not Applicable
$TR_{A/B}$	IRIS Carcinogen Class A/B target excess lifetime cancer risk	1.E-05	HSRA Rules
TR_C	IRIS Carcinogen Class C target excess lifetime cancer risk	1.E-04	HSRA Rules
SF_i	Inhalation cancer slope factor $(\text{mg/kg-dy})^{-1}$ $= IUR \times 1{,}000 \times BW\ 70\ \text{kg} / IR_{air}\ 20\ \text{m}^3/\text{dy}$	Chemical-Specific	Not Applicable
IUR	Inhalation unit risk $(\text{ug/m}^3)^{-1}$	Chemical-Specific	Not Applicable
SF_o	Oral cancer slope factor $(\text{mg/kg-dy})^{-1}$	Chemical-Specific	Not Applicable
AT_{car}	Averaging Time (yr)	70	HSRA Rules

RAGS Equation 7
Non-Residential Soil - Noncarcinogenic Effects

$$C_{noncar}\ \text{mg/kg} = \frac{THI \times BW \times AT_{noncar} \times 365\ \text{days/year}}{ED \times EF \times [\{(1/RfD_o) \times 10^{-6}\ \text{kg/mg} \times IR_{soil}\} + \{(1/RfD_i) \times IR_{air} \times (1/VF + 1/PEF)\}]}$$

Parameter	Definition (units)	Default Value	Source
C_{noncar} =	Concentration in soil (mg/kg)	Calculated	Not Applicable
THI	Target hazard index (none)	1	HSRA Rules
RfD_o	Oral chronic reference dose (mg/kg-dy)	Chemical-Specific	Not Applicable
RfD_i	Inhalation chronic reference dose (mg/kg-dy) $= RfC_i \times IR_{air}\ 20\ \text{m}^3/\text{dy} / BW\ 70\ \text{kg}$	Chemical-Specific	Not Applicable
RfC_i	Inhalation reference concentration (mg/m^3)	Chemical-Specific	Not Applicable
AT_{noncar}	Averaging time (yr)	25	HSRA Rules

Standard Assumptions			
BW	Body weight (kg)	70	HSRA Rules
EF	Exposure frequency (dy/yr)	250	HSRA Rules
ED	Exposure duration (yr)	25	HSRA Rules
IR_{air}	Inhalation rate (m^3/dy)	20	HSRA Rules
IR_{soil}	Soil ingestion rate (mg/dy)	50	HSRA Rules
PEF	Particulate emission factor (m^3/kg)	4.63E+09	HSRA Rules

Soil-to-Air Volatilization Factor (VF)

$$VF\ (m^3/kg) = \frac{(LS \times V \times DH) \times (\pi \times \alpha \times T)^{1/2}}{A \times 2 \times D_{ei} \times E \times K_{as} \times 10^{-3}\ kg/g}$$

Parameter	Definition (units)	Default Value	Source
LS	Length of side of contaminated area (m)	45	HSRA Rules
V	Wind speed in mixing zone (m/s)	2.25	HSRA Rules
A	Area of contamination (cm^2)	2.03E+07	HSRA Rules
DH	Diffusion height (m)	2	HSRA Rules
α	$(D_{ei} \times E)/[E + (\rho_s \times (1-E)/K_{as})]$ (cm^2/s)	Chemical-specific	HSRA Rules
T	Exposure Interval (s)	7.90E+08	HSRA Rules
ρ_s	Density of soil solids (g/cm^3)	2.65	HSRA Rules
D_{ei}	Effective diffusivity (cm^2/s)	$D_i \times E^{0.33}$	HSRA Rules
D_i	Molecular Diffusivity (cm^2/s)	Chemical-specific	Not Applicable
E	Total soil porosity	0.35	HSRA Rules
K_{as}	Soil-air partition coefficient (g soil/cm^3 air)	$(H/K_d) \times 41$	HSRA Rules
H	Henry's Law Constant (atm-m^3/mole)	Chemical-specific	Not Applicable
K_d	Soil-water partition coefficient (cm^3/g)	$K_{OC} \times OC$	HSRA Rules
K_{OC}	Organic carbon partition coefficient (cm^3/g)	Chemical-specific	Not Applicable
OC	Soil Organic Carbon Content (none)	2.0E-02	HSRA Rules

HSRA Rules: Georgia Hazardous Response Act Rules, 391-3-19, Appendix III, Media Target Concentrations and Standard Exposure Assumptions.

http://rules.sos.state.ga.us/docs/391/3/19/Appendix%20I-IV.pdf

RAGS: Risk Assessment Guidance for Superfund, Volume I - Human Health Evaluation Manual (Part B, Chapter 3, Development of Risk-Based Preliminary Remediation Goals), U.S. Environmental Protection Agency, December 1991.

http://www.epa.gov/oswer/riskassessment/ragsb/pdf/chapt3.pdf

IRIS: U.S. Environmental Protection Agency Integrated Risk Information System
https://cfpub.epa.gov/ncea/iris2/atoz.cfm

Substance	CAS No.	VF m^3/mg	Carcinogenic Effects: Car. Class	TR	Sf_i $(mg/kg\text{-}dy)^{-1}$	IUR $(ug/m^3)^{-1}$	Sf_o $(mg/kg\text{-}dy)^{-1}$	C_{car} mg/kg	Noncarcinogenic Effects: THI	RfD_o mg/kg-dy	RfC_i mg/kg-dy	RfD_i mg/kg-dy	C_{noncar} mg/kg	Type 4 RRS mg/kg
Arsenic, Default	7440382	NA	A	1.E-05	1.51E+01	4.30E-03	1.50E+00	3.82E+01	1	3.00E-04	1.50E-05	4.29E-06	6.13E+02	**3.82E+01**
Arsenic, RBA Adjusted	7440382	NA	A	1.E-05	1.51E+01	4.30E-03	2.03E-01	2.83E+02	1	2.22E-03	1.50E-05	4.29E-06	4.54E+03	**2.83E+02**

Data Input
Database look up values
Spreadsheet calculation

NA - Not Applicable, applies to inhalation for nonvolatile substances and substances not Classified as Class A, B, or C carcinogens.

	Sf_o $(mg/kg\text{-}dy)^{-1}$	RfD_o mg/kg-dy
Arsenic Default	1.50E+00	3.00E-04
Arsenic, RBA Adjusted	2.03E-01	2.22E-03

Sf_o, RBA Adjusted = Sf_o Default x RBA

RfD_o, RBA Adjusted = RfD_o Default / RBA

RBA = 13.5%

CDM Smith

Table C-2: Type 4 Soil RRSs
July 2017

Type 5 Risk Reduction Standard
CONSTRUCTION WORKER EXPOSURE ASSUMPTIONS

RAGS Equation 6
Non-Residential Soil - Carcinogenic Effects

$$C_{car}\ \text{mg/kg} = \frac{TR \times BW \times AT_{car} \times 365\ \text{days/year}}{EF \times ED \times [(SFo \times 10^{-6}\ \text{kg/mg} \times IR_{soil}) + (SF_i \times IR_{air} \times \{1/VF + 1/PEF\})]}$$

Parameter	Definition (units)	Default Value	Source
C_{car} =	Concentration in soil (mg/kg)	Calculated	Not Applicable
$TR_{A/B}$	IRIS Carcinogen Class A/B target excess lifetime cancer risk	1.E-05	HSRA Rules
TR_C	IRIS Carcinogen Class C target excess lifetime cancer risk	1.E-04	HSRA Rules
SF_i	Inhalation cancer slope factor $(\text{mg/kg-dy})^{-1}$ = IUR x 1,000 x BW 70 kg / IR_{air} 20 m^3/dy	Chemical-Specific	Not Applicable
IUR	Inhalation unit risk $(\text{ug/m}^3)^{-1}$	Chemical-Specific	Not Applicable
SF_o	Oral cancer slope factor $(\text{mg/kg-dy})^{-1}$	Chemical-Specific	Not Applicable
AT_{car}	Averaging Time (yr)	70	HSRA Rules

RAGS Equation 7
Non-Residential Soil - Noncarcinogenic Effects

$$C_{noncar}\ \text{mg/kg} = \frac{THI \times BW \times AT_{noncar} \times 365\ \text{days/year}}{ED \times EF \times [\{(1/RfD_o) \times 10^{-6}\ \text{kg/mg} \times IR_{soil}\} + \{(1/RfD_i) \times IR_{air} \times (1/VF + 1/PEF)\}]}$$

Parameter	Definition (units)	Default Value	Source
C_{noncar} =	Concentration in soil (mg/kg)	Calculated	Not Applicable
THI	Target hazard index (none)	1	HSRA Rules
RfD_o	Oral chronic reference dose (mg/kg-dy)	Chemical-Specific	Not Applicable
RfD_i	Inhalation chronic reference dose (mg/kg-dy) = RfC_i x IR_{air} 20 m^3/dy / BW 70 kg	Chemical-Specific	Not Applicable
RfC_i	Inhalation reference concentration (mg/m^3)	Chemical-Specific	Not Applicable
AT_{noncar}	Averaging time (yr)	1	HSRA Rules

Standard Assumptions			
BW	Body weight (kg)	70	HSRA Rules
EF	Exposure frequency (dy/yr)	174	Site-Specific
ED	Exposure duration (yr)	1	Site-Specific
IR_{air}	Inhalation rate (m^3/dy)	20	HSRA Rules
IR_{soil}	Soil ingestion rate (mg/dy)	330	Site-Specific
PEF	Particulate emission factor (m^3/kg)	4.63E+09	HSRA Rules

Soil-to-Air Volatilization Factor (VF)

$$VF\ (m^3/kg) = \frac{(LS \times V \times DH) \times (\pi \times \alpha \times T)^{1/2}}{A \times 2 \times D_{ei} \times E \times K_{as} \times 10^{-3}\ kg/g}$$

Parameter	Definition (units)	Default Value	Source
LS	Length of side of contaminated area (m)	45	HSRA Rules
V	Wind speed in mixing zone (m/s)	2.25	HSRA Rules
A	Area of contamination (cm^2)	2.03E+07	HSRA Rules
DH	Diffusion height (m)	2	HSRA Rules
α	$(D_{ei} \times E)/[E + (\rho_s \times (1-E)/K_{as})]$ (cm^2/s)	Chemical-specific	HSRA Rules
T	Exposure Interval (s)	7.90E+08	HSRA Rules
ρ_s	Density of soil solids (g/cm^3)	2.65	HSRA Rules
D_{ei}	Effective diffusivity (cm^2/s)	$D_i \times E^{0.33}$	HSRA Rules
D_i	Molecular Diffusivity (cm^2/s)	Chemical-specific	Not Applicable
E	Total soil porosity	0.35	HSRA Rules
K_{as}	Soil-air partition coefficient (g soil/cm^3 air)	$(H/K_d) \times 41$	HSRA Rules
H	Henry's Law Constant (atm-m^3/mole)	Chemical-specific	Not Applicable
K_d	Soil-water partition coefficient (cm^3/g)	$K_{OC} \times OC$	HSRA Rules
K_{OC}	Organic carbon partition coefficient (cm^3/g)	Chemical-specific	Not Applicable
OC	Soil Organic Carbon Content (none)	2.0E-02	HSRA Rules

HSRA Rules: Georgia Hazardous Response Act Rules, 391-3-19, Appendix III, Media Target Concentrations and Standard Exposure Assumptions.

http://rules.sos.state.ga.us/docs/391/3/19/Appendix%20I-IV.pdf

RAGS: Risk Assessment Guidance for Superfund, Volume I - Human Health Evaluation Manual (Part B, Chapter 3, Development of Risk-Based Preliminary Remediation Goals), U.S. Environmental Protection Agency, December 1991.

http://www.epa.gov/oswer/riskassessment/ragsb/pdf/chapt3.pdf

IRIS: U.S. Environmental Protection Agency Integrated Risk Information System

https://cfpub.epa.gov/ncea/iris2/atoz.cfm

Substance	CAS No.	VF m^3/mg	Carcinogenic Effects: Car. Class	TR	Sf_i $(mg/kg\text{-}dy)^{-1}$	IUR $(ug/m^3)^{-1}$	Sf_o $(mg/kg\text{-}dy)^{-1}$	C_{car} mg/kg	Noncarcinogenic Effects: THI	RfD_o mg/kg-dy	RfC_i mg/kg-dy	RfD_i mg/kg-dy	C_{noncar} mg/kg	Type 4 RRS mg/kg
Arsenic, Default	7440382	NA	A	1.E-05	1.51E+01	4.30E-03	1.50E+00	2.08E+02	1	3.00E-04	1.50E-05	4.29E-06	1.33E+02	**1.33E+02**
Arsenic, RBA Adjusted	7440382	NA	A	1.E-05	1.51E+01	4.30E-03	2.03E-01	1.54E+03	1	2.22E-03	1.50E-05	4.29E-06	9.89E+02	**9.89E+02**

Data Input
Database look up values
Spreadsheet calculation

NA - Not Applicable, applies to inhalation for nonvolatile substances and substances not Classified as Class A, B, or C carcinogens.

	Sf_o $(mg/kg\text{-}dy)^{-1}$	RfD_o mg/kg-dy
Arsenic Default	1.50E+00	3.00E-04
Arsenic, RBA Adjusted	2.03E-01	2.22E-03

Sf_o, RBA Adjusted = Sf_o Default x RBA

RfD_o, RBA Adjusted = RfD_o Default / RBA

RBA = 13.5%

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Table C-4: Type 5 Soil RRSs
July 2017

Attachment D
Soil Arsenic Data

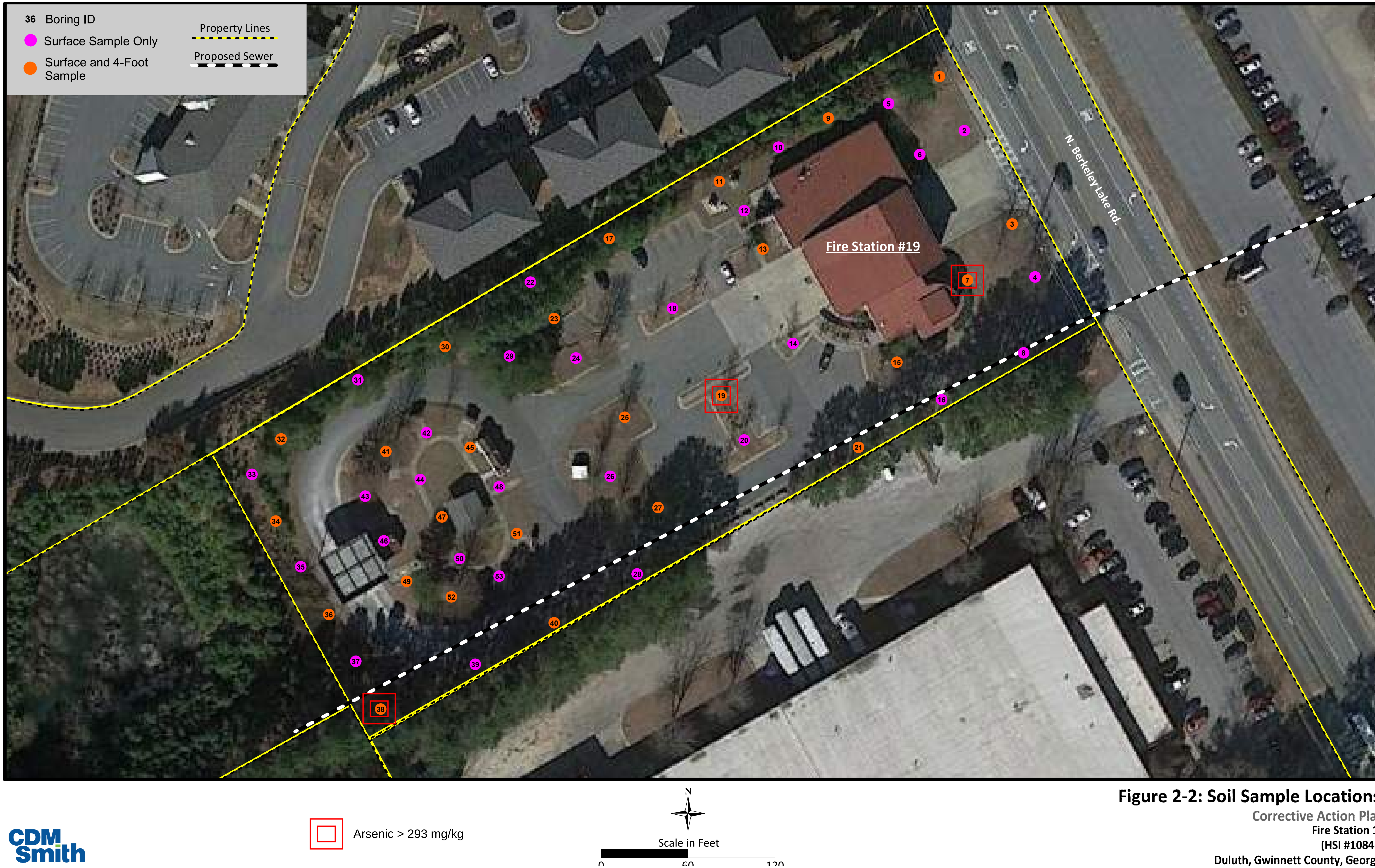

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Figure 2-2: Soil Sample Locations

Corrective Action Plan
Fire Station 19
(HSI #10844)
Duluth, Gwinnett County, Georgia

Table 2-1: Surface and Subsurface Soil Arsenic Results

Corrective Action Plan
Fire Station 19
(HSI #10844)
Duluth, Gwinnett County, Georgia

Sample ID	Sample Depth (ft bgs)	Arsenic Result (mg/kg)	PQL (mg/kg)
EPD Notification Concentration		**41**	
EPD Type 1 RRS		**20**	
SB-1	0.5 - 2	97.2	6.13
	4	73.2	5.46
DUP-7	4	70.4	5.47
SB-2	0.5 - 2	74.8	5.6
SB-3	0.5 - 2	112	5.63
	4	35.2	5.97
SB-4	0.5 - 2	61.9	5.33
SB-5	0.5 - 2	59.9	5.42
SB-6	0.5 - 2	47.1	5.47
SB-7	0.5 - 2	14.5	5.61
	4	491	28
SB-8	0.5 - 2	107	5.29
SB-9	0.5 - 2	129	6.13
	4	105	5.92
SB-10	0.5 - 2	40.4	5.59
SB-11	0.5 - 2	54.2	6.14
	4	32.6	5.81
SB-12	0.5 - 2	57	5.82
SB-13	0.5 - 2	37.4	5.56
	4	274	11.5
SB-14	0.5 - 2	153	5.73
SB-15	0.5 - 2	98.1	5.61
	4	5.55	5.4
SB-16	0.5 - 2	61.5	5.24
SB-17	0.5 - 2	31.2	5.99
	4	17.7	5.72
DUP-6	4	29.1	5.98
SB-18	0.5 - 2	89.7	5.71
SB-19	0.5 - 2	99.3	5.55
	4	52.5	5.48
DUP-5	4	337	10.9
SB-20	0.5 - 2	96.7	5.7
SB-21	0.5 - 2	96.2	5.48
	4	10.3	5.36
SB-22	0.5 - 2	80.3	5.73
SB-23	0.5 - 2	80.7	5.95
	4	32.1	5.4
SB-24	0.5 - 2	83.3	5.55

Table 2-1: Surface and Subsurface Soil Arsenic Results

Corrective Action Plan

Fire Station 19

(HSI #10844)

Duluth, Gwinnett County, Georgia

Sample ID		Sample Depth (ft bgs)	Arsenic Result (mg/kg)	PQL (mg/kg)
EPD Notification Concentration			**41**	
EPD Type 1 RRS			**20**	
SB-25		0.5 - 2	93.6	5.69
		4	85	5.68
	DUP-4	4	107	5.61
SB-26		0.5 - 2	93.6	5.71
SB-27		0.5 - 2	90.4	5.9
		4	31.1	6.11
SB-28		0.5 - 2	31.3	5.54
SB-29		0.5 - 2	71.9	5.56
SB-30		0.5 - 2	90.1	5.54
		4	106	5.8
SB-31		0.5 - 2	20.7	5.45
SB-32		0.5 - 2	78.7	5.38
		4	60.5	5.18
SB-33		0.5 - 2	76.1	5.65
SB-34		0.5 - 2	95.6	5.7
		4	33.4	6.06
SB-35		0.5 - 2	102	6.19
	DUP-1	0.5 - 2	42.5	6.08
SB-36		0.5 - 2	69.9	6.21
		4	103	6.11
SB-37		0.5 - 2	83.6	6.13
SB-38		0.5 - 2	98.3	6.23
		4	371	31.8
SB-39		0.5 - 2	97.7	6.08
SB-40		0.5 - 2	60.4	5.98
		4	54.5	6.32
SB-41		0.5 - 2	76.6	5.62
		4	70.2	5.25
SB-42		0.5 - 2	67.2	5.86
	DUP-2	0.5 - 2	75.9	5.86
SB-43		0.5 - 2	79.9	5.59
SB-44		0.5 - 2	63.1	5.81
SB-45		0.5 - 2	64.6	5.88
		4	45.3	5.67
SB-46		0.5 - 2	71.6	5.89
SB-47		0.5 - 2	64.7	6.21
		4	21.8	5.83
SB-48		0.5 - 2	63	5.72

Table 2-1: Surface and Subsurface Soil Arsenic Results

Corrective Action Plan

Fire Station 19

(HSI #10844)

Duluth, Gwinnett County, Georgia

Sample ID	Sample Depth (ft bgs)	Arsenic Result (mg/kg)	PQL (mg/kg)
EPD Notification Concentration		**41**	
EPD Type 1 RRS		**20**	
SB-49	0.5 - 2	73.4	5.42
	4	77.5	5.48
SB-50	0.5 - 2	76.6	5.88
SB-51	0.5 - 2	60.2	6.05
	4	218	12.6
SB-52	0.5 - 2	68.8	5.64
DUP-3	0.5 - 2	88.1	5.62
	4	138	5.61
SB-53	0.5 - 2	50.9	6.01

Notes:

DUP-X - duplicate sample where X corresponds to duplicate sample ID

ft bgs - feet below ground surface

PQL - Practical Quantitation Limit

All samples collected on July 29, 2014

Sample results are on a dry weight basis

Table 2-2: Soil Arsenic Statistical Summary

Corrective Action Plan

Fire Station 19

(HSI #10844)

0.5 - 2 Foot Depth, FS 19	
Mean	71
Median	75
Standard Deviation	29
Minimum	0.5
Maximum	153
Count	61
95% UCL of the Mean	79

4 Foot Depth, FS 19	
Mean	98
Median	62
Standard Deviation	112
Minimum	5.55
Maximum	491
Count	34
95% UCL of the Mean	138

Combined Depths, FS 19	
Mean	81
Median	72
Standard Deviation	72
Minimum	0.5
Maximum	491
Count	95
95% UCL of the Mean	96

Attachment E
Professional Certification

Professional Certification

I certify under penalty of law that this report and all attachments were prepared by me or under my direct supervision in accordance with the Voluntary Remediation Program Act (O.C.G.A. Section 12-8-101, et seq.). I am a professional engineer / professional geologist who is registered with the Georgia State Board of Registration for Professional Engineers and Land Surveyors / Georgia State Board of Registration for Professional Geologists and I have the necessary experience and am in charge of the investigation and remediation of this release of regulated substances.

Furthermore, to document my direct oversight of the Voluntary Remediation Plan development, implementation of corrective action, and long term monitoring, I have attached a monthly summary of hours invoiced and description of services provided by me to the Voluntary Remediation Program participant since the previous submittal to the Georgia Environmental Protection Division.

The information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

J. Tom Duffey, GA P.G. 000899
Vice President
CDM Smith

July 7, 2017
Date

JAMES T. DUFFEY
No. 000899
REGISTERED PROFESSIONAL GEOLOGIST

Summary of Oversight Provided by Georgia Licensed Engineers and Geologists

Engineer / Geologist	License Type and No.	Week Ending Date	Number of Hours	Description of Hours
Tom Duffey	Geologist PG000899	5/27/17	3	Senior hydrogeologist and Project Manager. Assessment of approved EPA method and derivation of revised RRSs.
		6/3/17	4.5	
		6/30/17	5	
John Reichling	Engineer PE017367	6/30/17	1	CDM Smith Officer in Charge and person overall responsible for project execution and quality.