United States

Environmental

Protection Agency

Guidance for Sample Collection for *In Vitro* Bioaccessibility Assay for Arsenic and Lead in Soil and Applications of Relative Bioavailability Data in Human Health Risk Assessment

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ACRONYMS AND ABBREVIATIONS

ABA	Absolute bioavailability
AF_{S+D}	Parameter representing the absorption fraction for soil and dust in the ALM
AFP_{soil}	Parameter representing the absorption fraction percent for soil lead in the IEUBK model
AFP_{water}	Parameter representing the absorption fraction percent of lead in drinking water in the IEUBK model
AL	Action level
ALM	Adult Lead Methodology
APHIS	Animal and Plant Health Inspection Service
ASTM	American Society for Testing and Materials
BAC	Bioavailability Committee (of the OSRTI TRW)
CERCLA	Comprehensive Emergency Response and Liability Act
CI	Confidence interval
CL	Confidence limit
CV	Coefficient of variation
DI	Daily intake of a soil contaminant
DQO	Data quality objective
EPC	Exposure point concentration
H_0	Null hypothesis
H_1	Alternative hypothesis
HDPE	High density polyethylene
HHRA	Human health risk assessment
IC	Incremental composite
ICS	Incremental composite sampling
IEUBK model	Integrated Exposure Uptake Biokinetic Model for Lead in Children
INAA	Instrumental neutron activation analysis
ISM	Incremental sampling method
ITRC	Interstate Technology and Regulatory Council
IVBA	*In vitro* bioaccessibility
LCL95	95% lower confidence limit on the mean
M_{SD}	fraction of door dust contributed by soil
OSHA	Occupational Safety and Health Administration
OSRTI	Office of Superfund Remediation and Technology Innovation
PCT95	95^{th} percentile
QAPP	Quality Assurance Project Plan
RBA	Relative bioavailability
RfD	Reference Dose
SD	Standard deviation
SE	Standard error
SOP	Standard operating procedure
TRW	Technical Review Workgroup
TWA	Time-weighted average
95UCL	95% upper confidence limit
U.S. EPA	United States Environmental Protection Agency
USDA	U.S. Department of Agriculture
XRF	X-ray fluorescence

EXECUTIVE SUMMARY

The U.S. Environmental Protection Agency (U.S. EPA) *Guidance for Sample Collection for In Vitro Bioaccessibility Assay for Lead (Pb) in Soil* (U.S. EPA, 2015) focused on sample collection for assessment of *in vitro* bioaccessibility (IVBA) and relative bioavailability (RBA) of lead. This 2020 guidance supplements U.S. EPA (2015) to include both arsenic and lead, and to address in greater detail the evaluation and analysis of IVBA and RBA data, and applications of RBA to human health risk assessment (HHRA). The purpose of the guidance is to provide information that will assist risk assessors and risk managers in collecting and effectively utilizing data on IVBA and RBA of arsenic and lead for use in HHRA. The guidance provides recommendations on the following major topics:

(1) rationale for collecting RBA data to support HHRA;

(2) application of IVBA and RBA data in HHRA;

(3) evaluation and analysis of IVBA and RBA data for use in HHRA;

(4) systematic planning for collection of RBA data; and

(5) collection and processing of soil samples for measurement of arsenic and lead IVBA at sites.

Topics 1 through 4 are addressed sequentially in Sections 3, 4, 5, and 6 of the guidance. Collection and processing of soils are addressed in Sections 7–10. Appendix A describes an approach to estimating minimum sample numbers needed for RBA assessments and provides examples of sample number calculations for various sampling designs, including discrete sampling and incremental sampling methods (ISM). A list of frequently asked questions about bioavailability sampling and assessment is provided in Attachment A. Attachments B–H provide practical examples of applications of RBA data to site-specific HHRAs.

Reference:

U.S. EPA (U.S. Environmental Protection Agency). (2015) Guidance for Sample Collection for *In Vitro* Bioaccessibility Assay for Lead (Pb) in Soil. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response: Washington, DC. OSWER 9200.3-100. March. Available online at: http://semspub.epa.gov/src/document/HQ/100000002.

1.0 PURPOSE AND ORGANIZATION OF THIS GUIDANCE

The purpose of this guidance is to provide information that will assist risk assessors and risk managers in collecting and effectively utilizing data on *in vitro* bioaccessibility (IVBA) and relative bioavailability (RBA) of arsenic and lead for use in human health risk assessment (HHRA). The guidance provides recommendations on the following major topics:

(1) rationale for collecting RBA data to support HHRA;

(2) application of IVBA and RBA data in HHRA;

(3) evaluation and analysis of IVBA and RBA data for use in HHRA;

(4) systematic planning for collection of RBA data; and

(5) collection and processing of samples for measurement of arsenic and lead IVBA at sites.

Topics 1 through 4 are addressed sequentially in Sections 3, 4, 5, and 6 of the guidance. Collection and processing of soils are addressed in Sections 7–10. Appendix A describes an approach to estimating minimum sample numbers needed for RBA assessments and provides examples of sample number calculations for various sampling designs, including discrete sampling and incremental sampling methods (ISM). A list of frequently asked questions about bioavailability sampling and assessment is provided in Attachment A. Attachments B–H provide practical examples of applications of RBA data to site risk assessments. Additional information and assistance with RBA assessments can be found at the U.S. Environmental Protection Agency (U.S. EPA) Technical Review Workgroup (TRW) Bioavailability Committee (BAC) website (https://www.epa.gov/superfund/soil-bioavailability-superfund-sites-technical-assistance) or can be obtained by contacting the BAC through its email or hotline (bahelp@epa.gov; 1-866-282-8622).

2.0 BIOAVAILABILITY TERMINOLOGY USED IN THIS GUIDANCE

Absolute bioavailability (ABA): Fraction of an ingested dose of the contaminant (arsenic or lead) that is absorbed from the gastrointestinal tract and enters the blood and tissues.

Relative bioavailability (RBA): Ratio of the ABA of the contaminant in the medium of interest to that of the same contaminant in the medium used to dose the test organism in the oral toxicity studies.

In vitro bioaccessibility (IVBA): Fraction of total amount of arsenic or lead in a soil sample that is soluble in a gastric-like (i.e., low pH) extraction medium.

RBA-adjusted action level (AL): Soil AL for the contaminant after adjustment relative to the RBA assumed in the AL.

RBA-adjusted concentration: Concentration of contaminant in soil after adjustment for RBA; distinguished from the unadjusted or *total concentration* of the contaminant in soil.

RBA-adjusted daily intake (DI): Estimated DI of the contaminant after adjustment for RBA.

3.0 RATIONALE FOR ASSESSING SOIL ARSENIC OR LEAD RBA

Soil RBA is dependent on physical and chemical properties of the arsenic or lead species, and co-occurring elements at any particular site or location within a given site. Accordingly, site-specific estimates of arsenic or lead RBA in soil from representative exposure areas of the site will increase confidence in estimates of risk related to site-specific exposures (U.S. EPA, 1989, 2007a, 2007b, 2012b, 2017b).

Health risk from ingestion of arsenic-contaminated soils is estimated by comparing the estimated daily soil arsenic ingestion intake to a chronic oral Reference Dose (RfD) or to an intake corresponding to a specific cancer risk defined by a cancer oral slope factor. The toxicity values (slope factors, RfD) were derived from human studies in populations chronically exposed to arsenic in drinking water. However, oral bioavailability of arsenic in soil can be substantially lower than soluble arsenic in drinking water (U.S. EPA, 2012b). RBA assessments provide information needed to adjust risk estimates to account for the differences in bioavailability of arsenic in water and soil. If these adjustments are not made, human health risk from ingestion of arsenic-contaminated soils will be overestimated.

Human health risk for lead in soil, where the probability of exceeding a blood lead concentration is used as a proxy for risk, is estimated by applying either the Integrated Exposure Uptake Biokinetic Model for Lead in Children (IEUBK model) for residential exposure scenarios or the Adult Lead Methodology (ALM) for non-residential exposure scenarios to predict blood lead concentrations (U.S. EPA, 2003c, 2007a). Embedded in these models is a default assumption that the RBA of lead in soil is 0.6 (RBA of lead in soil is 60% of the RBA of lead in drinking water). This assumption results in absorption fraction values of 0.3 for soil (at lead intakes below saturation) in the IEUBK model and 0.12 in the ALM. However, soil lead RBA at specific sites can vary considerably from the default RBA value used in the models. RBA assessments provide information needed to adjust bioavailability parameters in models to account for the site-specific difference between bioavailability of soil lead at the site and the value assumed in the models. If these differences are ignored, human health risk from soil ingestion may be under- or overestimated, depending on the actual RBA at the site.

Estimates of RBA can be used in various ways to adjust human health risk estimates for ingestion of arsenic or lead in soil. This guidance specifically addresses four types of RBA adjustments applied to the following risk estimation or risk-based decision parameters (see Section 7):

- soil and dust lead bioavailability parameters in the IEUBK model and ALM
- exposure point concentrations (EPCs);
- DIs of arsenic; and
- soil action levels (ALs) (or other risk-based levels, such as screening levels).

4.0 LABORATORY METHODS FOR MEASURING RBA

Various animal models (e.g., monkey, mouse, rabbit, rat, swine) have been used to study oral bioavailability of arsenic or lead in soil. Information on appropriate bioassays and pertinent primary literature can be found in U.S. EPA (2019a, 2019b). Bioassays using these models estimate RBA from measurements of tissue levels or urinary levels in relation to the oral dosage of arsenic or lead.

U.S. EPA has validated an IVBA assay for predicting soil arsenic and lead RBA for use in HHRA and recommends using the IVBA assay for characterizing site-specific soil arsenic or lead RBA (U.S. EPA Method 1340; U.S. EPA, 2017b, 2017c). The assay involves a simulated gastric-phase extraction of arsenic or lead from soil in a relatively simple extraction medium. Information on these assays and pertinent primary literature can be found in U.S. EPA (2019a, 2019b). In brief, after drying and sieving, 1 g of soil sample is rotated with 100 mL of buffered extraction fluid at 37 ± 2 °C for 1 hour. The supernatant is separated from the soil sample by filtration and analyzed for arsenic and lead by an appropriate analytical method (e.g., U.S. EPA Method 6010 or 6020). Recommendations for sample collection and preparation are provided in Sections 7 and 8 of this guidance. Information on arsenic or lead concentration is used to select samples for the assay to ensure that concentrations do not exceed method limits (see Section 7.1). Results from the IVBA assay (percent or fraction of arsenic or lead that is bioaccessible) are used to predict RBA from a regression model relating IVBA to RBA (described below).

The IVBA assay is a substantially less expensive alternative to an animal bioassay for assessing RBA. The relatively low cost of the IVBA assay compared to an animal bioassay, availability of standard operating procedures (SOPs), and availability of public and commercial laboratories where it can be performed, allows soil samples to be processed more rapidly for the same cost as a single animal bioassay while reducing animal testing. Using the IVBA assay to evaluate multiple soil samples at a site can provide a more thorough assessment of site RBA. However, it is prudent to conduct confirmatory animal RBA bioassays before using an IVBA assay to assess RBA of novel soil types that were not represented in the data used to validate the IVBA assay. These may include soils with chemical and physical characteristics outside the domain of soils used to develop and validate the IVBA assay. It may also include soils that have received treatments with amending agents that alter mobility or solubility of arsenic or lead. For example, IVBA methods have not been validated for predicting RBA of lead in soils amended with high levels of phosphate to reduce lead bioavailability. Additional information on limitations of the IVBA assays can be found in the technical literature available on the U.S. EPA TRW BAC website or can be obtained by contacting the BAC through its email or hotline (bahelp@epa.gov; 1-866-282-8622).

RBA is predicted from IVBA using a regression model (U.S. EPA, 2017b). The regression model for converting arsenic IVBA to arsenic RBA is as follows:

$$\textit{arsenic RBA percent} = 0.79 \times \textit{IVBA percent} + 3$$

The regression model for converting lead IVBA to lead RBA is as follows:

$$\textit{lead RBA percent} = 0.878 \times \textit{IVBA percent} - 2.8$$

Note that, in both of the above equations, RBA and IVBA and the regression intercept are expressed as percents. If the IVBA data from the laboratory are reported as fractions, rather than as percents, then the corresponding equation for arsenic RBA, expressed as a fraction, is as follows:

$$\textit{arsenic RBA fraction} = 0.79 \times \textit{arsenic IVBA fraction} + 0.03$$

and the corresponding equation for the RBA fraction for lead is as follows:

$$\textit{lead RBA fraction} = 0.878 \times \textit{lead IVBA fraction} - 0.028$$

5.0 APPLICATION OF RBA TO HHRA

5.1 RBA Adjustments of Bioavailability Parameters in Lead Risk Models

The IEUBK model includes a parameter that is used in the calculation of the absorption fraction for soil lead (AFP_{soil}, U.S. EPA, 1994). Users adjust this parameter for RBA when site-specific RBA is to be included in the IEUBK model prediction of the child blood lead distribution. The adjustment is as follows:

$$adjusted\ AFP_{soil} = RBA\ fraction \times 50$$

where RBA is expressed as a fraction, and 50 is the IEUBK model assumption for the absorption fraction percent of lead in drinking water (AFP_{water}). The IEUBK model includes a default value of 0.3 for AFP_{soil}, which is equivalent to a default RBA fraction of 0.6 multiplied by the AFP_{water} (50%). A detailed explanation of how to make an RBA adjustment of the IEUBK model is provided in Attachment B (*Calculation of IEUBK Model and Adult Lead Methodology (ALM) Absorption Fraction Parameters from IVBA Results of EPA Method 1340*). An example of an assessment of RBA for the purpose of adjusting the soil absorption parameter in the IEUBK model is provided in Attachment G (*Relative Bioavailability Adjustment of Absorption Fraction Parameters in the Integrated Exposure Biokinetic Model for Lead in Children and Adult Lead Methodology: Cherokee County Railroad Site Case Study*).

The ALM includes a parameter that represents the absorption fraction of ingested lead in soil and dust lead. Users adjust this parameter for RBA when site-specific RBA is to be included in the ALM prediction of the fetal blood lead distribution. The adjustment is as follows:

$$adjusted\ AF_{S+D} + dust = RBA\ fraction \times 0.2$$

where AF_{S+D} is the ALM parameter for the gastrointestinal absorption fraction of lead in soil and dust, RBA is expressed as a fraction, and 0.2 is the ALM default assumption for the absorption fraction of soluble lead (U.S. EPA, 2003c). A detailed explanation of the adjustment of how to make an RBA adjustment of the ALM is provided in Attachment B (*Calculation of IEUBK Model and Adult Lead Methodology (ALM) Absorption Fraction Parameters from IVBA Results of EPA Method 1340*). An example of an RBA assessment of RBA for the purpose of adjusting the soil absorption parameter in the IEUBK model is provided in Attachment G (*Relative Bioavailability Adjustment of Absorption Fraction Parameters in the Integrated Exposure Biokinetic Model for Lead in Children and Adult Lead Methodology: Cherokee County Railroad Site Case Study*).

5.2 RBA Adjustment of a Soil Exposure Point Concentration (EPC)

The EPC should represent the average exposure experienced by the receptor within the exposure unit or decision unit (U.S. EPA, 2002b). For contaminants other than lead, removal and remedial decisions are often made at sites based, in part, on a calculation of the risk from the EPC using a toxicity value (e.g., oral RfD, oral cancer slope factor), which represents an upper limit of the DI of the contaminant in soil that poses negligible risk. The EPC can be adjusted to account for differences between the bioavailability of the contaminant in soil and the bioavailability assumed in the derivation of the toxicity value or screening level. This adjustment facilitates comparisons of EPCs to screening levels that are based on specific RBA assumptions. In lead risk assessments, RBA-adjusted EPCs can be used in batch file processing of input data for the IEUBK model. The adjustment is as follows:

$$adjusted\ EPC = EPC \times RBA\ fraction$$

where RBA is expressed as a fraction. An example of an assessment of RBA for the purpose of adjusting an EPC for arsenic and lead is provided in Attachment C (*Relative Bioavailability Adjustment of Decision Unit Exposure Point Concentrations for Arsenic and Lead: Upper Columbia River Case Study*).

5.3 Adjustment of a Soil Contaminant Daily Oral Intake

For contaminants other than lead, removal and remedial decisions are made at sites based, in part, on comparison of the oral DI of a contaminant to a toxicity value such as a chronic oral RfD, which represents an upper limit of the contaminant intake soil that poses negligible risk. The DI for arsenic can be adjusted to account for differences between the bioavailability of the contaminant in soil and the bioavailability assumed in the derivation of the RfD. The adjustment is as follows:

$$adjusted\ DI = DI \times RBA\ fraction$$

where RBA is expressed as a fraction. An example of an assessment of RBA for the purpose of adjusting an oral DI for soil arsenic is provided in Attachment D (*Bioavailability Adjustment of Daily Oral Intake of Arsenic in a Baseline Human Health Risk Assessment: A Case Study*). An example of how to adjust a time-weighted soil lead concentration is provided in Attachment H (*Relative Bioavailability Adjustment of Soil Lead Exposure Point Concentrations for a Time-Weighted Exposure to Soil*).

5.4 RBA Adjustment of a Soil Risk-based Screening Level or Action Level (AL)

At sites where removal and remedial decisions are made based, in part, on comparison of the EPC to an AL or risk-based concentration or screening level, the AL can be adjusted to account for differences between the bioavailability of the contaminant in soil and the bioavailability assumed in the derivation of the AL. The adjustment should be made to the AL or to the EPC (see Section 5.3), but not to both. The exact adjustment to be made will depend on what assumptions about RBA are incorporated into the AL. For example, if a soil AL for arsenic has been derived assuming an RBA for arsenic of 1.0, then a site-specific RBA adjustment of the AL must be a value relative to 1. For example:

$$adjusted\ AL = AL \times 1.0/RBA\ fraction$$

where RBA expressed as a fraction. An example of adjustment of a soil AL for arsenic is presented in Attachment E (*Retrospective Relative Bioavailability Assessment in Support of a Removal Decision: A Case Study)*. Lead ALs derived from the IEUBK model that assume that the default model RBA value of 0.6 (absorption fraction for lead in soil = 0.3, absorption fraction for lead in drinking water = 0.5) would be adjusted as follows:

$$adjusted\ AL = AL \times 0.6/RBA\ fraction$$

An example for the adjustment of a risk-based concentration for lead is provided in Attachment F (*Relative Bioavailability Adjustment of a Risk-Based Concentration for Lead: A Case Study – Adjusting RBA in the IEUBK Model and ALM*).

6.0 SYSTEMATIC PLANNING FOR COLLECTION OF RBA DATA

6.1 Data Quality Objectives for RBA Assessment

A Data Quality Objective (DQO) process is used to establish performance or acceptance criteria, which serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support site assessment and remedial decision making. As with planning any environmental sampling,

DQOs should be developed for RBA data collection. See the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (U.S. EPA, 2006) for further discussion. The development of DQOs is a 7-step process:

(1) state the problem;

(2) identify the goal of the study;

(3) identify information inputs;

(4) define the boundaries (in space and time) of the study;

(5) develop the analytical approach;

(6) specify the performance criteria; and

(7) develop a detailed plan for obtaining the data.

The final step of the DQO process is to develop a sampling and analysis plan. This plan should consider potential soil exposure pathways for the site and any existing site data. If existing sampling data are available for a site, the information could assist in understanding the variability of data at the site and in planning a representative sampling design. Samples collected to assess RBA and total metal concentrations should be representative of the bioavailability throughout the area of exposure (i.e., the exposure unit). The *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan* is a useful resource for selecting a design to meet the project DQOs and provide representative data (U.S. EPA, 2002a). An example of application of DQOs to RBA assessment is presented in Attachment C (*Relative Bioavailability Adjustment of Decision Unit Exposure Point Concentrations for Arsenic and Lead: Upper Columbia River Case Study*). Consultation with a qualified statistician who has experience with sampling design is recommended.

6.2 Retrospective RBA Assessments of Archived Soil Samples

Retrospective RBA assessments are sometimes undertaken at sites based on RBA measurements made on archived soils collected for some other purpose (e.g., discovery, preliminary site characterizations, assessments to support removal decisions). In these instances, the original sampling design may not have considered DQOs for characterizing RBA. Therefore, development of a DQO for RBA assessment based on the archived soils is advised so that an appropriate approach to selecting soils for RBA measurement may be developed. For example, if the DQO is to estimate a site-wide RBA value, then consideration should be given to whether or not the archived soils actually provide a representative sample of RBA at the site. If not, sources of sampling bias should be identified and incorporated into the approach to selecting soils for RBA measurements. If these biases cannot be controlled with the method used to select samples, then they should be considered in the interpretation of the results and in any decisions that are made based on the results. In the absence of a DQO and appropriate sampling design, RBA assessments would be based on a "convenience sample" (e.g., random sample of the archive), rather than on a statistical sample of the site. Use of convenience samples to estimate a site-wide or area-wide RBA introduces larger uncertainty into the RBA estimate. For this reason, the selection of the statistic to represent the site or area RBA may need to recognize greater uncertainty in the mean. For example, rather than using a mean or 95% upper confidence limit of the mean (95UCL) of the mean, an upper percentile or maximum might be considered to represent RBA at the site. An example of a retrospective RBA assessment at a site based on measurement of the RBA using archived samples is provided in

Attachment E (*Retrospective Relative Bioavailability Assessment in Support of a Removal Decision: A Case Study*).

6.3 Evaluation of RBA Data Adequacy

Evaluation of adequacy of RBA data begins with a thorough evaluation of the data against the quality control limits for the methods used to collect the data. Quality control criteria of arsenic and lead IVBA assays can be found in the SOPs for the assay (U.S. EPA, 2017b). Quality evaluation of RBA data also includes evaluation of the implementation of sample collection methods to determine whether or not the sample design was followed and, if not, the causes, effects, and implications of deviations from the plan. Provided that quality control requirements for sampling and analysis have been achieved, adequacy of the RBA data should be evaluated against the DQO for RBA at the site. The DQO should specify performance and acceptance criteria of the data. More information on DQOs and performance criteria can be found in the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (U.S. EPA, 2006). For DQOs that test hypotheses such as, "is the EPC greater than an AL," the collected data should result in acceptable false compliance decision error (Type 1) and false exceedance decision error (Type 2) probabilities. A false compliance decision error occurs if it is concluded that the EPC is less than the AL, when it is actually greater than the AL. This outcome is also referred to as a false rejection error (U.S. EPA, 2006). A false compliance decision error could result in underestimating risk at the site and/or not taking an action when action is needed to reduce risk. A false exceedance decision error occurs if it is concluded that the EPC exceeds the AL, when it is actually less than the AL. This outcome is also referred to as a false acceptance error (U.S. EPA, 2006). A false exceedance decision error could result in overestimating risk at the site and/or taking action at the site to reduce risk when no action is needed. An example of how to estimate decision error probabilities that rely on estimates of RBA-adjusted EPCs is provided in Appendix A (*Guidance for Sample Collection for Estimating an RBA-adjusted Exposure Point Concentration for Soil*). The example is presented from the perspective of systematic planning for data collection; however, the data collected can be analyzed using the same methods to evaluate whether data collected were within acceptable limits of decision error.

6.4 Selection of Appropriate Statistic to Represent RBA at the Site

Selection of a statistic to represent the RBA for a decision unit will depend on the DQO established for the decision. If the RBA is to be used to adjust the EPC for the decision unit (i.e., adjusted EPC = EPC × RBA), the statistic selected to represent the RBA should be consistent with the definition of the EPC [see Attachment C (*Relative Bioavailability Adjustment of Decision Unit Exposure Point Concentrations for Arsenic and Lead: Upper Columbia River Case Study*)]. Often, in HHRA, the decision unit represents an exposure unit, within which the receptor has an equal probability of being exposed to soil contaminants anywhere within the decision unit. In this context, the EPC should be the average concentration in the decision unit, estimated as the arithmetic mean or the 95UCL of the mean, from a representative set of soil samples collected from the decision unit (U.S. EPA, 1989, 2002b, 2019c). If the EPC is intended to represent the average exposure concentration at the decision unit, then, consistent with the EPC representing the average exposure, the RBA-adjusted exposure should also represent the average and the statistic to be used to represent the RBA should be the mean or 95UCL of the mean.

The RBA may also be used to adjust the AL applied to evaluating the decisions such as whether or not to remediate at the decision unit [e.g., adjusted AL = AL/RBA; Attachment E (*Retrospective Relative Bioavailability Assessment in Support of a Removal Decision: A Case Study*)]. This adjustment, conceptually, also represents an adjustment of the EPC, in that an upward adjustment of the AL implies that the EPC can be higher without exceeding the AL. Therefore, the adjustment of the AL should also be

consistent with the definition of the EPC. If the EPC is intended to represent the average exposure concentration at the decision unit, then the mean or 95UCL should be selected to represent the RBA.

In some circumstances, it may be prudent to consider statistics other than the mean (or 95UCL) to represent the RBA [see Attachment E (*Retrospective Relative Bioavailability Assessment in Support of a Removal Decision: A Case Study*)]. For example, heterogeneity in RBA within the decision unit, if detected from sampling or inferred from other information about sources of contamination, may prompt consideration of a percentile to represent the RBA. The selection of the percentile will depend on the observed distribution of RBA within the decision unit. The RBA distribution can be estimated from a properly designed discrete sampling plan. In selecting a percentile rather than a mean to represent the RBA, the resulting adjusted EPC or AL will no longer represent the average adjusted exposure. This bias may be warranted on the basis of ensuring that risk is not underestimated at a decision unit in which there is high variability in RBA. Selection of an upper percentile to represent the RBA at the decision unit will decrease false compliance decision error and increase false exceedance decision error [see Appendix A (*Guidance for Sample Collection for Estimating an RBA-adjusted Exposure Point Concentration for Soil*), for further explanation of decision errors].

6.5 Estimation of a Site-wide RBA from RBA Data for Multiple Decision Units

A site-wide RBA may be estimated to simplify risk assessment calculations at sites where RBA is found be (or is assumed to be) homogenous across decision units. The method used to estimate a site-wide RBA will depend on the DQO and the conceptual site model (i.e., how well decision units represent the site), as well as the distribution of observed RBAs in the decision unit.

Use of a site-wide RBA to adjust decision EPCs or decision unit ALs: Often, in HHRA, the decision unit represents an exposure unit, within which the receptor has an equal probability of being exposed to soil contaminants anywhere within the decision unit. In this context, the EPC representing exposure within the decision unit should be the average concentration in the decision unit, estimated as the arithmetic mean or the 95UCL of the mean. The assumption of equal probability of exposure may not apply across decision units. If it did, the entire site could be considered a single decision unit. If exposure cannot be assumed to be random across the site, then use of a site-wide RBA to adjust decision unit EPCs or ALs is not advised, and these adjustments should be made at the decision unit level. If a site-wide RBA is to be used to assess risk at the decision unit level, and exposure is not random across the site, then some form of spatial or activity weighting of the decision units should be considered in the calculation of a site-wide RBA. However, it must be kept in mind that a weighted or unweighted estimate of a site-wide RBA (e.g., weighted mean) may over- or underestimate RBA at any given decision unit and, as a result, there will be lower confidence in the resulting adjusted EPC or adjusted AL for the decision unit if adjusted by a site-wide RBA. For this reason, consideration should be given in decision unit-level assessments for measuring RBA at each decision unit being assessed. If only a subset of decision units is assessed for RBA, then the DQO should address the following: (1) plan for selecting decision units for RBA measurement that ensures that resulting data can be used to predict RBAs at these decision units that are not selected for RBA measurement and (2) statistic to be used to represent the RBA at decision units not selected for measurement of RBA [see Attachment C (*Relative Bioavailability Adjustment of Decision Unit Exposure Point Concentrations for Arsenic and Lead: Upper Columbia River Case Study]*).

Use of a site-wide RBA to characterize RBA variability at the site: Assessment of site-wide variability in RBA can support decisions to assess RBA at the decision unit level. It may also reveal heterogeneity in RBA across the site that may be related to multiple sources of contamination with materials that have different RBA. If the objective is to understand variability in RBA at the site, then decision unit RBAs can be analyzed in a variety of ways, including probability plots and spatial

distribution plots. The outcome of these analyses will determine how the site-wide RBA is to be estimated (e.g., unweighted or spatially weighted statistics).

6.6 Use of the Conceptual Site Model to Inform RBA Sampling

Selection of an appropriate sampling design and sample numbers used to assess RBA at a site will depend, in part, on the RBA variability at the site. Often, in developing sampling design to support a DQO, accurate information of RBA variability may not be available (e.g., if site was not previously sampled) and would have to be assumed. These assumptions can be informed by the conceptual site model, which may identify factors that could contribute variability of RBA across the site. Examples of these factor include:

- Would the source(s) of contamination be expected to result in low or high variability in RBA? For example, multiple sources may release different forms of arsenic or lead, which could have different RBAs, depending on the initial source of contamination, timing of release, and environmental conditions that affect leaching and redistribution of the contamination and mixing with background sources.

- Does the soil or sediment geochemistry vary across the site? For example, local and regional variability in soil characteristics could contribute to RBA variability across the site.

- What are the expected soil concentrations? For example, decisions about contaminant concentrations that are more than 100 times the AL may not be appreciably affected by RBA assessments.

6.7 Use of Soil Concentration Data to Select Samples for RBA Measurement

RBA of soil arsenic and lead can be expected to range from 0 to 100%. Over the RBA range of 1 to 100%, adjustments of the EPC or AL to account for RBA will be less than a factor of 100, and decisions about contaminant concentrations (removal, remediation, control) that are more than 100 times the AL may not be appreciably affected by RBA assessments.

Large variations in concentrations across the site may also be indicative of multiple sources of contamination and, possibly, associated variation in RBA. This information may be useful for developing sampling designs in the DQO process. However, selection of soils for RBA assessment based on contaminant concentrations should be done in a manner that avoids biasing the data. The DQO planning process should be used to ensure that the resulting data can satisfy the DQO. For example, if the DQO is to estimate a site-wide RBA, selection of soils based on concentration may bias the site-wide estimate if some areas are sampled much less densely than others. This consideration is particularly important if the RBA results are to be used to predict RBA based on concentrations at locations where RBA was not measured.

An alternative to selection of soils for RBA assessment based on concentration is to select a random sample of soils and then analyze the data for RBA variance attributable to concentration (e.g., analysis of variance, regression modeling). Often, this approach may be preferable, given the relatively low additional expense of IVBA assays, the importance of understanding variability, and the need for samples to be representative (i.e., in addition to the expense of contaminant concentration measurements).

6.8 Use of Information on Mineralogy and Speciation to Select RBA Samples and Methods

Information on mineralogy and speciation can be useful to explain RBA variability at the site. This information may be useful for developing sampling designs in the DQO process. Speciation of soil metals is a technically complex and is often applied to a small subset of samples for the purpose of explaining observed RBA rather than for predicting RBA in advance of measurements. For example, unusual or unexpected RBA values may be followed up with speciation measurements to better understand why the RBA values were observed or to improve predictions of RBA from IVBA.

7.0 SAMPLE COLLECTION

7.1 Data Collection Requirements

The final step of the DQO process is to develop a sampling and analysis plan. This plan should consider potential soil exposure pathways for the site and any existing site data; for example, if the site is a residential area, then evaluation of exposure pathways in children's play areas, gardens, and the drip lines of homes should be given special attention (U.S. EPA, 2003a). If existing sampling data are available for a site, the information could assist in targeting the sampling locations where there is likely exposure to these contaminated areas. Measurements of RBA and total arsenic or lead concentrations should be representative of the area of exposure (i.e., the exposure unit), as well as the depth of exposure for the receptor. The *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan* is a useful resource for selecting a design to meet the project DQOs and provide representative data (U.S. EPA, 2002a).

Samples for the IVBA assay should have a total lead concentration of less than 50,000 mg/kg and a total arsenic concentration of less than 13,000 mg/kg (U.S. EPA Method 1340). If the IVBA assay needs to be performed on a sample with a lead concentration greater than 50,000 mg/kg or an arsenic concentration greater than 13,000 mg/kg, the laboratory performing the assays should be informed of the sample concentrations so that the amount of soil used in the IVBA assay can be adjusted to be within the appropriate concentration range. Often, soil samples are collected, submitted for metals analysis, and archived while data are collected and reviewed. Based on the analytical results, a subset of the samples is selected for the IVBA assay. This approach allows the site team to target specific areas within a sampling unit that are suspected of having different bioavailability. It also allows the IVBA samples to target specific total lead or arsenic concentrations that are relevant to decision making. For example, the site team may categorize samples into low, medium, and high total lead concentrations and select a representative subsample from each of those categories for IVBA analysis to evaluate if bioavailability is consistent across the concentration range at the site or to target total lead or arsenic concentrations that are particularly relevant to decision making. At other sites, sample locations could be identified in the sampling plan, and samples for measurement of total arsenic or lead concentration would be collected concurrently and analyzed without prior knowledge of concentrations at the site. Regardless of whether the IVBA sample locations are selected *a priori* or after the total metals concentration analysis is complete, incorporating RBA needs into the DQO process prior to sampling and/or IVBA analysis makes the field effort more efficient, simplifies the data analysis, and clarifies how the data will be used.

The use of portable X-ray fluorescence (XRF) is recommended to screen samples in the field because there is significant savings related to time and financial resources by eliminating the collection of samples that do not meet *a priori* criteria for IVBA analysis (such as concentrations that are below the decision range). There are many advantages of field screening for lead and other metals including a reduction of both laboratory and field work. Soils with little to no metals contamination would not be collected for IVBA analysis, shipped, or processed by laboratory staff. Highly variable soil lead concentrations within a site may be identified in real time by portable XRF in the field, allowing for the

immediate collection of additional samples to better characterize the variability or to form composite samples in the laboratory. Field screening with portable XRF therefore reduces the turnaround time required to generate IVBA results, the need for additional field deployments, and waste generation. Field operators of portable XRF instruments should ensure that they are following appropriate protocols to obtain reliable results (SW-846, Method 6200, U.S. EPA, 2007b). The U.S. EPA *Region 4 Superfund X-Ray Fluorescence Field Operations Guide* provides additional information on the use of portable XRF instruments (U.S. EPA, 2017a). It should be noted that the presence of lead can interfere with XRF measurements of arsenic. Method 6200 states "Arsenic concentrations cannot be efficiently calculated for samples with lead-arsenic ratios of 10:1 or more. This high ratio of lead may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for arsenic, regardless of the actual concentration present (U.S. EPA, 2007b)."

When collecting samples for IVBA assay, it is important to note site and soil sample characteristics that may suggest differences in the bioavailability of the arsenic or lead or indicate that interferences might be present. For example, the lead IVBA assay (U.S. EPA Method 1340) may not reliably predict RBA of lead in soils that have been amended with phosphate (Scheckel et al., 2013). If phosphate at a site is of concern, the phosphate concentration should be measured. Generally, this interference occurs at phosphate concentrations typical for treating a soil to bind lead and reduce its bioavailability. Naturally occurring levels of soil phosphate are not expected to interfere with Method 1340, and most fertilizers contain little, if any, phosphate. However, soil samples from a garden generally should not be composited with samples from the surrounding land use areas, because a garden exposure pathway would be expected to differ from exposure to the rest of the property and there is some possibility that a garden may have elevated phosphate levels. Likewise, it may not be advisable to composite soil samples from the drip line of a home with the remainder of the property, as lead within the drip line may be from lead paint and warrant special consideration (e.g., unrelated to the Comprehensive Environmental Response, Compensation and Liability Act; CERCLA).

In addition to the total metals and IVBA analyses, speciation analysis and animal bioavailability studies might also be considered. Speciation analysis is meant to determine the exact chemical/ mineralogical form(s), or species, of lead or arsenic in a sample. While speciation analysis is not necessary, it may be informative in explaining variability in IVBA across the site, identifying sources of contamination of the soil, and assessing the potential mobility of arsenic or lead in the soil (see Section 6.8). The IVBA assay is meant to be a faster and less expensive alternative to *in vivo* animal bioavailability studies. However, there may be cases (such as potential interference from soil amendment applications [e.g., phosphate], untested lead phases, etc.) when an animal study may be necessary. It is important to ensure that sufficient material is collected for each soil sample so that additional analyses could be performed. If additional analyses are determined to be necessary, such as lead speciation analysis or *in vivo* animal bioavailability studies, consultation with the TRW BAC is recommended.

Prior to sampling, a determination must be made as to whether the soil is regulated or quarantined by the U.S. Department of Agriculture (USDA) Animal and Plant Health Inspection Service (APHIS)/ Plant Protection and Quarantine (USDA, 2014). Special care should be taken to segregate regulated or quarantined soil samples from the non-regulated or non-quarantined samples. To determine if the soils collected are regulated or quarantined, contact the State Plant Health Director (https://www.aphis.usda.gov/aphis/ourfocus/planthealth?1dmy&urile=wcm%3apath%3a%2Fap%20his_content_library%2Fsa_our_focus%2Fsa_plant_health%2Fsa_program_overview%2Fct_sphd).

7.2 Number of Samples

The number of samples to collect and analyze for IVBA will depend on the DQO for the study. Factors that should be considered in estimating the number of samples include the following:

- goals of the RBA assessment;
- size and characteristics of the decision units at the site;
- expected variability in RBA within decision units, based on available data or bounding assumptions (U.S. EPA, 2007c); and
- acceptable limits on decision errors (false compliance decision error, false exceedance decision error, and the minimum detectable difference).

Project managers should consult with U.S. EPA *Guidance on Systematic Planning Using the Data Quality Objectives Process* or other appropriate guidance when developing DQOs (U.S. EPA, 2006). In general, sample size estimates for RBA assessments can be based on the same types of power analyses used to evaluate statistical hypotheses in estimating EPCs at decision units (see Appendix A). Chapters 6 and 7 of the DQO Guidance discuss selecting appropriate error rates and the minimum detectable difference, as well as estimating the number of samples needed to achieve those specifications (U.S. EPA, 2006). The anticipated variability of the data determines the number of samples that will be required to achieve the DQOs. Where the data set is expected to be highly variable, more samples will be necessary. Alternatively, the ISM (also referred to as Incremental Composite Sampling, ICS) may be used. This is a type of sample designed to reduce data variability, increase data representativeness, and reduce analytical costs (Brewer et al., 2017; ITRC, 2012). Data generated from ISM tend to be normally distributed, which also simplifies the data analysis. Appendix A discusses sample number calculations for both discrete samples and ISM.

7.3 Sampling Depth

The appropriate sampling depth for a site will depend on the expected exposure pathway for that site. For most scenarios involving exposure to contaminated surface soil, U.S. EPA generally recommends a sampling depth of the top 0–1 inches of soil below organic litter and sod for lead exposure analysis (U.S. EPA, 2020). With this shallow sample depth, obtaining sufficient sample mass for discrete samples may require collecting a larger mass of soil than is typical, especially if the material is particularly coarse. ICS can provide larger masses for shallow samples. If there are other exposure scenarios for a site, other sampling depth intervals that would represent these scenarios should be collected.

7.4 Field Sample Preparation

To ensure that sufficient sample material is available for analysis, the field samplers should consider sieving the material in the field to remove larger debris. Sieve screens No. 4 (4.72 mm) or No. 10 (2.0 mm) would be sufficient for removing larger debris in the field.

7.5 Sample Mass

For metals analysis, SW-846 recommends that a minimum of 200 g of soil be collected and that 2 g of sample be used for the digestions (SW-846, Chapter 3 Inorganic Analytes, Table 3-2, U.S. EPA, 2007a). Method 1340 specifies that 1 g of dried and sieved soil sample be used for IVBA assay of lead for a single replicate (U.S. EPA, 2013a). Additional replicates may be required if the assay does not meet performance specifications for IVBA. The amount of sample required will depend on the particle size distribution of the soil and the moisture content of the soil following coarse sieving in the field. If the

samples will be submitted for animal bioavailability studies or speciation analysis, the laboratories that will be conducting these analyses should be consulted on the amount of sample materials they will require to determine the sample mass needed. For further assistance in determining the sample mass for *in vivo* bioavailability and IVBA assays, please contact the TRW BAC.

8.0 SAMPLING EQUIPMENT AND HANDLING

8.1 Sample Containers

The analytical laboratory/program that will be conducting the metals analysis should be consulted about the appropriate sample container and size required. For the IVBA assay, there are no specific sample container requirements. If no sample container is specified by the metals analysis laboratory, then appropriate containers include glass jars, wide-mouth high density polyethylene (HDPE) jars, plastic zippered bags, or any other container that is clean and free of contaminants can be used. A single one-gallon plastic zippered bag (e.g., plastic freezer bag) should provide sufficient sample material for at least the metals analysis and IVBA assay for most soils. Two-gallon plastic zippered bags may be required for sandy soils and soils with rocks passing through the sieve in the field. If using wide-mouth HDPE jars, a 1000-mL jar should provide sufficient sample, but collect multiple jars per sample if the soil is particularly coarse. There will be considerable cost reduction using a plastic zippered bag compared to a HDPE bottle (both cost of sample containers and shipping).

8.2 Sampling Equipment

For most scenarios involving exposure to contaminated surface soil, U.S. EPA recommends a sampling depth of the top 0–1 inches of soil below organic litter and sod (U.S. EPA, 2020). Collection of surface soil samples may be accomplished with a stainless-steel cylindrical punch, which will capture a constant diameter core for the sampling depth of interest. Sampling using a kick-style cylindrical punch may reduce sample time in the field due to the ease of use. Kick-style punches are not recommended for sandy soils because the soil readily falls out of the probe. Likewise, soils with heavy clay content or rocks are not recommended due to the difficulty in removing clay soils from the equipment and rocky soil will be rejected at the soil surface. For these reasons, using plastic or stainless-steel spades, trowels, or spoons may be preferable, but the sampler should ensure that a sample is collected evenly across the sampling depth. Once the samples are collected, they should be placed in suitable containers for shipment. Any equipment that is not disposable should be thoroughly decontaminated between samples to maintain sample representativeness and prevent cross-contamination, and appropriately stored after sampling. If the exposure pathway being investigated requires deeper sampling depths than 0–1 inches, equipment such as augers, split spoon samplers, and backhoes may be necessary (U.S. EPA, 2000). If sampling at depth, care should be taken during sampling to account for any soil compaction as a result of sampling.

8.3 Field Sieving

Field sieving soils prior to shipment to laboratories decreases the amount of time needed for fine sieving (next section) and reduces the weight of soils and shipping costs through the removal of large soil fractions. Additionally, to help ensure that sufficient sample material is available for analysis, the field samplers should consider sieving the material in the field to remove larger debris (e.g., rocks, grass, sticks). Sieve screens No. 4 (4.76 mm) or No. 10 (2.0 mm) would be sufficient for removing larger debris in the field. The soil that passes through the No. 4 or No. 10 sieve can be collected in new plastic bags (e.g., Ziploc) or if larger amounts of soils are needed, clean plastic buckets. The field-sieved soil must then be sent to the laboratory for fine sieving, drying, and homogenization.

8.4 Fine Sieving

Samples should be fine-sieved to a particle size limit appropriate to the exposure scenario (e.g., <150 µm for dermal contact with surface soil (U.S. EPA, 2016). Personal protection equipment (e.g., face mask, lab coat, gloves) should be worn when fine sieving soils in the laboratory. If **possible**, a dust containment system such as a vent hood should be utilized to reduce exposure when sieving highly contaminated soils.

Once in the laboratory, the soil samples should be homogenized and completely dried in an air-drying oven at <40ºC for up to 5 days or until a constant mass. After drying, any clumps in the sample should be gently broken or declumped using a gloved hand in preparation for passing through a No. 10 (2 mm) standard test sieve. **Samples should NOT be ground by ball mill, mortar and pestle, or any other grinding method that could result in reduction in the particle sizes of the collected soils.**

***For sieving bulk soils (not field sieved)*:** Affix a No. 10 (2 mm) stainless steel test sieve on top of a No. 100 (149 µm) standard test sieve, with a receiver pan at the bottom. For soils or field samples with pebbles or conglomerated soil, a No. 3.5 (5.66 mm) sieve can be placed on top of the No. 10 sieve to separate these materials. In cases where clogging of the No. 100 sieve is suspected or observed, sieves of intermediate size (No. 30 or No. 40) may be placed between the No. 10 and No. 100 sieves as needed. **Note**: Brass sieves or sieves with lead solder should NOT be used as they can contaminate samples with trace amounts of heavy metals.

***For fine sieving field soils that were previously sieved*:** Affix a No. 100 (149 µm) standard test sieve, with a receiver pan at the bottom. Fill the attached topmost No. 100 sieve half full with unsieved soil. Disaggregate any large clumps of soil as needed using a gloved hand. Attach cover over top sieve. Place sealed, stacked sieves on the sieve shaker. Power on sieve shaker and sift to <150 µm until visual inspection of the soil indicates that it has been sufficiently sieved (approximately 5–10 minutes for sandy soils and 20–30 minutes for heavy clay soils).

After passing soil though No. 100 sieve: Transfer sieved soils into clean, pre-weighed individual polyethylene bags, or similar toxic element-free storage containers (wide-mouth HDPE jars, aluminum pan, etc.). Label all storage bags/containers with date, soil ID, soil particle size, and personnel initials plus any other information deemed relevant. Repeat sieving until the remaining soil sample is satisfactorily processed. Weigh (± 0.01 g per container) all sieved soils. Record weights of all sieved and collected soils (± 0.01 g per container) in laboratory notebook or electronic database.

To ensure that composite samples are representative of all of the component locations, the entire field sample should be processed (i.e., dried and fine sieved). Following sieving, each sample should be thoroughly mixed using American Society for Testing and Materials (ASTM) standard D6051-96 (ASTM 2006) or Interstate Technology and Regulatory Council (ITRC) Incremental Sampling Methodology (ITRC 2012), and then transferred to a suitable storage container (U.S. EPA, 2013b).

Extractable metals and metalloids analysis using methods appropriate digestion methods (e.g., U.S. EPA Methods 3051a, 3050) or direct metal concentrations (e.g., instrumental neutron activation analysis [INAA], XRF) and other analyses should be conducted on the same dried, sieved, and homogenized sample material that will also be used for the IVBA assay. To split a sample into equivalent aliquots for the different analyses, the processed soil should be passed through a riffle splitter and the aliquots collected in clean, 250-mL high-density polyethylene bottles (U.S. EPA, 2003b). Samples that have been dried and sieved can be submitted for total metals analysis, metals speciation, IVBA assay, and *in vivo* animal bioavailability studies, but should not be used for analysis of other contaminants of concern.

8.5 Labeling, Shipping and Storage Temperature, and Hold Time

Sample ID numbering, labeling, documentation, and chain of custody should follow the requirements of the analytical laboratory/program that will be conducting the metals analysis. The samples may be shipped at ambient temperature unless otherwise specified by the analytical laboratory/ program.

U.S. EPA recommends a holding time of 6 months for metals samples. The Method 1340 SOP recommends that all samples be archived after metal analysis and retained for further analysis, including *in vivo* bioavailability assay, for 6 months (U.S. EPA, 2012a, 2017b). The samples may be stored at ambient temperature unless specified otherwise by the analytical laboratory/program.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The field samplers should consult with the metals analysis laboratory or the U.S. EPA program to determine in advance the requirements for blanks, duplicates, and matrix spikes for the metals analysis samples. For the IVBA assay, Method 1340 does not require field blanks, field replicates, or matrix spikes to be prepared or collected by field samplers. However, the site team may collect or require these quality assurance samples where appropriate, based on consultation with the analytical laboratory, U.S. EPA program, or a qualified chemist. Material for the matrix spike and replicates for Method 1340 may be taken from the samples at the laboratory's discretion and may not require that samplers collect and designate separate matrix spike and duplicates in the field.

Samplers should take thorough field notes and retain any photographs taken, logbooks, and notes following the sampling event. The field group should make note of any differences in the media between the sample locations and indicate if there are any potential interferences (e.g., phosphate-amended soils) present.

10.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, Occupational Safety and Health Administration (OSHA), and any contractor's corporate health and safety procedures, in addition to the procedures specified in the site-specific Health and Safety Plan.

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APPENDIX A: Guidance for Sample Collection for Estimating an RBA-adjusted Exposure Point Concentration for Soil

The minimum sample number needed to estimate the relative bioavailability (RBA)-adjusted mean soil concentration of a contaminant will depend on the data quality objective (DQO). Data can be collected for the purpose of estimating soil concentrations and/or RBA at a site (estimation study) or for the purpose of supporting decision making (hypothesis testing; U.S. EPA, 2006). An example of an estimation study would be collection of data on soil concentrations or RBA to estimate a mean concentration or RBA and its variance. This might be done to characterize the site or to evaluate heterogeneity in concentration or RBA at the site. An example of hypothesis testing would be collecting data on concentration and RBA, in order to determine whether the mean concentration exceeds an action level (AL). This might be done to decide if an action (e.g., removal, remediation, control) is needed. For estimation studies, sample number predictions are targeted to obtain results that are within acceptance criteria; for example, to achieve a targeted level of confidence in the estimate of the mean or standard deviation (SD). For hypothesis testing, sample number predictions are targeted to obtain acceptable Type 1 (false compliance decision) and Type 2 (false exceedance decision) errors in evaluating validity a null hypothesis (e.g., H_0: concentration exceeds AL) against an alternative hypothesis (H_1: concentration does not exceed AL).

This appendix provides an example of how to estimate sample numbers needed for hypothesis testing; in this case, whether or not the estimated mean adjusted soil concentration for a contaminant exceeds an AL. A similar approach could be used to determine sample numbers needed to support estimation; in this case, the sample numbers would be evaluated in the context of with estimation acceptance criteria, rather than by null and alternative hypotheses related to decision making. The example described in this appendix is for an unspecified contaminant and could be customized for a specific contaminant (e.g., arsenic or lead) by assigning values appropriate for input parameters (e.g., mean and SDs for concentration and RBA, relevant AL). The example evaluates sample number requirements for two types of sampling designs: discrete sampling and incremental composite sampling (ICS). Discrete sampling designs collect individual soil samples and measure concentration in each sample, and RBA in each sample, or in a subset of the samples. Discrete sampling locations are either randomized or gridded, so that the resulting mean (or other statistics) can represent the area being sampled (e.g., decision or exposure unit). ICS designs create multiple composite samples composed of individual randomly located soil samples (referred to as increments). Concentration and RBA are then measured for the composites. Each ICS composite is intended to represent a single estimate of the area mean. Determination of whether discrete or ICS designs are used at a site will depend on the DQO as each offers certain advantages. For example, discrete sampling can provide estimates of variance in the concentration in the area of interest. ICS designs are intended to provide estimates of the area mean and confidence in the mean consistent with estimating an exposure point concentration (EPC) or comparison to a cleanup goal, and do not provide estimates of concentration variance.

Hypothesis to be tested for decision making: We define the null hypothesis (H_0) and alternative hypothesis (H_1) regarding whether the true mean adjusted soil concentration (*adjusted concentration*) is below or above an AL. The AL could be a risk-based concentration or some other soil concentration boundary established for decision making at the site (e.g., removal, remediation, control). The H_0 and H_1 can be defined as follows:

H_0: adjusted mean soil concentration $\geq$ AL

H_1: adjusted mean soil concentration $<$ AL

A Type 1 error occurs if we reject H_0 when it is true; we conclude that the mean adjusted soil concentration is less than the AL, when it is actually greater than or equal to the AL. This is also referred to as a false compliance decision error or false rejection error (U.S. EPA, 2006). A Type 1 error could result in underestimating risk at the site and/or not taking an action when action is needed to reduce risk.

A Type 2 error occurs if we accept H_0 when it is false; we conclude that the mean adjusted soil concentration is above or equal to the AL, when it is actually less than the AL. This is also referred to as a false exceedance decision error or false acceptance error (U.S. EPA, 2006). A Type 2 error could result in overestimating risk at the site and/or taking action at the site when it is not needed to reduce risk.

The objective of a sample number assessment is to identify sample numbers that are expected to satisfy specified requirements for Type 1 and Type 2 error rates. These error rates depend on several factors:

- the difference between the mean adjusted soil concentration and the AL;
- the variability in the soil concentration;
- the mean and variability of the soil RBA; and
- the sampling design used to estimate the mean adjusted soil concentration.

Larger sample numbers will be required to achieve a given error rate when the actual mean adjusted soil concentration is closer to the AL, or when variability (i.e., SD) of the soil concentration or RBA at the site is higher.

Assumptions for calculating sample number: Type 1 and Type 2 error rates were calculated for different numbers of discrete and composite samples having different numbers of contributing increments for RBA and total metals. The calculation method was a Monte Carlo simulation in which concentration and RBA are represented as probability distributions defined by a mean and SD.

A generic example of sample number calculation is presented here. It could be applied to any contaminant, including arsenic or lead, if the appropriate values for the contaminant are used in the calculation. Assumptions in the analysis are as follows:

(1) The underlying distribution of measured concentrations in discrete soil samples at the decision unit is lognormal (the ICS design should collect adequate samples to ensure a normal distribution of the concentrations of multiple composites).

(2) Distribution of measured RBA within a decision unit is normal (e.g., single source of contamination and uniform soil characteristics).

(3) The adjusted soil concentration for the decision unit is:

$$\textit{adjusted soil concentration} = \textit{soil concentration} \times \textit{soil RBA}$$

(4) For evaluating Type 1 error, we assume that the adjusted mean soil concentration at the decision unit exceeds the AL. For evaluating Type 2 error, we assume that the adjusted mean soil concentration at the decision unit is below the AL.

(5) An acceptable Type 1 error rate is 5% (i.e., the probability of concluding that the adjusted mean soil concentration is less than the AL when it is actually equal to or greater than the AL, is equal to or less than 5%).

(6) An acceptable Type 2 error rate is 20% (i.e., the probability of concluding that the adjusted mean soil concentration is equal to or greater than the AL, when it is actually less than the AL, is equal to or less than 20%). We are typically less concerned about a Type 2 error (overestimating risk) than a Type 1 (underestimating risk).

(7) The ICS design consists of n = C composites collected at the decision unit with each composite consisting of n = I increments, of which, n = R composites are randomly selected for RBA analysis (e.g., *in vitro* bioaccessibility [IVBA]). In this example, we have assumed that the RBA of every composite sample or discrete sample was measured; however, the same approach could be used to estimate sample numbers if RBA was measured in a subset of soil samples.

(8) The estimated mean soil concentration for the decision unit is the mean of measured concentrations of n = C composites.

(9) The estimated mean RBA for the decision unit is based on the mean of measured RBA of n = R randomly selected composites.

(10) Values assumed for soil concentration, AL, and RBA for evaluating Type 1 and Type 2 error rates are presented in Table A-1.

Sample size predictions: Type 1 and Type 2 error rates for various sample designs are presented in Tables A-2 to A-10. These tables provide predictions for a range of variability of soil concentration (coefficient of variation [CV] 0.5, 1.0, 3.0) and RBA variability (CV 0.05, 0.10, 0.30). The magnitude of Type 1 and Type 2 errors depends on the AL and the variability in the soil concentration and soil RBA (represented in the CV), as well as on the sample design. An example is illustrated in Figure A-1, which shows the predicted Type 1 error rate (%) for various numbers of discrete or ICS samples. In the case illustrated in Figure A-1, the SD of the soil concentration was assumed to be 3 times the mean and the SD for RBA was assumed to be 0.3 times the mean. These assumptions represent conditions of relatively high variability in the soil concentration and RBA. An acceptable Type 1 error ($\leq$5%) is predicted for ICS sampling in which 5 ICS composites are collected, with each composite consisting of 100 increments. Figure A-2 shows the prediction for the same sampling designs, at a lower variability in soil concentration (SD equals the mean, or CV=1). In this case, an acceptable Type 1 error is predicted for ICS designs that have 3 composites consisting of 30 increments, or for a discrete sampling design consisting of 100 random samples. Thus, how well a given sampling design performs depends, in part, on the variability in RBA and concentration.

In general, a larger sample size is needed to achieve acceptable Type 1 error when there is a smaller difference between the mean soil concentration and the AL. This is because the Type 1 error also depends on the difference between the actual adjusted mean soil concentration and the AL. For any given sampling design, error rates will increase as the actual mean soil concentration decreases and approaches the AL. This is illustrated in Figures A-3 and A-4, which show the probability of rejecting H_0 (H_0 = adjusted soil concentration is at or above the AL) as the ratio of the mean soil concentration to the AL (mean/AL) changes. Probabilities at soil concentrations that exceed the AL (to the right of the vertical line representing the AL) are Type 1 errors (reject H_0 when it is true). Type 1 errors increase as the mean/AL ratio increases. Figure A-3 shows this relationship for three levels of variation in RBA

(CV 0.05, 0.15, 0.3) and Figure A-4 shows the relationship for three levels of variation in the soil concentration (CV 0.5, 1, 3).

The predictions presented in Tables A-2 to A-10 apply to a sampling design that is intended to estimate the mean adjusted soil concentration for use in risk assessment. For most contaminants (other than lead), the EPC is considered to be the 95% upper confidence limit (95UCL) on the mean. Use of the 95UCL for the EPC will increase the Type 2 error for any given sampling design.

Conclusions:

(1) An objective in sample design is to ensure a Type 1 error (false compliance decision) of ≤5% without exceeding a Type 2 error (false exceedance decision) of 20%.

(2) If the variability in the soil concentration and RBA can be estimated, then Type 1 and Type 2 errors can be predicted for alternative sampling designs. This of course means that some data are available for estimating the soil concentration and RBA variability. Ideally, these data would be for the site; however, data from a surrogate site may have to be used if no site data are available. Note that RBA variability is a function of both site conditions and sampling design.

(3) Type 1 and Type 2 errors will depend on the variability in the soil concentration and RBA, sample numbers, sampling design (discrete or ICS), and how close the actual soil concentration mean is to the AL to be evaluated.

(4) Higher variability in soil concentration or RBA will require a larger number of increments per composite or number of discrete samples to achieve an acceptable Type 1 error rate.

(5) A larger number of increments or discrete samples will be needed if the actual mean soil concentration is closer to the AL, and fewer will be needed if the actual mean concentration is further from the AL.

(6) Tables A-2 to A-10 can be used to find an acceptable sampling design to achieve a Type 1 error of ≤5% for a range of expected variabilities in soil concentration and soil RBA.

Table A-1. Parameter Values for Sample Number Calculation		
Parameter	**Type 1 Error Assessment**	**Type 2 Error Assessment**
Ratio: mean adjusted soil concentration/AL[a]	1.25[a]	0.75[a]
Soil concentration CV	0.5. 1.0, 3.0[b]	0.5. 1.0, 3.0[b]
Mean soil RBA	0.60	0.60
Soil RBA CV	0.05, 0.15, 0.30[c]	0.05, 0.15, 0.30[c]

[a]Adjusted soil lead = soil concentration × soil RBA.
[b]Soil lead distribution: lognormal (mean, SD).
[c]RBA distribution normal (mean, SD, minimum, maximum), with minimum = 0, maximum = 1.

AL, action level; CV, coefficient of variation (SD/mean); RBA, relative bioavailability; SD, standard deviation

Table A-2. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 3 and RBA CV is 0.3

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
3	0.3	D	30	42	17
3	0.3	D	50	36	14
3	0.3	D	100	26	9.7
3	0.3	3	30	27	10
3	0.3	3	50	20	7.7
3	0.3	3	100	9.4	3.7
3	0.3	4	30	23	8.4
3	0.3	4	50	15	5.6
3	0.3	4	100	5.9	2.3
3	0.3	5	30	20	7.5
3	0.3	5	50	12	4.5
3	0.3	5	100	3.9	1.7

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above or equal to the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above or equal to the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-3. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 3 and RBA CV is 0.15

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
3	0.15	D	30	40	17
3	0.15	D	50	34	15
3	0.15	D	100	24	10
3	0.15	3	30	26	11
3	0.15	3	50	17	8.5
3	0.15	3	100	7.7	3.9
3	0.15	4	30	21	9.0
3	0.15	4	50	14	6.4
3	0.15	4	100	5.1	2.9
3	0.15	5	30	17	8.0
3	0.15	5	50	11	4.9
3	0.15	5	100	3.1	1.6

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-4. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 3 and RBA CV is 0.05

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
3	0.05	D	30	41	17
3	0.05	D	50	33	15
3	0.05	D	100	23	10
3	0.05	3	30	25	11
3	0.05	3	50	18	7.4
3	0.05	3	100	8.1	3.7
3	0.05	4	30	21	9.4
3	0.05	4	50	13	6.2
3	0.05	4	100	5.3	2.7
3	0.05	5	30	17	8.0
3	0.05	5	50	10	4.8
3	0.05	5	100	3.4	1.9

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-5. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 1 and RBA CV is 0.3

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
1	0.3	D	30	15	4.9
1	0.3	D	50	8.1	2.1
1	0.3	D	100	1.9	0.3
1	0.3	3	30	3.0	0.2
1	0.3	3	50	0.6	0.0
1	0.3	3	100	0.0	0.0
1	0.3	4	30	1.4	0.1
1	0.3	4	50	0.2	0.0
1	0.3	4	100	0.0	0.0
1	0.3	5	30	0.7	0.1
1	0.3	5	50	0.1	0.0
1	0.3	5	100	0.0	0.0

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-6. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 1 and RBA CV is 0.15

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
1	0.15	D	30	12	4.9
1	0.15	D	50	5.8	1.8
1	0.15	D	100	1.4	0.2
1	0.15	3	30	2.3	0.3
1	0.15	3	50	0.4	0.0
1	0.15	3	100	0.0	0.0
1	0.15	4	30	0.9	0.1
1	0.15	4	50	0.1	0.0
1	0.15	4	100	0.0	0.0
1	0.15	5	30	0.4	0.0
1	0.15	5	50	0.0	0.0
1	0.15	5	100	0.0	0.0

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-7. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 1 and RBA CV is 0.05

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
1	0.05	D	30	12	4.4
1	0.05	D	50	6.1	1.6
1	0.05	D	100	1.2	0.2
1	0.05	3	30	1.6	0.4
1	0.05	3	50	0.3	0.0
1	0.05	3	100	0.0	0.0
1	0.05	4	30	0.9	0.1
1	0.05	4	50	0.1	0.0
1	0.05	4	100	0.0	0.0
1	0.05	5	30	0.2	0.0
1	0.05	5	50	0.0	0.0
1	0.05	5	100	0.0	0.0

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-8. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 0.5 and RBA CV is 0.3

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
0.5	0.3	D	30	2.3	0.2
0.5	0.3	D	50	0.5	0.0
0.5	0.3	D	100	0.0	0.0
0.5	0.3	3	30	0.0	0.0
0.5	0.3	3	50	0.0	0.0
0.5	0.3	3	100	0.0	0.0
0.5	0.3	4	30	0.0	0.0
0.5	0.3	4	50	0.0	0.0
0.5	0.3	4	100	0.0	0.0
0.5	0.3	5	30	0.0	0.0
0.5	0.3	5	50	0.0	0.0
0.5	0.3	5	100	0.0	0.0

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-9. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 0.5 and RBA CV is 0.15

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
0.5	0.15	D	30	1.1	0.1
0.5	0.15	D	50	0.2	0.0
0.5	0.15	D	100	0.0	0.0
0.5	0.15	3	30	0.0	0.0
0.5	0.15	3	50	0.0	0.0
0.5	0.15	3	100	0.0	0.0
0.5	0.15	4	30	0.0	0.0
0.5	0.15	4	50	0.0	0.0
0.5	0.15	4	100	0.0	0.0
0.5	0.15	5	30	0.0	0.0
0.5	0.15	5	50	0.0	0.0
0.5	0.15	5	100	0.0	0.0

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

Table A-10. Error Rates for Discrete or Composite Sample Designs if Soil Concentration CV is 0.5 and RBA CV is 0.05

Concentration CV	RBA CV	Number of ICS Composites	Number of Discrete Samples or ICS Increments	Type 1 Error[a] Rate (%)	Type 2 Error[b] Rate (%)
0.5	0.05	D	30	0.9	0.1
0.5	0.05	D	50	0.1	0.0
0.5	0.05	D	100	0.0	0.0
0.5	0.05	3	30	0.0	0.0
0.5	0.05	3	50	0.0	0.0
0.5	0.05	3	100	0.0	0.0
0.5	0.05	4	30	0.0	0.0
0.5	0.05	4	50	0.0	0.0
0.5	0.05	4	100	0.0	0.0
0.5	0.05	5	30	0.0	0.0
0.5	0.05	5	50	0.0	0.0
0.5	0.05	5	100	0.0	0.0

[a]Type 1 error is an observed mean RBA-adjusted soil concentration that is below the risk-based concentration (AL), when the true RBA-adjusted concentration is above the AL.
[b]Type 2 error is an observed mean RBA-adjusted soil concentration that is above the risk-based concentration (AL), when the true RBA-adjusted concentration is below the AL.

AL, action level; CV, coefficient of variation (SD/mean); D, discrete samples; ICS, incremental composite sample; SD, standard deviation

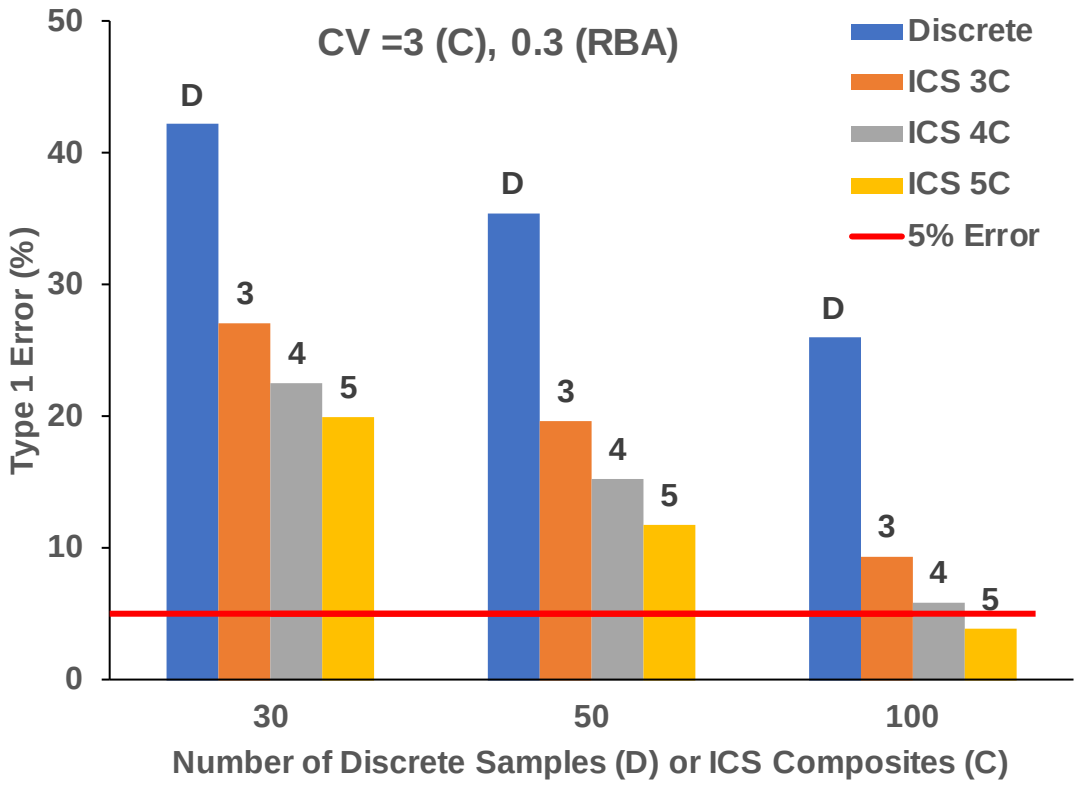

Figure A-1. Effect of Sample Size on False Negative Error Rates for Discrete or ICS Sampling if the CV for the Soil Concentration is 3 and the CV for RBA is 0.3.

Each bar represents the error rate for a combination a specified number of discrete or ICS increments (30, 50, or 100) and ICS composites (3, 4, or 5), and CV (SD/mean) for concentration and RBA. The horizontal line represents the upper end of the target error rate (≤5%). False negative error (Type 1) was estimated for the condition in which the actual mean RBA-adjusted concentration is 25% above the AL.

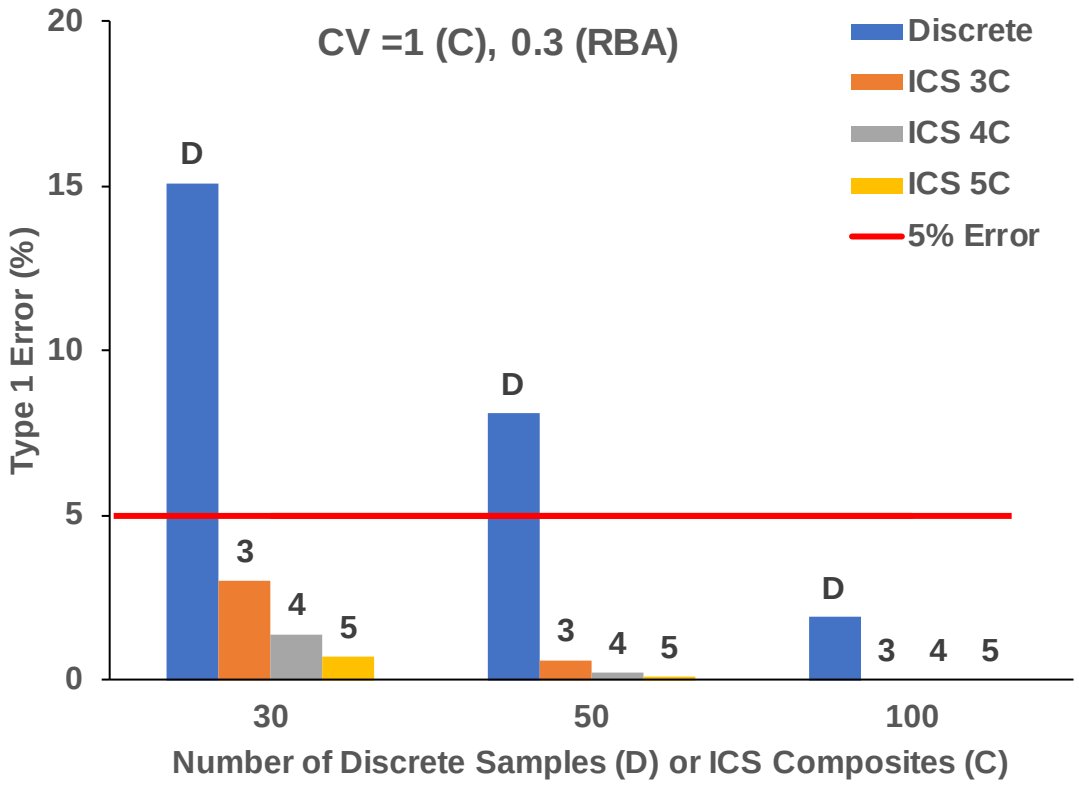

Figure A-2. Effect of Sample Size on False Negative Error Rates for Discrete or ICS Sampling if the CV for the Soil Concentration is 1 and the CV for RBA is 0.3.

Each bar represents the error rate for a combination a specified number of discrete or ICS increments (30, 50, or 100) and ICS composites (3, 4, or 5), and CV (SD/mean) for concentration and RBA. The horizontal line represents the upper end of the target error rate (≤5%). False negative error (Type 1) was estimated for the condition in which the actual mean RBA-adjusted concentration is 25% above the action level (AL). False negative error is the probability that sampling would result in an mean RBA-adjusted concentration that is less than the AL, when it is actually 25% greater than the AL.

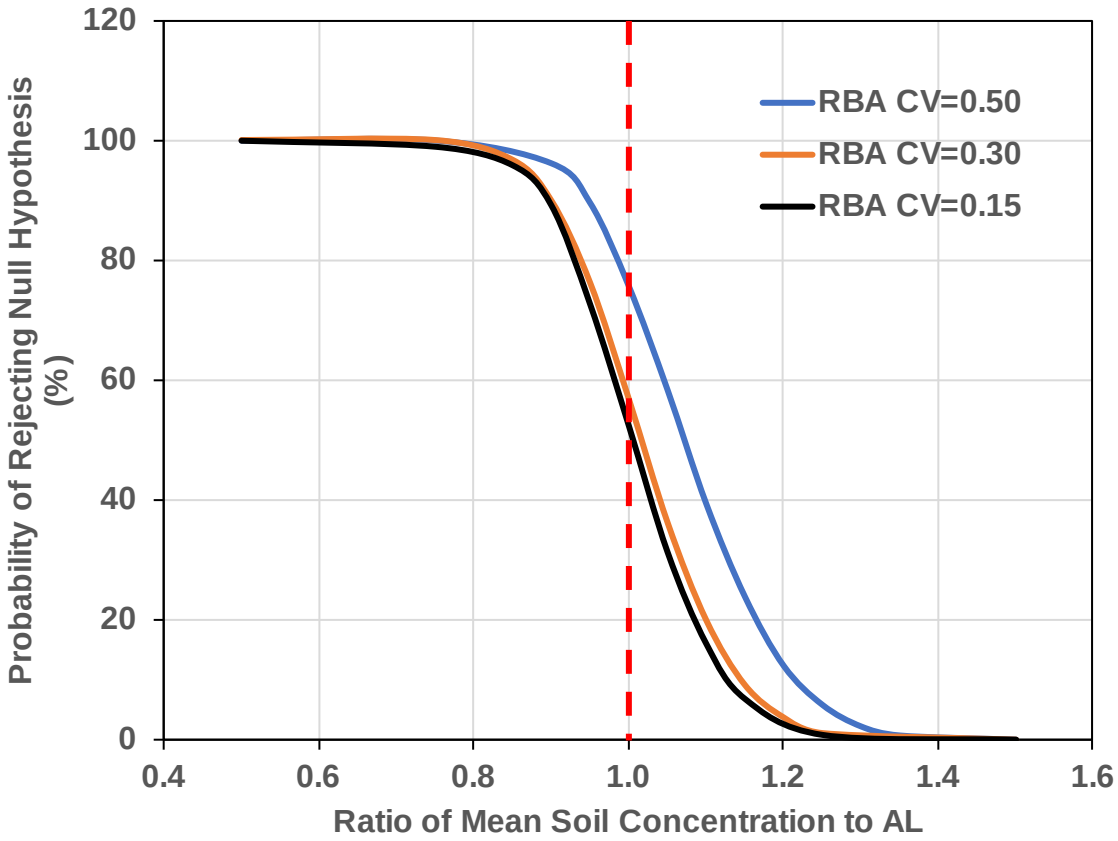

Figure A-3. Probability of Rejecting Null Hypothesis (H_0 = Adjusted Soil Concentration is Above the Action Level) as the Mean Adjusted Soil Concentration Increases when the CV of RBA is 0.15, 0.30, or 0.50.

Soil concentration CV = 1.0. Sample design is ICS 3 composites composed of 30 increments each. The vertical line represents the AL. Probabilities to the right of the vertical line are Type 1 errors (reject H_0 when it is true).

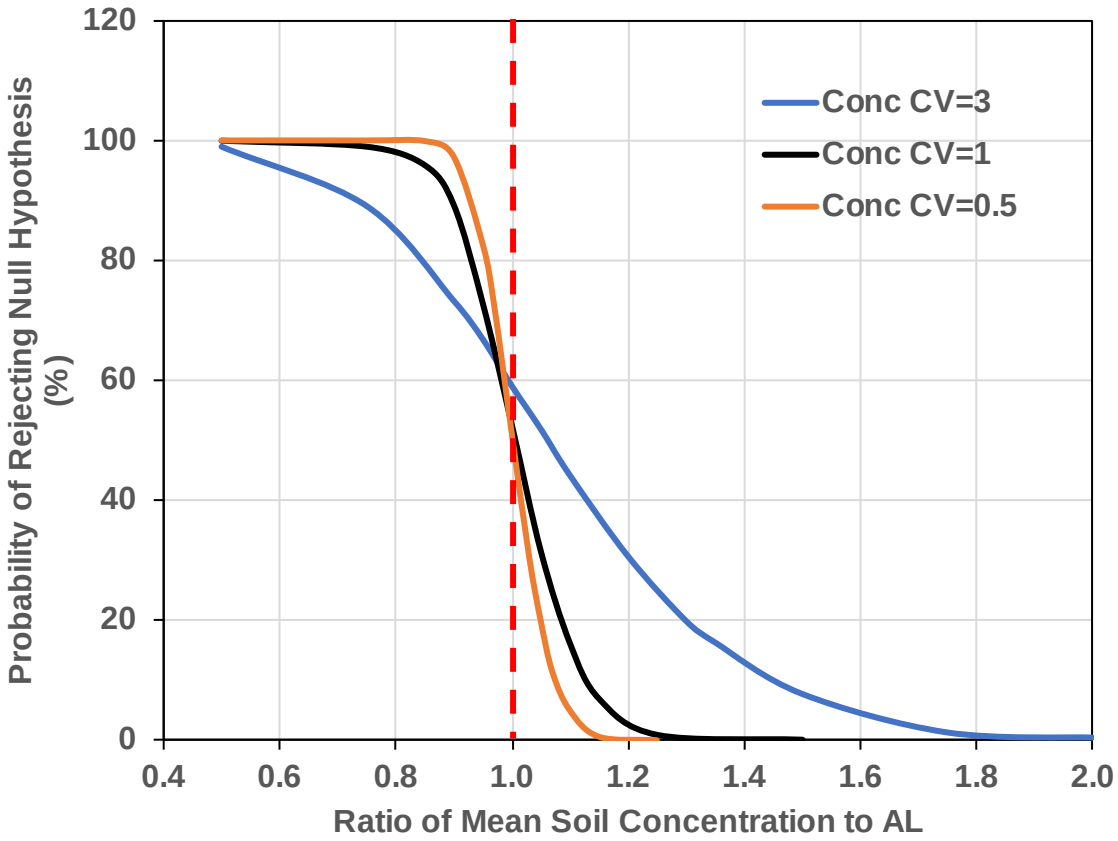

Figure A-4. Probability of Rejecting Null Hypothesis (H_0 = Adjusted Soil Concentration is Above the Action Level) as the Mean Adjusted Soil Concentration Increases when the CV of Soil Concentration is 0.5, 1, or 3.

RBA CV is 0.15. Sample design is ICS 3 composites composed of 30 increments each. The vertical line represents the AL. Probabilities to the right of the vertical line are Type 1 errors (reject H_0 when it is true).

Reference:

U.S. EPA (U.S. Environmental Protection Agency). (2006) Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4. U.S. Environmental Protection Agency, Office of Environmental Information: Washington, DC. EPA/240/B-06/001. February. Available online at: https://www.epa.gov/sites/production/files/2015-06/documents/g4-final.pdf.