



TETRA TECH, INC.

November 6, 2020

Lynn Nakashima
Department of Toxic Substances Control
700 Heinz Avenue, Suite 200C
Berkeley, California 94710

Sara Ziff
U.S. Environmental Protection Agency, Region 9
75 Hawthorne Street
San Francisco, California 94105

**Subject: Corporation Yard, Triplicates Sampling Approach, Revised
Richmond Field Station
University of California, Berkeley**

Dear Ms. Nakashima and Ms. Ziff:

On behalf of the University of California Berkeley, Tetra Tech, Inc. proposes to conduct additional data gap sampling as a follow-up to the removal action conducted at the Corporation Yard in 2017-2018 and data gap sampling presented in the Corporation Yard Data Gaps Sampling Results letter, dated November 22, 2019. At the request of DTSC, this letter also provides clarifications regarding the results presented in the November 2019 letter and discussions at a meeting conducted on May 8, 2020 regarding relative standard deviations (RSD) and the calculations of a weighted 95 upper confidence limit of the mean (weighted 95UCL).

This letter has been updated to incorporate comments received from DTSC on October 27, 2020. The comments and response-to-comments are provided as Attachment A.

PROPOSED SUPPLEMENTAL TRIPLICATE ANALYSIS

The purpose of this investigation is to further determine the mean concentrations of polychlorinated biphenyls (PCB) within the near surface (0-2 inches below ground surface) within the Corporation Yard between Building 120 and the fence line south of Building 185. This area is covered by DU09 through DU17, shown on Figure 1. In response to DTSC recommendations for additional triplicate analysis provided on May 15, 2020 and comments provided on June 17, 2020, UC Berkeley proposes to collect laboratory and field triplicates at DU09, DU10, and DU17. Total PCB sample results presented in the November 2019 letter are presented on Figure 1.

Sampling Approach

Incremental sampling methodology (ISM) will be used to collect soil samples from DU09, DU10, and DU17. ISM was selected for this project to provide a comprehensive and thorough evaluation of chemical concentrations in a specific volume of soil, or decision unit. ISM triplicate sample results enable the

quantification of field and laboratory measurement variability, which is not typically quantified in projects with discrete sample results. While ISM presents measured variability, the ISM sampling procedure is designed specifically to reduce field and laboratory variability when compared to discrete sampling.

The result of each ISM sample will be used as the mean concentration for the decision unit it was collected from. The approach presented below is consistent with the ISM sampling presented in the November 2019 letter.

- Field triplicates will be collected from each of the three decision units. A field triplicate consists of the collection of a minimum of 75 increments thrice within the same decision unit from different locations. In addition to providing chemical results, the field triplicate results help evaluate the effectiveness of the ISM sample to capture any PCB contaminant variability within the decision unit. The field triplicate results will also inherently include any laboratory variability.
- The corners and edges of each decision unit will be marked with flags to identify the grids by which the triplicate increments will be collected. The number of grids for DU9, DU10, and DU17 are 84, 85, and 82, respectively, based on the geometry of each decision unit. The triplicate increments for DU9 and DU10 will be placed equidistant in a triangle formation within each grid. DU10 contains two grids which are not square, and therefore the triplicates will be spaced equidistantly along a line within the two grids. Field conditions at DU17 differ from DU9 and DU10 in that DU17 is covered with 3-6 inches of 3-inch river rocks. The majority of the area has been used for heavy truck parking, and the river rocks have been compacted in place. The river-rocks cannot be easily removed by hand or shovel and require a small backhoe bucket to uncover the original ground surface. UC Berkeley conducted several test excavations on August 25, 2020, and concluded that three separate excavations per grid introduces concerns regarding cross-contamination within triplicate locations, given the amount of movement of the river rocks within a small area. As a result, the triplicates be collected along a single, linear excavation per grid. This method would minimize potential cross contamination between the triplicates since it minimizes the movement of river rocks within the unit. Grids and increment triplicate locations for each decision unit are included on Figures 2, 3, and 4.
- Increments will be collected from the top 2 inches of the native surface with a disposable scoop or other disposable sampling apparatus. In some areas, the native surface is the current surface cover; however, where gravel is present, the gravel will be removed prior to collecting the increment. Each increment will be approximately 20 grams of soil.
- Increments from each decision unit will be placed directly into freezer-grade, 1-gallon ziplocking bags. The target weight of each ISM sample is approximately 1.5 kilograms. Each bag will be labeled and packed into an insulated cooler; the use of ice packs is not necessary for the preservation of samples analyzed for PCBs. The samples will be transported under chain-of-custody procedures to Agriculture & Priority Pollutants Laboratories, Inc. (APPL) in Clovis, California.

Health and safety measures will adhere to the *Final Field Sampling Workplan, Appendix B, Health and Safety Plan*, dated June 2, 2010. Protocols to be followed specific to COVID-19 protections are included as Attachment B.

Laboratory Analyses

Soil samples will be processed according to APPL's internal ISM protocol. The 1.5 kilogram sample will be ground and subsampled to a final analytical aliquot of 30 grams. Samples will be analyzed for PCBs by EPA Method 8082 with 3540C Soxhlet extraction.

One laboratory triplicate will be identified from each field triplicate set and analyzed for PCBs by the laboratory three times. APPL has confirmed that the field sample mass is processed through sieving and grinding, with the triplicate subsampling and segregation occurring following this process. Each laboratory triplicate will be ground, subsampled, and evaluated separately. Following the sieve and grinding process, the remaining mass is laid out in a slab cake. The slab cake is divided into 30 grids and each grid is subsampled three separate times to compose each triplicate sample. The APPL standard operating procedure has been included as Attachment C.

The primary purpose of the laboratory triplicate is to evaluate the effectiveness of the subsampling protocol and any laboratory variability. Together, the field and laboratory triplicates constitute a nested triplicate.

Laboratory Triplicate Evaluation

RSDs will be calculated from each triplicate set. RSD values will be reviewed in conjunction with other pertinent data, including the relationship of the concentrations to action levels as well as the range of concentration values (low, moderate, or elevated). The data usability will be determined by the difference between the weighted mean and the weighted 95%UCL, and whether that distance introduces significant uncertainty into the decision-making process. Too much uncertainty, for example a mean-to-UCL difference that is too large and straddling the action level, may mean that additional data may need to be collected to increase n which decreases the mean-to-UCL distance. It is also possible that data need to be collected using modified sample collection, processing, subsampling, and/or analytical procedures in order to reduce random variability in the data set.

Field Triplicate Evaluation and Weighted 95UCL Calculations

The field triplicate results will be used to calculate RSDs for DU09, DU10, and DU17 in further support of the weighted 95UCL for the area defined above. The general approach is to apply pooled variances from the DUs with triplicates to obtain an average RSD that is applied to calculate 95%UCLs for the singlet DUs, and subsequently calculating a weighted 95UCL for the area encompassed by DU9 through 17.

The weighted 95UCL will apply the first laboratory triplicate sample reported, unless the laboratory triplicate results yield poor precision, then the triplicate mean will be used if the results support such as the most representative value. Evaluation of all triplicate results and application of RSDs should always be evaluated on a case-by-case basis, and will be discussed with EPA and DTSC.

The weighted 95UCL will be calculated consistent with the information presented at the May 8, 2020 meeting and as summarized in the section below.

UC Berkeley will provide preliminary recommendations regarding the appropriate use of the triplicate results in conjunction with all previous sampling results for the area, including discrete samples presented in the Site Characterization Report, Figure 6-8, attached to this letter. The calculation of a weighted 95UCL for this area is consistent with the Corporation Yard boundary evaluated in the risk assessment conducted in support of the RAW, as shown on RAW Figure 2-3, included as an attachment to this letter. The proposed approach will be discussed with EPA and DTSC prior to issuance of a sample results summary or formal weighted 95UCL calculations to ensure concurrence regarding the approach.

Following discussion with DTSC and EPA, the sample results will be presented in a sampling letter report providing complete details regarding the updated weighted 95UCL. Methods and equations and calculation results will be presented within the sample results summary.

The data collected during this investigation will ultimately be presented with the comprehensive data following completion of all Corporation Yard removal action activities.

NOVEMBER 2019 LETTER CLARIFICATIONS

The Corporation Yard Data Gaps Sampling Results letter, dated November 22, 2019, provided a summary of data gaps investigation and the sampling event conducted at the East Meadow, adjacent to the Corporation Yard Boundary, as defined by the Final Removal Action Workplan, dated July 18, 2014. The purpose of the letter was to provide the mean concentrations of PCBs within the near surface (0-2 inches below ground surface) within the entire Corporation Yard, Building 185, and north of Building 197.

The November 2019 letter included a discussion of quality assurance based on the ISM results of the laboratory and field triplicate sample results. The collection of triplicates allowed for the calculation of the RSD for each triplicate set. The laboratory RSD provides an indication of the variability associated with subsampling and analytical procedures. The field RSD provides an indication of how well the sample result represents the average concentration of the area sampled. The field RSD inherently includes variability associated with subsampling and analytical procedures.

The November 2019 letter provided a qualitative summary of the triplicate results and RSDs using several lines-of-evidence to support the conclusions. The evaluation was not intended to provide the basis for applying a confidence interval to a risk-based evaluation.

Subsequent to submittal of the November 2019 letter, EPA, DTSC, and UC Berkeley have conducted several meetings to discuss the strategies and technical approaches for transitioning from a “not-to-exceed” PCB concentration compliant with the Toxic Substances Control Act (TSCA) Section 761.61(a) presented in the Removal Action Workplan (RAW) to a risk-based approach compliant with TSCA Section 761.61(c). As a part of those discussions, UC Berkeley has proposed the calculation of a weighted 95UCL to meet the needs of a risk-based approach with a confidence interval applied. DTSC requested that UC Berkeley provide clarification regarding the proposed approach and discrepancies with the November 2019 letter, as presented below.

RSD Calculations

The weighted 95UCL calculation: (1) normalizes the areal dimensions of the sample results to ensure that results from larger areas are more represented than smaller areas, and (2) incorporates the measured

variability measured in triplicate results to the singlet sample results. The application of the triplicate results within the weighted 95UCL calculation is independent of the qualitative analysis of the triplicate results presented in the November 2019 letter. The approach for calculating a weighted 95UCL for the area south of Building 120 and outside of the previous excavation boundary was presented to EPA and DTSC on May 8, 2020, and is summarized and updated below. The area to be evaluated is represented by sample results from DU09 through DU17. The calculation of a weighted 95UCL for this area is consistent with the Corporation Yard boundary evaluated in the risk assessment conducted in support of the RAW, as shown on RAW Figure 2-3, included as an attachment to this letter.

- The weighted 95UCL calculation applies triplicate results from decision units which are most representative of the decision units they will be applied to. For DU09, DU10, and DU12 through 17, triplicate results from DU11 were selected because they best represent the conceptual site model for contaminant release as the other decision units, and the concentrations are similar with regards to concentrations. The triplicate results from DU9, DU10, and DU17 will be evaluated in conjunction with DU11 triplicate results and other DU results to determine the optimal application of the triplicate results.
- The weighted 95UCL applies only the field triplicate results and not the laboratory triplicate results, since the field triplicates best represent how well the sample results represent the average concentration of the area sampled.
- The weighted 95UCL applies the first laboratory triplicate sample reported, not the average of the three laboratory triplicates. This supports the statistical evaluation of the three field triplicate results since they are all singlet results, and the third is not an average. The first laboratory triplicate reported is always the result selected, regardless of concentration. If laboratory triplicate results yield poor precision, then the triplicate mean can be used if the site-specific data support such as the most representative value.

Consequently, the RSD values presented in the November 2019 letter are different than the RSD values presented in the weighted 95UCL equations presented during the May 8 meeting. The letter presented the average of the three lab results for DU11 (0.23 micrograms per kilogram [$\mu\text{g/kg}$]) to estimate the value of the third field triplicate to calculate the field RSD; however, the weighted 95UCL uses only the first lab triplicate DU11-T3A, which was reported as non-detect. RSD values will be reevaluated following the results of DU09, DU10, and DU17 triplicates.

During the May 8 meeting, simulated triplicate values were discussed in order to meet the requirements of the weighted 95%UCL calculator tool. The calculator tool has been updated so that the calculations no longer require the simulation of simulate triplicate data sets in order to calculate the weighted 95%UCL. The updated calculator tool will be provided to DTSC and EPA upon request.

The surrogate value for the non-detect result is based on an evaluation of half the reporting and method detection limits for Aroclor 1254 and 1260, which are the primary detected PCBs.

- The Aroclor 1254 half reporting limit was 65 $\mu\text{g/kg}$ and the method detection limit was 54 $\mu\text{g/kg}$. If the actual concentration was 65 $\mu\text{g/kg}$, then the method detection limit of 54 $\mu\text{g/kg}$ would have resulted as 65 J $\mu\text{g/kg}$, which it was not. As a result, half the method detection limit of 27 $\mu\text{g/kg}$

is the appropriate surrogate concentration for Aroclor 1254.

- The Aroclor 1260 half reporting limit was 65 µg/kg and method detection limit was 90 µg/kg. If the actual concentration was 65 µg/kg, then the method detection limit of 90 µg/kg would have resulted as a non-detect, which it was. As a result, half the reporting limit of 65 µg/kg is the appropriate surrogate concentration for Aroclor 1260.

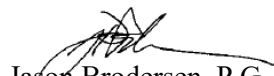
The surrogate sample result used for the weighted 95UCL for DU11-T3A is $27 \mu\text{g/kg} + 65 \mu\text{g/kg} = 92 \mu\text{g/kg}$.

The RSD based on the field triplicate set from DU11-T1 (0.060 µg/kg), DU11-T2 (0.070 µg/kg), and DU11-T3A (0.092 µg/kg) is 21%, which differs from the November 2019 letter presenting 80% RSD.

Note that the calculations provided in the November 2019 letter will be updated with the triplicate data collected during this investigation, including an evaluation of the pooled variances and a reevaluation of the applicability of DU11 triplicate results. Attachment D provides supporting information regarding the use of RSDs and pooled RSDs in ISM projects.

If you have any questions or comments regarding this submittal, please call me at (415) 497-9060 or Greg Haet at (510) 812-1541.

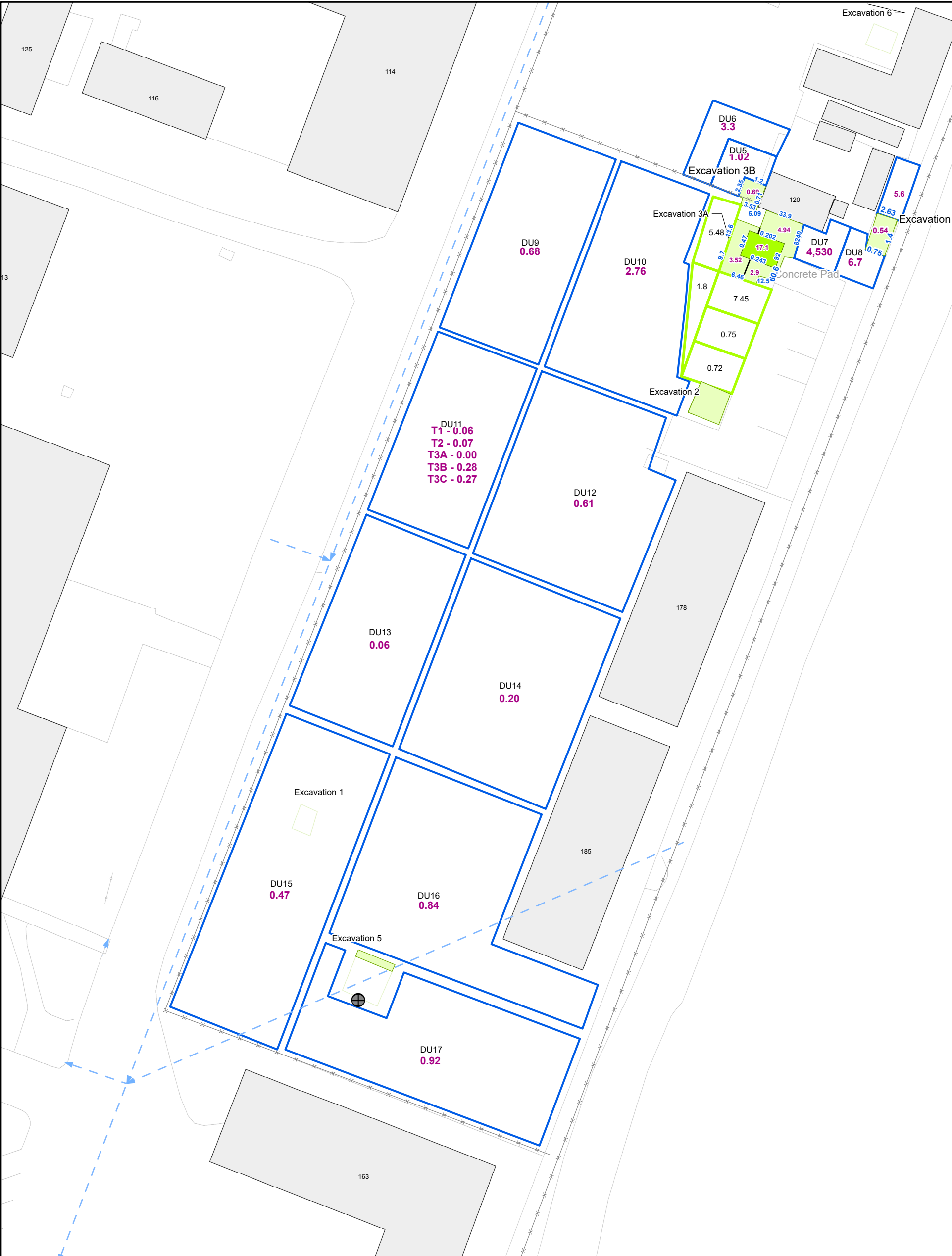
Sincerely,

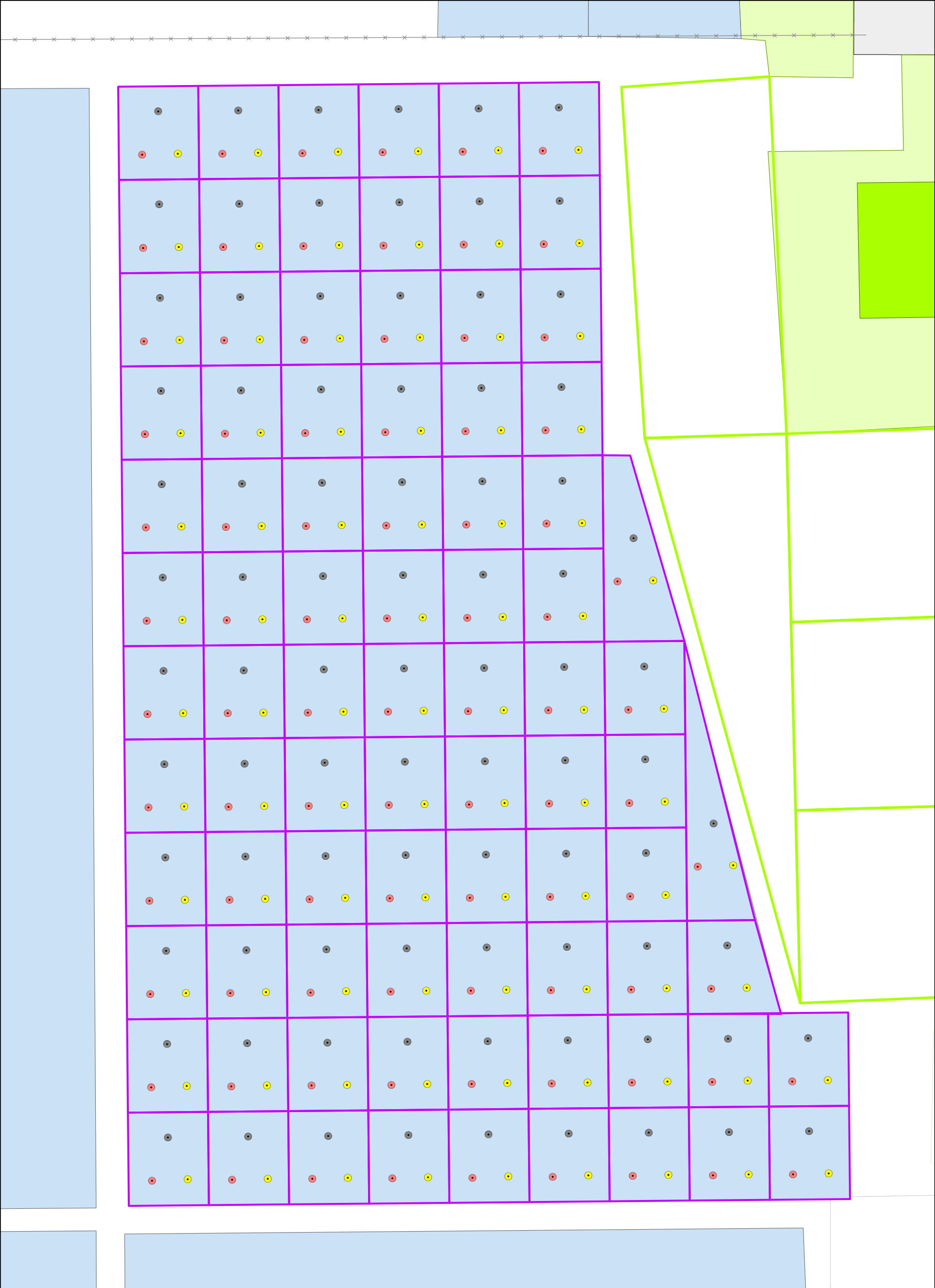


Jason Brodersen, P.G.
Project Manager

Attachments: Figure 1: Site Map
Figure 6-8, Site Characterization Report
Figure 2-3, Removal Action Workplan
Attachment A: Comments and Response-to-Comments
Attachment B: COVID-19 Activity Hazard Analysis
Attachment C: APPL Standard Operating Procedure
Attachment D: Incremental Sampling Methodology, RSD Calculations and Uses

cc: Greg Haet, UC Berkeley EH&S
Bill Marsh, Edgcomb Law Group





Decision Unit

85 Sampling Increments

Final Excavation Depth 1.5 feet

Final Excavation Depth 3.5 feet

Buildings

Sampling Replicates:

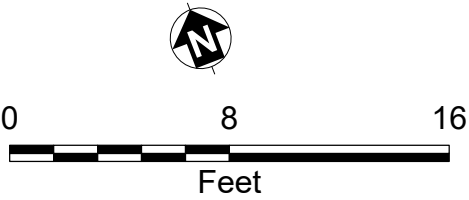
Replicate 1

Replicate 2

Replicate 3

Fenceline

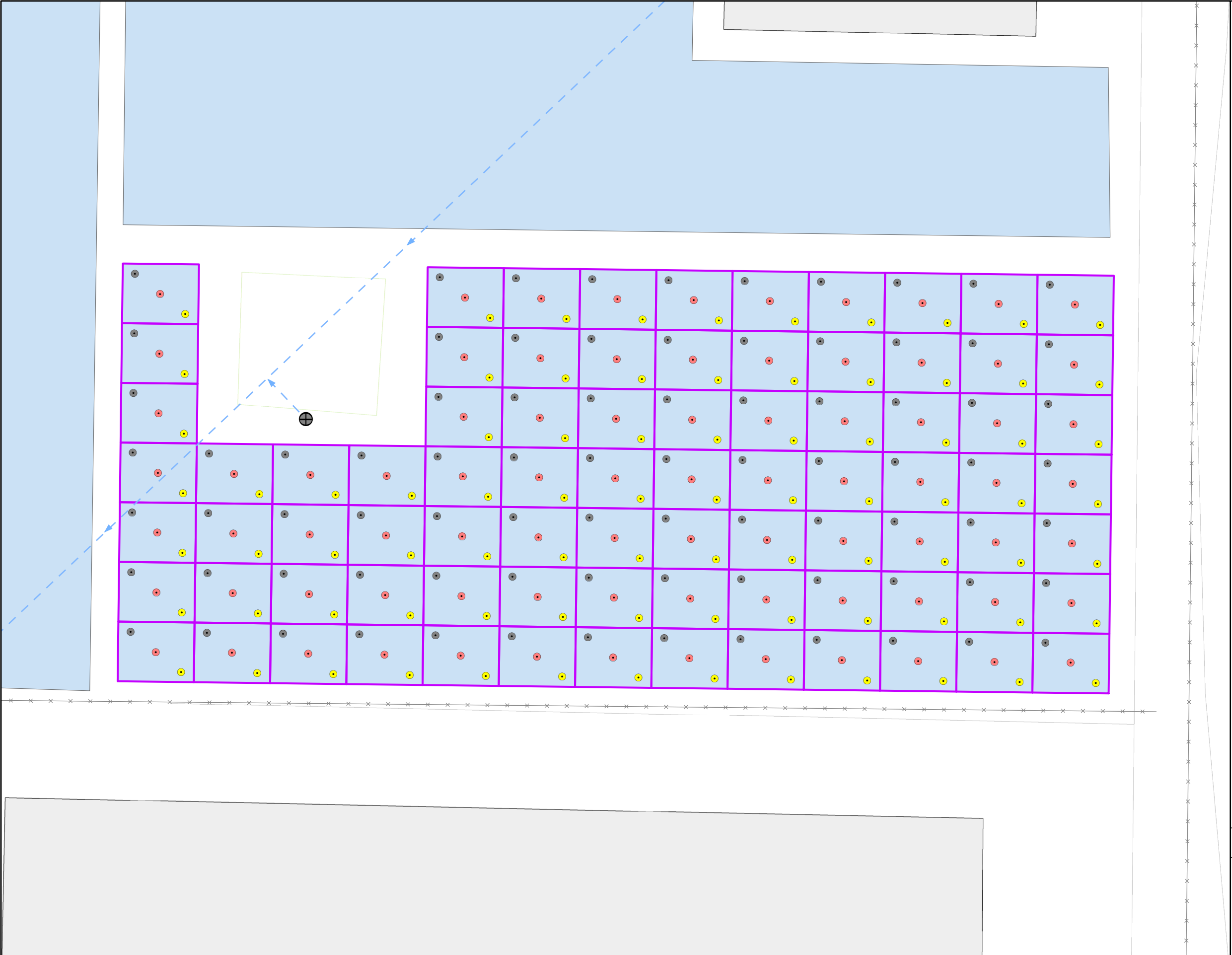
Roads and other Landscape Features



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Richmond Field Station Site
University of California, Berkeley

FIGURE 3
DECISION UNIT 10
INCREMENT LOCATIONS



Decision Unit

82 Sampling Increments

Sampling Replicates:

Replicate 1

Replicate 2

Replicate 3

Buildings

Fenceline

Roads and other Landscape Features

Storm Water Lines and Direction of flow

Storm Water Inlet

0

10

20

Feet

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Richmond Field Station Site

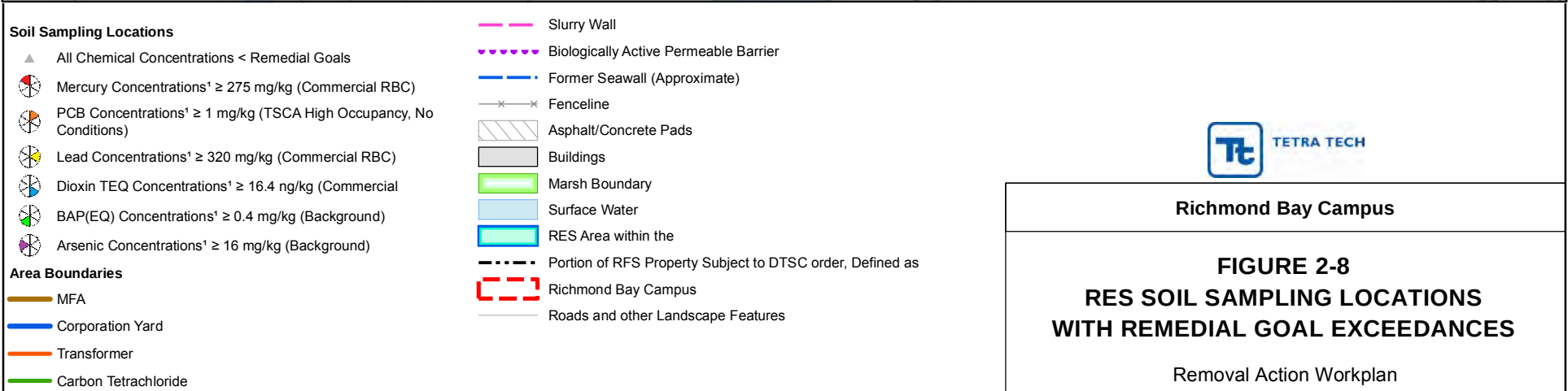
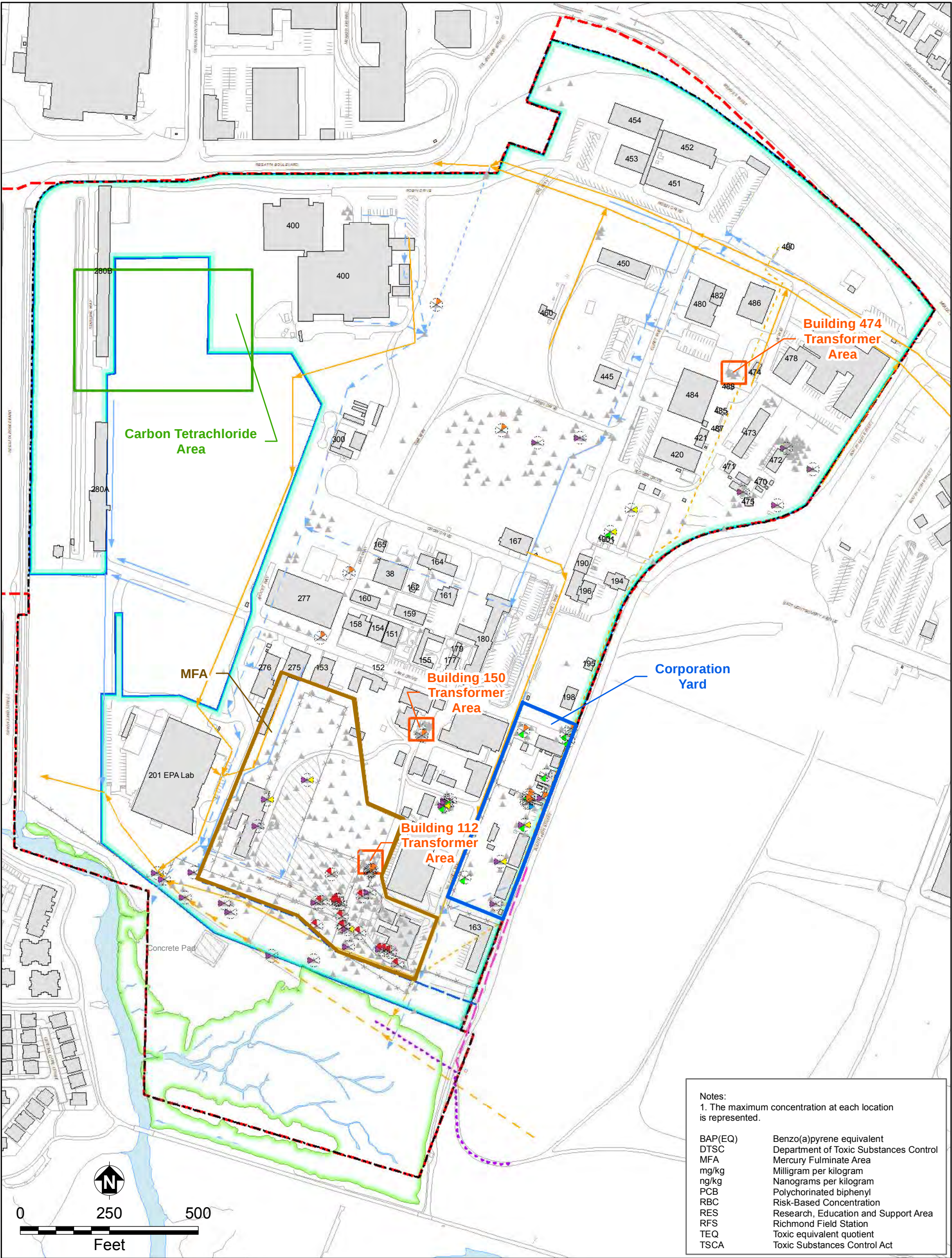
University of California, Berkeley

FIGURE 4

DECISION UNIT 17

INCREMENT LOCATIONS





ATTACHMENT A
COMMENTS AND RESPONSE-TO-COMMENTS



Jared Blumenfeld
Secretary for
Environmental Protection



Department of Toxic Substances Control

Meredith Williams, Ph.D., Director
700 Heinz Avenue
Berkeley, California 94710-2721



Gavin Newsom
Governor

October 27, 2020

Greg Haet, P.E.
EH&S Associate Director, Environmental Protection
Office of Environment, Health & Safety
University of California, Berkeley
University Hall, 3rd Floor, #1150
Berkeley, California 94720
ghjaet@berkeley.edu

Dear Mr. Haet:

The Department of Toxic Substances Control (DTSC) received the July 16, 2020 *Corporation Yard, Triplicate Sampling Approach* letter (Sampling Approach) for the Richmond Field Station site, located at 1301 South 46th Street in Richmond, California. The Sampling Approach prepared by Tetra Tech, Inc. on behalf of the University of California, Berkeley clarifies information previously provided in a November 2019 letter and during discussions at a meeting held on May 8, 2020 and responds to DTSC's June 17, 2020 comment letter. The Sampling Approach proposes to conduct additional data gap sampling at the Corporation Yard using the incremental sampling method. DTSC program, Human and Ecological Risk Office (HERO) and Geologic Services Unit staff have reviewed the proposal and have the following comments. Also enclosed is a memorandum with comments prepared by Dr. Karen DiBiasio of HERO.

1. Page 7 of 7: The surrogate sample result for DU11-T3A should be 92 µg/kg, not 0.092 µg/kg. Please correct this value.

The Sampling Approach needs to be revised to address the above comment and those found in the enclosed memorandum. Please submit a revised document within 30 days of the date of this letter.

Mr. Greg Haet, P.E.

October 27, 2020

Page 2

If you have any questions regarding this letter, please contact Lynn Nakashima at lynn.nakashima@dtsc.ca.gov.

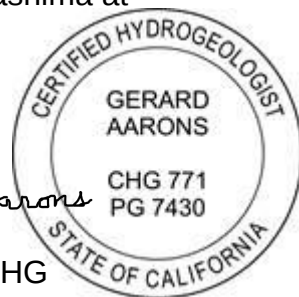
Sincerely,

Lynn Nakashima

Lynn Nakashima, Project Manager
Senior Hazardous Substances Scientist
Site Mitigation and Restoration Program
Berkeley Office - Cleanup Operations

Gerard F. Aarons

Gerard F. Aarons, PG, CHG
Senior Engineering Geologist
Site Mitigation and Restoration Program
Geological Services Branch



Enclosure

cc: (via email)

Sara Ziff, P.E.
US Environmental Protection Agency
Region IX
Land, Chemicals and Redevelopment Division
Ziff.Sara@epa.gov

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Karen DiBiasio, Ph.D.
Staff Toxicologist
Human and Ecological Risk Office
Department of Toxic Substances Control
Karen.DiBiasio@dtsc.ca.gov

From: DiBiasio_Karen@DTSC
To: Nakashima_Lynn@DTSC
Cc: Mathrani_Vivek@DTSC; Endlich_Brian@DTSC; Sorrentino_Claudio@DTSC
Subject: HERO comments on UC Berkeley Richmond Field Station, Corp Yard - PCBs ISM sampling plan Project Code: 201605-00 Activity Code: 11018 MPC: TECHMEMO
Date: Tuesday, August 11, 2020 4:20:59 PM

TO: Lynn Nakashima
Project Manager
Site Mitigation and Restoration Program
700 Heinz Avenue
Berkeley, California 94710-2721

FROM: Karen W. DiBiasio, Ph.D.
Staff Toxicologist
Human and Ecological Risk Office (HERO)
Site Mitigation and Restoration Program

DATE: August 11, 2020

SUBJECT: UC BERKELEY – RICHMOND FIELD STATION, CORPORATION YARD,
RICHMOND, CALIFORNIA

INCREMENTAL SAMPLING METHOD (ISM) SAMPLING PLAN

Project Code: 201605-00 Activity Code: 11018 MPC: TECHMEMO

DOCUMENT REVIEWED

HERO reviewed the July 16, 2020 memorandum with the subject “Corporation Yard, Triplicates Sampling Approach, Richmond Field Station, University of California, Berkeley” (Tech Memo) prepared by Tetra Tech in Oakland, California.

BACKGROUND

The Richmond Field Station (RFS) Corporation Yard (Corp Yard or Site) had surface releases of PCBs in transformer oil and is currently used primarily for parking of PG&E trucks. Additional sampling for PCBs is proposed using the incremental sampling method (ISM) as a follow-up to the removal action conducted at the Corporation Yard in 2017-2018 and data gap sampling presented in the Corporation Yard Data Gaps Sampling Results letter dated November 22, 2019. Previously, in a June 16, 2020 email HERO provided comments on the June 3, 2020 tech memo with the subject “Corporation Yard, Triplicates Sampling Approach, Richmond Field Station, University of California, Berkeley” (Tech Memo) prepared by Tetra Tech in Oakland, California. The Tech Memo reviewed herein also provides clarifications regarding the ISM results presented in the November 22, 2019 letter and recent teleconferences on the relative standard deviation (RSD) of laboratory and field replicates and the calculation of the weighted 95 percent upper confidence limit of the arithmetic mean (95%UCL). The Tech Memo reviewed herein has been updated to incorporate DTSCs June 17, 2020 comments (which contain HERO’s June 16, 2020 comments) and includes response-to-comments in Attachment A.

SCOPE OF REVIEW

The review comments herein focus solely on the ISM sampling for PCBs from 0-2 inches below ground surface and use of the ISM results to calculate a 95%UCL. The intent of HERO’s comments

is to yield a transparent and scientifically defensible work plan.

COMMENTS

1. Work Plan Insufficient: The Tech Memo incompletely presents the proposed activities. In addition, the Tech Memo contains some internal inconsistencies and ambiguities. Some aspects of the proposal within the Tech Memo are insufficient and lack transparency, as detailed in the below comments. HERO does not fully concur with some of the technical aspects of the proposed sampling, as described below. HERO recommends revising the proposed ISM sampling plan per the below comments.
2. Increment Locations – Within each Decision Unit (DU) 75 increments are proposed for collection. The Tech Memo states on page 2 that the spacing of increments will be determined in the field, whereas both further down on page 2 as well as on page 2 of Attachment A containing response to comments state *“The locations of increments comprising the first triplicate will be placed at 75 locations based on equally spaced grid nodes. The second and third triplicate increments will be collected 3 ft away from the each of the first triplicate increment locations.”* ISM guidance (ITRC, 2012) recommends systematic planning and random locations. To reduce potential error in the estimate of the mean, to guard against bias in increment sampling locations and to provide even spatial coverage in each DU, HERO recommends use of a systematic random sampling approach using a random number generator to determine placement of replicates 1, 2 and 3 within the first grid and applying those relative locations to the remaining 74 grids. For singlet DUs, it is only replicate 1 that will be randomly assigned a location in the first grid, then apply the same relative location to the remaining 74 grids. For transparency, HERO recommends providing a figure to demonstrate the proposed locations of the increments within each DU.
3. Field Triplicates – The Tech memo is insufficient in its presentation on aspects of the field triplicates. HERO recommends the following:
 - Include in the Sampling Approach section that one of the purposes of collecting field triplicates is to enable calculation of 95%UCLs from singlet DUs.
 - Include in the Field Triplicate Evaluation section the RSD criteria for acceptability for data usability.
4. Laboratory Subsampling – The Tech Memo is unclear whether the laboratory subsampling is one 30 gram aliquot or multiple aliquots from the full depth of a Japanese slab cake or some other method. HERO recommends transparently presenting the proposed laboratory subsampling procedure.
5. Laboratory Triplicate Processing – The Tech Memo is ambiguous on whether the field sample that will be processed and analyzed as a lab triplicate is homogenized/ground before or after separating the field sample into lab triplicates. HERO recommends transparently describing when grinding is proposed, and preferably grinding before segregating the soil from the field sample into lab triplicates to reduce variability in lab triplicate results.
6. Laboratory Triplicate RSD Evaluation – While HERO does not fully concur with the use of the lab RSD goals in the Tech Memo, HERO concurs with deferring discussion on the subject to after the analytical data are produced. HERO notes that of the scenarios evaluated in the lab RSD simulations presented in the Tech Memo only those with total PCB concentrations in the 0.2 – 0.7 and 0.7 – 2 mg/kg ranges (scenarios 2 and 3) are potentially subject to decision errors.
7. Inconsistency in Field Triplicates for Pooled Variance – It is unclear whether DU11 field triplicates will be used in calculating the pooled variance that will be applied to singlet DUs to derive their 95%UCLs. HERO recommends clarifying whether triplicate results from DU11 are proposed for inclusion in calculating the pooled variance because page 4 (Field Triplicates

Evaluation and Weighted 95UCL Calculations section) only specifies DUs 9, 10 and 17, whereas Attachment A response to comments page 4 states DU11 will also be included. If DU11 is used for calculating the pooled variability, provide justification for its use with non-detected concentrations of PCBs.

8. **95%UCL** – Since the exposure area for risk-based decision making (exposure unit) is the entire Corp Yard, a weighted 95%UCL is proposed from the ISM data collected from DUs 9 through 17. The Tech Memo proposes using pooled variances from the DUs with triplicates to obtain an average RSD and subsequently calculating a weighted 95%UCL. Applying pooled variance from triplicates to calculate 95%UCLs for the singlet DUs is appropriate for CSM-equivalent DUs where a statistical test that compares variances demonstrates that the differences in variances are not statistically significant (e.g., at the 95% level of confidence). While the concept of a weighted 95%UCL is appropriate for the Corp Yard, the proposed methods and equations are not presented. The Tech Memo is unclear on whether the proposal is to use the pooled variance to calculate 95%UCLs for the singlet DUs or to derive surrogate replicate values for replicates 2 and 3 of singlet DUs so each DU will have 'data' for triplicates to then calculate the weighted 95%UCL. HERO recommends using the pooled variance to calculate 95%UCLs for the singlet DUs, then applying the spatial area weighting factors to generate the overall 95%UCL for Corp Yard consisting of DUs 9 through 17. HERO recommends transparently providing the proposed weighted 95%UCL methodology with all equations.
9. **RSD Calculations** – The Tech Memo on pages 6 - 7 provides clarification on RSD calculations in the November 2019 letter and notes those RSDs were not intended for use in calculating 95%UCLs for risk-based decisions. However, it is unclear what procedure is intended for the application to the proposed field triplicate ISM results. HERO recommends setting RSD limits in the Data Quality Objectives for data usability determination. HERO recommends transparently presenting (a) the proposal for field triplicate RSD calculations and all associated equations, and (b) the pooled variance calculations and all associated equations.

CONCLUSIONS

HERO reviewed the July 16, 2020 Tech Memo for additional ISM sampling and analysis at the Corp Yard. HERO does not concur with the proposed sampling, primarily due to insufficient presentation of the proposal and internal inconsistency. HERO recommends addressing the comments above to improve transparency and scientific defensibility in a revised ISM sampling plan submission.

Please contact me at (916) 255-6633 or Karen.DiBiasio@dtsc.ca.gov if you have any questions.

Reviewed by: Vivek Mathrani, Ph.D., DABT
Staff Toxicologist
Human and Ecological Risk Office
Brownfields and Environmental Restoration Program

Brian P. Endlich, Ph.D.
Senior Toxicologist
Chief, Central California Unit
Human and Ecological Risk Office
Brownfields and Environmental Restoration Program

Concur: Claudio Sorrentino, Ph.D.
Senior Toxicologist
Chief, Northern California Unit
Human and Ecological Risk Office
Brownfields and Environmental Restoration Program

Karen W. DiBiasio, Ph.D.
Staff Toxicologist
Human and Ecological Risk Office
Department of Toxic Substances Control
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Sacramento, CA 95826-3268

Karen.DiBiasio@dtsc.ca.gov
(916) 255-6633

**Removal Action Workplan
Research, Education, and Support Area and Groundwater
Corporation Yard Triplicate Sampling Approach
July 16, 2020**

**Response to Comments from
Department of Toxic Substances Control
October 27, 2020**

Page 1 of 5

DTSC Comment	Comment	UC Berkeley Response
1	Page 7 of 7: The surrogate sample result for DU11-T3A should be 92 µg/kg, not 0.092 µg/kg. Please correct this value.	Text has been updated to 92 µg/kg.
DTSC HERO Comment No.	Comment	UC Berkeley Response
1	<u>Work Plan Insufficient</u> : The Tech Memo incompletely presents the proposed activities. In addition, the Tech Memo contains some internal inconsistencies and ambiguities. Some aspects of the proposal within the Tech Memo are insufficient and lack transparency, as detailed in the below comments. HERO does not fully concur with some of the technical aspects of the proposed sampling, as described below. HERO recommends revising the proposed ISM sampling plan per the below comments.	Responses are provided to specific comments detailed below.

**Removal Action Workplan
Research, Education, and Support Area and Groundwater
Corporation Yard Triplicate Sampling Approach
July 16, 2020**

**Response to Comments from
Department of Toxic Substances Control
October 27, 2020**

Page 2 of 5

2	<u>Increment Locations</u> – Within each Decision Unit (DU) 75 increments are proposed for collection. The Tech Memo states on page 2 that the spacing of increments will be determined in the field, whereas both further down on page 2 as well as on page 2 of Attachment A containing response to comments state <i>“The locations of increments comprising the first triplicate will be placed at 75 locations based on equally spaced grid nodes. The second and third triplicate increments will be collected 3 ft away from the each of the first triplicate increment locations.”</i> ISM guidance (ITRC, 2012) recommends systematic planning and random locations. To reduce potential error in the estimate of the mean, to guard against bias in increment sampling locations and to provide even spatial coverage in each DU, HERO recommends use of a systematic random sampling approach using a random number generator to determine placement of replicates 1, 2 and 3 within the first grid and applying those relative locations to the remaining 74 grids. For singlet DUs, it is only replicate 1 that will be randomly assigned a location in the first grid, then apply the same relative location to the remaining 74 grids. For transparency, HERO recommends providing a figure to demonstrate the proposed locations of the increments within each DU.	Replicate triplicate samples are collected from within similar sized grids within each DU; the number of grids is determined by the number of increments selected for each project. For this project, a minimum of 75 increments has been selected, therefore a minimum of 75 grids will be identified. There are several acceptable methods for determining the spacing of triplicate samples. One method involves identifying three random locations within each grid, as suggested by the DTSC comment. Another method consists of selecting the first triplicate location randomly, and then selecting the two subsequent triplicates at predetermined distances from the first. A third method consists of spacing the triplicates equidistantly within each grid. All methods are considered acceptable and valid. Given the small sizes of the grids for this project (approximately 7 feet by 7 feet), the triplicate spacing is not critical to the overall objectives, since each triplicate will be no more than 2 to 3 feet apart. UC Berkeley recommends spacing the triplicate increments for DU9 and DU10 equidistant in a triangle formation within each grid. DU10 contains two grids which are not square, and therefore the triplicates will be spaced equidistantly along a line within the two grids. Field conditions at DU17 differ from DU9 and DU10 in that DU17 is covered with 3-6 inches of 3-inch river rocks. The majority of the area has been used for heavy truck parking, and the river rocks have been compacted in place. The river-rocks cannot be easily removed by hand or shovel and require a small backhoe bucket to uncover the original ground surface. UC Berkeley conducted several test excavations on August 25, 2020, and concluded that three separate excavations per grid introduces concerns regarding cross-contamination within triplicate locations, given the amount of movement of the river rocks within a small area. UC Berkeley instead recommends that each of the triplicates be collected along a single, linear excavation per grid. This method would eliminate potential cross contamination between the triplicates since it minimizes the movement of river rocks within the unit.
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**Removal Action Workplan
Research, Education, and Support Area and Groundwater
Corporation Yard Triplicate Sampling Approach
July 16, 2020**

**Response to Comments from
Department of Toxic Substances Control
October 27, 2020**

Page 3 of 5

		The sampling letter has been amended to include this information, and Figures 2, 3, and 4 demonstrate the increment grids and triplicate locations for DU9, DU10, and DU17.
3	<u>Field Triplicates</u> – The Tech memo is insufficient in its presentation on aspects of the field triplicates. HERO recommends the following: • Include in the Sampling Approach section that one of the purposes of collecting field triplicates is to enable calculation of 95%UCLs from singlet DUs. • Include in the Field Triplicate Evaluation section the RSD criteria for acceptability for data usability.	Text has been amended to clarify that the intent of collecting additional triplicate samples is to improve the confidence in the calculation of a weighted 95% UCL for DU9 through DU17. The triplicates will not be used to calculate 95% UCLs for existing singlet DU results. Text has been amended to eliminate the numeric RSD goals, and text has been added to clarify that RSD values will be reviewed in conjunction with other pertinent data, including the relationship of the concentrations to action levels as well as the range of concentration values (low, moderate, or elevated). Text has been added to clarify that the data usability is ultimately determined by the difference between the weighted mean and the weighted 95%UCL, and whether that distance introduces significant uncertainty into the decision-making process. Too much uncertainty, for example a mean-to-UCL difference that is too large and straddling the action level, may mean that additional data may need to be collected to increase n which decreases the mean-to-UCL distance. It is also possible that data need to be collected using modified sample collection, processing, subsampling, and/or analytical procedures in order to reduce random variability in the data set.
4	<u>Laboratory Subsampling</u> – The Tech Memo is unclear whether the laboratory subsampling is one 30 gram aliquot or multiple aliquots from the full depth of a Japanese slab cake or some other method. HERO recommends transparently presenting the proposed laboratory subsampling procedure.	The laboratory APPL has confirmed that the received sample mass is sieved and ground, and then the remaining mass is laid out in a slab cake. The slab cake is divided into 30 grids and each grid is subsampled three separate times to compose each triplicate sample. The APPL standard operating SOP is included as Attachment C.
5	<u>Laboratory Triplicate Processing</u> – The Tech Memo is ambiguous on whether the field sample that will be processed and analyzed as a lab triplicate is homogenized/ground before or after separating the field sample into lab triplicates. HERO recommends transparently describing when grinding is proposed, and preferably grinding before segregating the soil from the field sample into lab triplicates to reduce variability in lab triplicate results.	APPL has confirmed that the field sample mass is processed through sieving and grinding, with the triplicate subsampling and segregation occurring following this process. The APPL standard operating procedure is included as Attachment C.

**Removal Action Workplan
Research, Education, and Support Area and Groundwater
Corporation Yard Triplicate Sampling Approach
July 16, 2020**

**Response to Comments from
Department of Toxic Substances Control
October 27, 2020**

Page 4 of 5

6	<u>Laboratory Triplicate RSD Evaluation</u> – While HERO does not fully concur with the use of the lab RSD goals in the Tech Memo, HERO concurs with deferring discussion on the subject to after the analytical data are produced. HERO notes that of the scenarios evaluated in the lab RSD simulations presented in the Tech Memo only those with total PCB concentrations in the 0.2 – 0.7 and 0.7 – 2 mg/kg ranges (scenarios 2 and 3) are potentially subject to decision errors.	As discussed in response to HERO Comment 3, text has been amended to eliminate the numeric RSD goals, and text has been added to clarify that RSD values will be reviewed in conjunction with other pertinent data, including the relationship of the concentrations to action levels as well as the range of concentration values (low, moderate, or elevated). The adequacy of data precision will be evaluated by its effect on the 95%UCL in relation to the cleanup level and recommendations.
7	<u>Inconsistency in Field Triplicates for Pooled Variance</u> – It is unclear whether DU11 field triplicates will be used in calculating the pooled variance that will be applied to singlet DUs to derive their 95%UCLs. HERO recommends clarifying whether triplicate results from DU11 are proposed for inclusion in calculating the pooled variance because page 4 (Field Triplicates Evaluation and Weighted 95UCL Calculations section) only specifies DUs 9, 10 and 17, whereas Attachment A response to comments page 4 states DU11 will also be included. If DU11 is used for calculating the pooled variability, provide justification for its use with non-detected concentrations of PCBs.	Text has been clarified that the use of DU11 triplicate data will be evaluated following receipt of the new triplicate data. Note that the term "pooled" applies when two or more field triplicate sets are available to predict the variability of conceptual site model-equivalent DUs. To take advantage of all available information, the variabilities can be "averaged" into a single value that is used as the "borrowed" variability for singlet DUs. If only a single triplicate DU is available, there is no pooling. Attachment D provides an overview of the calculation of RSD and pooled RSDs. As discussed in response to HERO Comment 3, the purpose is not to derive 95%UCLs for the singlet DUs. An expression of variability or RSD for each DU is needed in order to calculate the overall weighted 95%UCL for DU9 through DU17.

**Removal Action Workplan
Research, Education, and Support Area and Groundwater
Corporation Yard Triplicate Sampling Approach
July 16, 2020**

**Response to Comments from
Department of Toxic Substances Control
October 27, 2020**

Page 5 of 5

8	<u>95%UCL</u> – Since the exposure area for risk-based decision making (exposure unit) is the entire Corp Yard, a weighted 95%UCL is proposed from the ISM data collected from DUs 9 through 17. The Tech Memo proposes using pooled variances from the DUs with triplicates to obtain an average RSD and subsequently calculating a weighted 95%UCL. Applying pooled variance from triplicates to calculate 95%UCLs for the singlet DUs is appropriate for CSM-equivalent DUs where a statistical test that compares variances demonstrates that the differences in variances are not statistically significant (e.g., at the 95% level of confidence). While the concept of a weighted 95%UCL is appropriate for the Corp Yard, the proposed methods and equations are not presented. The Tech Memo is unclear on whether the proposal is to use the pooled variance to calculate 95%UCLs for the singlet DUs or to derive surrogate replicate values for replicates 2 and 3 of singlet DUs so each DU will have ‘data’ for triplicates to then calculate the weighted 95%UCL. HERO recommends using the pooled variance to calculate 95%UCLs for the singlet DUs, then applying the spatial area weighting factors to generate the overall 95%UCL for Corp Yard consisting of DUs 9 through 17. HERO recommends transparently providing the proposed weighted 95%UCL methodology with all equations.	Text has been clarified that the proposed weighted 95%UCL evaluation is for the geographic area covered by DU9 through DU17 only. This area is a subset of the exposure area identified for the Corporation Yard within the risk assessment presented in the Site Characterization Report, dated May 28, 2013. Completion of the response action for the entire Corporation Yard will incorporate the results of the evaluation of DU9 through DU17. In regards to use of a statistical test, such as an F test, to evaluate the equality of variances, given that n is only 3 for triplicate sets, the F test will have a low power to determine a quantifiable difference between variances, even if a meaningful difference exists. As a result, a statistical test is not recommended. As discussed in response to HERO Comment 3, the purpose is not to derive 95%UCLs for the singlet DUs, but instead to calculate the weighted 95%UCL for the area represented by DU9 through DU17. The text has been amended to clarify that the calculations no longer require the simulation of simulate triplicate data sets in order to calculate the weighted 95%UCL.
9	<u>RSD Calculations</u> – The Tech Memo on pages 6 – 7 provides clarification on RSD calculations in the November 2019 letter and notes those RSDs were not intended for use in calculating 95%UCLs for risk-based decisions. However, it is unclear what procedure is intended for the application to the proposed field triplicate ISM results. HERO recommends setting RSD limits in the Data Quality Objectives for data usability determination. HERO recommends transparently presenting (a) the proposal for field triplicate RSD calculations and all associated equations, and (b) the pooled variance calculations and all associated equations.	As discussed in response to HERO Comment 3, text has been amended to eliminate the numeric RSD goals, and text has been added to clarify that RSD values will be reviewed in conjunction with other pertinent data, including the relationship of the concentrations to action levels as well as the range of concentration values (low, moderate, or elevated).

ATTACHMENT B
COVID-19 ACTIVITY HAZARD ANALYSIS



ACTIVITY HAZARD ANALYSIS (AHA)

Tetra Tech Inc.

Procedures for Working in Areas Potentially Impacted by COVID-19

Task Description

This Activity Hazard Analysis (AHA) applies to field work in areas potentially impacted by COVID-19. The “essentiality” of field work should be determined by the office manager and Health & safety Department (HSD). It has been developed and approved by the HSD for Tetra Tech EMI. This AHA contains potential hazards posed by working in the field during pandemic conditions, lists procedures to control hazards to minimize possible exposure, and presents required safety equipment, inspections, and training.

Overall Job Risk Assessment code (RAC)

Low

Hazards		Actions	
Task Steps	Potential Hazards	Critical Safety Procedures and Controls	Risk Assessment Code (RAC)
Determine work essentiality. Work with your OM, PM and HSD to determine if the work is essential.	Exposure to persons or locations potentially impacted by COVID-19	DO review SWP 5-55, <i>Infectious Disease Guidance</i>, and Tetra Tech EMI <i>COVID-19 Response and Contingency Plan</i> (attached) DO NOT just show-up. These are trying times, and some may not appreciate it. The client should be amenable to this. DO determine if work can be done virtually or remotely. DO call ahead and tell them who you are, what you would like to do, why you need to do it, and what time to expect you. DO ask the clients or persons you may be working with: - Has anyone working/residing in or that recently visited the facility travelled out of the country or to any location (i.e. nursing home, daycare center, etc.) where someone has been diagnosed with COVID-19 in the past three weeks? - Has anyone working/residing in or that recently visited the facility been diagnosed with COVID-19? - Has anyone working/residing in or that recently visited the facility experienced any of the following symptoms? Fever? Sore throat? Cough? New shortness of breath? - If YES to ANY, inform the client and work with your OM, PM, supervisor and Safety Manager to determine essentiality of trip.	Low

Work deemed essential. Travel to the worksite,	Exposure to persons or locations potentially impacted by COVID-19	• DO travel in separate vehicles if possible. • DO voluntarily use of respirators IAW 29 CFR 1910.134 Appendix D (attached) with P-100 cartridges if you have one and prefer to wear it. • DO have all Tetra Tech personnel perform self-evaluations each day PRIOR to work. If any new symptoms or if any potential exposures have occurred, the employee should STAY HOME or in the hotel if on travel. • DO maintain a supply of soap and water, alcohol-based hand sanitizer (ABHS), AND sanitizing wipes at all times.	Low
Arrive at facility and preparing to conduct assigned tasks.	Exposure to persons or locations potentially impacted by COVID-19	• DO voluntarily use of respirators IAW 29 CFR 1910.134 Appendix D (attached) with P-100 cartridges if you have one and prefer to wear it. • DO maintain social distancing from everyone, including the client and your coworkers, even during safety briefings and planning sessions. • DO NOT shake hands or touch anyone. • DO NOT touch your face. • DO wash your hands with soap and water for at least 20 seconds or use ABHS PRIOR to donning Nitrile gloves. • DO don Nitrile gloves PRIOR to approaching other persons. This will keep them from wanting to shake hands. • DO wear two pair of Nitrile gloves and keep the inner pair on continuously while replacing the outer pair after each sampling event. • DO place all supplies in a 5-gallon bucket or similar non-porous container. • DO NOT approach if you hear, see, or suspect that someone at the site may be ill and inform the client. • DO NOT attempt to persuade someone that you need to enter if they disagree with the essentiality of why you are there. Thank them, leave, and inform the client. • DO NOT use conversation to explain your visit. You explained this when you called. If appropriate, print any documentation explaining why you are there and present it when you arrive. • DO NOT accept any food or drinks offered to you by unknown persons. • DO proceed immediately to the area to conduct required tasks.	Low

Conduct assigned tasks.	Exposure to persons or locations potentially impacted by COVID-19	• DO voluntarily use of respirators IAW 29 CFR 1910.134 Appendix D (attached) with P-100 cartridges if you have one and prefer to wear it. • DO keep everything (i.e. sampling supplies, tools, logbook, pens etc.) you have in the bucket or similar non-porous container and NEVER set anything down inside buildings or on the ground except the container. • DO conduct required tasks. • DO thank the client or persons you worked with and depart. • DO continue wearing gloves to manage the samples. • DO wipe the outside and bottom of the bucket with a sanitizing wipe prior to placing it back in the vehicle. • DO wash your hands with soap and water for at least 20 seconds or use ABHS immediately AFTER doffing Nitrile gloves. • DO wash your hands with soap and water for at least 20 seconds or use ABHS and re-don Nitrile gloves while handling and documenting the samples at the end of the day. • DO update this procedure as you learn more or recognize additional steps or controls are needed.	LOW
<u>Equipment to be Used</u> • Ensure that you have the minimum PPE and supplies for ALL other assigned tasks (see task specific AHAs) • Minimum PPE: steel-toed boots, safety glasses, nitrile gloves and Type 2 or better reflective safety vest • Disinfecting hand soap • Alcohol-based hand sanitizer (>60%) • Sanitizing wipes • First Aid Kit • 5-gallon bucket or similar non-porous container	<u>Inspection Requirements</u> • Inspect all PPE for proper operation, wear and defects	<u>Training Requirements</u> • See task specific AHAs	



[By Standard Number](#) / 1910.134 App D - (Mandatory) Information for Employees Using Respirators When not Required Under Standard.

▪ Part Number:	1910
▪ Part Number Title:	Occupational Safety and Health Standards
▪ Subpart:	1910 Subpart I
▪ Subpart Title:	Personal Protective Equipment
▪ Standard Number:	1910.134 App D
▪ Title:	(Mandatory) Information for Employees Using Respirators When not Required Under Standard.
▪ GPO Source:	e-CFR

Appendix D to Sec. 1910.134 (Mandatory) Information for Employees Using Respirators When Not Required Under the Standard

Respirators are an effective method of protection against designated hazards when properly selected and worn. Respirator use is encouraged, even when exposures are below the exposure limit, to provide an additional level of comfort and protection for workers. However, if a respirator is used improperly or not kept clean, the respirator itself can become a hazard to the worker. Sometimes, workers may wear respirators to avoid exposures to hazards, even if the amount of hazardous substance does not exceed the limits set by OSHA standards. If your employer provides respirators for your voluntary use, or if you provide your own respirator, you need to take certain precautions to be sure that the respirator itself does not present a hazard.

You should do the following:

1. Read and heed all instructions provided by the manufacturer on use, maintenance, cleaning and care, and warnings regarding the respirators limitations.
2. Choose respirators certified for use to protect against the contaminant of concern. NIOSH, the National Institute for Occupational Safety and Health of the U.S. Department of Health and Human Services, certifies respirators. A label or statement of certification should appear on the respirator or respirator packaging. It will tell you what the respirator is designed for and how much it will protect you.
3. Do not wear your respirator into atmospheres containing contaminants for which your respirator is not designed to protect against. For example, a respirator designed to filter dust particles will not protect you against gases, vapors, or very small solid particles of fumes or smoke.
4. Keep track of your respirator so that you do not mistakenly use someone else's respirator.

[63 FR 1152, Jan. 8, 1998; 63 FR 20098, April 23, 1998]

TETRA TECH EMI COVID-19 RESPONSE AND CONTINGENCY PLAN

INTRODUCTION

The health and safety (H&S) of Tetra Tech employees is our number one priority. During this world-wide crisis, Tetra Tech has taken actions to inform and protect our employees at their local offices and field worksites. We have established a dedicated [COVID-19 Information and Guidance page](#) on My.TetraTech.com to provide the most recent company guidance and policies regarding our response to Coronavirus Disease 2019 (COVID-19).

COVID-19 is a respiratory illness that can spread from person-to-person. The virus that causes COVID-19 is a novel (newly discovered) coronavirus that was first identified during an investigation into an outbreak in Wuhan, China and has only been known to spread in people since December 2019. For the latest summary on the COVID-19, visit the [CDC Situation Summary page](#).

The purpose of this Tetra Tech EMI COVID-19 Response and Contingency Plan is to ensure that EMI employees are prepared to respond to a potential outbreak within our work environments. This situation is very fluid, and you are advised to stay updated by checking the [Coronavirus Disease CDC website](#) frequently. In addition, continually monitor the emails shared by your leadership and Safety Managers on this topic, as well as on the employee [COVID-19 Information and Guidance page](#) on the Tetra Tech intranet.

SYMPTOMS AND DISEASE TRANSMISSION

Person-to-person contact is the primary mode of transmission. Respiratory droplets from coughs and sneezes can infect others within close contact – about 6 feet. Touching contaminated surfaces then touching your own mouth, nose, or eyes is a possible route, but is not considered as significant as close contact with infected people; however, exposure pathways are still being studied by the Centers for Disease Control and Prevention (CDC). People are thought to be most contagious when they are most symptomatic (the sickest). Some exposure might be possible before people show symptoms (asymptomatic); there have been reports of this, but this is not thought to be the primary way the virus spreads. Monitor the CDC [How COVID-19 Spreads](#) site for up-to-date information on transmission.

The CDC believes the typical incubation period before symptoms appear is 2 to 14 days after infection. An analysis of publicly available data on infections estimated **5.1 days** for the median disease incubation period, according to a study led by Johns Hopkins Bloomberg School of Public Health. Symptoms include:

- Fever, usually over 100.4° F
- Cough, usually dry
- Shortness of breath

Check the CDC COVID-19 [Symptoms](#) page for updates.

TREATMENT AND PREVENTION

There is currently no FDA-approved medication or vaccine available for COVID-19. People infected with this virus should receive supportive care such as rest, fluids, and fever control, to help relieve symptoms. However, hospital care, including use of ventilators may be required for severe cases.

Steps to prevent the spread of COVID-19 are:

- Tetra Tech's corporate work-at-home policy has been revised to encourage all staff to work at home, whenever feasible, and in communication with project managers, their supervisor, and Operations Manager (OM), as appropriate.
- **Stay home** when you are sick.
- If you are sick, follow the CDC [Prevention Measures for Persons Under Investigation](#).
- Wash your hands often with soap and water for at least 15-20 seconds. If soap and water are not available, use a hand sanitizer with at least 60% alcohol.
- Avoid touching your eyes, nose, and mouth with unwashed hands.
- Avoid crowds and close contact (within 6 feet) with others who may be infected.
- Cover your cough or sneeze with a tissue, then throw the tissue in the trash.
- Standard household cleansers and wipes are effective in cleaning and disinfecting frequently touched objects and surfaces.
- As it is currently flu and respiratory disease season, CDC recommends getting vaccinated for flu, taking [everyday preventive actions](#) to stop the spread of germs, and taking flu antivirals if prescribed.

**TETRA TECH EMI
COVID-19 RESPONSE AND CONTINGENCY PLAN**

REGARDING DOMESTIC AND INTERNATIONAL BUSINESS TRAVEL

Travelers

All non-essential domestic travel is prohibited. All travel will be limited to essential matters only with appropriate approvals by the EMI President, Jeremy Travis. Specific requests for travel approval should be routed through the appropriate supervisor and OM. Questions regarding definitions of “non-essential” or “essential matters” shall be determined by your OM.

International travel to any countries identified by the CDC as either Level 2 or Level 3 is **currently prohibited**. For the most current list of Level 2 and Level 3 countries see: <https://wwwnc.cdc.gov/travel/notices>. Any international travel must be approved by the EMI President, Jeremy Travis, and completion of a hazard assessment by H&S on a case-by-case basis. Check this [site](#) to determine if your planned international travel may involve countries with travel restrictions **before** you travel.

Level 3 Countries: Warning	Level 2 Countries: Alert	Level 1 Countries: Watch
<i>Prohibited</i>	<i>Prohibited</i>	No non-essential travel

All non-essential travel has been cancelled. Essential travel requests must be approved by Jeremy Travis.

Essential travel approved by Jeremy Travis should be limited.

To protect yourself during approved, essential travel:

- Travel **MUST** be booked using the [Tetra Tech Travel Hub Dashboard](#).
- For international travel, have the [International SOS \(ISOS\) app](#) on your phone and check frequently for updates.
- Avoid contact with sick people.
- Avoid touching your eyes, nose, or mouth with unwashed hands.
- Discuss travel plans with CORE (855-683-9006), Tetra Tech’s occupational medical consultant, and your personal provider.
- Older adults and travelers with chronic medical conditions may be at risk for more severe disease.
- Clean your hands often by washing them with soap and water for at least 20 seconds or using an alcohol-based hand sanitizer that contains at least 60%-95% alcohol.
- Sanitizer wipes are recommended for air travel.
 - It is especially important to clean hands after going to the bathroom; before eating; and after coughing, sneezing, or blowing your nose.

If you have spent time in a Level 2 or 3 location during the past 14 days (for work OR personal reasons) and feel sick with fever, cough, or have difficulty breathing:

- **Do not come to work!** Avoid public places and public transportation. Notify your supervisor, Human Resources (HR) representative, and H&S representative of your health condition.
- Seek medical advice. Call ahead before you go to a doctor’s office or emergency room. Tell them about your recent travel and your symptoms.
- Use Tetra Tech’s [Teladoc service and app](#) or similar telemedicine services to consult with physicians.
- Avoid contact with others.
- **Do not travel while sick.**

If you have spent time in a Level 2 or 3 location during the past 14 days (for work OR personal reasons) and are asymptomatic:

- **Do not come to work!** Avoid public places and public transportation. Notify your supervisor, HR, and H&S representatives of your health condition.
- Be sure you have your laptop and charger with you to facilitate working from home if necessary.
- Continue communicating with your supervisor on your status.
- After completing the self-quarantine 14-day period and if you do not exhibit any signs or symptoms mentioned above, you may be allowed to return to work.

TETRA TECH EMI COVID-19 RESPONSE AND CONTINGENCY PLAN

PREVENTING OUTBREAKS IN THE WORKPLACE

The Tetra Tech Safe Work Practice, *Infectious Disease Guidance (SWP 5-55)*, provides guidance to identify risk management techniques to protect employees who may be at increased risk of infection, address related complications, and maintain business operations. Tetra Tech has additionally eliminated in-person meetings of over 10 persons and is using “virtual meetings” whenever possible.

OMs are encouraged to work with their building management to ensure an appropriate cleaning schedule. Work surfaces should be regularly cleaned to maintain good housekeeping in the work environment. Clean surfaces that are touched by the hands or face diligently; such as, but not limited to: doorknobs, light switches, elevator buttons, remote controls, handrails, computer keyboards, mice, telephones, microphones, tables and chairs, coffeemakers, vending machines, etc.

If building management is non-responsive to our cleaning requests, OMs are encouraged to implement regular cleaning schedules of office space and restrooms using outside contracted janitorial personnel.

OMs should procure facial tissue, hand sanitizer (greater or equal to 60% alcohol), and disposable disinfectant wipes for employees to facilitate self-cleaning of frequent hand-contact surfaces (e.g., doorknobs, light switches, computer keyboards, telephones, vending machines). Employees or designated persons should inspect common areas and frequent hand-contact surfaces for cleanliness. If necessary, clean these areas with available disinfectant wipes. OMs should encourage personnel to clean their own workstation surfaces with available disposable disinfectant wipes.

Common areas should be regularly checked to ensure dishwashing detergent, sponges, and cleaning cloths are available and replaced as necessary.

Posters communicating COVID-19 prevention strategies shall also be posted in common areas throughout all office locations, including satellite offices and field sites with office trailers or facilities. Web resources for these posters can be found here:

- CDC Print Resources: <https://www.cdc.gov/coronavirus/2019-ncov/communication/factsheets.html>,
- ISOS Education and Communication: <https://pandemic.internationalsos.com/2019-ncov/ncov-education-and-communication>

Recommendations for EMI worksites include:

- Consider Skype meetings, use of Microsoft Teams, or SharePoint sites as opposed to meetings.
- Tetra Tech personnel should perform self-evaluations each day PRIOR to work. If any new symptoms or if any potential exposures have occurred, the employee should STAY HOME or at the hotel.
- Field workers should consider texting or emailing daily communications, such as safety briefings, to increase social distancing.
- Maintain soap and water, alcohol-based hand sanitizer (ABHS), AND sanitizing wipes in the vehicle.
- Do not shake hands. Maintain social distancing from everyone, including clients and your coworkers.
- Ensure workspaces are cleaned frequently.
- Ensure all staff members are provided information on disease transmission, symptoms, and prevention as discussed above.
- Supervisors shall work with OMs to determine the applicable actions regarding work arrangements other than normal work environments. This includes employees who may need to be home to care for children or other family members who are sick or affected by institutional closures.
- Managers and supervisors should be flexible with work at home assignments.
- All employees are responsible for notifying their supervisors or project/program managers if project work will be affected during absence.

TETRA TECH EMI COVID-19 RESPONSE AND CONTINGENCY PLAN

REPORTING AND MONITORING SUSPECTED CASES

Employees who become ill should report their illness to their Project Manager, OM, and H&S immediately. All employees absent from work three or more days because of their health, or to care for a family member, should report the absence to HR and may be eligible for [Family Medical Leave Act](#). Contact your personal physician and consider using Tetra Tech's [Teladoc service and app](#) or similar telemedicine services. See the [COVID-19 General Guidelines for Response flowchart](#) at the end of this plan.

OMs should verify that more than one method of communicating with staff is available. Please ensure all telephone numbers / email distribution lists are up to date.

If your risk profile includes recent foreign travel, close contact with infected individuals, or a household member diagnosed with COVID-19, or you experience symptoms:

- Isolate – if at home, stay at home. If at the office, go home immediately and notify your supervisor of your health condition. If you are on business travel, isolate in the hotel and contact CORE and your supervisor immediately for guidance. Continue isolation until cleared by your physician or state or local health department.
- Seek medical attention as described above.
- Report to your supervisor, HR, and H&S representative. Report confirmed COVID-19 cases to help us track and monitor for possible workplace outbreaks. A suspected/confirmed case register will be maintained. The case register will include employee's name, dependent name, if applicable, current location, contact information, and emergency contact information. Actions outlined below may be necessary. CORE Occupational Medicine will be contacted to verify test results performed by an employee's physician or state or local health department. ***All personally identifiable information must be kept confidential.***
- Notify HR if an employee requests to self-quarantine because they have reason to believe that reporting to work would pose an imminent or serious danger to themselves or others.

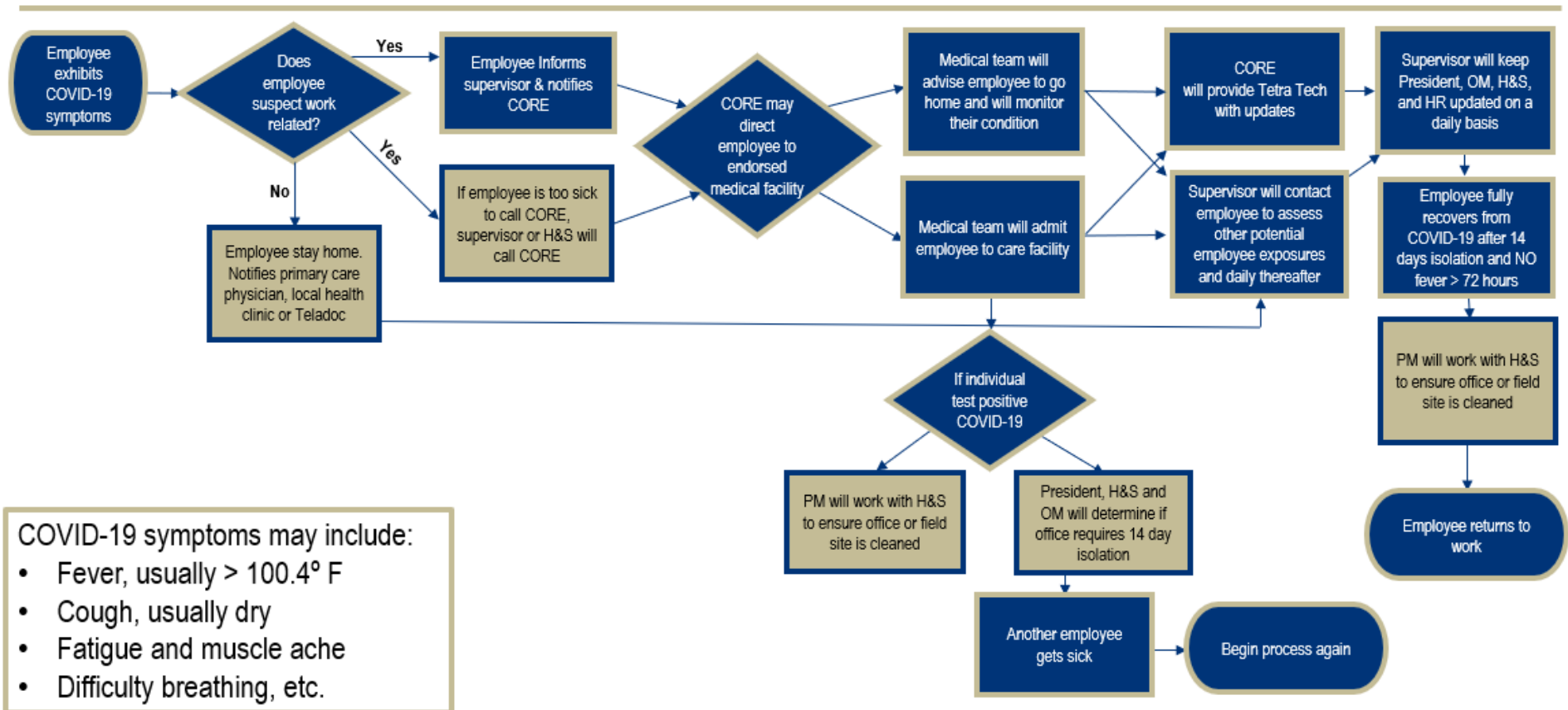
Response to Possible Outbreak

In the event of a confirmed case of COVID-19 in the workplace:

- OM and H&S representative will provide for guidance on cleaning procedures for a confirmed employee's work environment, including offices, field worksites, and hotel rooms.
- OM, H&S, and HR will coordinate notice to staff of the confirmed case and possible exposure to the virus, without revealing the individual's identity.
- Ask all employees to remain vigilant and immediately isolate and report any symptoms.
- The OM for the office location will notify the building landlord.
- Field team leaders will notify the client and any others that have been in contact with a potentially infected employee.
- Notify Jeremy Travis. Executive leadership will determine appropriate contingency plan for the specific location with local OMs. This may include shutting down the site or office to minimize the spread of COVID-19.
- H&S representative will notify the hotel and any local or state public health agencies and complete reporting requirements, if any.
- Executive leadership and H&S will coordinate care for employees quarantined in hotels or areas apart from their families as necessary.

If a worksite or office is closed, EMI leadership will continue to monitor and communicate with affected work site or office leadership during closure.

COVID-19 General Guidelines for Response



CORE: 1-855-683-9006

H&S: Chris Draper – 615-969-1334; Denny Cox – 816-668-7464; or Dave Brown – 619-446-7261

HR: Shannon Stuver – 541-482-8938 or Diane Stopa – 703-885-5518

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	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 1 of 9

INTRODUCTION

Tetra Tech recognizes the need to prepare for and minimize the impact of either a localized outbreak of serious infectious disease, pandemic disease events or other events that may present a health risk to employees. The objective of this guidance is to identify risk management techniques and coordinate response, protect employees who are at increased risk of infection, address related complications, and maintain business operations.

Given the diversity in the size and nature of Tetra Tech operations, appropriate responses to these health events will depend on several key indicators such as:

- Disease severity in general and high risk populations;
- Extent of disease at the location;
- Amount of worker absenteeism; and
- Other factors that may affect an employee's ability to get to work (restrictions on travel, school closures, care for sick family members, conflicts, etc.).

Tetra Tech offices and project locations are encouraged to take appropriate actions based on conditions at each location.

In the event the severity of a pandemic event increases and key business operations are impacted, Tetra Tech may elect to activate its Business Continuity Plan (BCP) to maintain enterprise essential business functions. The decision to activate the BCP will be at the discretion of Tetra Tech's executive management.

This guidance outlines measures to identify risk in the workplace, appropriate work practice control measures, work policies, continuity of business operations, and communication methods. **While these general guidelines have been established, Tetra Tech may modify this guidance as needed based on current recommendations from public health authorities, Tetra Tech clients or specific business needs.**

RESPONSIBILITIES

Executive Management

Tetra Tech Management has the overall responsibility for effective and appropriate response to pandemic or disease outbreak events, including assuring that necessary resources are provided and that line managers and employees are held accountable for their responsibilities under this guidance.

	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 2 of 9

Line Management (Chief of Party, Program Managers)

Line Management is responsible to evaluate the current situation based on their detailed knowledge of the project, location and available resources.

Line Management is responsible for ensuring that all project personnel are aware of and abide by company and project specific guidelines.

Line Managers must also be familiar with signs and symptoms of disease infection and ensure that the appropriate work practices and guidelines have been addressed for operations and tasks conducted by the employees they manage.

Health and Safety

Health and Safety personnel are responsible to provide overall direction for the health related components of this guidance at individual operating units. They will assure response effectiveness and act as a resource regarding health guidelines. Health and Safety may also consult with Tetra Tech's Medical Director or other medical resources regarding medical issues as appropriate.

Human Resources

Human Resource personnel will be responsible to provide direction for workplace policies related to this guidance at individual operating units. They will also assure response effectiveness and act as a resource regarding these issues.

Employees

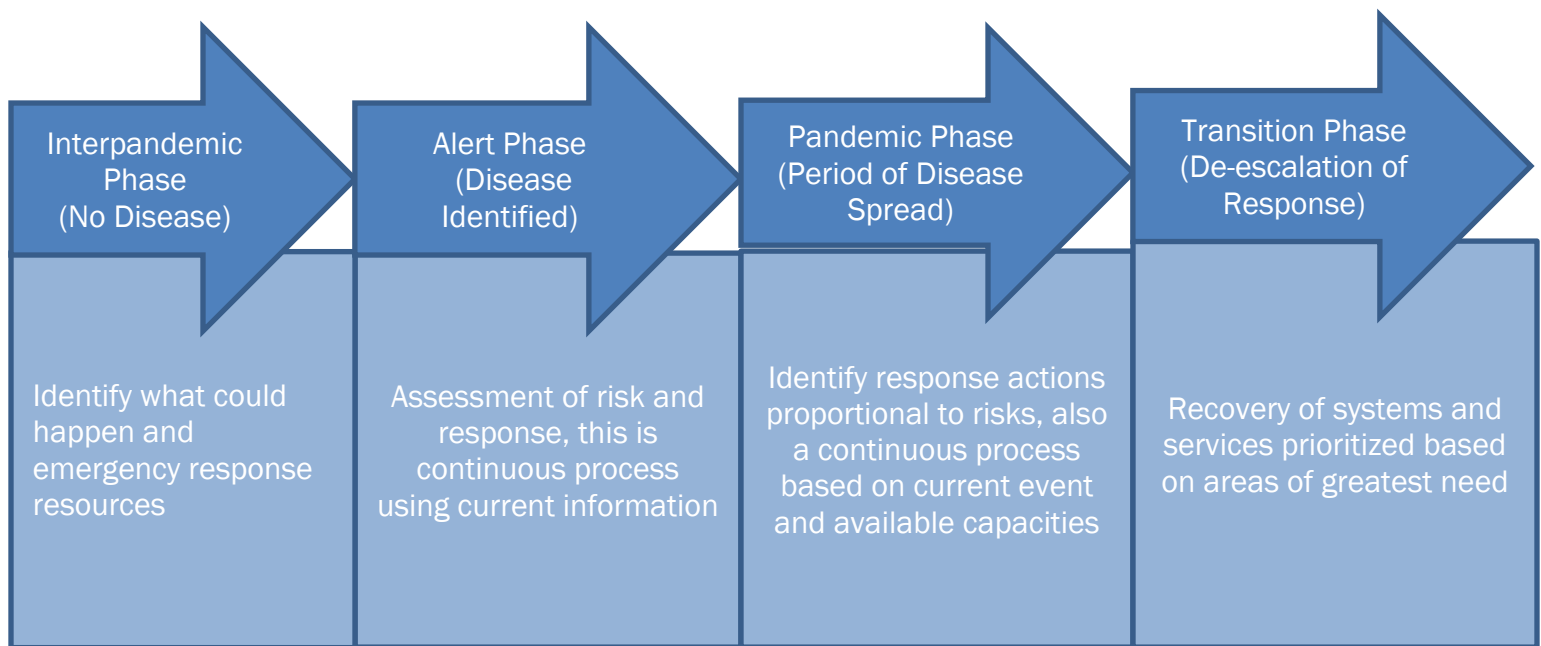
Employees are responsible for performing their job duties in a manner that is compliant with guidance established. During infectious disease events, employees are encouraged to report relevant health symptoms to either their appropriate line manager or, if they prefer, to their Human Resources or Health and Safety contacts so that proper control methods can be implemented.

RISK ASSESSMENT

The World Health Organization (WHO) has developed an interim guidance document that addresses the management of pandemic influenza events. As part of this guidance, WHO has identified pandemic phases that identify the continuum of pandemic disease in the context of preparedness, response and recovery. This guidance will be used to frame the company's risk based response to these types of events.

	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 3 of 9

The following figure identifies broad categories of risk assessment actions addressed at each phase:





**TETRA TECH, INC.
SAFE WORK PRACTICE
INFECTIOUS DISEASE GUIDANCE**

Revision Date: 07/30/2014

Document Control Number:

SWP 5-55

Page 4 of 9

The US Centers for Disease Control (CDC) has adopted a classification system to address international travel when impacted by global health events. This system identifies levels of risk for the traveler and recommended preventive measures to take at each level. Established levels, definitions and with specific examples are listed below. Tetra Tech will rely on both the WHO and CDC guidance when responding to global health events.

Notice Level	Traveler Action	Risk to Traveler	Outbreak/Event Example
Level 1: Watch	Reminder to follow usual precautions for this destination	Usual baseline risk or slightly above baseline risk for destination and limited impact to the traveler	Dengue in Panama-Outbreak Watch: Because dengue is endemic to Panama, this notice most likely would signify that there is a slightly higher rate of dengue cases than predicted. Travelers are to follow “usual” insect precautions. Olympics in London-Event Watch: There may be possible health conditions in London that could impact travelers during the Olympics, such as measles. Travelers are to follow usual health precautions making sure they are up to date on their measles vaccine, follow traffic safety laws and use sunscreen
Level 2: Alert	Follow enhanced precautions for this destination	Increased risk in defined settings or associated with specific risk factors	Yellow Fever in Brazil-Outbreak Alert: Because an outbreak of yellow fever was found in areas of Brazil outside of the reported yellow fever risk areas, this would be a change in “usual” precautions. Travelers should follow “enhanced precautions” for that risk area by receiving the yellow fever vaccine.
Level 3:	Avoid all non-	High risk to	SARS in Asia-Outbreak Warning:



**TETRA TECH, INC.
SAFE WORK PRACTICE
INFECTIOUS DISEASE GUIDANCE**

Revision Date: 07/30/2014

Document Control Number:

SWP 5-55

Page 5 of 9

Notice Level	Traveler Action	Risk to Traveler	Outbreak/Event Example
Warning	essential travel to this destination	travelers	Because SARS spread quickly and had a high case fatality rate; a warning notice signifies there was a high chance a traveler could be infected. Travelers should not travel if possible. Earthquake in Haiti-Event Warning: The destination's infrastructure (sanitation, transportation, etc.) cannot support travelers at this time.

Tetra Tech will also refer to US OSHA established various risk levels to address occupational exposure to infectious disease during a pandemic or disease outbreak event. These risk levels are based on the whether job assignments require close proximity to people potentially infected and whether they are required to have repeated or extended contact with known or suspected sources such as coworkers, the general public, outpatients, school children or other such individuals.

Typical work tasks conducted by Tetra Tech personnel are considered office employees with minimal occupational contact with the general public and other coworkers and present a low risk of exposure. The majority of Tetra Tech employees fall under this risk category. The intent and scope of this plan addresses this target population and associated risk level. Control measures for employees supporting contracts where the risk of exposure may be classified at higher designated levels will be evaluated and addressed on a case by case basis.

In these cases, Tetra Tech's Medical Director or other medical resources will be consulted to provide additional prevention measures that may include medical screening including the use of antiviral agents for prophylaxis or treatment of infection if available.

	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 6 of 9

WORK PRACTICE CONTROLS

Work practice controls are procedures that will reduce the duration, frequency or intensity of exposure. The following work practice controls shall be implemented at Tetra Tech work locations during pandemic flu or other infectious disease events:

- Provide resources to promote good personal hygiene. This includes tissues, hand soap, hand sanitizers, surgical masks, disinfectants and disposable towels so that employees can clean work surfaces.
- Communicate risk factors, signs and symptoms of illness and proper infection control behavior. Information specific to current health events will be developed and distributed to affected employees as needed.
- Employees with signs and symptoms of disease infection should remain at home until at least 24 hours after they are free of fever (100°F or greater) without the use of fever reducing medications.
- Employees are encouraged to report signs and symptoms of infection to either their immediate supervisor or Human Resources or Health and Safety personnel.
- Sick employees may be asked to go home. Employees who appear to have symptoms upon arrival or become ill during the day should be promptly separated from other workers and advised to go home. When possible and if tolerated, employees with illness symptoms should be given a surgical mask to wear before they go home if they cannot be placed in an area away from others.

	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 7 of 9

Employees exposed to a sick co-worker or who care for sick family members can report to work. However these employees should monitor their health every day. Before coming to work, employees should ask themselves:

- Do I have a fever?
- Do I have a sore throat?
- Am I coughing?
- Do my muscles ache?
- Do I feel ill?

If yes is answered to any of the above, employees should stay at home, notify their supervisor and seek medical guidance.

Employees who become ill and are at increased risk of complications from infectious diseases should call their health care provider for medical advice.

Encourage vaccinations if they are available.

In the event of health events with severe outcomes, Tetra Tech may elect to activate additional work practice control measures such as:

- Proactive screening of employee's health;
- Increase the number of days an employee may be required to stay at home when ill;
- Apply social distancing measures;
- Consider alternative work environments for employees at higher risk for complications of infection;
- Require travel approval to areas of high risk; and
- Restrict employee business travel to affected areas.

HUMAN RESOURCES POLICIES AND PROCEDURES

Impacted operating units shall maintain a current roster of affected employees, dependent names if applicable, current location, contact information and emergency contact information.

Notifications of potential exposure events will be sent by Human Resources to all affected employees when probable exposure events occur. At all times the confidentiality of the ill employee will be protected to the degree practical.

	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 8 of 9

Tetra Tech's standard sick leave and disability policies will apply in these events. Tetra Tech reserves the right to modify these policies as necessary to be consistent with public health guidance. As an example, a doctor's note may not be required to return to work as doctor's offices and medical facilities may be overcrowded. Human Resources is responsible for identifying legally mandated actions that are required in regard to regulations that may apply to the general workforce, US examples - the Family and Medical Leave Act, the Americans with Disabilities Act, etc.

The Tetra Tech Employee Assistance Program is available to all benefits eligible personnel. Human Resources will encourage employees to utilize these services to manage additional stressors related to the pandemic or other similar events. These are likely to include distress related to personal and family illness, life disruption, loss of routine support systems and similar challenges.

CONTINUITY OF BUSINESS OPERATIONS

Managers responsible for an office or project should plan for continuity of operations if there is significant absenteeism from sick workers. Contingency plans must be put in place to ensure that client-related work and deliverables are not impacted by employee absenteeism. Plans must be developed to notify key contacts including both customers and suppliers in the event an outbreak has impacted the company's ability to perform contracted services. All employees are responsible for notifying their immediate supervisor or office manager if project work will be affected during their absence. These plans may include:

- Identify essential business functions;
- Cross train employees in essential business functions;
- Establish flexible worksites and work hours, telecommuting, staggered shifts;
- Enhance where possible communications and IT technology as needed to support employee telecommuting;
- Identify sources of replacement employees; and
- Identify critical elements within supply chains as applicable.

In the event the severity of a health event escalates and key business operations are impacted, Tetra Tech may elect to activate its Business Continuity Plan (BCP) to maintain enterprise essential business functions. The decision to activate the BCP will be at the discretion of Tetra Tech's executive management. Tetra Tech's BCP is reviewed with key personnel and includes periodic testing of emergency communications procedures during table-top exercises.

	TETRA TECH, INC. SAFE WORK PRACTICE INFECTIOUS DISEASE GUIDANCE	Revision Date: 07/30/2014
		Document Control Number:
		SWP 5-55
		Page 9 of 9

COMMUNICATION METHODS

Tetra Tech has established several methods of communication to ensure that timely information is received and communicated as appropriate.

Tetra Tech has partnered with several resources such as International SOS to provide real time medical updates and alerts. Employees can elect to directly receive these alerts via their email address. The International Assistance wallet card lists the contact information needed to access these resources.

Up to date disease guidance and illness information and training material are available on the ISOS website and can be accessed using the Tetra Tech member number 11BCMA000238. Depending on current events and circumstances, information may also be posted on the My.TetraTech main landing page or included in the Health and Safety portion of the site.

For US based employees, Tetra Tech has partnered with the National Safety Council and participates in a real time health alert system that is directly linked to the US Centers for Disease Control. These alerts are distributed as applicable to H&S staff for publication or response.

Tetra Tech also relies on our medical surveillance provider to provide periodic updates and medical guidance on specific health care issues.

Employees will be provided information regarding the relevant components of this guidance, as well as local instructions through various methods such as safety meetings, newsletters, posters, and employee training, etc. Information and training will include illness prevention topics, how to avoid the spread of disease, and company policies concerning illness.

Email communication is the most direct method to reach the majority of Tetra Tech employees and will be utilized in the event critical information must be distributed. Tetra Tech has the ability to send All Tetra Tech or all unit email notifications. Tetra Tech also has the ability to send SMS text messages to traveling employees that may be at risk. Line managers are responsible for having alternative means of communications available to them in order to communicate with employees who do not readily have access to these systems.

ATTACHMENT C

APPL STANDARD OPERATION PROCEDURE



Standard Operating Procedure

Incremental Sampling (IS) Techniques for Explosives with Mechanical Shaker Extraction and (IS) Techniques for Other Target Analytes EPA METHOD 8330B, Appendix A

STATEMENT OF PURPOSE

The purpose of this SOP is to describe the procedure for the extraction of solid samples that are to be analyzed for explosives by EPA 8330B or other target analytes using DoD QSM guidelines.

INSTRUCTIONS

1.0 Scope and Application

This SOP applies to all personnel involved in the mechanical shaker extraction of explosives in solid samples using IS procedures from EPA method 8330B, Appendix A, IS (Incremental Sampling), as well as QSM v5.3 (or later) Table B-3 and Table B-23.

2.0 Method Summary

- 2.1 Soil samples for explosives analysis are dried to a constant weight, sieved, ground using a mechanical grinder (for firing ranges) or mortar and pestle (for ammunitions depots), incrementally sampled and extracted by mechanical shaker with Acetonitrile solvent.
 - 2.1.1 The client should advise whether mechanical grinding is required for the project, depending on the type of site.
 - 2.1.2 The samples are prepared for injection on HPLC instrumentation by filtering through an Acrodisc PSF GHP 0.2 um. They may also be centrifuged if necessary.
- 2.2 Soil samples for analytes other than explosives may be incrementally sampled either dry or wet, depending on the client's project requirements.
 - 2.2.1 The samples are prepared by the designated digestion or extraction method, depending on the target analyte.

3.0 Detection Limits - NA

4.0 Definitions

- 4.1 Batch - Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)
- 4.2 Calibration standard - A solution prepared from the primary dilution standard solution or stock standard solution and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration.



- 4.3 Field Reagent Blank - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.
- 4.4 Instrument Performance Check (IPC) - A solution of one or more compounds (analytes, surrogate, internal standard, or other test compounds) used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
- 4.5 Laboratory control spike (LCS) - An aliquot of reagent water or other matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 4.6 Laboratory Reagent Blank - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 4.7 Limit of Detection (LOD) - An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent. The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%).
- 4.8 Limit of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence. The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. This also equates with the term Practical Quantitation Limit (PQL).
- 4.9 Matrix - A surrounding substance within which something originates, develops, or is contained, such as: drinking water, saline/estuarine water, aqueous substance other than drinking water or saline/estuarine water, non-aqueous liquid, biological tissue, solids, soils, chemical waste, and air.
- 4.10 Matrix duplicate (DUP) - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a matrix sample and matrix sample duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 4.11 Matrix spike (MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the matrix spike corrected for background concentrations.
- 4.12 Matrix spike duplicate (MSD) - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a matrix spike and matrix spike duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.



- 4.13 Method blank - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 4.14 Method detection limit (MDL) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero, as determined from analysis of a sample containing the analyte in a given matrix, as described in 40 CFR Part 136, Appendix B, 1 July 1995 edition.
- 4.15 Practical quantitation limit (PQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The practical quantitation limit is generally three to ten times greater than the method detection limit.
- 4.16 Primary Dilution Standard - A solution of several analytes prepared in the laboratory from stock solution and diluted as needed to prepare calibrations solutions and other needed analyte solutions.
- 4.17 Quality Control Sample (QCS) - A solution of method analytes of known concentrations which is used to fortify an aliquot of LCS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.
- 4.18 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials purchased from a reputable commercial source.

5.0 Interferences and Potential Problems

Special care must be taken in the handling of explosive samples. DO NOT CONCENTRATE the final extract using a heated water bath, due to the unstable nature of explosive compounds. THEY MAY DETONATE! For safety reasons, sample extracts must be stored in a refrigerator until instrument analysis. If extracts require concentration, a six-port nitrogen gas manifold may be used for gentle solvent blowdown.

6.0 Health and Safety

DO NOT CONCENTRATE the final extract using a heated water bath, due to the unstable nature of explosive compounds. THEY MAY DETONATE! For safety reasons, sample extracts must be stored in a refrigerator until instrument analysis. If extracts require concentration, a six-port nitrogen gas manifold may be used for gentle solvent blowdown.

7.0 Sample Preservation, Containers, Handling and Storage

A pre-cleaned plastic bag with zipper seal (for incremental sampling) may be used as the sample container for explosive analysis. Sample containers must be stored at or below 4° and handled carefully, due to the potential for detonation at high sample concentrations. If samples do not arrive to the laboratory chilled on ice, then it is not necessary to store them in the refrigerator. In such cases the client has determined it more beneficial to keep the sample at ambient temperatures to reduce condensation issues within the sample bag.



8.0 Quality Control

- 8.1 One method blank, one lab control spike and a set of matrix spikes must accompany each set of twenty samples (or fewer). For DoD projects, an LCSD is required, if there is insufficient volume for MS/MSD. When mechanical grinding is employed, the method blank should be ground after at least one sample has been ground.
- 8.2 Puck Mill grinder verification: Use a mechanical Puck mill grinder to grind 500g sand to particle size <75um in diameter and passing the ground sand through a #60, 200 mesh sieve.) Document in the mechanical grinder logbook.

9.0 Equipment/Apparatus

- 9.1 Mechanical extraction shaker
- 9.2 2.5oz jars
- 9.3 8mL screw-cap vials
- 9.4 2mL injection vials
- 9.5 Laboratory Centrifuge
- 9.6 Mechanical ring-puck grinding mill
- 9.7 1000 gram grinder bowls with puck and lid
- 9.8 Soil Sieves (brass) – 2mm (#10 mesh)
- 9.9 Soil drying racks and trays
- 9.10 20mL volumetric pipettes
- 9.11 Silica sand
- 9.12 Variable-volume pipettors with tips
- 9.13 Laboratory Analytical Balance
- 9.14 Laboratory Balance (capacity ~ 15kg)
- 9.15 Laboratory Hood
- 9.16 Hepa-filter Vacuum
- 9.17 Flat/Spoon Stainless Steel spatulas
- 9.18 Ceramic mortar and pestles
- 9.19 Rotary Splitter

10.0 Reagents and Standards

Reagent grade chemicals are used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the analysis. All reagents used are traceable at all steps of the procedure. Reference standards must be calibrated by a body that can provide ILAC-signatory (MRA) traceability.

- 10.1 Acetonitrile Burdick and Jackson HPLC Grade Cat# AH015-4.
- 10.2 Millipore Water from the Laboratory Source.

11.0 Calibration and Standardization – NA

12.0 Procedure

Sample Drying to a Constant Weight:

- 12.1 Place the entire contents of the client's plastic bag sample container onto a baker's tray labeled with the sample number and lined with aluminum foil.
- 12.2 If the client requires "wet" Incremental Sampling (prior to sample drying) for such analyses as metals (particularly mercury), PAH, fuels, or SVOCs, then a portion of the sample may be removed through incremental sampling techniques listed below in



- section 13.0, **before** the drying process has begun, and **after** the sample has been weighed. Consult the ARF comments and the project manager on special client ISM requests.
- 12.3 This step is not necessary if the client sends multiple containers for “wet” and “dry” IS techniques. If only one container is sent, then thoroughly homogenize the wet soil with a gloved hand prior to removing a portion before drying. The remaining soil for 8330B explosives or other target analytes “dry” IS techniques may then be placed on the drying tray.
- 12.4 If the client requires Metals or SVOC analysis and does not want the sample to be ground prior to IS, then place a colored sticker containing the letters “NG” (for no grind) on the tray to indicate that the sample should not be ground (due to the contribution of metals from the grinder bowl and potential breakdown of SVOCs during grinding).
- 12.4.1.1 The proper sticker placement on the tray will be verified by a supervisor or his/her designee who dates and initials the “I.S. Preparation Log Book”. Verification will be based on the Extraction Backlog and whether or not IS Labworks codes are listed. It is also a good idea to verify the requirements for a particular project with the project manager prior to drying samples.
- 12.5 Use a gloved hand to break apart large soil agglomerates. Turn the plastic bag inside out and place on top of the tray to dry along with the sample.
- 12.6 Record the date / time and the weight of the tray plus sample in the “I.S. Preparation Log Book”. Dry the sample trays using a baker’s stackable rack at room temperature to a “constant weight” as described below: The room temperature should not exceed 22°C +/- 6°C. The daily room temperature is electronically recorded. If temperature criteria are exceeded, then the client should be notified in order to decide whether new aliquots of samples must be sent to the laboratory from the client.
- 12.7 The following morning visually examine the soil. If the soil appears to contain moisture then break up any soil clumps with a gloved hand and visually examine the soil again later. If not then weigh the tray containing the sample and record the weight, date and time in the “I.S. Prep Log”, and place the trays back in the rack. After approximately one to three hours reweigh the tray and record the weight, date and time again.
- 12.8 If the weight is consistent with the previous weighing (within +/- 3%), then this step is complete. The technician should visually inspect the soil for dryness by using a gloved hand to break apart some of the soil clumps, making sure the soil within is dry and that the soil at the bottom of the tray is dry. Extra care must be taken for clay-like samples that tend to retain moisture by breaking up the soil clumps to ensure complete dryness. If the weight is still not constant, then place the sample trays back in the rack for additional drying and subsequent weighing until a constant weight is achieved before proceeding to the next step. Soil with residual moisture will not grind sufficiently, and soil clumping may be observed.
- 12.9 A tray containing blank commercial ground silica sand is placed on the baker’s rack at all times while samples are being dried. This sand is to be used for method blanks (and LCS’s) and will help monitor the presence of potential laboratory contaminants during the drying step. Since the sand is commercially ground it is not passed through the sieve.
- NOTE: Explosive materials may appear as gray lumps in the soil sample. TNT-based explosives that have been exposed to sunlight may appear as reddish-orange lumps. The soil should be examined for such materials, since mechanical



grinding is not recommended for pure explosive pieces, as they may detonate. Remove these particles and place in a Ziplock bag labeled with the sample ID and the following statement - "Hazardous-Potential Explosive"

Sample Sieving and Grinding:

- 12.10 Pass the entire dried sample through a metal 2mm (#10 mesh) screen sieve to eliminate coarse rocks, sticks and leaves. Fine vegetation (such as mosses or grass) should be physically shredded during sieving by applying pressure with a gloved hand to press the fine vegetation through the screen. (Some clients may request gross vegetation to be removed prior to sieving.) Do not intentionally include vegetation in the portion of the sample that passes through the sieve, unless required by a specific project.
- 12.11 Examine the "non-passing" portion of the sample. Hard dirt clods should be manually ground using a ceramic mortar and pestle, in order to insure passage through the sieve. Repeat sieving and manual grinding of the non-passing portion until only material that cannot be manually ground remains (such as rocks or metal fragments).
- 12.12 Place the non-passing sample portion onto a clean tared zippered plastic bag on top of a balance. Record the passing and non-passing weights in IS log book. If the client requests, take a picture of the pass and non-passing together on the original tray showing the sample number sticker.
- 12.13 Reserve the passing portion on the original tray for mechanical grinding if requested by the client, or for further sieving if requested by the client (such as a smaller #60-250um sieve). Consult the ARF comments and the project manager on special client ISM requests.
- 12.14 Wipe down the tabletop with soap and water after processing samples. Wash the sieve in between each sample with soap and water and rinse with acetone. Allow the sieves to dry thoroughly, especially at the seal between the screen and the metal cylinder.
- 12.15 For sample analyses that do not require mechanical grinding, such as metals, PAH, SVOCs, perchlorate etc., proceed to the Incremental Sampling section of this SOP. For samples that do require mechanical grinding, proceed to the next step.
- 12.16 Prior to grinding, use the last column in the "I.S. Prep Log Book" to document, date and initial that the samples have been incrementally sampled for metals and other analyses that do not require grinding.
- 12.17 Transfer the sieved soil portion into a metal grinder bowl. Place the puck on top of the soil and place the lid on top of the bowl. Use a mechanical Puck mill grinder to grind the entire sample to particle size <75um in diameter. (As was initially demonstrated by passing approximately 500g of ground sand through a #60, 200mesh sieve.) Depending on the amount of sample, several grindings may be required, since the grinding bowl may only hold up to 1000g at a time.
- 12.18 Use a single 90second grind cycle to pulverize the sample, unless the client requests a special 60 second grind cycle that is repeated five times (with a 2 min cool down period between grinds).
- 12.19 If the sample has been split into two or more bowls, then combine the portions back together into a labeled clean zippered plastic bag and thoroughly mix before incremental sampling. Place the contents of the bag back onto the tray before incremental sampling. **If the client requests**, take a digital photo of the ground sample and save to the "IS Photos" subdirectory sorted by ARF number and located on the shared documents T:\ drive.



- 12.20 In the mechanical grinder log book, record the sample ID, date, analyst initials, grind cycle time and serial number of the grinder bowl used to process each sample.
- 12.21 The lab technician should wear a lab coat, gloves and safety goggles for the grinding process. Due to the potential lead particles from ballistic material in the samples, the handling of finely ground soil samples exposed to open air should be done in the fume hood as much as possible (i.e.: transferring samples from bowl to tray, cleaning the bowls and incremental sampling).
- 12.22 Thoroughly clean the grinder bowl, puck and lid in between each sample with warm soapy water and rinse with clean water and then acetone. The Teflon lid seal should be removed with a small spatula and cleaned separately.
- 12.23 Grind clean commercial silica sand for the method blank and lab control spike with each batch of 20 field samples. The blank should be ground after at least one field sample has been ground.
- 12.24 The DoD QSM requires a composite grinder blank, in which silica sand can be ground in between each sample for version 4.2 and once after every 10 samples and at the end of the batch for version 5.0 or later. Each grinder blank can be analyzed individually or composited, and will be analyzed according to SOP HPL8330B. The method blank will consist of a composite sample from all the grinder blanks performed for a given extraction batch. The analyst should change gloves in between processing each sample, in order to reduce cross contamination. The technician should ask the project manager regarding the grinder blank requirement for each particular project.
- 12.25 The DoD QSM requires a pre-grind LCS using a SRM (solid reference material) or (PE sample) to be analyzed with the extraction batch, in addition to the post-grind routine LCS. The PT sample is ground with each analytical batch of 20 or less field samples. The PT sample is prepared in exactly the same manner as a field sample (e.g. sieved, ground and sub-sampled). The technician should ask the project manager regarding the PT sample requirement for each particular project, and refer to the ARF comments for project specific PE-LCS requirements.
- 12.25.1 When a new SRM (PE LCS) is received by the lab, the QAU will have a lot check ARF generated, in order to verify the un-ground concentration prior to use with a 10 gram aliquot of the un-ground sand. Tetryl tends to degrade faster than the other target analytes, and is purchased in a separate SRM. Once the new lot has been verified, it is released for use.
- 12.25.2 A 100 gram aliquot is mechanically ground with each ISM extraction batch. The ground aliquot may be incrementally sampled multiple times, as long as the percent recoveries continue to meet acceptance criteria. The 100g ground aliquot should be stored in a ziploc bag and labeled with the date of grinding and supplier's lot number.

Incremental Sampling (IS)

- 13.0** For Dried Incremental sampling, spread the dried, pulverized sample (from the grinder bowl or ceramic mortar, depending on the client's requirements) onto the original foil (or plastic bag)-lined drying tray to a depth of approximately 1-2 cm. For samples where multiple bowls are used to grind a sample, thoroughly mix the portions from each bowl on the tray before incrementally sampling. Use the flat end of a flat/spoon stainless steel spatula to scoop appropriate amount of soil from 30 random areas to reach the total needed for the analyses to be performed. The spatula is used to draw a 6 rows x 5 columns into the soil, which creates a two-dimensional grid of 30 squares. The subsample amounts are taken from within each of the 30 grid squares. For example, for the method 8270B take approximately



- 1g of soil from random locations inside each of the 30 grid squares throughout the tray. Place it into a tared jar for a total weight of at least 30g. If the sample weight is above the amount needed, do not remove soil from the jar and put it back onto the tray. Record the exact weight to the nearest 0.01 grams on the extraction sheet. Continue this process for each analysis needed.
- 13.1 Matrix spike / Matrix Spike Duplicate and Sample Triplicate analysis are required by the DoD for each analytical batch. The technician should ask the project manager regarding the MS/MSD and Triplicate analysis for each particular project.
- 13.1.1 After the sample has been dried, sieved and ground, perform the necessary MS/MSD and Triplicate incremental sampling separately into their own labeled jars. Do NOT further homogenize the soil in between replicate incremental samplings, as this may cause the large heavier soil particles to sink to the bottom of the tray.
- 13.1.2 For samples requiring MS/MSD, perform the 30 increment MIS procedures once for the parent sample and then two subsequent times for the MS and the MSD, for a total of 3 jars to be extracted.
- 13.1.3 For samples requiring Triplicate analysis, perform the 30 increment MIS procedures for the designated sample three times for a total of 3 jars to be extracted.
- 13.2 For samples needing IS without mechanical grinding (such as SVOCs or Metals), spread the sieved "passing" portion of the sample on a clean labeled tray and perform the IS procedures listed above. For metals samples, IS at least 2.5 grams of sample. For SVOC samples, IS at least 30 grams of sample. Sample triplicates and matrix spikes are IS'd in separate jars.
- 13.3 After subsampling is complete, the remaining soil from the tray may be placed back into the original client's plastic bag sample container and stored at the temperature appropriate for the analyses required, along with the "non-passing" portion of the soil. When IS is complete the sample is checked back into the COC database into an IS box# and put in receiving. After samples have been IS'd, the metals and extraction department will initial the IS log book. The EPA 8330B method does not require the dried, pulverized portion of the soil to be stored under refrigeration. Clean the fume hood in between each sample by using a Hepa-filter vacuum to remove soil particles. The analyst should change gloves in between processing each sample, in order to reduce cross contamination.
- 13.4 For projects requiring the use of a rotary splitter, refer to SOP HPLSplitter for more information.

Samples for analytes other than explosives are digested / extracted according to their method procedures and APPL SOPs

Explosives Sample Extraction by Mechanical Shaker

- 13.5 One method blank and one LCS are prepared with every analytical batch of 20 samples, using silica sand. The LCS is spiked after sieving and grinding. The blank and LCS are taken through the exact sample procedures as the samples, including sieving, grinding, incremental sampling and extraction. The method blank should be processed through the mechanical grinder after at least one sample has been processed.
- 13.6 One triplicate sample will be prepared with each analytical batch of 20 samples from a parent designated by the laboratory or the client. The triplicate sample may not be



- taken from any type of blank sample. One of the client's sample matrices is chosen by either the client or the laboratory for the triplicate. Check with the project manager to make sure that the client is aware of the triplicate requirement.
- 13.7 Spike soils with appropriate analytes and surrogates. One LCS and MS/MSD is required per analytical batch of 20 samples. If the client has not designated an MS/MSD on the COC, then check with the project manager before designating an MS/MSD in the laboratory.
 - 13.8 Add 60 μL of the 8330 Soil Surrogate (See SOP HPL002 Standard and Spike Prep) to the prepared jars for the Blank and all field samples.
 - 13.9 Add 200 μL of the 8330 Soil Spike (See SOP HPL002 Standard and Spike Prep) to the prepared jars for the LCS and MS/MSD.
 - 13.10 One PT sample is required by the DoD QSM with every extraction batch. Depending on the client's project, the PT sample may not be requested by the client. It is the client's responsibility to obtain a variance for the PT sample. APPL personnel should check with the project manager before extraction. The PT soil sample already contains the analytes of interest from the supplier, so only the surrogate is added prior to extraction (but after sieving, grinding and Incremental Sampling (IS)).
 - 13.11 Add 20mL Acetonitrile to each jar containing the spiked /surrogated soil. Place jars on a mechanical shaker for at least 18 hours.
 - 13.12 Allow the extracts to settle for 30minutes and remove approximately 8mL of the extract and transfer to 8mL Teflon-screw-cap glass vials.
 - 13.13 Store the samples in a refrigerator between 2°C and 6°C. The original extract in the 2.5oz jar may be stored at room temperature until disposal.
 - 13.14 Samples are be filtered through a 0.2- μm syringe filter (PALL GHP or equivalent) using a disposable PTFE syringe. All samples and QC in a batch should be filtered if any one of the samples is filtered.
 - 13.15 Using a variable-volume pipettor, remove a portion of the final extract and combine with 3 parts of millipore water in an injection vial. Store under refrigeration until HPLC analysis.

14.0 Data Analysis and Calculations - NA

15.0 Data Assessment and Acceptance Criteria for QC - NA

16.0 Corrective Actions and Contingencies for Out of Control Data or Unacceptable Data - NA

17.0 Method deviations

APPL Inc. will filter the extracts to remove suspended soil particles rather than centrifuge the extracts, as suggested in the EPA method 8330B.

APPL Inc will defer to the client's QAPP for the necessity of a ground SRM with each batch, and a lab triplicate with each batch.

18.0 Pollution Prevention

All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes- acidic, basic and solvents.

19.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land



Standard Operating Procedure

QA Control Copy # 100

SOP: MSE018IS

Section: 6

Revision: 10

Date: 08/19/20

disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.

20.0 Method Performance - NA

21.0 Equipment/Instrument Maintenance and Troubleshooting - NA

22.0 Computer hardware and software - NA

23.0 References

- 23.1 EPA Method 8330B, Revision 1, Oct 2006 "Nitroaromatics, Nitramines and Nitrate Esters by HPLC"
- 23.2 DoD QSM v. 5.3, May 2019
- 23.3 Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples (EPA No. EPA/600/R-03/027 November 2003) Section 4
- 23.4 ISO/IEC 17025:2017

24.0 Any tables, diagrams, flowcharts, validation data

Diagram 1: Incremental Sampling Preparation LogBook.

Appendix 1: DoD QSM Table B-3 and Table B-23

SALUTATION

This procedure is applicable to all personnel who extract explosives from solid samples using the orbital shaker for EPA 8330B and the IS techniques described in EPA 8330B, Appendix A and DoD QSM.

Section Manager Name: Hart Hedgepeth

Section Manager Signature:  Date: 08/19/20

QAU Director Name: Sharon Dehmlow

QAU Director Signature  Date: 08/19/20



Standard Operating Procedure

QA Control Copy # 100

SOP: MSE018IS

Section: 6

Revision: 10

Date: 08/19/20

Diagram 1

Incremental Sampling Preparation Log Book # 1 Page #								
ARF # / Project	Sample #	1st Weight *	2nd Weight *	3rd Weight *	4th Weight * (If Needed)	Passing Sample (weight of foil negligible)	Tarred Container + Non-passing Sample	LS. before grind (Initial Box when Complete)
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____
AY	NG sticker verified	Weight: _____ g	Weight: _____ g	Weight: _____ g	Weight: _____ g			Metals: _____
		Date: _____	Date: _____	Date: _____	Date: _____	g	g	8270: _____
		Time: _____	Time: _____	Time: _____	Time: _____			8290: _____
		Date/Initials	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Initials: _____	Other: _____

* Each recorded weight includes the foil-lined tray + the sample. NG = No Grind (applies to metals, PAH, Perchlorate, other methods etc.)



Standard Operating Procedure

QA Control Copy # 100

SOP: MSE018IS

Section: 6

Revision: 10

Date: 08/19/20

Appendix 1

DoD QSM Table B-3 and Table B-23

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil drying procedure	Each sample, LCS, and Method Blank. The appropriateness of the drying step is determined by each project.	Laboratory must have a procedure to determine when the sample is dry to constant mass. Entire sample must be air dried at room temperature.	NA.	Flagging is not appropriate.	Commercial PT samples must reflect the grinding, extraction, and analysis steps as a minimum. Record date, time, and ambient temperature on a daily basis while drying samples. If a laboratory utilizes a self-spiked LCS, the fortification must be performed prior to any preparation steps performed (drying, grinding, etc.) Drying may introduce a bias and is not recommended for certain compounds. Drying should be performed in the laboratory, not the field. LCS reference material is not required to be air dried if the vendor specifies that drying is not required. LCS and Blank matrix can be Ottawa sand, clean soil, or other vendor provided clean matrix.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil sieving procedure	Each sample, LCS, and Method Blank. The appropriateness of the sieving step is determined by each project.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging is not appropriate.	Do not include vegetation or debris in the portion of the sample that passes through the sieve unless that is a project specific requirement. Projects may require an alternate sieve size.
Soil grinding procedure	Initial demonstration at start up and any time major equipment is changed or when a reduction in the number or time of grinding cycles occurs. Each required sample, LCS, Blank, and Matrix Spike sample. The appropriateness of the grinding step is determined by each project.	Initial demonstration of grinding equipment: The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging is not appropriate.	Grinding and sieving is an iterative process, so cycles and duration can be varied to reduce heat if all samples are treated the same. Grinding may introduce a bias and is not recommended for certain compounds. Each sample, LCS, and Method Blank must use the same grinding process (i.e., same time intervals and number of grinding cycles).



Standard Operating Procedure

QA Control Copy # 100

SOP: MSE018IS

Section: 6

Revision: 10

Date: 08/19/20

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil sieving procedure	Each sample, LCS, and Method Blank. The appropriateness of the sieving step is determined by each project.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging is not appropriate.	Do not include vegetation or debris in the portion of the sample that passes through the sieve unless that is a project specific requirement. Projects may require an alternate sieve size.
Soil grinding procedure	Initial demonstration at start up and any time major equipment is changed or when a reduction in the number or time of grinding cycles occurs. Each required sample, LCS, Blank, and Matrix Spike sample. The appropriateness of the grinding step is determined by each project.	Initial demonstration of grinding equipment: The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging is not appropriate.	Grinding and sieving is an iterative process, so cycles and duration can be varied to reduce heat if all samples are treated the same. Grinding may introduce a bias and is not recommended for certain compounds. Each sample, LCS, and Method Blank must use the same grinding process (i.e., same time intervals and number of grinding cycles).
Soil Sample Triplicate	At the subsampling step, performed on one sample per batch. Cannot be performed on any sample identified as a blank (e.g., Field Blank, Method Blank, Grinding Blank).	The RSD for results above the LOQ must not exceed 20%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reported per the client, apply J-flag to all samples within that batch if acceptance criteria are not met and explain in the Case Narrative.	Sample triplicates are randomly selected unless the project specifies the sample to be used.



Standard Operating Procedure

QA Control Copy # 100

SOP: MSE018IS

Section: 6

Revision: 10

Date: 08/19/20

Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Preparation Processing	Each required ISM sample, PT, LCS, and Blank. Entire sample must be dried, ground, and sieved unless otherwise stipulated in the QAPP.	Refer to method specific tables for blank and other QC acceptance criteria. Where method specific tables require the use of DoD/DOE QSM Appendix C limits, those limits are not applicable to analysis after preparation using table B-23 and laboratories shall develop in-house acceptance criteria. Laboratories shall develop in-house surrogate acceptance criteria.	Refer to method specific tables for respective corrective actions.	Refer to method specific tables for respective flagging criteria.	Surrogates are added prior to sample preparation procedures. Holding times begin with the collection of the first increment. At least one Grinding Blank per batch must be processed after the LCS or after a client identified sample with known contamination, or at the end of the batch. A Grinding Blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as an ISM sample. Do not include vegetation or debris in the portion of the sample that passes through the sieve unless that is a project specific requirement.

Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Preparation Processing	Each required ISM sample, PT, LCS, and Blank. Entire sample must be dried, ground, and sieved unless otherwise stipulated in the QAPP.	Refer to method specific tables for blank and other QC acceptance criteria. Where method specific tables require the use of DoD/DOE QSM Appendix C limits, those limits are not applicable to analysis after preparation using table B-23 and laboratories shall develop in-house acceptance criteria. Laboratories shall develop in-house surrogate acceptance criteria.	Refer to method specific tables for respective corrective actions.	Refer to method specific tables for respective flagging criteria.	Surrogates are added prior to sample preparation procedures. Holding times begin with the collection of the first increment. At least one Grinding Blank per batch must be processed after the LCS or after a client identified sample with known contamination, or at the end of the batch. A Grinding Blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as an ISM sample. Do not include vegetation or debris in the portion of the sample that passes through the sieve unless that is a project specific requirement.



Standard Operating Procedure

QA Control Copy # 100

SOP: MSE018IS
Section: 6
Revision: 10
Date: 08/19/20

Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
ISM laboratory replicates	At the subsampling step, performed on one ISM sample per batch. Cannot be performed on any sample identified as a blank (e.g., Field Blank, Method Blank, Grinding Blank) or other QC.	For results above the LOQ, RSD or RPD must not exceed 20%.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	If reported per the client, apply J-flag to all samples within that batch if acceptance criteria are not met and explain in the Case Narrative.	Sample selection for performing replicates is based upon samples expected to contain the highest concentrations or as specified for the project. Percent RSD can be used for 3 or more replicates, otherwise use RPD or other appropriate metric. Field replicates do not replace the requirement for the laboratory to generate laboratory duplicates during subsampling.

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ATTACHMENT D
INCREMENTAL SAMPLING METHODOLOGY
RSD CALCULATIONS AND USES

Incremental Sampling Methodology

RSD Calculations and Uses

1.0 Introduction to the RSD

A relative standard deviation (RSD) is a measure of data variability, which serves as a reverse measure of precision. In other words, “precision” (how closely replicate values agree) is measured by determining how much disagreement there is. The less disagreement, the better the precision. Therefore, the larger the RSD value, the larger the variability, and the worse the precision. A low RSD value reflects low data variability, which implies good precision.

2.0 Understanding Data Variability

Concentration data are necessarily produced by some measurement technique (such as an analytical chemistry method). Although overall data precision is affected by several factors, the most fundamental factor is the ability of the measurement technique to produce the “same” result from repeated measurements of the “same” sample.

2.1 Zero Variability Equals Perfect Precision

As an example, assume a 1-mL vial of liquid is completely analyzed by injecting 10 0.1-mL aliquots into an ideal analytical instrument 10 times. The 10 ideal results are

20.0, 20.0, 20.0, 20.0, 20.0, 20.0, 20.0, 20.0, 20.0, and 20.0 (i.e., all are 20.0 ppm) Data Set #1

The statistics for the above data set are a mean of 20.0 ppm with a variability of 0. Since the vial was “exhaustively” measured (i.e., the entire vial was analyzed), we can be confident the true concentration is 20.0 ppm. Variability must intuitively be 0 because there is no variation at all. Thus, this ideal instrument shows perfect measurement precision.

2.1 Low Variability Equals Good Precision

Perfect analytical instruments do not exist in the real world. If another 1-mL vial of the exact same liquid were measured in the real world, an accurate method with good precision might give 10 results that look like this:

19.8, 20.1, 19.5, 20.4, 20.3, 19.5, 19.9, 20.2, 20.0, and 20.3. Data Set #2

The mean of this exhaustive data set is also 20.0 ppm; however, most individual results vary from the true 20.0 ppm concentration. The variations are small, so intuitively the variability is evaluated as low and precision was good. The statistic commonly used to measure variability is the standard deviation (SD). The SD for Data Set #1 was 0 ppm, and the SD for Data Set #2 is 0.3 ppm. More details about how to calculate the SD and its statistical relevance are easily found on the Internet.

2.3 High Variability Equals Poor Precision

The good precision shown in Data Set #2 can be contrasted with the poorer precision of the following data set:

24.6, 17.8, 15.4, 20.4, 14.2, 28.7, 19.9, 22.5, 17.8, and 19.0 Data Set #3

The mean of this data set is also 20.0 ppm, but individual results often vary quite a bit from 20.0. Intuitively the variability of Data Set #3 is larger than that of Data Set #2: the SD of Data Set #3 is 4.3 ppm, over 10 times larger than the SD of 0.3 for Data Set #2.

3.0 Relative Standard Deviation (RSD)

The RSD is useful because it “normalizes” the SD for easier comparisons among different data sets. As the name suggests, the magnitude of variability is assessed “relative to” something else, which is the mean. The RSD is calculated by dividing the standard deviation by the mean. The RSD for Data Set #2 is $0.3/20.0 = 0.016$. For Data Set #3, the RSD is $4.3/20.0 = 0.216$. For convenience, RSDs are often communicated as a percent: 1.6% for Data Set #2, and 21.6% for Data Set #3. The magnitude of variability in Data Set #2 is very small in proportion to the mean, whereas Data Set #3’s variability is 1/5th of the mean’s magnitude.

3.1 Low RSDs and High Concentrations

When the means of data sets are very similar, there is little advantage to using the RSD rather than the SD to compare the degree of variability among data sets. The RSD becomes more useful when comparisons involve data sets where the concentrations differ. Consider Data Set #4 from the complete analysis of another 1 mL vial, but one with a different concentration from the previous examples.

145.4, 150.8, 134.2, 167.4, 184.2, 158.7, 139.3, 156.0, 140.8, 166.7 Data Set #4

Because this data set is from an exhaustive analysis, the arithmetic mean of 154.4 ppm for the 10 analyses must be the vial’s true mean. The SD of the data set is 15.4 ppm—almost four times larger than the SD for Data Set #3 (which was 4.3 ppm).

Which data set is more variable?

If judged by the SD, Data Set #4 is more variable since its SD is greater. However, if the degree of variability is considered relative to concentration, Data Set #4 has the lower variability, since $15.4/154.4 = 10.0\%$ RSD, compared to Data Set #3’s RSD of 21.6%.

3.2 Calculating the RSD in Excel

The RSD for triplicate subsampling sets is calculated in Excel using the following notation:

=STDEV.S(C3:C5)/AVERAGE(C3:C5)

The %RSD is simply the RSD decimal value times 100.

Note that Excel’s “sample” standard deviation (STDEV.S) function is used, not the “population” standard deviation (STDEV.P). The same formula can be used to calculate the RSD directly for duplicate sets. Note also that RSD is unitless because both SD and mean have the same units and division cancels out the units.

3.2.1 Relationship between RSD and Relative Percent Difference (RPD)

When measuring the difference between duplicates, most practitioners are accustomed to using the relative percent difference (RPD). As suggested by the name, the RPD is a measure of the difference (as found by subtraction) between two values and expressed relative to (i.e., divided by) the average of the two values. Like RSD, RPD has no units.

Although both RPD and RSD measure variability, it is important to realize they are different “units.” For the same amount of variability, the RPD value is higher than the RSD value. For example, for the two-number data set (10, 15), the RPD is 40.0% and the RSD is 28.3%.

An analogy is the English vs. metric measures of distance: 1 inch is the same magnitude of length as 2.54 cm. Just as length in cm can be converted to inches units by dividing by 2.54, RPD and RSD are mathematically related by the square root of 2. An RPD of 35 can be converted into RSD units by dividing 35 by $\sqrt{2}$ to give an RSD of 25% (i.e., $35\% \text{ RPD} / \sqrt{2} = 25\% \text{ RSD}$).

3.2.2 Use the Same Measure of Variability to Avoid Confusion

ISM QC practices usually measure variability using three repeat measures (i.e., triplicates) as a more robust measure of variability than just two (i.e., duplicates). Because an RPD involves subtraction, it cannot be calculated on more than two replicates. That is one reason why ISM projects use RSDs to measure and express data variability. Another advantage is that the RSD is much easier to integrate into calculations of other important statistics, such as the upper confidence limit (UCL), than the RPD is. To avoid confusion, it is best to choose just one (either RSD or RPD) to use throughout a project or report so all measures of variability are in the same “units.” If necessary, RSD values can be converted to RPD values by multiplying by $\sqrt{2}$.

3.3 RSDs at Very Low Concentrations

As mentioned above, the larger the RSD value, the larger the variability and the poorer the precision. However, the usefulness of this relationship breaks down when concentrations are very low. Since dividing by a small number increases the quotient, the RSD value will be larger at lower concentrations, even if the absolute difference among the replicate values is very small.

The example data sets at right illustrate this effect. Although the absolute difference between the values in the two sets is the same ($4-3 = 1$ and $10-9 = 1$), their RSDs are quite different. An *absolute* difference of 1 may be insignificant to decisions and well within the range of simple analytical variability. But the *relative* difference causes the RSD to be significantly higher for the lower concentration set. This fact is important when setting limits on RSDs for QC purposes: *any limits placed on RSDs must consider the concentrations likely to be obtained*. A QC limit that may be achievable at higher concentrations may be mathematically unachievable at lower concentrations.

Comparison of RSDs for Data Sets of Lower vs. Higher Concentrations

2 sets of duplicate results			
	Set 1	Set 2	
Value 1	4	10	
Value 2	3	9	
RSD =	20.2%	7.4%	

3.3.1 RSD Limits at Low Concentrations

If RSD limits need to be set for analytes that are typically reported at low concentrations (such as PCBs, dioxins, dioxin TEQ, and pesticides), the absolute magnitude of differences (in terms of concentration) that might be expected due to analytical variability alone should be considered before setting an RSD limit. Specifically, what analytical precision can be expected when sample concentrations are near the detection or reporting limits. An analytical chemist familiar with the method and soil data may be able to determine what degree of absolute precision can be expected at different concentration levels. Mathematically feasible RSDs can then be predicted.

3.3.2 Use UCL Calculations to Determine Acceptable RSD For a Given DU Mean Concentration

The best approach is to determine the amount of absolute variability that is acceptable or reasonable from a decision-making purpose. When UCLs are the basis for decisions, limits on field replicate RSDs can be expressed in the context of limits on decision uncertainty. As an expression of variability, RSD interacts with concentration to determine whether the UCL exceeds a screening level. The closer the concentration is to the action level, the less the RSD can be before it causes the UCL to exceed the screening level.

A calculator can be set up in Excel so that trial and error entering numbers for triplicate results can find the maximum variability possible so that the UCL will not exceed the action level at a given mean concentration. Refer to the table at right for examples:

triplicate ave =	0.90	0.80	0.70
ISM Rep 1	0.85	0.71	0.60
ISM Rep 2	0.90	0.80	0.65
ISM Rep 3	0.96	0.90	0.84
Mean =	0.90	0.80	0.70
Std Dev =	0.06	0.10	0.13
n =	3	3	3
%RSD =	6.1	11.8	18.2
1-sided 95% t-UCL =	1.00	0.96	0.91
1-sided Chebyshev 95% UCL =	1.04	1.04	1.02

- When the mean of triplicate field samples is 0.90 ppm, the RSD cannot be higher than 6.1% (since at that RSD the 1-sided 95% t-UCL is exactly 1.0).
- If the mean concentration is 0.8 ppm, the highest allowable RSD is 11.8%.
- When there are 75 increments per DU and the field triplicate RSD exceeds 14.5%, the UCL must switch to the Chebyshev, and the maximum RSD is 18.2% if the DU mean is 0.70 ppm.

4.0 Pooling of RSDs

When there are two or more RSDs with relevant concentrations, those RSDs may be “pooled” to produce the equivalent of an “average” RSD. Combining multiple RSDs from equivalent SUs with similar concentrations produces a more representative RSD to apply to singlet SUs (i.e., SUs with only a single field sample). The equation used to pool two RSDs in Excel is:

$$\text{Pooled RSD} = \sqrt{(\text{RSD\#1}^2 + \text{RSD\#2}^2)/2}.$$

The equation for pooling three RSDs is:

$$\text{Pooled RSD} = \sqrt{(\text{RSD\#1}^2 + \text{RSD\#2}^2 + \text{RSD\#3}^2)/3}.$$

These equations are applicable where

- All replicate sets have the same n from which the RSD is calculated (e.g., n = 2 for all SU field sampling replicates),
- The concentrations of the SUs being pooled are similar enough so that the DUs can be considered CSM-equivalent, and
- The standard deviation (SD) values for the SUs are similar (so that the DUs can be considered CSM-equivalent).

4.1 Pooled RSD as Parallel to Pooled Standard Deviation (SD)

The concept of pooling RSDs to obtain an “average” of two or more RSDs mirrors the pooling of SDs which is a standard statistical calculation. The following text is copied from the statistical website called “Statistics How To” (accessed 3/3/2020) (<https://www.statisticshowto.datasciencecentral.com/pooled-standard-deviation/>).

What is a Pooled Standard Deviation?

The Pooled Standard Deviation is a **weighted average** of [standard deviations](#) for two or more groups. The individual standard deviations are averaged, with more “weight” given to larger sample sizes.

Once the pooled standard deviation has been calculated, SD_{pooled} is used in place of SD_1 and SD_2 in the formula for [standard error](#). Along with an updated [degrees of freedom](#) formula ($df = n_1 + n_2 - 2$), the idea is that you would be able to get a better model for the [sampling distribution of the sample mean](#).

Pooled standard deviations are used in many areas in statistics, including: [effect size calculations](#), [t-tests](#), and [ANOVAs](#). They are also used in lab-based sciences like biology and chemistry, where they can be an indication for repeatability of an experiment.

How to Calculate the Pooled Standard Deviation

Cohen (1988) offers a couple of options for calculating the pooled standard deviation. The simplest is:

$$SD_{pooled} = \sqrt{\frac{(SD_1^2 + SD_2^2)}{2}}$$

Where:

- SD_1 = [standard deviation](#) for group 1 (as long as both data sets have the same n)
- SD_2 = standard deviation for group 2

RSDs can be pooled In a similar way:

$$\text{Pooled RSD} = \sqrt{(\text{sumsq}(\text{RSD\#1}, \text{RSD\#2})/2)},$$

Remembering that RSDs must be converted to relative variance by squaring before averaging can be performed. After the average relative variance is obtained, its square root is taken to get back to standard deviation.

4.2 Derivation of the Pooled RSD Equation from Ingersoll's Work

As described below, Ingersoll explained the usefulness of partitioning variability when assessing data quality and identifying corrective actions (Ingersoll, 2001). He used an equation to derive an averaged variability term on page 44 of his paper:

$$s_r = (\sum(d_r^2)/2k)^{1/2}$$

This formula uses rather esoteric notation but is translated this way:

$$\text{Pooled RSD} = \sqrt{\{[\text{the sum of the (relative differences}^2)]/(2*k)\}},$$

where k is the number of replicate sets whose variability is being pooled.

Notice that Ingersoll uses the “relative difference (d_r)” in his equation to accommodate traditional laboratory duplicate sets whose variability was calculated using the RPD (relative percent difference). “Relative difference” is the same as RPD except that it is expressed as a decimal rather than as the percent. As discussed earlier, RPD and RSD are related by the $\sqrt{2}$ in that $\text{RPD}/\sqrt{2} = \% \text{RSD}$. Therefore, the term “ $d_r/\sqrt{2}$ ” equates to RSD (expressed as a decimal).

Isolating the $d_r/\sqrt{2}$ term in $s_r = (\sum(d_r^2)/2k)^{1/2}$ and using the plus sign rather than the summation sign arranges Ingersoll's equation to mirror the pooled SD equation: $\text{Pooled RSD} = \sqrt{[(d_{r1}^2/2k) + d_{r2}^2/2k]}$, where $k = 2$ when two RSD are being pooled.

$$\text{The equation now becomes } \text{Pooled RSD} = \sqrt{[(d_{r1}^2/2*2) + d_{r2}^2/2*2)]}. \quad \text{Eqn. 1}$$

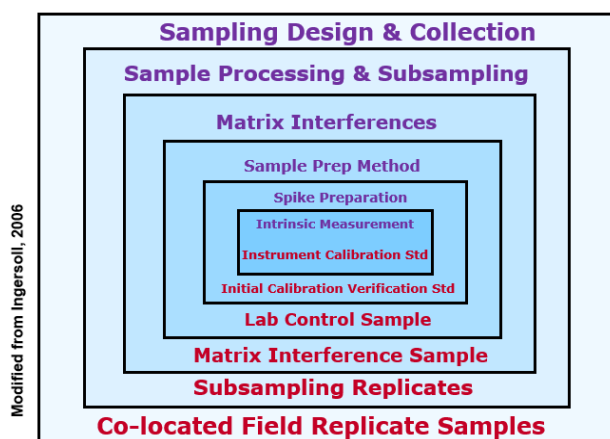
Since $d_r/\sqrt{2} = \text{RSD}$, squaring both sides gives $d_r^2/2 = \text{RSD}^2$. Substituting RSD^2 for $d_r^2/2$ in Eqn 1 gives $\text{Pooled RSD} = \sqrt{(\text{RSD}_1^2/2 + \text{RSD}_2^2/2)}$, which can be rewritten as $\text{Pooled RSD} = \sqrt{[(\text{RSD}_1^2 + \text{RSD}_2^2)/2]}$, or generalized as

$$\text{Pooled RSD} = \sqrt{[(\text{RSD}_1^2 + \dots + \text{RSD}_k^2)/k]}. \quad \text{Eqn. 2}$$

5.0 “Measurement Method” Includes More than Just the Laboratory Analysis

A “measurement method” encompasses all activities involved in generating a data result. For soil data, the measurement method includes activities on both the sampling side and analytical side. The sampling side includes sampling design (how many samples from what locations), sample collection, and sample processing. The analytical side includes subsampling of the processed sample, analytical preparation of the subsample to produce an extract, possibly cleanup of the extract, and instrumental analysis of the extract. Each step offers an opportunity for data variability to creep in and magnify. Data variability attributable to each of these steps can be represented as a “nested configuration.” QC checks are targeted to each step in order to separate out the variability contributed by each step (Ingersoll, 2001 and 2006).

Conceptual Model for Data Uncertainty Steps Introducing Variability and QC to Measure



5.1 Assessing Data Variability and Its Components

The consensus of experts is that activities on the sampling side contribute much more heavily to data uncertainty than does the analytical side: “It has been estimated that up to 90 percent of all environmental measurement variability can be attributed to the sampling process.” (Homsher, 1991). ISM address this by routinely using QC replication to measure variability stemming from field sampling and sample handling. Field triplicates serve as co-located field samples (USEPA, 2006). Since they are at the “top of the chain,” they measure all components, but allow estimation of variability attributable to the field sampling design. The difference among field replicates is calculated as a total RSD.

If DU-level RSDs are elevated, the reasons for elevation are explored by calculating the RSDs for QC replicates for nested steps. For example, variability attributable to the processing/subsampling procedures will be measured using subsampling triplicates. Laboratory control sample (LCS) data can be used to evaluate analytical variability, and surrogate recovery QC data can be used to extend the evaluation of analytical variability to include matrix-specific sample preparation effects. If corrective action is required to reduce data variability, these variability evaluations can help pinpoint which corrective actions would be most effective for reducing data uncertainty, as reflected by the width of confidence intervals around individual sample results.

5.2 Measuring Variability Components

As illustrated by the above nested variability diagram, field sample precision is dependent on analytical precision. If the analytical process is not precise, different results can be expected even if field replicate samples were to have identical concentrations. For that reason, subsampling variability (which

incorporates all components of analytical variability “downstream” of sample processing) must be known to have adequate precision. If subsampling variability is not measured, it cannot be known whether field sample variability stems from field heterogeneity or poor sample processing. If QC checks show good analytical precision, but field sample replicates are too variable, the problem is isolated to some aspect of the field sample collection design.

5.2.1 Key Variability Components and Their QC Measures

QC checks should be structured to provide QC data that quantifies the major components of data variability.

- Laboratory control samples (LCS) can quantify analytical variability that is independent of the sample matrix. Other QC measures, such as surrogate compounds, might be used for this purpose if LCS data are not available. Such measures of analytical variability incorporate the extraction step, as well as extract cleanup and concentration steps, through to the last step which is instrumental determination/quantitation,
- Subsampling QC triplicates quantify the combined variabilities from sample processing through analysis.
- Field sample triplicates add in the variability from sampling design and field collection procedures so that all contributions to data variability are encompassed.

5.2.2 Literature References for the Mathematical Strategy Used in this Project

The above relationships can be expressed mathematically using the RSDs from replicate QC. This process is described in technical detail in various documents:

- Ingersoll, 2001: Environmental Analytical Measurement Uncertainty Estimation: Nested Hierarchical Approach, available through the Defense Technical Information Center (<https://discover.dtic.mil/>); also see References for the direct URL
- Ingersoll, 2003: *Standard Operating Procedure Estimation of Analytical Measurement Uncertainty*
- Ingersoll's uncertainty calculation strategy was referenced in the Department Of Defense Quality Systems Manual for Environmental Laboratories (Prepared by the DoD Environmental Data Quality Workgroup, Final Version 3, January 2006), page 61.

5.3 Mathematical Strategy for Partitioning Variability

Ingersoll's calculation strategy and equations can be adapted and simplified to meet specific projects needs. For the purpose of this project, the simplified equation is based on the key variability components listed in 5.2.1. The mathematical strategy is shown in the graphic below:

Total variability = Field variability + Subsampling variability
 Subsampling variability = Within-sample variability + Analytical variability

equivalent to:

$$(\text{Total \%RSD})^2 = (\text{Field \%RSD})^2 + (\text{Subsampling \%RSD})^2$$

rearrange equation to solve for field component:

Eqn 1: $\text{Field \%RSD} = \sqrt{[(\text{Total \%RSD})^2 - (\text{Subsampling \%RSD})^2]}$

=====

If subsampling RSD too high, the analytical portion should be
 subtracted to evaluate the within-sample RSD:

Eqn 2: $\text{Within-sample \%RSD} = \sqrt{[(\text{Subsampling \%RSD})^2 - (\text{Analytical \%RSD})^2]}$

The overall mathematical strategy can be summarized as:

The total variability (as measured by DU field replicates) is the sum of two major components: field heterogeneity and subsampling/analysis variability (as measured by subsampling triplicates).

Although field heterogeneity cannot be measured directly, it can be partitioned (i.e., separated or isolated) out of the total. This is done by subtracting the subsampling/analytical variability from the total variability per Equation 1. The remainder is attributed to the field component. In this way components that cannot be measured directly can be isolated from an aggregated variability.

Similarly the portion of variability attributable solely to sample processing and slab cake subsampling can be backed out from the analytical variability (Eqn 2 above). It is the remainder after the analytical variability is subtracted from the total within-sample variability (i.e., subsampling/analytical component measured by the subsampling triplicates).

Remember that RSDs cannot be added or subtracted directly since they are values obtained as square roots. The RSDs must be squared first to obtain relative variances (USEPA, 2002, p. 197). The relative variances are used to carry out the math, then the square root of the equation's output is taken to convert back to an RSD value.

5.3.1 Indications for Corrective Action

If the field component is identified as an issue, corrective action would be to evaluate field sample collection procedures such as the number of increments per field sample, the efficiency and consistency of sample collection tools and potential losses during transfer of increment masses into the sample container. Identified deficiencies would be corrected and samples recollected if necessary.

If the subsampling component is identified as an issue, corrective action is to re-evaluate sample processing and subsampling procedures. More rigorous sample processing may be needed (such as milling of the field sample, rather than sieving alone). Slabcake subsampling procedures (number of increments, the tools used) may need revision. It is also important to check the laboratory reports to make sure that the designated subsampling mass (30 grams) was actually used. Deficiencies should be corrected. If necessary, samples may need to be reprocessed or re-subsampled and reanalyzed.

If the pure analytical component is higher than desired, corrective action involves close review of the data validation reports and laboratory packages for any problems identified by laboratory QC such as

surrogate recoveries, blanks, calibration issues and clerical errors. Reanalysis of processed samples may be needed.

5.3.2 Dioxin Project Example of Partitioning to Identify Corrective Action

An example of this partitioning strategy and corrective action is provided by a past dioxin TEQ project to which the author provided assistance. The project consisted of two nearby areas (referred to here as “Site A” and (Site B”) which were sampled at different times. The results of QC variability partitioning are summarized in the table below:

Replication QC Summary for Dioxin Site	Total RSD (%)	Field RSD (%)	Within-sample RSD (%)	Analytical RSD (%)
Site A Data (Oct 2011)	Subsampling done in Lab from single 100-g sample jar			
QC calculations	31	18	25	2.5
Site B Data (Jun 2012)	15-g analytical subsampling done in field-1 analysis/jar			
QC calculations	16	15	5.4	2.7

Site A was sampled in 2011. Its QC data showed a higher RSD for sample processing (25%) than for field variability (18%), which is unusual and a cause for concern. Fortunately, TEQ concentrations were well below the screening level (51 ppt) so that the added data uncertainty caused by the total RSD of 31% did not compromise decision-making, and reanalysis of samples was not needed.

A follow-up query to the laboratory revealed that their subsampling method involved simple scooping off the top of the sample in its jar. Although the field sample had been correctly subsampled by field staff, 100-gram jars were filled for the laboratory. Therefore, the laboratory was scooping off the top of stratified/segregated soil samples, which introduced the data variability that was isolated to the subsampling step. The lab was unwilling to implement slabcake subsampling for the project’s future work at Site B.

When Site B was sampled in 2012, corrective action was implemented by sending no more sample than the analytical mass (15 grams) used by the laboratory, with the instructions that the complete mass inside the jar was to be analyzed. Site B’s QC results and variability partitioning showed great improvement: the processing RSD was down to 5.4% (from Site A’s 25%), which was only twice the LCS RSD (2.7%) and much less than the field RSD (15%). This reduction in subsampling variability brought total data variability down to 16% from the previous 31% RSD observed in Site A’s sampling effort.

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