

December 18, 2014

## MEMORANDUM

To: Kris McCaig and Dave Enos – Teck American Incorporated (TAI)  
From: Dina Johnson and Emma McConnell  
Subject: UCR Upland Soil Study - Proposed Sample Selection Approach and Identification of Samples for Lead IVBA Analyses

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This memorandum summarizes a proposed approach for identifying a subset of Upland Soil Study samples for lead in vitro bioaccessibility assay (IVBA) analysis.

### Background:

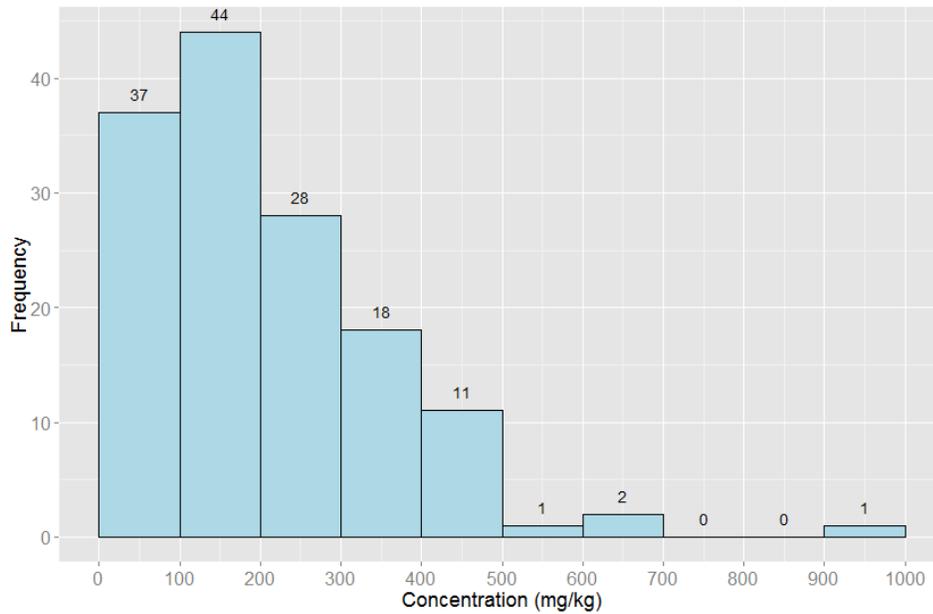
Section B.4.2 of the Final Soil Study quality assurance project plan (QAPP) (TAI 2014) specifies:

*Twenty percent of samples having lead concentrations greater than 100 mg/kg will be analyzed for lead bioaccessibility using an in vitro extraction method (USEPA 2012). These samples will be selected in consultation with EPA so that they provide a representative distribution of samples with lead concentrations greater than 100 mg/kg.*

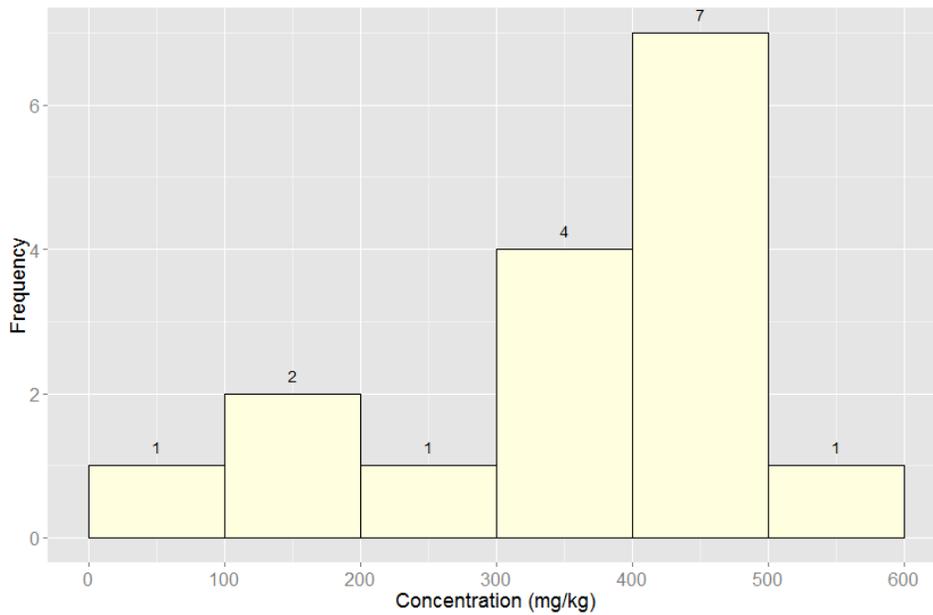
Upland soil samples (<149 µm size fraction) for IVBA analysis will be selected from the aerial deposition area (ADA) and the relict floodplain (RF) deposition areas. A total of 218 individual preliminary sample results for lead corresponding to 158 decision units (DU) are available for these areas. Of these 158 DUs, 142 DUs are from the ADA and 16 are from RF. The combined dataset includes 60 triplicates (i.e., 20 sets of 3 replicates) and 20 field splits (10 sets of field splits). The dataset also includes 28 samples for which EPA splits were specified; results for EPA's split samples have not been received at this time.

For each DU with triplicate sample results, the relative standard deviation (RSD) is within the QAPP-specified control limit of 35 percent (QAPP sections B.4.5 and D.1). The relative percent difference (RPD) for each pair of field splits is also within the control limit (RPD = 20 percent) used by the laboratory for laboratory duplicates. Laboratory data validation for the Upland Soil Study samples is currently underway.

Substituting the average of each triplicate set or field split pair, the distribution of lead concentrations for the 142 ADA DUs is shown below:



Similarly, the distribution of lead concentrations for the 16 RF DUs is:



As shown by these histograms, the ADA distribution appears to be lognormal while the RF data appear to be normally distributed. Goodness-of-fit testing in ProUCL (ver. 5.0.00) supports this conclusion.

Summary statistics for the two areas are shown below:

| Statistic          | ADA, Averaged Triplicates and Splits | RF, Averaged Triplicates and Splits |
|--------------------|--------------------------------------|-------------------------------------|
| n                  | 142                                  | 16                                  |
| Mean (mg/kg)       | 211                                  | 353                                 |
| SD                 | 147                                  | 135                                 |
| Minimum (mg/kg)    | 35                                   | 72                                  |
| 25%ile(Q1) (mg/kg) | 98                                   | 287                                 |
| 50%ile(Q2) (mg/kg) | 171                                  | 388                                 |
| 75%ile(Q3) (mg/kg) | 289                                  | 442                                 |
| 90%ile (mg/kg)     | 404                                  | 467                                 |
| 95%ile (mg/kg)     | 456                                  | 492                                 |
| 99%ile (mg/kg)     | 663                                  | 551                                 |
| Maximum (mg/kg)    | 988                                  | 565                                 |
| CV                 | 0.7                                  | 0.4                                 |

Considering the data distributions and summary statistics, as well as the nature of potential lead sources contributing to each area, it is recommended that the process for selecting IVBA samples for analysis be applied to the ADA subset and RF subset separately.

Further, it is recommended that each triplicate and duplicate set be pre-averaged prior to consideration of the 100 mg/kg lead threshold. This is appropriate because each set represents the exposure point concentration for an individual DU and preliminary results for the replicate sets have been determined to be within QAPP-specified control limits. However, it should be noted that when screening is based on averages for each set of replicates, the resulting DUs do not differ from those which would be retained if screening was based on the individual replicate samples instead.

**Proposed Approach for Identification of Representative 20% of Upland Soil Sample DUs for Lead IVBA Analysis:**

1. Separate triplicate sets and field split pairs from non-replicate samples in the dataset.
2. Screen out non-replicate samples with "EDD\_Result" concentrations less than 100 mg/kg.
3. For each triplicate set, calculate average lead concentration and screen out average concentrations less than 100 mg/kg.
4. For each field split pair, calculate average lead concentration and screen out average concentrations less than 100 mg/kg.
5. Randomly select one replicate sample from each remaining triplicate and field split set (from steps 3 and 4).
6. Combine randomly selected replicate samples (from step 5) with the remaining non-replicate samples (from step 2). Steps 1 – 6 yields a total of 105 samples within the ADA and 15 samples within the RF from which the 20% for IVBA will be selected.
7. Assign a random number to each sample in the ADA dataset (from step 6) and sort the rows of sample data based on random number (from low to high). Instruct the laboratory to process samples corresponding to the first 21 rows of sorted data (i.e., 20% of 105 samples) for IVBA analysis.

8. Assign a random number to each sample in the RF dataset (from step 6) and sort the rows of sample data based on random number (from low to high). Instruct the laboratory to process samples corresponding to the first 3 rows of sorted data (i.e., 20% of 15 samples) for IVBA analysis.

**Results of Proposed IVBA Sample Identification Approach:**

Based on execution of the proposed IVBA sample identification approach, the following 21 samples from the ADA have been identified for IVBA analysis:

| Sample ID       | Lab_Code     | Component | Units | EDD_Result | location_id |
|-----------------|--------------|-----------|-------|------------|-------------|
| DIRT-011-150um  | K1410739-012 | Lead      | mg/Kg | 164        | ADA-141     |
| ADA-081-150um   | K1411306-018 | Lead      | mg/Kg | 166        | ADA-081     |
| ADA-145-150um   | K1410738-018 | Lead      | mg/Kg | 318        | ADA-145     |
| ADA-160-150um   | K1411144-012 | Lead      | mg/Kg | 282        | ADA-160     |
| ADA-047-150um   | K1410744-021 | Lead      | mg/Kg | 269        | ADA-047     |
| ADA-076-150um   | K1411669-006 | Lead      | mg/Kg | 215        | ADA-076     |
| ADA-152-150um   | K1411309-024 | Lead      | mg/Kg | 221        | ADA-152     |
| DIRT-015-150um  | K1411144-009 | Lead      | mg/Kg | 359        | ADA-161     |
| ADA-061-150um   | K1410077-018 | Lead      | mg/Kg | 137        | ADA-061     |
| ADA-059-150um   | K1411305-006 | Lead      | mg/Kg | 129        | ADA-059     |
| ADA-126-150um   | K1410072-006 | Lead      | mg/Kg | 340        | ADA-126     |
| ADA-016-A-150um | K1410738-009 | Lead      | mg/Kg | 211        | ADA-016     |
| ADA-048-150um   | K1411987-018 | Lead      | mg/Kg | 124        | ADA-048     |
| ADA-125-150um   | K1411987-003 | Lead      | mg/Kg | 122        | ADA-125     |
| ADA-142-150um   | K1410742-009 | Lead      | mg/Kg | 466        | ADA-142     |
| ADA-144-150um   | K1410741-009 | Lead      | mg/Kg | 668        | ADA-144     |
| ADA-096-150um   | K1410743-009 | Lead      | mg/Kg | 287        | ADA-096     |
| ADA-035-150um   | K1411144-027 | Lead      | mg/Kg | 183        | ADA-035     |
| ADA-150-150um   | K1411142-027 | Lead      | mg/Kg | 559        | ADA-150     |
| ADA-001-150um   | K1410069-021 | Lead      | mg/Kg | 288        | ADA-001     |
| ADA-057-150um   | K1411311-024 | Lead      | mg/Kg | 134        | ADA-057     |

Additionally, the following 3 samples from the RF have been identified for IVBA analysis:

| Sample ID       | Lab_Code     | Component | Units | EDD_Result | location_id |
|-----------------|--------------|-----------|-------|------------|-------------|
| RFA-005-150um   | K1409766-009 | Lead      | mg/Kg | 362        | RFA-005     |
| RFD-002-150um   | K1409759-003 | Lead      | mg/Kg | 413        | RFD-002     |
| RFA-001-C-150um | K1410071-021 | Lead      | mg/Kg | 492        | RFA-001     |

Summary statistics for the randomly selected samples are compared to summary statistics for the full datasets (from step 6) below. These summaries suggest the approach results in a representative selection of the distribution of lead concentrations for samples equal to or greater than 100 mg/kg.

|                 | Step 6 Dataset | Random 20% for IVBA |
|-----------------|----------------|---------------------|
| <b>ADA</b>      |                |                     |
| n               | 105            | 21                  |
| Minimum (mg/kg) | 102            | 122                 |
| Maximum (mg/kg) | 956            | 668                 |
| Average (mg/kg) | 262            | 269                 |
| Median (mg/kg)  | 218            | 221                 |
| <b>RF</b>       |                |                     |
| n               | 15             | 3                   |
| Minimum (mg/kg) | 187            | 362                 |
| Maximum (mg/kg) | 581            | 492                 |
| Average (mg/kg) | 381            | 422                 |
| Median (mg/kg)  | 407            | 413                 |

Details of the steps and data used to arrive at the proposed sample selections are documented in an Excel workbook (“Upland IVBA Sample Selection\_121814.xlsx”), which is being transmitted with this memorandum. It is important to note, that repeat execution of the process outlined would not result in the same set of samples identified for IVBA analysis as noted above due to the use of formulas to randomly select individual replicates (step 5) and to randomly identify 20% of samples for IVBA (steps 7 and 8). Each run of the random selection formulas will yield a different result.

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**From:** McCaig Kris SPOK [<mailto:Kris.McCaig@teck.com>]  
**Sent:** Wednesday, January 14, 2015 10:50 AM  
**To:** Buelow, Laura  
**Cc:** Dina Johnson - ENVIRON ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)) ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)); [rschoof@environcorp.com](mailto:rschoof@environcorp.com); [gardn@exponent.com](mailto:gardn@exponent.com); Wilkening, Matt; Stifelman, Marc; [thayer@srcinc.com](mailto:thayer@srcinc.com); [diamond@srcinc.com](mailto:diamond@srcinc.com); John Toll; Enos Dave SPOK; Anne Fairbrother ([afairbrother@exponent.com](mailto:afairbrother@exponent.com))  
**Subject:** RE: Proposed IVBA sample selection approach for UCR upland soil study

Thank you, Laura. We will proceed.

Kris

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**From:** Buelow, Laura [<mailto:Buelow.Laura@epa.gov>]  
**Sent:** Wednesday, January 14, 2015 9:48 AM  
**To:** McCaig Kris SPOK  
**Cc:** Dina Johnson - ENVIRON ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)) ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)); [rschoof@environcorp.com](mailto:rschoof@environcorp.com); [gardn@exponent.com](mailto:gardn@exponent.com); Wilkening, Matt; Stifelman, Marc; [thayer@srcinc.com](mailto:thayer@srcinc.com); [diamond@srcinc.com](mailto:diamond@srcinc.com); John Toll ([JohnT@windwardenv.com](mailto:JohnT@windwardenv.com)); Enos Dave SPOK; Anne Fairbrother ([afairbrother@exponent.com](mailto:afairbrother@exponent.com))  
**Subject:** RE: Proposed IVBA sample selection approach for UCR upland soil study

Kris,

I agree that this reflects EPA's request.

Laura

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**From:** McCaig Kris SPOK [<mailto:Kris.McCaig@teck.com>]  
**Sent:** Friday, January 09, 2015 4:15 PM  
**To:** Buelow, Laura  
**Cc:** Dina Johnson - ENVIRON ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)) ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)); [rschoof@environcorp.com](mailto:rschoof@environcorp.com); [gardn@exponent.com](mailto:gardn@exponent.com); Wilkening, Matt; Stifelman, Marc; [thayer@srcinc.com](mailto:thayer@srcinc.com); [diamond@srcinc.com](mailto:diamond@srcinc.com); John Toll ([JohnT@windwardenv.com](mailto:JohnT@windwardenv.com)); Enos Dave SPOK; Anne Fairbrother ([afairbrother@exponent.com](mailto:afairbrother@exponent.com))  
**Subject:** RE: Proposed IVBA sample selection approach for UCR upland soil study

Laura,

Based on our call with EPA this past Tuesday (1/6/15) and clarification of your comments below, it is our understanding that EPA has requested the following 5 samples be submitted for IVBA analysis in addition to

the 24 samples we proposed in our December 18, 2014 memorandum (see attached):

- ADA-016-B-150um (replicate for memo proposed sample ADA-016-A-150um)
- ADA-016-C-150um (replicate for memo proposed sample ADA-016-A-150um)
- RFA-001-A-150um (replicate for memo proposed sample RFA-001-C-150um)
- RFA-001-B-150um (replicate for memo proposed sample RFA-001-C-150um)
- ADA-162-150um (randomly selected field split for DU with highest average lead concentration)

We also understand EPA requests that for samples submitted for IVBA analysis, concentrations for all metals (i.e., TAL metals which include aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc) be reported for the IVBA for potential use in the BERA. Although we agree to this request it is important to note the following:

There are two major variables that affect bioaccessibility of metals in non-ruminant animals – the pH of the gastric fluid and gut retention time. The write-up that EPA provided emphasizes only pH differences. Some of the researchers cited in that write up recognized the gut retention time issue as well, and modified their procedures to be representative of the species they were interested in simulating.

So, the problem is that there is not a “one size fits all” (or even “most”) approach, if an accurate representation of bioaccessibility is desired for all birds and mammals (or for those found at the UCR site). Ruminants (deer, elk, big horn sheep) and hind-gut fermenters (horses, rabbits, quail, pheasants) will most definitely not be represented by the IVBA methodology.

With your concurrence we will direct ALS laboratory to begin analysis on the samples identified in the attached memo and listed above for IVBA analysis and that concentrations for all TAL metals be reported for the IVBA.

Regards,

Kris

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**From:** Buelow, Laura [<mailto:Buelow.Laura@epa.gov>]  
**Sent:** Monday, January 05, 2015 12:07 PM  
**To:** Enos Dave SPOK; McCaig Kris SPOK  
**Cc:** Dina Johnson - ENVIRON ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)) ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)); [rschoof@environcorp.com](mailto:rschoof@environcorp.com); [gardn@exponent.com](mailto:gardn@exponent.com); Wilkening, Matt; Stifelman, Marc; [thayer@srcinc.com](mailto:thayer@srcinc.com); [diamond@srcinc.com](mailto:diamond@srcinc.com)  
**Subject:** RE: Proposed IVBA sample selection approach for UCR upland soil study

Dave and Kris,

EPA has reviewed the updated IVBA sample selection approach. Here are our comments:

Add the max sample from the ADA (988 ppm) is also run as part of the IVBA analysis.

Choose 3 of the DUs that were run in triplicate to have all three incremental samples run for IVBA. We acknowledge that the %RSD was met for all of the metals analysis, however we believe that running all triplicates for 3 samples would provide more confidence in the data.

EPA requests that concentrations for all metals be reported for the IVBA for potential use in the BERA. We recognize that lead is the only validated method for IVBA, however it may be possible to develop site-specific bioaccessibility estimates for these other metals. Further discussions will be needed to determine the usability of the data. We would rather request the results be presented rather than hold up running the IVBA to make a final determination of how we can use the results from the other metals now.

Laura

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**From:** Enos Dave SPOK [<mailto:Dave.Enos@teck.com>]  
**Sent:** Monday, December 22, 2014 2:38 PM  
**To:** Buelow, Laura; Wilkening, Matt  
**Cc:** Dina Johnson - ENVIRON ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)) ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)); McCaig Kris SPOK; [rschoof@environcorp.com](mailto:rschoof@environcorp.com); [gardn@exponent.com](mailto:gardn@exponent.com)  
**Subject:** FW: Proposed IVBA sample selection approach for UCR upland soil study  
**Importance:** High

Laura: attached is a revised memorandum describing our proposed sample selection approach for IVBA analysis for the Uplands Soil Study. This approach incorporates your suggestions from December 17. Also, we are providing a list of samples selected by using this approach, and associated unvalidated (preliminary) lead data.

Please let us know if you have further comments. Thank you and Happy Holidays!

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**Dave Enos, LHG, RG**  
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**From:** Dina Johnson [<mailto:DLJohnson@environcorp.com>]  
**Sent:** Thursday, December 18, 2014 6:37 PM  
**To:** McCaig Kris SPOK; Enos Dave SPOK  
**Cc:** Emma McConnell; Rosalind Schoof  
**Subject:** FW: Proposed IVBA sample selection approach for UCR upland soil study  
**Importance:** High

Hi Kris and Dave,

In response to Laura's comments below, attached is a revised memorandum. The attached replaces the histograms previously run in ProUCL with ones run using R given that EPA's ProUCL support staff will not be able to follow up on the glitch we identified until early 2015.

The selection approach has also been modified to incorporate the request to select specific replicates randomly rather than

defaulting to the primary sample in each set. Note, this step is now applied to triplicates and field splits prior to random selection of 20% of the ADA and RF samples greater than or equal to 100 mg/kg.

As noted in the attached and on the call earlier this week, screening of DUs with triplicates based on the average concentration of the triplicates rather than the maximum triplicate for each DU does not alter the results of the screening for the Upland Soil Study. This is also true for field splits screened on average vs. maximum concentrations. Given that all replicate samples were within QAPP-specified compliance limits, this finding is not unexpected.

The attached memorandum has also been expanded to include a summary of the results from execution of the revised approach. Summary statistics comparing the subset of samples identified for IVBA analysis with the datasets from which they were randomly selected are included in the memorandum. The means and medians of the samples proposed for IVBA analysis are similar to, but higher than those corresponding to the datasets from which they were selected suggesting the approach has resulted in identification of a representative distribution of the samples for IVBA analysis.

Lastly, an Excel workbook referenced in the memorandum is attached. It provides the data and details most of the steps described in the memorandum.

I will be on vacation beginning tomorrow (12/19/14) through 12/26/14. Emma will be in the office tomorrow through 12/23 and has been assisting me with some of this work so she may be able to assist you if you have any questions during my absence.

Happy Holidays!  
Dina



**Dina L. Johnson** | Senior Manager

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**From:** Buelow, Laura [<mailto:Buelow.Laura@epa.gov>]

**Sent:** Wednesday, December 17, 2014 10:59 AM

**To:** McCaig Kris SPOK

**Cc:** Dina Johnson; Rosalind Schoof; Nicholas Gard - Exponent ([gardn@exponent.com](mailto:gardn@exponent.com)); Enos Dave SPOK; Wilkening, Matt

**Subject:** RE: Proposed IVBA sample selection approach for UCR upland soil study

Kris,

In response to the memo, I have a few follow up questions/comments.

I would like to confirm that there is not a case where the maximum Pb result is > 100 mg/kg but the average for the DU is <100 mg/kg (sending us the raw data for Pb would help us confirm).

I would like to see the change in the average lead concentration graph when the glitch is worked out (or if it's going to take some time, hopefully there is a different way to graph the results).

We believe that the increment to be sampled should not always be the primary ("A"), but randomly chosen from A, B or C.

Has Teck randomly selected the samples yet? We would like to see what samples were selected and how their summary

statistics compare to the total samples.

Thank you,

Laura

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**From:** McCaig Kris SPOK [<mailto:Kris.McCaig@teck.com>]

**Sent:** Thursday, December 11, 2014 11:06 PM

**To:** Buelow, Laura

**Cc:** Dina Johnson - ENVIRON ([DLJohnson@environcorp.com](mailto:DLJohnson@environcorp.com)); Rosalind Schoof; Nicholas Gard - Exponent ([gardn@exponent.com](mailto:gardn@exponent.com)); Enos Dave SPOK; Wilkening, Matt

**Subject:** FW: Proposed IVBA sample selection approach for UCR upland soil study

Laura,

Please see attached for EPA's review and discussion, the above referenced. We will be available on the weekly project management call to answer questions.

Regards,

Kris

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**From:** Dina Johnson [<mailto:DLJohnson@environcorp.com>]

**Sent:** Thursday, December 11, 2014 2:20 PM

**To:** McCaig Kris SPOK; Enos Dave SPOK

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**Subject:** Proposed IVBA sample selection approach for UCR upland soil study

Hello Kris and Dave,

Per your request, attached is a memorandum summarizing ENVIRON's proposed approach for selection of samples from TAI's soil study for lead in vitro bioaccessibility assay (IVBA) analysis. Please let me know if you have any questions about this approach.

Best regards,

Dina



**Dina L. Johnson** | Senior Manager

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