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NEVADA DIVISION OF ENVIRONMENTAL PROTECTION
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STATE OF NEVADA
Department of Conservation & Natural Resources
DIVISION OF ENVIRONMENTAL PROTECTION

Brian Sandoval, Governor
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May 8, 2014

Jay A. Steinberg
Nevada Environmental Response Trust
35 East Wacker Drive, Suite 1550
Chicago, IL 60601

Re: **Tronox LLC (TRX) Facility**
Nevada Environmental Response Trust (Trust) Property
NDEP Facility ID #H-000539
Nevada Division of Environmental Protection (NDEP) Response to: *Quality Assurance Project Plan, Reversion 0, Nevada Environmental Response Trust Site, Henderson, Nevada*

Dated: January 24, 2014

Dear Mr. Steinberg,

The NDEP has received and reviewed the Trust's above-identified Deliverable and provides comments in Attachment A. A revised Deliverable should be submitted **by 06/09/2014** based on the comments found in Attachment A. The Trust should additionally provide an annotated response-to-comments letter as part of the revised Deliverable.

Please contact the undersigned with any questions at wdong@ndep.nv.gov or 702-486-2850 x252.

Sincerely,

Dong

Weiquan Dong, P.E.
Special Projects Branch
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NDEP-Las Vegas City Office

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Attachment A

Essential Corrections

1. Precision

Sec 1.6.2, pages 8 – 9, Measurement Performance Criteria - Precision contains conflicting information.

A) Paragraph 2 mentions both duplicate control samples and laboratory control standard duplicates (LCSD). These are the same thing and are typically referred to as laboratory control sample duplicates (LCSD). The reference to duplicate control sample should be removed.

B) Paragraph 2 discusses using percent relative standard deviation (%RSD) and relative percent difference (RPD) values to assess precision, but only discusses control limits for RPD values. For clarity, the section should note that %RSD values are calculated when there are more than two replicates, and the values are comparable to the RPD values.

C) Paragraph 2 gives objectives of 30% for waters and 50% for solids and airs; however, two paragraphs later, laboratory control limits are referenced. In Tables 2-5, laboratory precision limits are provided for LCS/LCSD and MS/MSD analyses, as well as the 30%/50% limits in the "Duplicate" column. It should be clarified which limits (laboratory or QAPP) should be used to evaluate data. It appears that the 30%/50% limits are meant to apply to sample duplicates and field duplicates.

D) At the end of paragraph 2, it is stated that data may be "excluded from the data set" if the precision criteria are not met. This implies rejection of the data, but no guidelines are given as to what outliers would trigger rejection of the data. Most validation guidance documents do not recommend rejection of data based on precision outliers. Paragraph 4 only discusses qualifying data based on precision outliers.

E) The laboratory QA manuals are referenced for QC sample frequency of analysis. The project required frequency is presented in Table 6, which should be referenced here.

2. Completeness

Sec 1.6.2, page 9, Measurement Performance Criteria - Completeness contains potentially misleading information:

A) It is stated that "...data failing to meet DQOs have been removed from the data set..." Most data that fail to meet DQOs are estimated, but are still usable. Only data that have been rejected should be removed from the data set.

B) Completeness for a project is not only based on collected data, but on the number of planned analyses. If planned samples could not be collected or if collected samples could not be analyzed for some reason, this would also impact overall completeness.

3. Section 4.0, pages 33 – 36, Data Validation and Usability

There are several places in the document where verification is confused with validation. Verification applies to the checking of the completeness and correctness of the laboratory

deliverables (data packages and EDDs). Verification should be done by the laboratory prior to releasing data and may also be done by ENVIRON upon receipt of the laboratory deliverables. Validation applies to the evaluation of the data to determine if the laboratory followed the analytical methods and the laboratory SOPs, and to evaluate data usability based on the project specific DQOs in the QAPP. As mentioned in Sec 1.3, validation will be performed by independent contractors.

A) Sec 4.1, p 33, Data Review, Validation, and Verification Requirements – This section should state that the laboratory and ENVIRON will perform data verification. Validation will be performed by LDC and Neptune.

B) Sec 4.2.2, p 33, Procedures Used to Validate Laboratory Data – The laboratory will perform verification, not validation. Independent validation will be performed by LDC and Neptune.

C) Not present: The levels of validation (90% EPA Stage 2B and 10% EPA Stage 3/4), the QC elements reviewed for the different validation levels, and the guidance documents for validation (NDEP 2009b, NDEP 2009c, and EPA National Functional Guidelines) should be included in the Data Validation and Usability section.

D) Sec 4.3.1, p 34, Precision – For three or more replicates percent relative standard deviation (RSD) is used to evaluate precision, not just 'relative standard' as stated in this section. Since the RSD is multiplied by 100, the final value is "%RSD". Immediately below the equation, the acronym definition should be changed to %RSD (from RPD).

E) Sec 4.3.2, p 34, Accuracy – The division line in the %R calculation is missing.

F) Sec 4.3.2, p 34, Accuracy – SRMs are discussed here, but not anywhere else in the QAPP. If there is a chance that reference materials will be analyzed, they should be discussed as appropriate in the rest of the QAPP.

G) Sec 4.3.3, p 35, Completeness – In order to evaluate overall completeness, "T" should be defined as the number of ***planned*** measurements.

Minor Corrections

1. Sec 1.6.2, p 8, Measurement Performance Criteria, Accuracy – laboratory control sample and laboratory control standard (LCS) are mentioned. These are the same thing and are typically referred to as laboratory control samples (LCS).
2. Sec 1.8.6, p 12, Verification of Electronic Data – this section contains a reference to validation levels which is not related to verification. This information should be moved to the validation section.
3. Sec 1.8.7, pages 12 – 14, Electronic Data Deliverables (EDD)

   A) Appendix C should be referenced for the EQuIS format requirements.

   B) It is also recommended that spike levels, percent recoveries, RPDs, and control limits for %R and RPD should be added to the list of requirements for alternate format EDDs.

C) The percentage of results that must be verified during validation by comparison to the hardcopy should be specified.

4. Sec 1.8.8, pages 14 – 15, Laboratory Documentation

   A) The following items should be added to the bulleted list of requirements for a Level IV data package: detection limits, initial calibration summaries, calibration verification summaries, internal standard summaries, interference check standard summaries (metals only), serial dilution summaries (metals only), post digestion spike summaries (metals only), dilution factors, initial sample aliquots (weights or volumes), final sample volumes, sample preparation logs, sample run logs/injection logs, total solids

   B) In the 2nd paragraph on p. 15, the word "organic" should be removed from the last sentence. Except for surrogates, the QA/QC results listed apply to all analyses. The surrogate section already notes that surrogates only apply to organics.

   C) The two "Precision and Accuracy" bullets should be changed to "Matrix Spike/Matrix Spike Duplicates" and "Laboratory Control Sample/Laboratory Control Sample Duplicates". Both sections should note that the spiked results, percent recovery values, RPD values, and the associated recovery and RPD control limits should be reported. For MS/MSD, the parent sample results should also be included on the summary form.

   D) A separate bullet item for "Laboratory Duplicates", with required information of sample results, duplicate results, RPD values, and RPD control limits is recommended.

5. Sec 2.5.2.1, p 25, Method Blanks

   A) DI water is not used as a method blank for all tests/matrices (for example soils for SVOC or air samples). A method blank is "...a sample of a matrix similar to the batch of associated samples....". Or the verbiage from sec 2.5.2.2 could be used.

   B) In addition to the frequency requirement of 1 in 20 samples, the requirement of "...or one per preparation batch, whichever is more frequent..." should be added.

6. Sec 2.5.2.2, p 26, Laboratory Control Samples - In addition to the frequency requirement of 1 in 20 samples, the requirement of "...or one per preparation batch, whichever is more frequent..." should be added.

7. Sec 2.5.2.3, p 26, Matrix Spikes and Blank Spikes

   A) This section should be titled "Matrix Spikes" only. Blank spikes are the same as laboratory control samples and are discussed in the previous section.

   B) In addition, the same frequency verbiage regarding preparation batches as mentioned above should be added.

8. Sec 2.5.2.4, p 26, Laboratory Duplicates

   A) There are more than two types of laboratory duplicates – sample duplicates should be added to laboratory control sample duplicates and matrix spike duplicates.

   B) In addition, the same frequency verbiage regarding preparation batches as mentioned above should be added.

9. Sec 2.5.2.5, p 26, Surrogates – The identification of surrogates as "analyte isomers" should be changed to "chemically similar compounds" (such as bromofluorobenzene, or analytes containing deuterium such as toluene-d8, or ^{13}C isotopes like ^{13}C-2,3,7,8-TCDD). These are not isomers.

10. Table 1 - Analytical Methods and Laboratories

    A) The analytical method for Organic Acids should provide a reference to the Lab SOP and/or HPLC.

    B) Total Dissolved Solids (TDS) is not a soil test. If it is associated with SPLP, then the matrix should be soil leachate.

    C) Total Suspended Solids (TSS) is not a soil test. If it is associated with SPLP, then the matrix should be soil leachate.

    D) A matrix of TCLP is only associated with EPA method 1311. EPA method 1312 is for the SPLP method.

    E) The analytical method for mercury for a leachate would be 7470A, not 7471A.

    F) Was the reference to EPA 600 series intentional for the water matrix SVOC and OP Tests? No other 600 series tests were specified, the SW846 methods are applicable to aqueous matrices and typically have more robust QC requirements, and the 600 series was not included in the references.

11. Table 2 – Soil Analytes and Analytical Quality Control Criteria

    A) It should be specified if the Duplicate RPD column applies only to sample duplicates and field duplicates. See the comments above on the precision section also.

    B) No surrogate is specified for the OP pesticides (Method 8141A).

    C) It is recommended that the BZ# be added for the PCB congeners to avoid confusion when dealing with the IUPAC names.

    D) The labeled compounds and recovery control limits for dioxins and PCB congeners should be added to the table.

    E) No surrogate is specified for the organic acids.

    F) Tests associated with SPLP analyses (TDS, TSS, etc.) should be removed from the soil table.

    G) A method reference, such as Lab SOP by HPLC, should be added for Organic Acids.

    H) In the field sampling plan (FSP), Section 3.1.5, under the paragraph for Area 6 there is a discussion of the analysis of hexachlorobenzene (HCB) analyzed as part of the

organochlorine pesticides (OCP) group. This is confirmed by Table 2 in the FSP. However, in the QAPP Table 2, HCB only mentioned as part of the SVOC (8270) analytical suite. HCB needs to be added to the OCP analytical suite.

I) Footnote (4) states that dioxins are reported to the EDL. This is typically true for PCB congeners, also. The footnote should include a reference to PCB congeners.

J) Footnote (4) also discusses the calculation of TEQ values for dioxins. Many users of PCB congener data also calculate TEQs for the 12 congeners specified by WHO. The end use of the PCB congener data should be considered, and if appropriate, PCB congeners should be added to the discussion in this footnote.

12. Table 3 - Soil Gas Analytes and Analytical Quality Control Criteria:

A) It should be stated that the Duplicate RPD criteria applies to sample duplicates and field duplicates.

B) The LCS/LCSD RPD criterion is "N/A" for several analytes. All other analytes have a criterion of 25%. This criterion should apply to all analytes.

C) The RPD criterion of 200,000 for the analytes referenced under method SW8260B does not make sense.

D) Surrogates and control limits should be added for the SW8260B analysis.

13. Table 4 - Soil Leaching Analytes and Analytical Quality Control Criteria

A) It should be specified if the Duplicate RPD column applies only to sample duplicates and field duplicates.

B) The SPLP soil leaching method (EPA 1312) should be noted to avoid confusion with TCLP (EPA 1311).

C) The labeled compounds and recovery control limits for dioxins and PCB congeners should be added to the table.

D) Surrogates and control limits should be added for methods 8141A, 8082, and Organic Acids.

E) A method reference, such as Lab SOP by HPLC, should be added for Organic Acids.

F) It is recommended that the BZ# for PCB congeners also be included to agree with how these analytes are listed as COPCs in the RIFS Work Plan.

G) See comment 11I above regarding PCB congener EDL values.

H) See comment 11J above regarding PCB Congener TEQ values.

14. Table 5 – Groundwater Analytes and Analytical Quality Control Criteria

A) It should be specified if the Duplicate RPD column applies only to sample duplicates and field duplicates.

B) See comment 10F above regarding the 600 series methods.

C) Surrogates and control limits should be added for the SVOCs, OC pesticides, and Organic Acids.

D) A method reference, such as Lab SOP by HPLC, should be added for Organic Acids.

15. Table 6 - Frequency of QA/QC Samples:

A) Performance/Blind Check Samples are listed in the Accuracy Control Sample Section, however these are not mentioned anywhere else in QAPP or FSP. If Performance/Blind Check Samples are not going to be submitted, they should be removed from this table.

B) Field replicate frequency is not necessarily related to analytical batches. The frequency is based on sample collection.

C) Matrix spikes and matrix spike duplicates should be analyzed in each batch, where applicable to the method.

D) The first sentence for footnote (2) does not make sense. Also, soil gas analyses are not the only tests that do not have matrix spikes (radionuclides, TSS, TDS, etc.).

16. Table 7 – Sample Preservation, Containers, and Holding Times

A) "Volatile Organic Acids" should be changed to "Organic Acids".

B) A method reference, such as Lab SOP by HPLC, should be added for Organic Acids.

C) TAT should be defined in the footnotes.

D) As a general comment, "plastic" should be replaced (or footnoted) with "HDPE" (high density polyethylene) or similar.

E) Soil VOC and GRO preservation requirements appear to be based on EPA Method 5035. That method also specifies sodium bisulfate as a preservative in addition to DI water for low level analyses. Freezing is only required for highly alkaline or calcareous samples, which may react with the preservative and keep the pH >2.

F) It should be specified that the VOC and GRO DI preserved samples are for low level analyses and methanol preserved samples are only appropriate for high level analyses.

G) EPA methods no longer have such restrictive holding times for PCBs and dioxin/furans. With proper storage, the sampling to extraction and extraction to analysis holding times can each be extended to one year. This should at least be added to the tables as a footnote.

H) TDS and TSS are not performed on soil matrices. As a general comment, it might make more sense to add "Leachate" as a matrix in this table where appropriate.

I) The sample matrix for TO-15 Tedlar bags should be Soil Gas.

J) Footnote (4) states that sulfur is a rare earth metal. Should platinum be added?

K) The footnotes have a definition for TCLP, however SPLP (method 1312) is the leaching method specified.

17. Table 9 - Analytical Laboratory Calibration Frequencies

A) Two different sets of calibration requirements are presented for VOC by 8260B. Even if different labs are analyzing samples by this method, the calibration requirements are the same. The second entry should be removed.

B) Same comment about the 600 series methods. Note that here Method 608 is not cited – if the 600 series methods are retained, then 608 should be cited for the OC pesticides to match Tables 1 and 5.

C) Method 7470A should be added for mercury. A separate entry is not needed as the calibration requirements are the same for both methods.

18. References - it is recommended that the following documents be added:

A) NDEP 2011 Guidance on Qualifying Data Due to Blank Contamination, July 18

B) NDEP 2012 Guidance on Qualifying Data Due to Blank Contamination, Rev 2, November 23

C) NDEP 2012 Guidance for Data Validation of Asbestos in Soils, July 24

D) EPA National Functional Guidelines for Superfund Organic Methods Data Review (June 2008)

E) EPA National Functional Guidelines for Inorganic Superfund Data Review (January 2010)

F) EPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDD) and Chlorinated Dibenzofurans (CDF) Data Review (September 2011)

Editorial Changes

A) FTable 2, p. 6 of 20 – EPN (Ethyl P-Nitorphenyl Benzenethiophosphate). Change to Nitro.

B) Table 2, p. 11 of 20 – 3,4,4',5-TeCB6. Remove the "6"

C) Table 4, p. 5 of 8 – DeCB3. Remove the "3".