Inspection of Post-Pilot Study Activities at the Disa Technologies, Inc. Process Laboratory

A. Before the Disa Technologies, Inc. (Disa) process laboratory inspection, Tetra Tech, Inc. (Tetra Tech) sent Disa an email indicating areas of interest during the facility visit as follows:

> *"For my facility visit next week, I will be reviewing compliance with processes and records at outlined in the project SAP/QAPP [sampling and analysis plan and quality assurance project plan]*
>
> - *Section 4.3 - Quality Assurance and Quality Control*
> - *Section 4.4 - Sample Handling and Custody*
> - *SAP/QAPP Attachment A-1 Field and DISA processing SOPs [standard operating procedures]*
>
> *I will also want to discuss how we will want to present results, as outlined in SAP Sections 5.4.2 to 5.4.4, in the treatability study report. We will want to take care of as much field and DISA facility documentation as possible while waiting on lab and data validation results.*
>
> *It would also be a good idea to provide in the treatability study report drawings, descriptions, and anticipated operational ranges for 5 ton/hour system, including feed, screen/crushed, water recycle, dewatering, etc.*
>
> *This will help bolster the economics study that you will be preparing and show path forward for technology."*

B. Tetra Tech conducted an inspection of the Disa process laboratory in Casper, Wyoming, on September 21 and 22, 2022 to:

- Observe facility equipment and cleanliness.
- Observe facility staff conducting treated material post-processing.
- Review sample custody procedures.
- Review logs tracking all sample mass through processing.
- Review X-ray fluorescence (XRF) calibration logs.
- Review gamma calibration logs.
- Review personal protective equipment (PPE) use and decontamination equipment.
- Review compliance with Disa SOPs in the SAP/QAPP.
- Review incoming and in-process materials handling and storage.
- Review post-processing waste material handling (filter, water, waste back to sites, and Pace Analytical Services, LLC (Pace) and Eagle Engineering materials return and disposal).

- Observe filtration, wet sieving, drying, and subsampling and compositing.
- Review reused item (sieves, ring and puck mill, trays, and pans) cleaning procedures.
- Review information records (logbook, forms and sheets, email communications, deviations from SAP/QAPP, and field logs).
- Review analytical priorities in the event of mass limitations.
- Take photographs and videos of equipment and material processing steps.
- Discuss the treatability study report outline.
- Discuss scale-up plans.

C. Inspection observations, findings, and corrective actions are discussed below. A photographic log of Disa processing laboratory equipment, operations, and processed materials is provided in Attachment D1.

1. Observe facility equipment and cleanliness.

   The Disa process laboratory is in a new building that contains new or well-maintained fixed equipment, field equipment, and high-pressure slurry ablation (HPSA) batch unit and full-scale component equipment. The facility is kept clean, and staff clean up as they work. Radiological samples are stored and processed in a dedicated area separate from other projects at the laboratory.

2. Observe facility staff conducting treated material post-processing.

   Tetra Tech observed Disa staff walking through steps conducted during material post-processing at the facility. Tetra Tech observed Disa staff opening post-treatment bulk sample buckets, conducting wet sieving of post-treatment samples, filtering fractionation water, handling filter paper with fines, adding passing 270-mesh material with pre-cut materials, drying samples in ovens, and discussing sample splitting. Tetra Tech observed cooler packing, chain-of-custody forms, and cooler seals for a water shipment ready to send to Pace were observed, decontamination procedures and the HPSA batch unit inside the facility and the full-scale vibratory screens outside the facility were inspected. No items requiring corrective actions were noted.

3. Review sample custody procedures.

   Disa provided chain-of-custody forms for post-treatment samples leaving the three pilot study sites and logging into the Disa facility. Disa also provided chain-of-custody forms for samples submitted to Pace. Tetra Tech suggested adding the purchase order number to the forms for proper tacking and billing by the laboratory and signing across closure tape. No other items requiring corrective actions were noted.

4. Review logs tracking all sample mass through processing.

   Disa maintains both written logs and electronic tracking spreadsheets of sample mass processed at the laboratory. Disa prepared a material tracking flowchart for the SAP/QAPP. Tetra Tech and Disa discussed deviations from the flowchart because of a pre-cut step (removal of passing 270-mesh materials) to improve removal efficiencies. The pre-cut step requires recombining pre-cut mass with other passing 270-mesh samples after fractionation processing. Disa will include a modified spreadsheet in the report. No other items requiring corrective actions were noted.

5. Review XRF calibration logs.

   Disa provided logs for inspection and described initial, ongoing, and final calibration and reference check procedures. No items requiring corrective actions were noted.

6. Review radiation frisking equipment calibration logs.

   Disa provided logs for inspection and described frisking procedures. No items requiring corrective actions were noted.

7. Review PPE use and decontamination equipment.

   Level D PPE was used except when handling dry materials capable of generating dust when a half-face respirator with P100 cartridges was used to prevent dust inhalation. Eye protection and nitrile gloves were used at all times when handling material in the laboratory. General decontamination was dry brushing followed by soap and water and a water rinse. No items requiring corrective actions were noted.

8. Review incoming and in-process materials handling and storage.

   Disa demonstrated the steps for acceptance, storage, and tracking of materials before processing at their facility. All intake materials were stored and clearly labeled in a dedicated project area. All materials were logged in following chain-of-custody procedures and also into Disa sample tracking spreadsheets. No items requiring corrective actions were noted.

9. Review post-processing waste material handling (filter, water, waste back to sites, and Pace and Eagle Engineering materials return and disposal).

   Tetra Tech observed the storage of fractionation and process water and disposal of filter paper and the process for returning unused waste materials to each site. Pace and Eagle Engineering will return soil material to Disa. Disa will, in turn, transport samples back to the sites for disposal. Pace will dispose of water samples. No items requiring corrective actions were noted.

10. Observe filtration, wet sieving, drying, and subsampling and compositing.

    Tetra Tech observed Disa staff conducting wet sieving using RO-TAP equipment and a range of sieve sizes for a post-treatment sample. Disa staff were careful to recover mass from each sieve through rinsing and vibration. Sieves were decontaminated between each treatment sample. Disa also sieved samples from low to high concentration to further reduce any carryover concerns.

    After fractionation, Tetra Tech observed Disa staff dry fractionation samples in dry ovens, set up the fractionation water filtration, and pull a filtered sample off of the filtration station. Tetra Tech documented 5-micron water filtration equipment and observed Disa staff set up equipment for filtering fractionation water and recover and process a filter containing passing 270-mesh material. Tetra Tech discussed subsampling with Disa, including use of a sample splitter as discussed in Item 11 below. No items, other than the sample splitter, requiring corrective actions were noted.

11. Review reused item (sieves, ring and puck mill, trays, and pans) cleaning procedures.

    Reusable equipment was decontaminated with a dry brush, soap and water with a wet brush, water rinse, and ultrasonic cleaning followed by a second water rinse. One concern noted was ensuring the adequacy of decontamination of the sample splitter and the potential loss of sample mass in its damp corners because it is difficult to dry without a drying oven between samples. Tetra Tech and Disa agreed upon a different method of sample splitting to avoid any potential cross-contamination, which is described in meeting notes and emails with Disa staff in Section D below. No other items requiring corrective actions were noted.

12. Review information records (logbook, forms and sheets, email communications, deviations from SAP/QAPP, and field logs).

    Disa provided all paperwork (field and laboratory logbooks, forms, and sheets) for inspection and demonstrated how they also track information in Excel spreadsheets. Reviewed information was ordered logically in a work-step manner. Disa also had workflow sheets of each step in the processes to ensure no step was skipped. All recorded information reviewed was satisfactory. Disa agreed to document in the treatability study report any deviations from SOPs and sample type, count, and methods from the SAP/QAPP. No other items requiring corrective actions were noted.

13. Review analytical priorities in the event of mass limitations.

    After discussions with the staff, Disa indicated the sample mass for some size fractions are inadequate to support analytical requirements of the Pace and Eagle Engineering methods. Disa agreed to prioritize analyses supporting primary project objectives before secondary objectives. Combining masses of size fractions to obtain the minimum mass requirements was also discussed and resolution of the issue is summarized in Section D.

14. Take photographs and videos of equipment and material processing steps.

    Tetra Tech documented equipment and material processing steps during the facility inspection. A photographic log is included in Attachment D1. Videos are not included in the report because of file size but are available upon request from the U.S. Environmental Protection Agency task order contract officer representative.

D. Summarized notes from conversations with Disa staff regarding observations, suggestions, and corrective actions are provided below along with an email record regarding sample mass issues and resolution.

   Action Items - September 21, 2022 Meeting:

   - If sample mass is low, prioritize the synthetic precipitation leaching procedure (SPLP) over the toxic characteristic leaching procedure (TCLP).
   - Analyze TCLP leachate for mercury but omit analysis for mercury in the solid samples.
   - Indicate that a 0.45-micron filter was used in the notes section of the CR-M-0-SL-01 fractionation sample where fractionation water was filtered.
   - For the report, split the current sample tracker Excel sheet for each site into 3 PDF pages.

- Add the purchase order number to future chains of custody for Pace.
- When the laboratory receipt form is filled out and emailed to Disa, combine the PDF chain-of-custody form, bill of lading for the shipment, and laboratory receipt form into a single document for tracking purposes.
- Attach FedEx receipts to the shipment document PDFs for samples shipped to Eagle Engineering.
- Sign across the duct tape on the coolers as a custody seal for future shipments. Continue to take pictures as with previous shipments.
- Calculate the mass required for minimum sample analyses (Pace needs 1 gram for metals, 200 grams for TCLP, 200 grams for SPLP, 300 grams for radium-226 (Ra-226); Pace provided a table summarizing the Ra-226 minimum detectable concentrations for corresponding masses on September 22, 2023).
- Note the samples for which no water was left over after filling water bottles to Pace. Take pictures of all water bottle samples.
- Note that the slurry samples retrieved in the field after HPSA processing have rip tabs to avoid tampering and ensure custody.
- Save all decision emails to document deviations from the SAP/QAPP.
- Create a document logging all deviations from the SAP/QAPP.
- Create a document summarizing key field notes that can then be analyzed easily.

<u>Action Items - September 22, 2022 Meeting:</u>

- Calculate the mass loss on rinsed and dried filter paper after caked solids were removed and combined with the QV-L-4-SY RO-TAP sample.
- Riffle splitting samples for the 0.25, 0.25, 0.5 mass split in the SAP/QAPP may cause some contamination, especially with the fine fraction material. Do not split the fine fraction material with this splitter. Coarse fraction material may be okay to split in this manner as long as the rifle splitter is cleaned and the material is split from lowest to highest potential concentration. The email dated September 28, 2022, below describes the revised sampling slitting approach.
- Further break down the flakes from the passing 270-mesh fraction samples before sample splitting. Disa staff concluded that ring and puck milling would work best for dried filter cake without losing sample mass or cross-contaminating samples.

<u>Email From Disa on September 28, 2022, Regarding Sample Splitting Methodology</u>

“As I see it, the manner in which the samples are split for analysis by Pace should be gauged on these three criteria in order of priority:

1. *Preventing cross contamination between samples*
2. *Getting the correct mass for the respective splits*

3. *Unbiased splitting of the samples*

While the riffle splitter will give us a higher probability of unbiased sampling, to your point I think it should be lower on our priority list due to the outweighing factor of potential for contamination. This is especially true due to the small sample masses we have, where a little bit of contamination in the nooks and crannies of the splitter could go a long way in throwing the results off.

As such, I propose a combination between Variant B and Variant C in our SOP ***without the use of the riffle splitter*** *which will call for the following:*

1. *Place a clean sample cup on the balance and tare the balance. Label the cup as "SPLP Charge" with the proper sample labeling before it. Ex.: "CR-L-4-SY Combined +25/+270 SPLP Charge"*
2. *Homogenize the sample bag containing the +25-mesh fractionated sample manually by mixing the material in the bag while the bag is closed.*
3. *Pour (or scoop with a clean/disposable spoon) the sample from the bag into the cup until the scale mass reading matches the mass needed for the required split of the fraction as detailed in the Excel document "2022 09 28 RAES TO33 Sample Splitting Goals." This should be half the mass of the originally collected sample for the +25-mesh fraction.*
4. *Record the actual mass as shown on the balance (nearest hundredth of a gram) for comparison against the goal mass.*
5. ***DO NOT at any point in this procedure, place material from the cup back into the bag which it came from.***
6. *Tare the balance with the cup containing the +25-mesh material split.*
7. *Homogenize the sample bag containing the +50-mesh fractionated sample manually by mixing the material in the bag while the bag is closed.*
8. *Pour (or scoop with a clean/disposable spoon) the sample from the bag into the cup until the scale mass reading matches the mass needed for the required split of the fraction as detailed in the Excel document "2022 09 28 RAES TO33 Sample Splitting Goals." This should be half the mass of the originally collected sample for the +50-mesh fraction.*
9. *Record the actual mass as shown on the balance for comparison against the goal mass.*
10. *Tare the balance with the cup containing the +25-mesh material split and +50-mesh material split.*
11. *Repeat this until the "SPLP Charge" cup contains the proper half splits for the size fractions of +25- through +270-mesh.*
12. *Label a bag with the proper nomenclature for the sample. Example as above: "CR-L-4-SY Combined +25/+270 SPLP Charge." On a zeroed scale, record the tare mass of the sample bag.*
13. *Pour the cup into the bag. Record the gross mass on the bag as well as the net mass by calculation. Compare this to the summation of the previous splits poured into the cup. Once clean, dispose of the sample cup used for combination of the size fractions.*

14. *Repeat the process with a new clean cup, splitting only one quarter of the mass as dictated by the excel document "2022 09 28 RAES TO33 Sample Splitting Goals." This sample will be labeled per the same example as: "CR-L-4-SY Combined +25/+270 MLA Charge."*
15. *The remaining sample in the original sample fractionation sample bag should be ¼ split and will be used in metals and Ra 226 analysis.*

Let me know what you think. I also think this will be more accurate in getting the correct masses for the respective splits to the second item on the priority list above. There is the most potential for a biased sample when splitting the +25-mesh fraction in this manner. However, the other size fractions should be easily homogenized for unbiased splitting directly from the sample bag."

Email From Tetra Tech on September 28, 2022, Accepting Sample Splitting Methodology

"I am fine with the proposed process, except that I would propose collecting volumes for lab assay of metals and Ra-226 first, MLA [mineral liberation analysis] second, and SPLP third. If anything gets short-changed on mass, it should be SPLP as it is already going to go through a dilution step and is not as critical and the lab assay."

ATTACHMENT D1: PHOTOGRAPHIC LOG

Photo 1 September 21, 2022 Receiving area for bulk and post-treatment samples	
Photo 2 September 21, 2022 Post-treatment sample buckets awaiting processing	

Photo 3 September 21, 2022 Intact zip seal on post-treatment slurry sample bucket	
Photo 4 September 21, 2022 Broken seal on post-treatment slurry sample bucket	

Photo 5 September 21, 2022 Five-micron filtered fractionation water in tote	
Photo 6 September 21, 2022 RO-TAP wet sieving setup: Oscillating motor creates a vibration; material is placed in the sieve, the water and finer material pass through the sieve into bucket, and the larger sized material stays on the sieve screen	

Photo 7 September 21, 2022 Transfer of solids captured on the top of the sieve into the red sample tray for drying; fines in bucket will be captured on 5-micron filter paper	
Photo 8 September 21, 2022 Wet fractionation samples retained on six sieve mesh sizes after wet sieving and before sample drying; coarsest samples in upper left, and finest in lower left	

Photo 9 September 21, 2022 Overview of water filtration station for fractionation water and solids-water separation of treated slurry samples	
Photo 10 September 21, 2022 Pressure filtration apparatus from bottom to top: base, mesh screen, filter support cloth, and 5-micron filter paper	

Photo 11 September 21, 2022 5-micron filter with filtered solids on top of paper before drying	
Photo 12 September 21, 2022 Overview of the wet filter cake removal from the filter paper after solid-water separation to be recombined with the applicable size fractions	

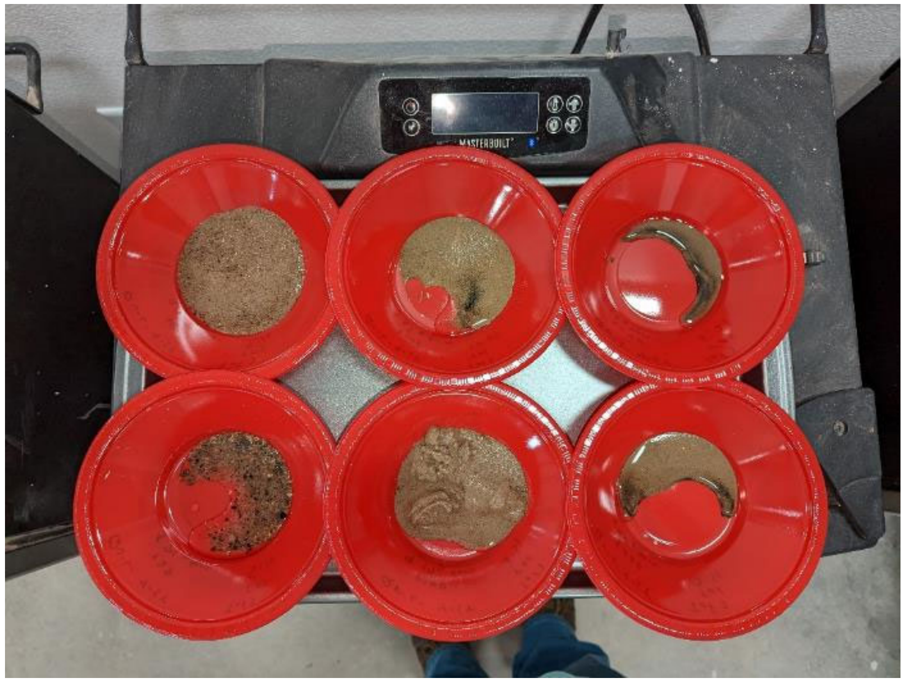
Photo 13 September 21, 2022 Wet fractionation samples to be weighed before oven drying; coarsest samples in the bottom left and finest in the upper right	
Photo 14 September 21, 2022 Drying oven with a sample tray added	

Photo 15 September 21, 2022 Inside of drying oven with three trays of dried samples in cups; bottom tray contains dried fine (passing 270 mesh) fraction on 5-micron filter paper	
Photo 16 September 21, 2022 Tray of dried samples to be weighed again to assess moisture loss and dry solids weight; coarse sand in lower left and very fine sand in upper right; silts and clays in passing 270-mesh size fractions	

Photo 17 September 21, 2022 X-ray fluorescence (XRF) testing stand with hood; the XRF analyzer is underneath the stand; the sample is placed on the stand with the hood closed and the XRF analyzer is activated	
Photo 18 September 21, 2022 Olympus Vanta Reflex series XRF analyzer attached below the testing stand	

Photo 19 September 21, 2022 XRF calibration blank (left) and uranium standard reference cup (right)	
Photo 20 September 21, 2022 Weighing of composite sample for synthetic precipitation leaching procedure (SPLP) extraction at Pace Analytical Services, LLC (Pace)	

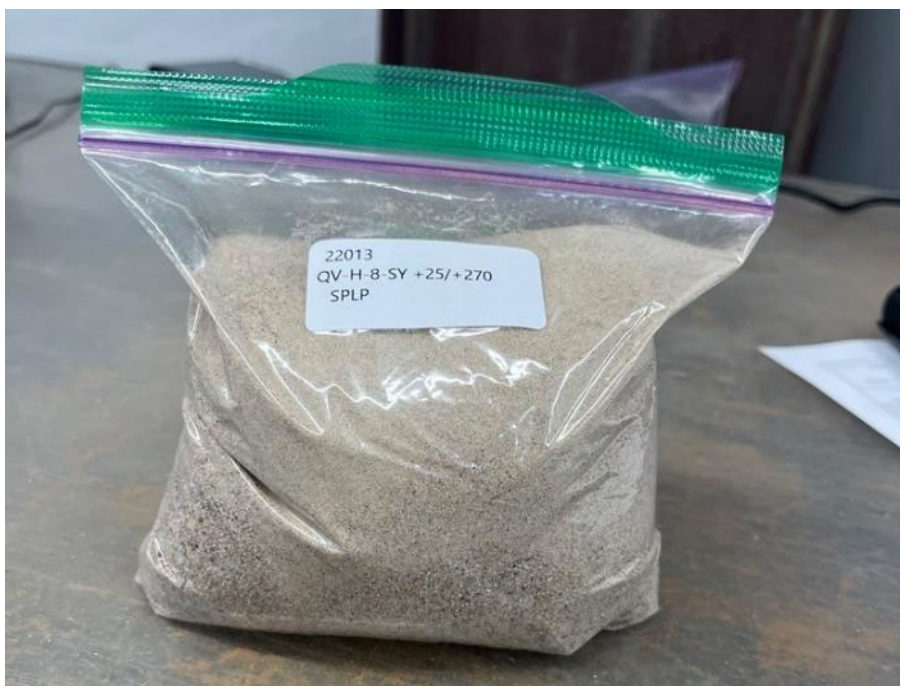
Photo 21 September 21, 2022 Fractionation samples from the Quivira Church Rock 1 Mine (CR-1), high concentration location prepared for shipment to Pace for analysis	
Photo 22 September 21, 2022 Composite sample from the Quivira CR-1 high concentration location ready for SPLP extraction at Pace	

Photo 23 September 21, 2022 Ring and puck grinder used to break up dried filter cake from a 5-micron filter	
Photo 24 September 21, 2022 Bulk samples ready for shipment to Eagle Engineering for mineral liberation and X-ray diffraction analysis	

Photo 25 September 21, 2022 Dried filter cake material passing 270-mesh sieves to be broken up with ring and puck mill before sending to Pace	
Photo 26 September 21, 2022 Filtered water samples from Quivira CR-1 low concentration material processing ready for shipment to Pace for analysis	

Photo 27

September 21, 2022

High-pressure slurry ablation (HPSA) batch treatment unit used during the pilot study; slurry transfer pumps are gold and blue at the bottom left and right of the skid

Photo 28

September 21, 2022

Slurry is pulled from the bottom of the cone tank and transferred via blue pipes to the collision chamber above the cone tank

Photo 29

September 21, 2022

Close up of one of the slurry transfer pumps

Photo 30

September 21, 2022

Side view of the blue transfer pipe to the collision chamber above the cone tank

Photo 31 September 21, 2022 Collision chamber above the cone tank with feed lines in the foreground and background	
Photo 32 September 21, 2022 Viewing panel on one side of the collision chamber	

Photo 33 September 21, 2022 Close up of the feed line and pressure sensor on one side of the collision chamber	
Photo 34 September 21, 2022 Side view of the vibratory wet screen used for full-scale fractionation used to segregate concentrates from the treated clean materials	

Photo 35

September 21, 2022

Front view of the vibratory wet screen; two vibratory motors (white) used for shaking the screen panels on the top of the screen; clean water flushes any contaminated process water from the clean material fraction using the green spray bar at the top of the screen

Photo 36

September 21, 2022

Disa Technologies, Inc.'s (Disa) in-house material processing equipment with various size reduction equipment

Photo 37 September 21, 2022 Disa’s in-house dry oversize material vibratory screen	
Photo 38 April 7, 2023 HPSA 5-ton per hour (TPH) skid-mounted continuous treatment unit during fabrication with three cone bottom process tanks; HPSA collision chambers mount on top of each tank	

Photo 39 April 7, 2023 HPSA 5-TPH skid-mounted process water tank and base for wet screen to be mounted on top of skid during fabrication