



EXHIBIT D:

Standard Operating Procedures for Sampling After RO-TAP Material Processing

1. Scope

This SOP covers the material preparation for assay analysis by a third-party lab of samples taken from the HPSA batch unit during operation and previously processed through the RO-TAP for size fraction separation. For the SOPs covering the performance of HPSA batch unit testing/collection of samples from the batch unit as well as the sieve separation of the material through RO-TAP sample processing, refer to Exhibit A and Exhibit C, respectively.

2. Terminology

Assay – Third-party analysis of collected samples. This may be through a variety of methods depending on the requirements for the testing.

Sample – Refers to an assayed sample as outlined in the procedure.

Sample Cup – Refers to the sample cups collected and correctly labeled as described in Exhibit C.

Sample Bag – Sandwich size bag for collection and storage of samples. Sample bags should be properly labeled with the test they were collected from, what time sample they are a part of, and if applicable the size fraction they represent from the sieved separate size fraction or combined size fraction of the sample.

Bulk Reserve – Denotation for samples to be kept at Disa's HQ if further replicates or other analyses are required for the sample.

Time Sample Bag – In assay of separate size fractions collected from samples, these gallon or larger Ziploc bags should contain the separate size fractions for the appropriate test and time sample they correspond to. In the case of separate size fraction assay of all samples at a collected time from the batch unit, the chain of custody (CoC) should be included in the shipment of the bag along with the individual size fraction sample bags.

Sieved Sample – Refers to the sample separated into the sieved size fractions as described in Exhibit C. This may be referred to throughout the procedure in the past tense (prior to sieve separation) or in the present tense (after sieve separation and drying) since the samples are identical and have only been separated through a physical process.

XRF – Refers to the bench top X-Ray Fluorescence device used throughout the initial spot check of the procedure.

XRF Sample – Refers to small sample split from original samples for the corresponding size fraction, contained in the XRF sample cup.

XRF Sample Cup – Small sample cup containing 5 to 10 grams of collected split sample for XRF spot check analysis.



Spot Check – Also referred to throughout the procedure as spot check analysis. This analysis is to determine a baseline understanding of the material to be sent for assay. It may educate the combination of size fractions for combined size fraction analysis or may be used as a comparison against the assays for assay of separated size fractions. Spot check analysis should never be used as a substitute for third-party assays and is often only used internally by Disa.

3. Summary of Test Method

This testing method comprises the standard operating procedures for collection of data for mass distributions from the prior sieve separations for the material fractions as well as assay of the sample after logging of mass distribution data by A: separate size assay of each individual fraction, B: combined size fraction assay, C: combination of separate size assay and assay of combined size fractions collected from the procedure described in Exhibit C. In general, all sample size fractions are initially analyzed using a bench top XRF for initial bearing on the concentrations of constituents, as well as verified either by assay of the individual size fraction or a combination of multiple size fractions for the specific class.

4. Apparatus

In general, a device (computer or other) is required for logging of data, a bench top Olympus Vanta XRF, and a weight scale for reporting of weight to the nearest hundredth (0.01) of a gram. Further, as described in Variant C of this procedure, a small bench-top riffle splitter may be required with a capacity of approximately 1000 grams of material in each side of the riffle split cups.

5. Sampling

Care should be taken when transferring the material from sample cups to reduce sample cross contamination for accurate assay results from a third-party laboratory. Sample bags should be properly labeled prior to transfer of the sample from the sample cup into the sample bag according to the test they were collected from, the time which they were collected at, and what size fraction the sample represents from the total sieved sample. Sample masses should be recorded while the samples still reside in the sample cups, factoring in the tare mass of the sample cups as originally labeled by Exhibit C for a total net mass of the sample, provided that the sample cups were labeled correctly. If the sample cups were labeled incorrectly or do not include labeling information on the tare mass of the sample cup, place the sample bag on the weight scale and tare the scale. Transfer, as best as possible, all the sample contained in the sample cup to the sample bag on the scale. Record this as the sample's net mass.

Gloves may remain on for the transfer of samples from the sample cups to the sample bags for the duration of a time sample (through the entire sieve set). However, between time samples, gloves should be exchanged for fresh gloves to ensure as little cross-contamination of time samples for a particular test as possible.

The XRF is used in this procedure only as a spot check of the material. No material is pulverized throughout the course of this SOP. While this may lead to inaccuracies from the XRF data due to non-uniformity of the sample in the XRF cup, the XRF is used as a baseline to understand the material and its constituent distributions and should not be used as a substitute for third-party assay analysis. By pulverizing the material prior to XRF and assay, the material will be altered and may provide inaccurate results for assay analysis, specifically for leaching tests that may be performed. When collecting samples in XRF cups for the initial spot checks, ensure that the sample bag is tightly closed. Mix this bag prior to collection of the XRF sample in a small XRF cup to ensure, as best as possible, uniformity prior to sampling and analysis.



As described in variant C of this procedure, some samples may require splitting through riffle splitting. Prior to riffle splitting, ensure that there are no contaminants on the riffle splitter or riffle splitter cups. If contamination is present that might lead to inaccurate assay results, wipe the riffle splitter thoroughly with disinfectant wipes until no more contamination can be visibly seen in the riffle splitter. Allow the riffle splitter to dry prior to riffle splitting to prevent riffle split material from becoming wet and sticking to the sides of the riffle splitter.

6. Procedure

General procedure for logging of mass distributions and XRF for separated sample size fractions through the procedure described in Exhibit C:

- a. Once material has been allowed to dry for a sufficient period, remove the sample cups from their drying racks in the oven.
- b. With a weight scale capable of reporting the mass in grams to the nearest hundredth of a gram (0.01), place the sample cup on the weight scale.
- c. Record the mass shown on the electronic display of the weight scale and subtract the tare mass of the sample cup from this mass for the net mass of the material (typically logged in Excel or other Microsoft processing datasheet for ease of data analysis) for the specific mass of the sample reporting to that size fraction.
- d. Record the time spent on screen as labeled on the sample cup per the instructions in Exhibit C.
- e. Prepare a sample bag by writing in permanent marker, the test which the sieved sample was collected from, the size fraction which it represents, and the time at which the sieved sample was collected. Write the net mass of the sample on the sample bag and transfer as much of the material as possible from the sample cup to the sample bag. Close this sample bag tightly and store the sample bag in the correctly labeled time sample bag.
- f. Once all net masses of samples have been collected and the sample bags transferred to the proper time sample bag, analyze the recorded mass distribution data using Excel or another appropriate program.
 - a. If the sieved sample was dried prior to sieving, perform a summation on the collected masses of the size fractions and compare this summation to the dried sample mass by: $(\text{mass pre-sieved sample} - \text{mass summation of size fractions}) / (\text{mass pre-sieved sample})$ for a percentage difference that may have contributed to error in the RO-TAP procedure. NOTE: For dry sieved samples this value should be less than 1%.
 - b. From the summation of masses collected on each sieve, perform a balance on the percentage report by mass of the sieved sample for the appropriate size fractions it was separated into. As an example $(\text{net mass} + 10\text{-mesh fraction}) / (\text{summation of size fractions}) = \text{percentage report by mass of the sieved sample to that fraction}$.
 - c. With this information further analysis can be performed for cumulative percent passing a certain size fraction or cumulative percent retained on a size fraction. These values can additionally be used for fitting of particle size distribution curves/particle size shift curves over processing time for the specific test.
- g. For each collected sample fraction, collect a split of the sample in an XRF sample cup. Cap the XRF sample cup with mylar film and place the cup with the mylar film facing down on the beam of the XRF setup.
- h. Using the Vanta Desktop App for the XRF, analyze the sample through two replicates and record the average in Microsoft Excel or another appropriate data analysis program with the correct labelling for the retrieved results.



- i. Upon conclusion of spot check on the sample with the XRF, remove the mylar film from the XRF cup and return the sample contained in the XRF sample cup to the sample bag.
- j. Dispose of the mylar film and clean the XRF sample cup with soap and water.
- k. The sample is now ready for assay by any of the following variants.

Procedure variant A: Assay of all separated size fractions.

- l. With the samples in their respective sample bags, fill out the third party CoC according to the directions on the CoC with correct required analyses and note the recorded mass of the sample that is labeled on the bag for the specified sample.
- m. Place the completed CoC in the Time Sample Bag to ensure that the correct analyses are performed for the correct CoC.

Procedure variant B: Assay of combined size fractions.

- n. Depending on the results of the XRF or required material for particular analyses, material may need to be combined from the sieve separated size fraction samples.
- o. When combining, label the sample bag according to the test from which it was sampled, the time the sample was taken, and the size fractions that were combined for the sample.
 - a. Example: for the combined size fractions of +10-, +25-, +50-, and +100-mesh, label in permanent marker "+10/+100-mesh."
- p. Ensure that samples are combined in the proper proportions from their sieved fractions by transferring all the material from the sieve separated sample bags to the combined fraction sample bags.
- q. Record the net mass of the combined sample bag and compare this net mass to the summation of the previously recorded individual fractions for analysis of error in the combination of the size fractions for assay.

Procedure variant C: Combination of assay for both combined and separated size fractions.

- r. If both separate size fraction assay and combined size fraction assay are required for the specific sieved sample, a riffle splitter must be used to split the samples into roughly equal and representative proportions.
- s. Pour a specific size fraction sample from its sample bag container through the riffle splitter three times to ensure adequate mixing and collection of a representative sample for assay. Once the material has been riffle split three times, riffle split a fourth time and collect the split size fractions.
- t. Transfer the split samples into their respective sample bags for either combined size fraction analysis or separate size fraction analysis. On the separate size fraction analysis sample bags, note the date and time when the sample was split and how much of the net original mass remains in that size fraction. This will be used along with the data collected from the masses from each split into the combined size fraction sample bag to determine propagation of error from this riffle splitting.
- u. If a bulk reserve sample is required for the sample, split the material into fourths and label the sample bags according to which material will be kept as bulk reserve, combined for assay, or kept in individual size fractions for assay.
- v. By splitting the material into halves and fourths, combined size fractions and individual separate size fractions should retain their same mass report throughout the sample and should decrease the amount of error propagation associated with sample variance.

Post-Report Data Analysis:



- w. Once reports have been issued for the assay of separate size fractions, determine the percentage report of the element(s) of interest throughout the sieved material.
 - a. Overall concentration throughout the sieved size fractions can be determined using excel by a mass average of the assay concentration results using the “sumproduct” function.
 - b. Once the overall concentration of the sieved sample has been determined from the assays, this may be used to determine the percentage report of the element(s) of interest by:
$$(\text{Mass\% of size fraction}) \times (\text{Concentration size fraction}) / (\text{Overall concentration}) = \% \text{ report of constituent to the analyzed size fraction.}$$
- x. These results will be compared across time samples and tests for determination of the test producing the best results of recovery of the constituent(s) of interest.
- y. Combined size fraction assay results will be analyzed on a case by case basis.