![USGS - science for a changing world]

U.S. Geological Survey
Mineral Resources Program – Quality Management System
Technical SOP

MRP-SPECLAB-SOP-02.01
CONDUCTING SPECTRAL REFLECTANCE MEASUREMENTS
WITH THE ASD SPECTROMETERS

Reviewed by:

Approved by:

Digitally signed by TERESA
KIRSCHLING
Date: 2020.08.18 07:38:14 -06'00'

Quality Management System Manager

AMY BERN
Digitally signed by AMY BERN
Date: 2020.08.21 11:27:16
-06'00'

Science Center Director

GREGG SWAYZE
Digitally signed by GREGG
SWAYZE
Date: 2020.08.17 17:24:26 -06'00'

Laboratory Manager

Digitally signed by MICHELE
WOLF
Date: 2020.08.17 14:03:00 -06'00'

Laboratory Quality Assurance Specialist

Effective Date: 8/21/2020

MRP-SPECLAB-SOP-02.01 – Conducting spectral measurements with the ASD spectrometers

1. SCOPE, APPLICATION, AND SUMMARY

1.1. Summary

Spectral reflectance measurements of minerals, ices, and chemical reagents are a necessary component of Earth and planetary studies.   The ASD FS3®, FS4®, and FS4-NG® ultraviolet to near infrared spectrometers can be used to measure reflectance of materials over the spectral range from 0.35 to 2.5 µm at spectral resolutions ranging from 3 to 12 nm. This SOP is based on modifications and improvements on Swayze and others (2014) and Kokaly and others (2017).

1.2. Trademark Disclaimer: The use of trade, product, or firm names in this SOP is for descriptive purposes only and does not imply endorsement by the U.S. Government.

1.3. Table of Analytes, CAS Numbers, Reporting units, and Operational Range

Values are reported as unit less reflectance (0 to 1).

1.4. Interferences - NA

1.5. Laboratory Personnel Required to Use the SOP.

This SOP must be used by all analysts operating the ASD spectrometers while making routine spectral measurements of samples.

1.6. Demonstration of Capability (DOC) Requirements for the SOP

In order to produce data using this procedure, analysts are required to demonstrate competence and attain demonstration of capability (DOC) by the Laboratory Manager. Subsequent DOCs must be performed annually. Competence will be met when the analyst demonstrates to the Laboratory Manager that they can follow the SOP step-by-step and produce two repeatable consecutive spectral measurements of the MCREEmylar wavelength standard in reflectance that

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

overlay for all channels within ± 5% over the 0.35 to 2.5 μm wavelength range starting from the beginning of the SOP for each measurement without input cues from the Laboratory Manager.

2. HEALTH, SAFETY, AND WASTE DISPOSAL INFORMATION

Refer to the individual laboratory Chemical Hygiene Plan (CHP). The development of a CHP by the lab supervisor for each individual laboratory under his/her jurisdiction is required (29 CFR 1910.1450) by the Occupational Safety and Health Administration (OSHA). A hardcopy is available for immediate use by lab personnel. It is required that all lab personnel read, discuss, practice safety procedures listed within the CHP, and sign the training log within the CHP, signifying that they have read and understand procedures and rules. All lab personnel must understand what to do in the event of an emergency, and where within the CHP to find hazard and/or chemical information, including SDS information.

3. DEFINITIONS, ABBREVIATIONS, AND ACRONYMS

ASD = Analytical Spectral Devices
GDS = Geophysics Denver Spectroscopy sample collection
HS = Hunt and Salisbury sample collection
MCREEmylar wavelength standard = Multicomponent REE Labsphere® puck with a mylar film cover
μm = microns
PI = Principle Investigator
REFL = reflectance
SOP = standard operating procedure
White reference panel = 99% reflectance NIST-traceable Labsphere Spectralon® panel
WS = Ward Science mineral collection

4. SAMPLE PRESERVATION, HANDLING, CONTAINERS, ANALYTICAL PROCESSING/HOLDING TIMES, AND DISPOSAL

4.1. Preservation Requirements

Preservation of geologic samples for spectral measurements is not required beyond storage for future use if so determined by the PI.

4.2. Time Limits for Critical Stages of Analysis

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

Samples must be measured within 1 hour of the white reference panel measurement. If more than 1 hour has lapsed between planned white reference panel and sample measurements, then the white reference panel (i.e., Spectralon® panel) must be re-measured prior to sample measurements to maintain a time lapse of ≤ 1 hour, unless warranted by special sample circumstances, which must be noted in the instrument log book or Analyst's scientific notebook.

4.3. Sample Locations, Sample Retention, and Sample Storage Conditions

If samples are part of the official spectral collections (e.g., GDS, HS, WS, or other mineral standard collection) they must be returned to their designated positions on shelves housing those collections in the spectral laboratory. Storage of field samples is the responsibility of the PI who requested or carried out spectral measurements.

4.4. Sample Disposal

Sample disposal is the responsibility of the PI. Most samples are part of the spectral laboratory collections and will not be disposed of.   If samples are no longer of value, then disposal is the responsibility of the PI.

5. PREPARATION OF REAGENTS, STANDARDS, AND SOLVENTS

5.1. Reagents, Standards and Solvents Preparation, Storage and Shelf Life

Sample preparation is the responsibility of the PI. Some samples are ground to a PI-specified grain size prior to measurement and others are measured whole depending on what is dictated by research needs.   Still other samples may be measured at cryogenic temperatures depending on research needs. Preparation, storage, and observance of shelf life for chemical reagents that are themselves samples for spectral measurement are the responsibility of the PI.

5.2. Reagents, Standards and Solvents Documentation

Reagents are not used in spectral measurements unless they themselves are the subject of the spectral measurements.   Their labeling, preparation, safe handling, and disposal are the

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

responsibility of the PI.

5.3. Verification of Reagents, Standards and Solvents

If necessary (see Section 7.3.9), a spectrometer wavelength accuracy test is done prior to spectral measurement of samples to ensure the ASD spectrometer is performing up to specifications defined by the spectrometer software. The wavelength standard is a Multicomponent REE Labsphere® puck with a mylar film cover. Reflectance measurement of the wavelength standard is described by MRP-SPECLAB-SOP-08, which generates a report that gives a Pass/Fail grade for wavelength accuracy. If the calibration run fails, the calibration process must be repeated a second time. If it fails again, contact the Laboratory Manager for assistance to determination if adjustments are needed or if instrument maintenance is necessary. Solids in this laboratory are normally used for comparison purposes and do not expire unless marked. Liquid organic reagents in the laboratory are normally used for cleaning. If spectral measurements are made on organic liquid reagents, they will be validated before use. If a solid or liquid is used for a mixture or for a chemical reaction it will be noted in the instrument logbook or Analyt's scientific notebook.

6. APPARATUS

6.1. Lab Ware

Sample cups

5x5 inch 99% Labsphere Spectralon® Panel (i.e., white reference panel)

Adjustable optical bench

Illumination lamp (e.g., twin or triple 50-watt halogen lamps)

12v DC power supply for lamps

Computer power

Spectrometer power

Ethernet cable

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020

A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

Instrument computer

Multicomponent REE Labsphere® 2.4 inch round puck with mylar film

30-degree incident fiber optic holder

6.2. Instrumentation

ASD spectrometers (e.g., 18011, 18388, 18594, and 18683), which are controlled by a computer running RS³ software. The instrument serial and calibration numbers are automatically recorded in the digital manual history of each spectrum. The following instrument labels can be used in the instrument notebook and as character availability in the spectrum titles allows.

18011 = MRP-SPECLAB-ASD-18011

18388 = MRP-SPECLAB-ASD-18388

18594 = MRP-SPECLAB-ASD-18594

18683 = MRP-SPECLAB-ASD-18683

7. SAMPLE PREPARATION, ANALYSIS, AND INSTRUMENT OPERATION AND SHUTDOWN

7.1. Sample Preparation

Minerals, man-made materials, chemical reagents, cryogenically formed frosts, and other materials may be ground to a grain size determined by the analyst to satisfy the needs of the PI's experimental requirements. Note the grain size in the spectrum title if known in the instrument log book. Larger samples such as rocks can also be measured when the distance between the rock surface and spectrometer fiber optic (e.g., dependent on measurement setup) is visually judged to be the same as that used to measure the white reference panel. <u>Be extremely careful not to scratch the polished surface of the instrument's fiber optic with the sample.</u>

7.2. Sub-sampling - NA

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

7.3. Sample Analysis

7.3.1   Plug the ASD spectrometer power cable into a power source (120-volt electrical outlet or ASD battery).

7.3.2   Plug the instrument computer power cable into a power source (120-volt electrical outlet or computer battery). Make sure the last series of numbers printed in large letters on the cover of the computer matches the serial number printed on the side of the spectrometer (e.g., 18011, 18388, 18594, or 18683).   If the numbers do not match seek help in locating the appropriate instrument controller from the Laboratory Manager.

7.3.3   Gently place the fiber optical cable into the 30-degree incident (off-nadir from vertical) fiber optic holder. Be careful not to scratch the tip of the fiber optic. The fiber optic cable is composed of glass surrounded by a metal sheath, but it is still fragile. Do not let it drop or curve tighter than a 10-inch radius. <u>Replacement fiber optic cables cost several thousand dollars so handle them with care.</u>

7.3.4   Turn the illumination lamp on by flipping the on/off switch on the 12-volt power supply.

7.3.5   Turn the ASD spectrometer on and let it warm up for ≥ 1 hour. Two hours warmup is optimal and will allow longer periods of sample measurement between white reference panel measurements as described below.

7.3.6   After the ASD spectrometer is warmed up, carefully place the Spectralon® panel (i.e., white reference panel) on the adjustable optical bench and adjust its height so the distance between the tip of the spectrometer fiber optic and surface of the Spectralon® panel is equal to the intended working distance (e.g., dependent on what is being measured) between the fiber optic cable and sample surface. Note the working distance in the instrument logbook or Analyst's scientific notebook. <u>Be careful not to scratch the polished surface of the fiber optic or put your fingers on the Spectralon® panel's surface. Avoid scratching the Spectralon® panel's surface or letting anything contact its surface.</u> Oil and dirt from skin can darken the Spectralon® panel causing inflated reflectance values for sample measurements. If any dirt or darkening is detected on the Spectralon<u>®</u> panel's surface under strong light then wash it under a continuous stream of DI water with a 220-240 grit waterproof emery cloth using a circular light sanding motion until the surface is totally hydrophobic (i.e., water beads and runs off immediately).

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

7.3.7   Turn the spectrometer off. Turn the controlling computer on. If necessary to establish a working Wifi connection allow approximately 20 seconds to lapse before turning the spectrometer back on. If needed, this spectrometer-off/controlling-computer-on step allows the proper sequencing between the computer and the spectrometer to establish a successful WiFi connection. If a WiFi connection error occurs, see step 7.5.2.

7.3.8   Start the RS³ program on the computer by clicking on the RS³ icon

7.3.9   A check of spectrometer wavelengths must be conducted at least once after the spectrometer has been returned from the manufacturer for maintenance or service and after returning from fieldwork and prior to use for laboratory measurements. Analysts need to check the instrument log book to confirm that the wavelength performance evaluation was completed after the spectrometer's last use in the field. If it has then subsequent checks are not required until the spectrometer is used for field work again. In addition to these required checks, a wavelength evaluation may be performed at any time at the analyst's discretion. If a check is required or elected, refer to MRP-SPECLAB-SOP-08 and follow instructions to run the wavelength performance check. If conducted, note in the instrument logbook that a wavelength performance check was run. Otherwise, continue with the next step. After a wavelength performance check is conducted and the spectrometer receives a "pass" grade then proceed with the next step.

7.3.10   Select "Control" on the top menu, then select "Adjust Configuration" from the drop-down menu (Fig. 1) Because of recent design changes, all of our ASD spectrometers are shutterless so the default dark current measurement is 100. Even if the Analyst sets this parameter at another value the software defaults to 100 counts for the dark current and the value listed in the configuration menu will show as "100."

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.
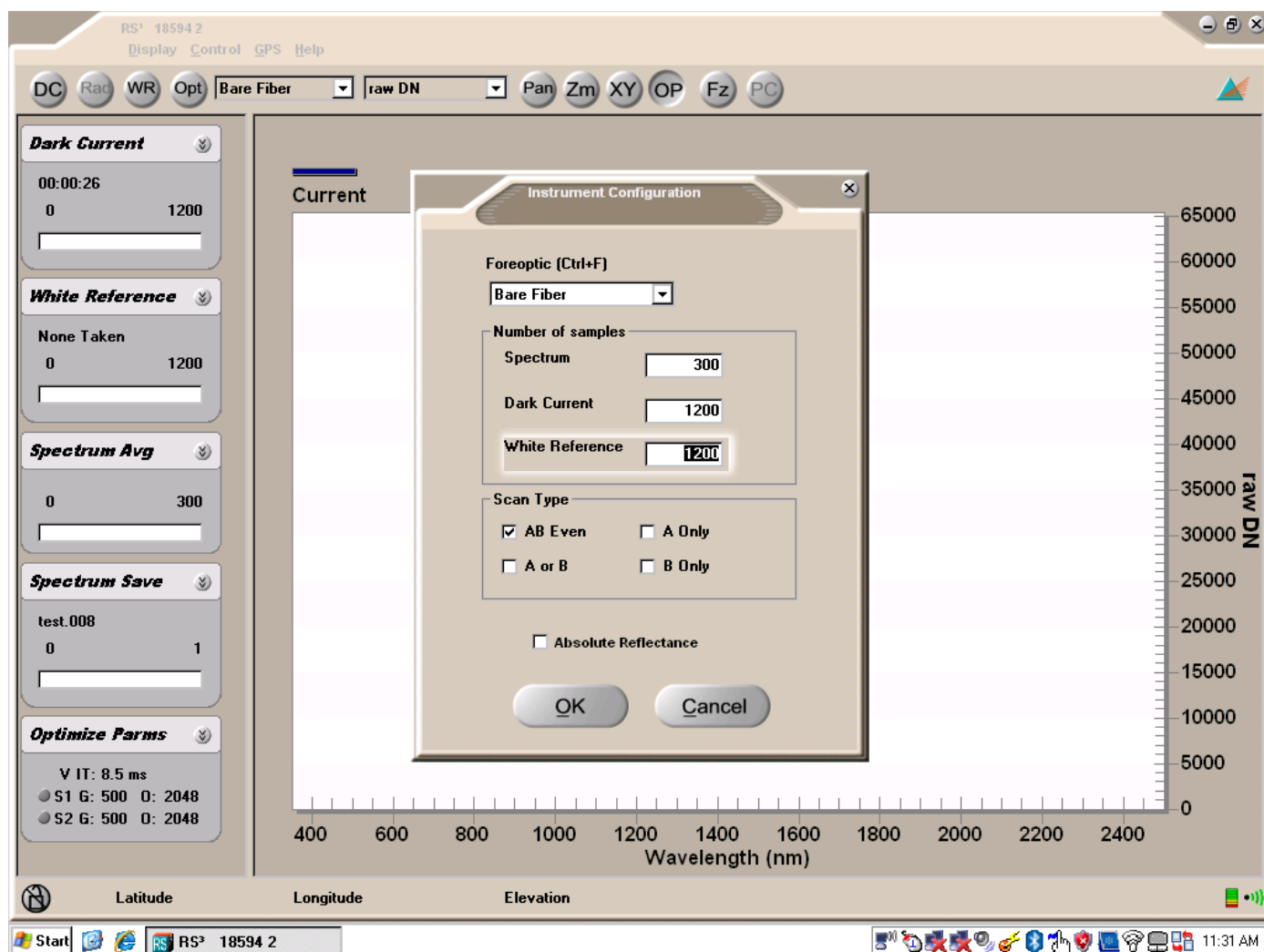
Figure 1. Spectrometer "Instrument Configuration" menu in the RS³ software.

7.3.11 On the resulting drop-down "Instrument Configuration" menu:

If using a normal resolution FS spectrometer enter the following under "Number of Samples":

Spectrum ≥ 60

Dark Current ≥ 100

White Reference ≥ 240

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

If using a high-resolution FS NG spectrometer enter the following under "Number of Samples":

Spectrum ≥ 300

Dark Current ≥ 100

White Reference ≥ 1200

Note any deviation from these settings in the instrument logbook or Analyst's scientific notebook.

7.3.12 While still on the "Instrument Configuration" drop down menu select:

For "Scan Type" select "AB Even"; leave fiber as "Bare"; make sure that the "Absolute Reflectance" box is NOT checked.

7.3.13 Select "Control" on the top menu, then select "Spectrum Save" from the drop-down menu (Fig. 2).

7.3.14 Make sure the "Save As New Format" box is checked at the bottom of the "Spectrum Save" menu.

7.3.15 Fill in the "Path Name" line with "C:\Spectraldata\ASD\date" where "date" follows the yyyy_mm_dd format (e.g., C:\Spectraldata\ASD\2018_03_14 for March 3, 2018).

7.3.16 Fill in the "Base Name" line. This will be the file name of each spectral measurement that will be incremented by consecutively numbered extensions (e.g., Alunite.00000, Alunite.00001, etc.).

7.3.17 Make sure the "Starting Spectrum Num" is "0".

7.3.18 Make sure the "Number of Files to Save" is 1 or more as discussed in step 7.3.26, depending on the overall brightness of the sample.

7.3.19 Make sure the "Interval between Saves" is "00:00:00" when there is no need for time lapses.

7.3.20 On the "Comment" line enter a title for the spectrum using the format:

Name of material, catalog number, grain size or other sample information, "Refl"

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy

Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

(e.g., "Alunite HS395.3B 74-250µm Refl" or "Muscovite GDS111 <250µm Refl")

7.3.21 Click "OK"; Say "Yes" to creation of a new directory if prompted.

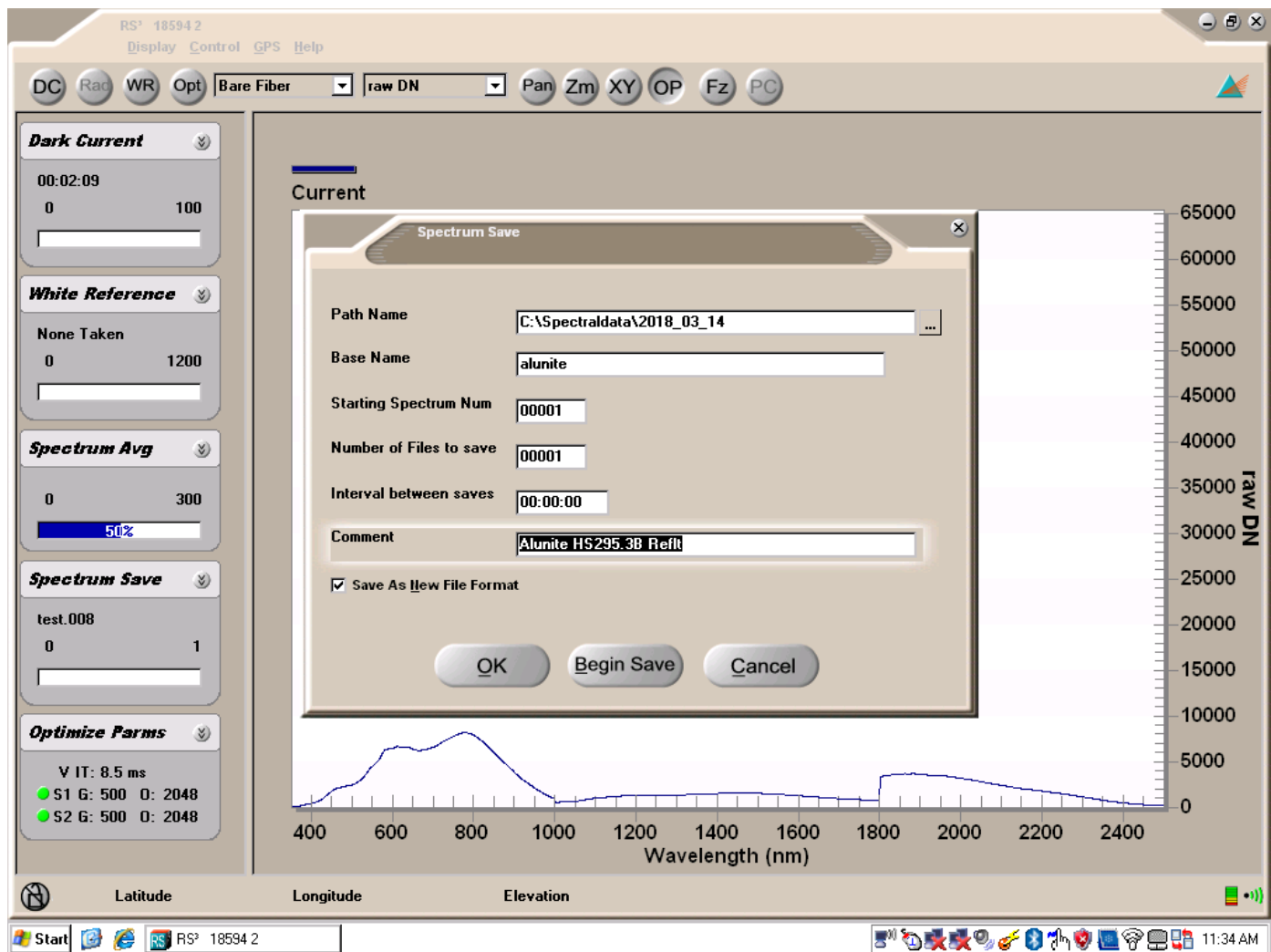7.3.22 Pour the sample into the sample cup and put aside in a secure place.

Figure 2. Spectrometer "Spectrum Save" menu in the RS³ software.

With the tip of the fiber optic cable pointed at the Spectralon® panel, press the optimization "Opt" button on the top menu. This will set the gains on the detectors by measuring the raw DN (Data Number) of light reflecting off the Spectralon® panel. Activating the optimization button causes the spectrometer to take a "Dark

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

Current" measurement and then a "White Reference" measurement. Progress on these measurements will be shown by growth of their status bars indicating the % of the spectrum collected. The optimization is complete once the "Spectrum Avg" status bar starts to function. Check to be sure the resulting "Spectrum Avg" (DN) spectrum has three humps, one for each spectrometer, and has no flat tops and no warning messages, or beeps, as seen in Figure 3. If these occur, re-optimize until these errors do not occur.
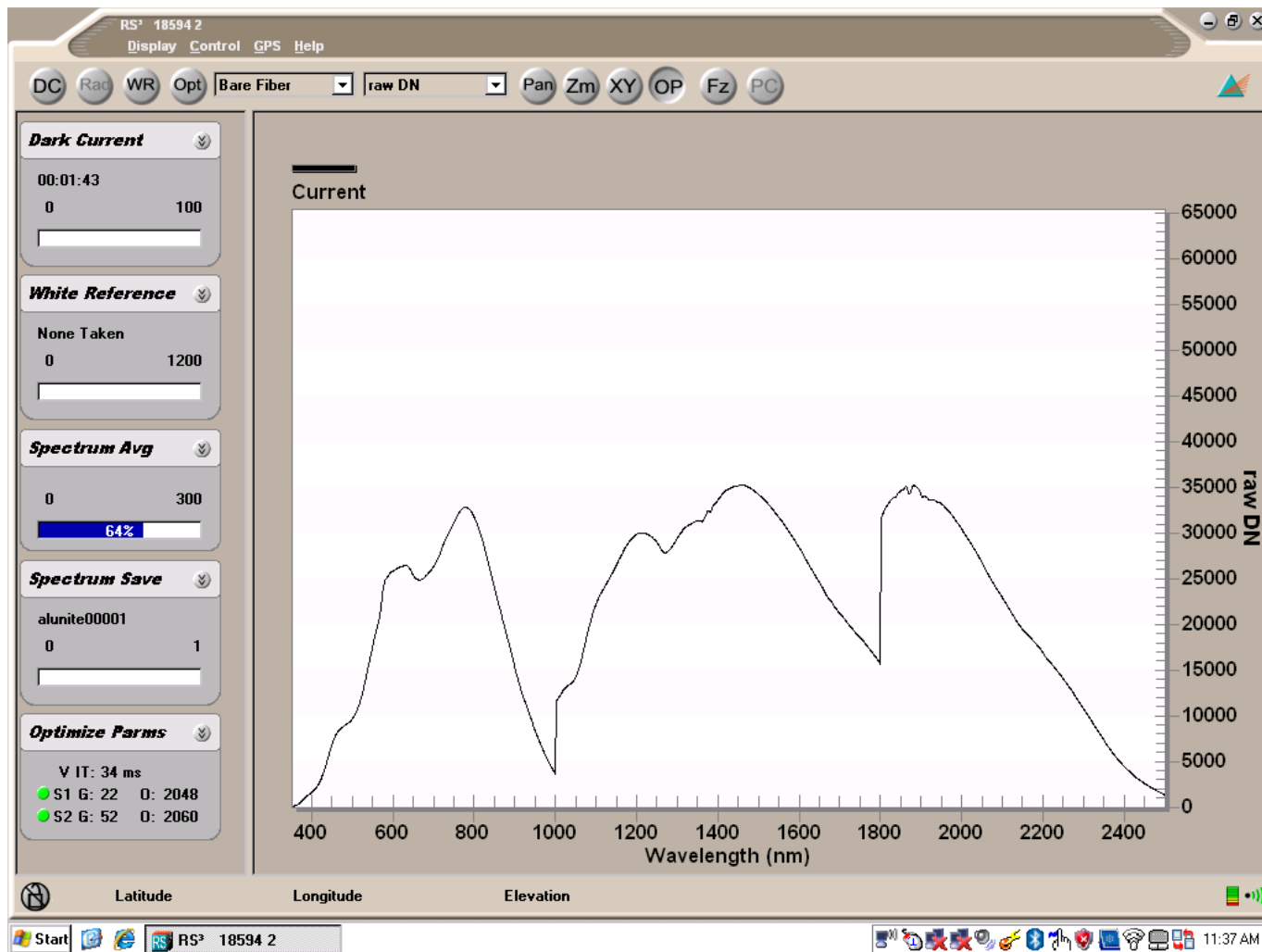

Figure 3. Spectrometer "optimization screen" in the RS³ software, showing an example optimization spectrum. Note each "hump" represents the raw DN values for one of the three internal spectrometers.

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

7.3.23   After optimization is 100% complete, press the White Reference "WR" button (to the left side of the optimize "Opt" button). This will collect a spectrum of the Spectralon® panel (i.e., white reference panel) to use for the denominator in calculating the reflectance of the samples (i.e., Refl = sample/white reference panel). Activating the "WR" button causes the spectrometer to take a "Dark Current" measurement, then a "White Reference" measurement, and finally a "Spectrum Avg" measurement. Progress on these measurements will be shown by growth of their status bars indicating the % of the spectrum collected. The result should be a flat line plotted in the spectral plot window (Fig. 4). If the line is not completely flat, without magnification, then repress the White Reference "WR" button to remeasure the white reference panel, discarding the previous measurement. Do not proceed with the sample measurements until a flat white reference spectrum is achieved within the highlighted "Spectrum Avg" status bar function (Fig. 4). A useable white reference panel spectrum will be visually no noisier than this spectrum (i.e, noise must not be greater than ± 0.2 reflectance units).

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.
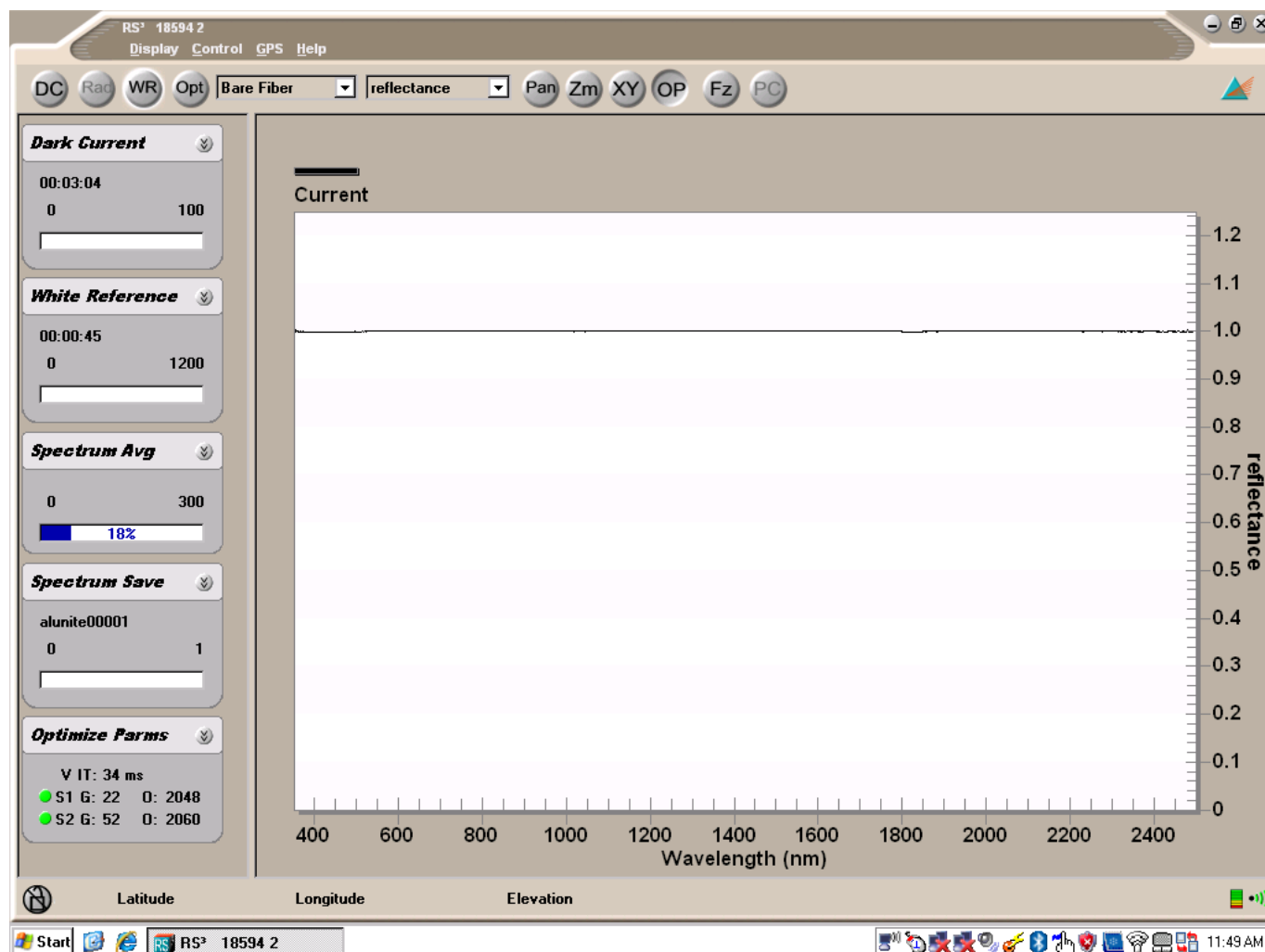
Figure 4. RS³ software white reference plot. Note the trace deviations from perfectly flat.   A useable white reference panel spectrum will be visually no noisier than this spectrum.

Several unsuccessful attempts to achieve a flat line white reference panel spectrum may indicate that the detectors are not at a stable temperature or that the optimization was not successful.   Re-optimize (step 7.3.22) and measure the white reference panel again. If this does not result in a flat "white reference" line, then wait an hour and retry starting at step 7.3.22. If this does not work, then consult the Laboratory Manager to determine if spectrometer maintenance is required.

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

7.3.24   After the "white reference" measurement is complete, remove the Spectralon®
            panel and place it under the glass cover away from the spectral measurement area.

7.3.25   Place the sample cup under the fiber optic cable on the adjustable optical bench.
            Make sure the distance between the tip of the fiber optic cable and sample surface
            is visually the same as that used when making the Spectralon® panel measurement.
            The fiber optic field of view is 22°. Because the fiber optic is mounted in a rod that
            holds it 30° off of nadir (i.e., 30° from the vertical), when the height of the fiber
            optic above the sample container is selected to be 1.5 cm, then the center of the
            field of view will be approximately 1 cm horizontally beyond the point on the
            sample container directly beneath the tip of the fiber optic. Make sure the center
            of the sample cup is offset approximately 1 cm horizontally in the direction of the
            dip of the fiber optic tip. Compensate as needed if the fiber optic is vertically closer
            or further than 1.5 cm from the sample surface.

7.3.26   Let the spectral plot refresh at least twice before saving the first spectrum. Each
            displayed spectrum is a running average over a set period of time (determined by
            0.1 seconds times the "Number of samples" set for spectra in step 7.3.18). When
            the sample is moved under the fiber optic, the first refreshed plot will include data
            from the portion of time when the sample was not below the fiber optic. To be sure
            that the recorded spectra only include measurements of the sample, wait for the
            spectral plot to refresh twice after moving the sample.

            Press the space bar to record a single sample spectrum ("Spectrum Avg") to the
            designated directory. If this sample has an average reflectance level greater than
            30% in the 2 to 2.5 µm region then record (collect) at least 10 spectra of the sample
            for co-averaging. If the sample has an average reflectance level less than 30% (or
            0.3 on a scale from 0 to 1) in this same spectral region, record at least 20 spectra of
            the sample for co-averaging. If the surface of the sample is not uniform, as happens
            with rock samples, record a single spectrum at between 10 to 20 different spots on
            the rock surface. Move the rock, not the light or fiber optic cable as these need to
            remain fixed in place during spectral measurements. If the light or fiber optic cable
            are disturbed during a set of measurements, spectra recorded after the disturbance
            will not be valid. The spectrometer will require a new optimization and white
            reference. Optionally, if the sample is fine grained and a single sample position will
            suffice to achieve the PI's measurement objectives, the software can be set to
            record the number of sample spectra required with only one press of the space bar.
            Depending on the average reflectance level of the sample in the 2 to 2.5 µm region,
            the "Number of Files to Save" variable can be set to at least 10 on the "Spectrum

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
*A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision
numbers match.*

Save" drop down menu. The PI determines if a greater number is needed based on the brightness and homogeneity/heterogeneity of the sample. Pressing the space bar once will then collect the requisite number of spectra and save them to the designated directory. The "Spectrum Save" status bar will display the percent of spectra collected.

7.3.27 If spectral artifacts appear in the spectra (e.g., "shark fin" shaped artifact at wavelengths just short of 1.0 µm) then it is time to perform new optimization and white reference operations (i.e., step 7.3.22). Optimally, perform new optimization and white reference operations every 10 to 30 minutes while measuring samples to compensate for detector drift (i.e., changes in spectrometer or illumination lamp temperature). Do not exceed more than 1 hour between "optimization" and "white reference" measurements. If a new white reference is required in less than 10 minutes because it is not a smooth horizontal line, allow the spectrometer to warm up longer.

7.3.28 When changing samples, repeat steps starting at 7.3.25. If a new "white reference" measurement is need repeat steps starting at 7.3.22 after replacing the sample cup with the Spectralon® panel. Let the spectral plot refresh at least once to make sure the instrument is still within optimization. If it is not, then a beep will sound and a warning message will appear. If the warning message appears, repeat steps from 7.3.22. Make sure to note the distance between the Spectralon® panel and the fiber optic and adjust the height as appropriate for the next sample that will be measured. Remove the sample from the sample cup and return the sample it to its container or dispose of according to PI instructions.

7.3.29   When sample measurements are complete, copy spectral measurement data and wavelength performance report (if measured, refer to MRP-SPECLAB-SOP-08) from the C:\Spectraldata\ASD\date directory to a thumb drive or other external drive for transfer to the /d1/Spectraldata/ASD/date directory on the Cassini server.

7.3.30 To shut down the ASD spectrometer (in any order):

  1) Remove the sample,

  2) Exit the computer program and shut the computer off,

  3) Turn the spectrometer power off,

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

4) Remove the fiber optic cable from the 30-degree incident fiber optic holder and place the fiber cap on it.

5) Turn the illumination lamp off.

7.4. Dilutions, Problematic Samples, Carryover - NA

7.5. Troubleshooting and Bench Notes

7.5.1   The instrument number printed on the top of the computer (e.g., 18388) must match the serial number on the side of the ASD spectrometer for proper wavelength calibration, because the RS^3 software runs an *.ini file that has wavelength calibration data specific to each ASD spectrometer. If a computer with a different ASD spectrometer's *.ini file is used to run a non-matching spectrometer (e.g., computer designated 18011 is used to run ASD spectrometer 18388), then the wavelengths of the data collected could be inaccurate.

7.5.2   Occasionally, the WiFi connection between the controlling computer and spectrometer fails. The RS^3 software will notify the Analyst of the loss/non-establishment of a WiFi connection with an error message. The spectrometer and computer must both be restarted. Start the computer first and then the spectrometer. Wait for the connection to be established (e.g., usually within a minute's time). If the WiFi connection is not re-established after trying this remedy three times, then connect the spectrometer to the computer with an Ethernet cable and restart the computer and then the spectrometer to establish communication. If this does not work contact the Laboratory Manager for assistance.

7.6. Maintenance

 Annual maintenance of each ASD spectrometer is performed at the ASD facility where the detector wavelengths are calibrated relative to noble gas emission lines using peak wavelength positions provided by NIST.   Internal USGS checks of wavelength and bandpass performance are done when the spectrometers are returned after factory calibration. The ASD spectrometers are evaluated for wavelength performance when they are returned from the manufacturer for maintenance or servicing and when they return from the field as described by MRP-SPECLAB-SOP-08.

8.   DATA ACQUISITION, PROCESSING, AND EVALUATION

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

8.1. Data Acquisition and Calculations

The RS^3 software automatically ratios the sample spectrum to the "white reference" spectrum, thus, providing a reflectance spectrum output. On the computer, the output reflectance spectrum is written to C:\Spectraldata\ASD\date.

8.2. Handling Sample Data That Is Outside the Calibration Range - NA

8.3. Data Processing

See 8.1 above.

9. QUALITY ASSURANCE (QA) AND QUALITY CONTROL (QC)

9.1.   Internal QC Samples

The spectrometer wavelength performance check, run after spectrometer field use, tests spectrometer wavelength accuracy. The wavelength standard is a NIST traceable Multipcomponent Labsphere® puck with a mylar film cover measured in reflectance and it is described in MRP-SPECLAB-SOP-08. The instrument software generates a report that gives a Pass/Fail grade for wavelength accuracy. If the performance check fails, the process needs to be repeated a second time. If it fails again contact the Laboratory Manager for assistance to determination if adjustments are needed or if instrument maintenance is necessary.

9.2.   External QC Samples - The Labsphere multipcomponent REE puck is NIST traceable and constitutes both an internal and external QC standard.   It is measured after the spectrometer is returned from fieldwork.

9.3. QC Charts - QC charts are not applicable as we measure comparison samples. The pass/fail wavelength performance check run after the spectrometer is returned from the field serves to reveal when and if an abnormal event has taken place that would potentially affect instrument performance.

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

10. DATA AND RECORDS MANAGEMENT

10.1. Data Packages

Spectral reflectance data is transferred by thumb drive or other external drive from the instrument computer (which is not connected to the internet) in "C:\Spectraldata\ASD\date" to the Cassini server and placed in "/d1/Sampledata/ASD/date". The data package from a spectral measurement session contains spectra of the samples measured, the wavelength performance check report if generated by measuring the MCREEmylar puck after the instrument is returned from the field, and the secondary reviewer checklist.

10.2. Archival and Storage Requirements

The spectral data on the Cassini Server in "/d1/Spectraldata/ASD/date" is backed up on a daily basis on USGS onsite and offsite hard drives.

11. REFERENCES

Kokaly, R.F., Clark, R.N., Swayze, G.A., Livo, K.E., Hoefen, T.M., Pearson, N.C., Wise, R.A., Benzel, W.M., Lowers, H.A., Driscoll, R.L., and Klein, A.J., 2017, USGS Spectral Library Version 7: U.S. Geological Survey Data Series 1035, 61 p. https://doi.org/10.3133/ds1035.

Swayze, G.A., Clark, R.N., Goetz, F.H., Livo, K.E., Breit, G.N., Kruse, F.A., Sutley, S.J., Snee, L.W., Lowers, H.A., Post, J.L., Stoffregen, R.E. and Ashley, R.P., 2014, Mapping advanced argillic alteration at Cuprite, Nevada using imaging spectroscopy: Economic Geology, v. 109, no. 5, p. 1179-1221. doi:10.2113/econgeo.109.5.1179

12. ATTACHMENTS – SECONDARY REVIEWER CHECKLIST

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

13. HISTORY OF CHANGES

Revision No.	Effective Date	Description of Changes
00	07/10/2019	Initial issue
01	8/21/2020	Corrected sections 7.1 and 7.3.6 to add dependent on sample set-up.

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020

A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.

ATTACHMENT 1

DATA PACKAGE UNIQUE ID:_____

DATE OF ANALYSIS: _____

SECONDARY DATA REVIEW CHECKLIST:

	Analyst		Reviewer	
	Yes	No	Yes	No
1. Did the ASD spectrometer pass the wavelength performance check if one was run after returning the unit from the field?				
2. Does the data package contain spectrum files and a wavelength performance report if one was run?				
3. Do spot checks of spectral files show their titles contain the names of material, catalog number or other sample information?				
4. Was an entry made describing the type of measurements and any deviations from this SOP in the instrument logbook or Analyst's scientific notebook?				

Comments: Secondary Reviewer
Analyst name (print):
Analyst signature: Date:
Reviewer name (print):
Reviewer signature: Date:

MRP-SPECLAB-SOP-02.01   Technical SOP   CONTROLLED Copy
Title: Conducting spectral measurements with the ASD spectrometers
Authors: Gregg Swayze
Effective Date: 8/21/2020
Approved By: Amy Bern   Revise or reapprove by: 8/21/2020
A paper copy of this document is only valid if its revision number matches the controlled copy posted. It is the user's responsibility to verify that these revision numbers match.