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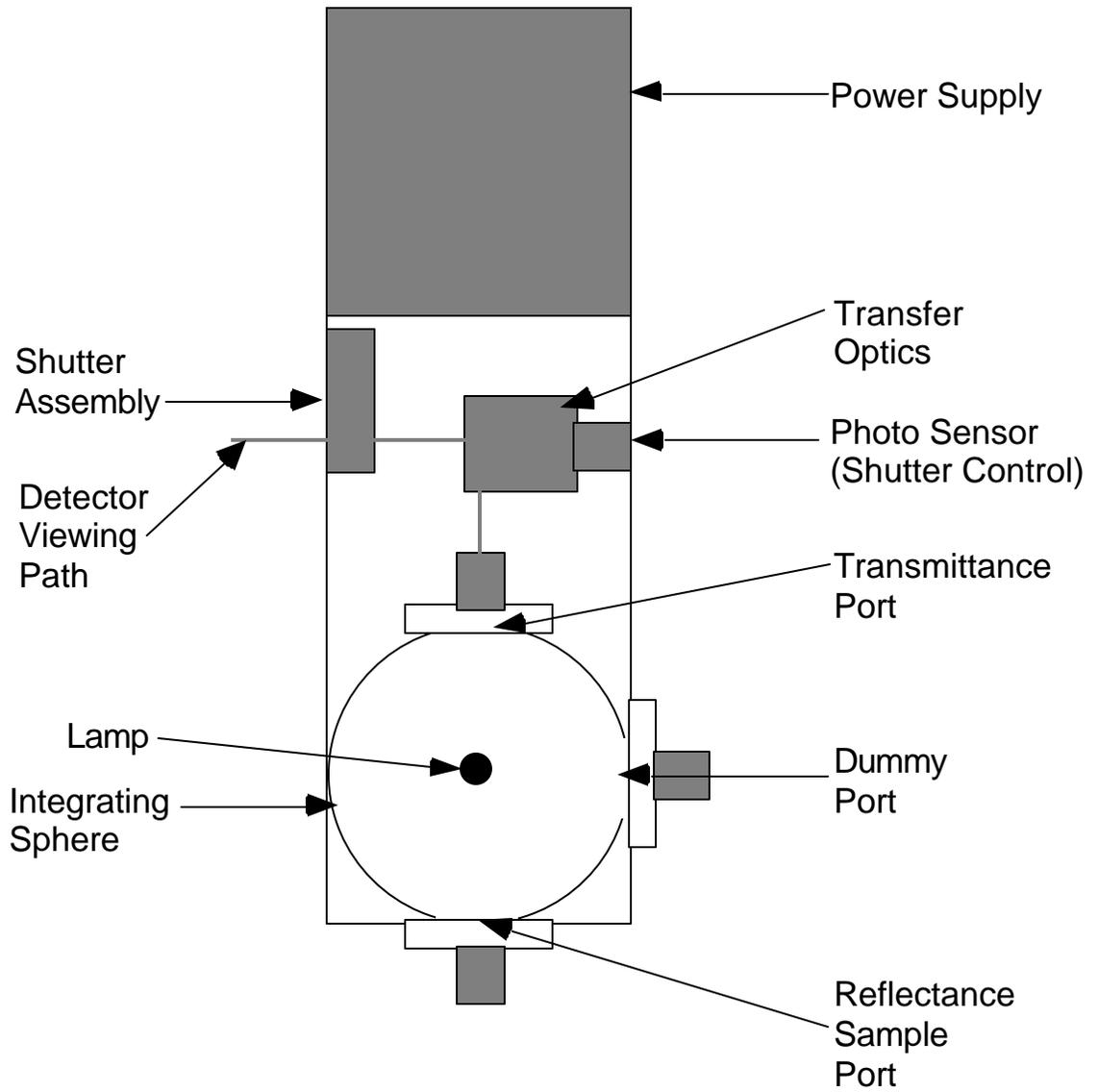
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I. INTRODUCTION

The Labsphere RSA-HP-84 is a diffuse reflectance and transmittance accessory designed exclusively for the Hewlett-Packard HP8452A diode-array spectrophotometer. The accessory expands the versatility and range of applications for the HP8452A, accommodating reflectance measurements of both diffuse and specular samples as well as the transmittance/absorbance of turbid or scattering samples. The integrating sphere geometry allows for easy measurement of total hemispherical or specular excluded reflectance.

The RSA-HP-84 is a modular unit complete with its own power supply, light source, transfer optics, optical shutter and sample holders in addition to the 95 mm diameter integrating sphere. (See RSA-HP-84 illustration, Figure 1. A detail representation of the RSA-HP-84 transfer optics appears in Figure 2.) Easily installed and simple to use, the RSA-HP-84 is compatible with all Hewlett-Packard operating software.

This manual is intended to provide installation procedures for the RSA-HP-84 accessory only. The "Operator's Manual" of the Hewlett-Packard HP8452A diode-array spectrophotometer should be consulted for measurement procedures, since the RSA-HP-84 does not modify normal operations.



Front of RSA-HP-84

Figure 1. Top View of the RSA-HP-84 Accessory.

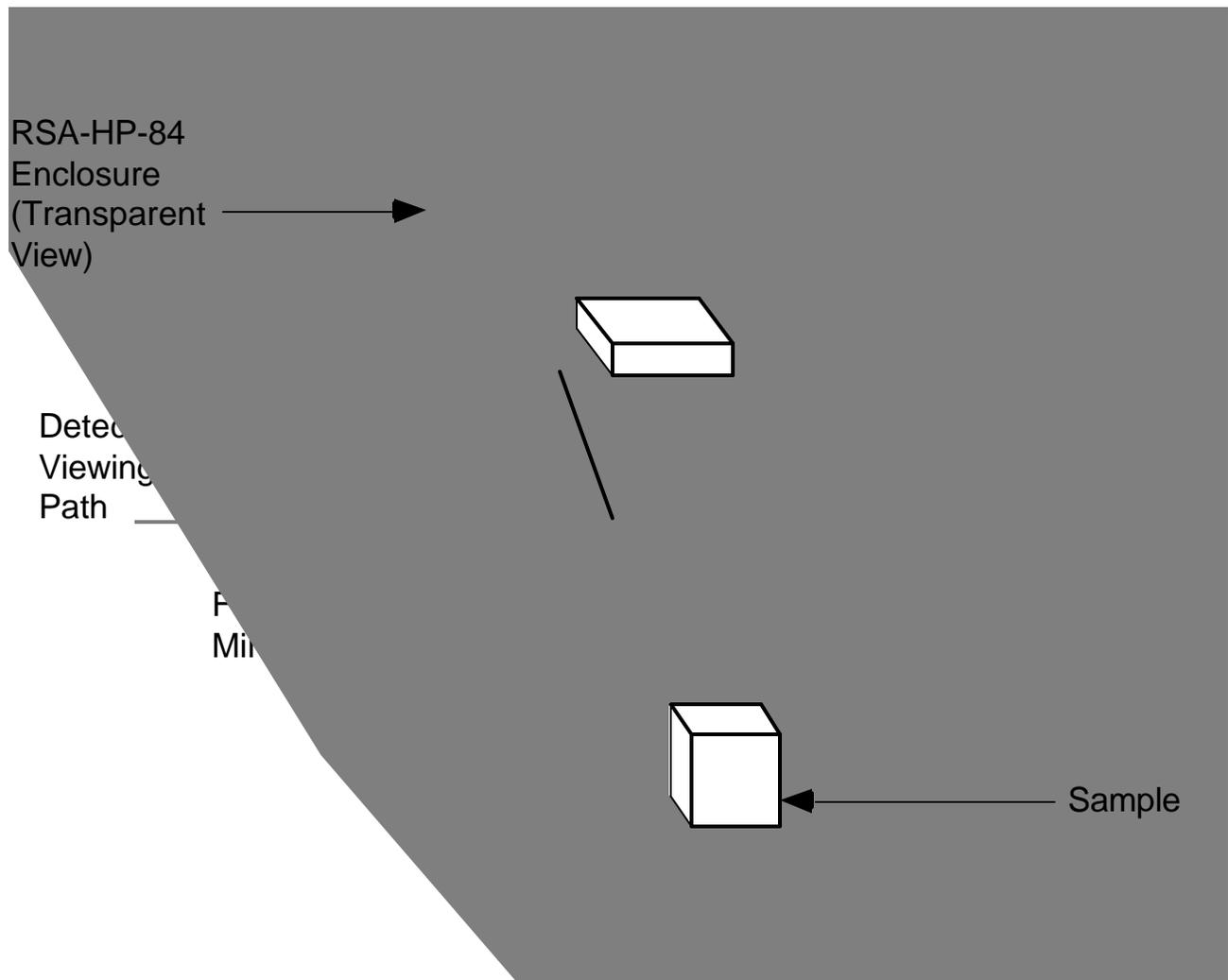


Figure 2. Transfer optics detail.

A. Components

- 1 RSA-HP-84 Reflectance Spectroscopy Accessory (with two mounting screws)
- 1 8 degree sample holder
- 1 0 degree transmittance sample holder
- 2 0 degree reflectance sample holders
- 1 Power cord
- 1 SRS-99-010 Calibrated Diffuse Reflectance Standard
- 1 Calibration certificate(SRS-99)
- 2 USRS-99-010 Uncalibrated Diffuse Reflectance Standard
- 1 RSA-HP-84 Instruction Manual
- 1 Calibration documents (RSA-HP-84)
- 1 Hex socket driver set

II. INSPECTION PROCEDURE

The Accessory was thoroughly inspected before shipping and should be ready to operate after completing the set-up instructions. It is packaged and shipped in a reinforced cardboard carton. Carefully check the assembly after unpacking it for any damage incurred during shipping. If there is any such damage, file a claim immediately with the involved carriers and contact Labsphere's Customer Service Department at (603) 927-4266.

III. INSTALLATION PROCEDURES

A. Initial Installation

1. Turn on the HP8452A and enter the HP software.
2. Remove the standard cell holder and/or any other accessories from the sample compartment of the HP8452A.
3. Attach an 8° sample holder to the reflectance (sample) port and a 0° sample holder to the dummy port, using a 0.35-inch hex wrench.
4. Orient the RSA-HP-84 such that the sphere ports are towards the front of the HP8452A.
5. Lower the accessory into the sample compartment until it rests firmly upon the mounting studs.
6. Open the top lid of the enclosure.
7. Position the two mounting screws into the holes at the base of the assembly, and tighten to secure the accessory.

B. Power Connections

1. Locate the AC power connector at the back of the RSA-HP-84.
2. Plug the female end of the power cord into the power input socket of the RSA-HP-84.
3. Plug the male end of the power cord into the electrical outlet.
4. Place an SRS-99-010 calibrated Spectralon (or other) diffuse reflectance standard into the reflectance (sample) port.
5. Place a USRS-99-010 uncalibrated Spectralon (or other) reflectance standard in the dummy port.
6. Turn the RSA-HP-84 power switch ON. The sphere should illuminate. If the sphere does not illuminate, recheck all connections.

C. Optical Alignment

The transfer optics of the RSA-HP-84 were factory aligned prior to shipping. However, you should verify the accessory's alignment *before first use* to ensure that a misalignment did not occur in transit.

WARNING: *Do not touch mirror or lens surfaces.* Skin oil will reduce performance. Clean gloves are recommended.

1. Make sure that a Spectralon reflectance standard has been placed in both the reflectance sample and dummy ports.
2. Configure the software parameters as follows: **Transmittance** mode, **Wavelength Range = 500 - 820 nm**.
3. Perform a Blank scan or background correction. The spectrum should appear as a flat 100% transmittance line.
4. Darken the room. This minimizes the ambient light that could reach the accessory.
5. Remove the Spectralon standard from the sample port and put a light trap in its place or leave the port open.
6. Initiate the sample SCAN, and observe the displayed spectrum. If the accessory is properly aligned, the open port zeroline scan should appear between 0.2 - 0.4% T. Otherwise, realignment of the accessory is necessary.
7. If the results appear above 1%, open the top lid of the enclosure to access the mirror adjustment screws of the transfer optics assembly.
8. Carefully tweak one adjustment screw on the top mirror mount in one direction only. Initiate the sample SCAN. Rescale the Y-axis as needed.
9. If the displayed scan is lower than the first, continue to tweak the adjustment screw in that same direction until the transmittance value is minimized. CLEAR the screen occasionally before continuing to scan.
10. If the displayed scan is higher than the first, tweak the screw in the opposite direction to obtain the minimum display result.
11. If changes of more than 5% occur during the alignment process, repeat the background correction of steps 1-3.
12. Repeat the alignment procedure with the remaining adjustment screw on the top mirror mount to obtain the minimum display result.
13. Using a 7/64" (5.5 mm) ball end hex driver, tweak each adjustment screw on the bottom mirror mount as described above until the minimum display result is achieved.
14. Repeat steps 8-13 until the results between the range of 500-820 nm are less than 1% T; ideally, the result should lie between 0.2 and 0.4% T.

15. Turn on the room lights after the alignment procedure is completed.

Note: Once the accessory is properly aligned, the HP8452A should initialize with the RSA-HP-84 installed in the instrument's sample compartment. In order to initialize the instrument properly, the lamp of the RSA-HP-84 should be illuminated and the Spectralon reflectance standards should be positioned over the reflectance sample port and dummy port *before* the instrument is turned ON. Upon initialization, the accessory will make a series of six clicking noises followed by a three second delay and then a seventh click. At this point, the system is fully initialized and ready for operation.

Note: An error message will appear in the HP8452 display panel (located in the upper right hand corner of the instrument) until the software has been entered. The message should disappear once the software and the RSA-HP-84 are fully initialized.

IV. OPERATING PROCEDURES

A. Theory of Operation

In a diode array spectrophotometer, the measurement of reflectance factor and transmittance involves the performance of a blank scan. The blank scan is used to establish a baseline for the instrument with the sphere in place, much as one would run a baseline with a solvent in a cuvette in a standard liquid measurement. This correction is performed automatically by the instrument in the blank scan mode.

The tungsten halogen source within the sphere illuminates the sample with diffuse radiation. The sample port is imaged onto the polychromator and then to the diode array. The blank scan records an intensity level for each diode when the reference (which is placed in the sample port) is measured. When the sample replaces the reference and a sample scan is performed, the instrument ratios the intensities at each diode with that of the stored blank. The accessory uses the Hewlett-Packard 8452's own array. It is assumed that the detector response is temporally stable while the source--a tungsten halogen lamp--has a steady, voltage-controlled power supply to ensure lamp stability.

Diode array instruments that employ an integrating sphere as an accessory operate in either the substitution mode or the comparison mode. That is, a blank scan is run with a sample of known reflectance positioned in the reflectance sample port during reflectance measurement, or air is used as the reference during transmittance measurements (or a cuvette in the transmittance port, if measuring liquids). The dummy port may contain the sample to be measured (if the comparison mode is used) or a white Spectralon plug (substitution mode). The flux measured by the detector within the sphere can be expressed as:

$$D_b = I_s \cdot R_d \cdot r_r \cdot k_r$$

Where: I_s = light incident into the sphere
 R_d = detector response,
 ρ_r = reflectance of reference for background, and
 κ_r = sphere efficiency function

The sphere efficiency factor (κ) for a given sample is a function of many variables including the spatial distribution of the energy reflected from the sample, the reflectance of the sphere wall, the geometry of the sphere (location of ports, baffles, etc.) and the efficiency of the detector itself. However, proper measurement procedures make it possible to greatly reduce or eliminate the effect of such factors on transmittance and reflectance factor measurements. I_s is dependent on the efficiency of the coupling optics and polychromator, and the efficiency of the diode array detector itself.

When one measures a sample of different reflectance, the flux measured by the detector can be expressed as:

$$D_s = I_s \cdot R_d \cdot r_s \cdot k_s$$

Assuming that the spatial distribution of energy reflected from this unknown sample is the same as that of the original standard, the efficiency factor (κ) will be the same for both materials:

$$\kappa_r = \kappa_s$$

Therefore,

$$\frac{D_s}{D_b} = \frac{I_s \cdot R_d \cdot r_s \cdot k_s}{I_s \cdot R_d \cdot r_r \cdot k_r}$$

and since, D_b is set to equal unity, then

$$D_s = \frac{r_s}{r_r}$$

Therefore, the value displayed by the instrument (D_s) will simply be equal to the ratio of the reflectance factor of the unknown sample to that of the original standard. If the reflectance factor of this original standard is known, it can be used as a reference standard and the reflectance factor of the unknown sample can be derived as follows:

$$r_s = D_s \cdot r_r$$

The foregoing account involves two simplifying assumptions which bear further discussion. First, the efficiency of the integrating sphere, represented by κ_s and κ_r , is not necessarily constant under changes in the system. The introduction of a reflectance or transmittance sample into the system may change the sphere efficiency. It is commonly assumed, however, that both κ_s and κ_r will change by the same factor. In a properly designed integrating sphere, the systematic uncertainty associated with this assumption is very small. For a diode array instrument, however, $\kappa_r \neq \kappa_s$, except where the reflectances of reference and sample are very closely matched. Non-matching of sample and reference reflectance may cause what is called 'single beam substitution error'. This is covered in some detail in the Labsphere publication [A Guide to Reflectance Spectroscopy](#) and in Appendix A. If the comparison method is used, then:

$$k_r = k_s$$

and

$$D_s = \frac{r_s}{r_r}$$

and, therefore,

$$r_s = D_s * r_r$$

exactly as it would in a double beam instrument with chopped, active referencing.

The second assumption applies to reflectance factor measurements. Here it is assumed that the geometric scattering properties of the sample and the reference standard are identical. Therefore, the efficiency factor for radiation reflected from each sample will be the same. If the geometric scattering properties of the sample differ greatly from those of the reference, significant systematic errors may be introduced into the measurements. Therefore, the geometric scattering properties of the sample and reference should match as closely as possible.

B. Diagnostic Scans

A series of diagnostic scans was performed on each accessory at the factory prior to shipping. The results of the diagnostic scans obtained on your accessory are provided in the Quality Assurance Document. Upon first installation of this accessory, or if problems with the accessory are suspected, repeat these diagnostics, as described below and compare the results with those provided by the factory. Consult Labsphere's Technical Support Department for help in interpreting these diagnostic scans.

To reproduce these diagnostic scans, first set the parameters in the instrument software to match those in the "STANDARD CHECKOUT" method, as given below:

- Transmittance
- 360 - 820 nm
- 1 second integration time
- overlay scans

NOTE: When the instrument is turned on with the accessory in place, an error message will appear stating that there is not enough dark current intensity. Ignore this message.

Energy Scan

1. Set up instrument parameters as in the STANDARD CHECKOUT method described in the Quality Assurance Document, using intensity as the data mode, rather than transmittance.
2. Place the *calibrated* Spectralon standard in the reflectance-sample port. Place the *uncalibrated* Spectralon standard in the dummy port.
3. Initiate the scan to record the measurement data. (It is not necessary to record a blank scan in this case.)
4. Compare the scan with that run in the instrument checkout. While the scans may vary slightly due to the individual instruments, the shape and intensities of the peaks should be quite similar. Spikes in the scan are normal. The instrument uses a deuterium lamp and the accessory uses a tungsten lamp. The instrument corrects scans for the spikes characteristic of deuterium lamps. When the accessory is used, these correction factors appear as spikes in the scan.

100% Baseline:

1. Place the calibrated Spectralon reflectance standard in the reflectance sample port and the uncalibrated standard in the dummy port.
2. Set the instrument parameters to:
 - Transmittance
 - 360 - 820 nm
 - Y-scale 95 - 105%T.
3. Perform a blank scan.
4. Without altering the instrument's configuration, initiate the sample scan to record the 100% baseline scan. The resulting scan should closely resemble that provided in the documents.

The baseline noise should fall below $\pm 2\%$ from 380 - 820 nm. The noise peaks that appear below 380 nm are the result of the tungsten lamp which does not produce energy at low wavelengths. If a quieter scan is required, reset the integration time to between 5 and 10 seconds. This allows the detector to collect more energy for each scan and will reduce the noise of the spectrum.

Blocked-Beam Zeroline:

1. Collect a blank scan as described in steps 1 - 3 of the 100% baseline procedure. Change the Y-scale to range from -5 to 5%T.
2. Open the lid of accessory and place a metal plate, or other non-transmitting sample, in the transmittance sample holder, so that the transmittance-sample port is completely blocked.
3. Close the accessory lid.
4. Initiate the scan. The resulting spectrum should closely match that provided in the Q.A. documentation.

Note: Be sure to remove the plate from the beam path when the scan is complete.

Open-Port Zeroline:

1. Remove the Spectralon standard from the dummy port and leave the port open. The Spectralon standard should remain in position in the reflectance sample port.
2. Turn off the room lights.
3. Scan the blank.
4. Remove the Spectralon standard from the reflectance port. Place the standard into the dummy port.
5. Initiate the scan with the room darkened. The resulting scan should closely match that provided in Q.A. documents.

Note: This scan may also be performed with a light trap substituting for the open sample port.

C. Hemispherical/8 Degree Reflectance Factor Measurements

Diffuse Samples - Comparison Method

1. Place the calibrated SRS-99-010 Spectralon standard, or other diffuse reflectance standard, in the reflectance sample port. Place the sample to be measured in the dummy port.
2. Scan the blank.
3. Reverse the locations of the standard and the sample.
4. Initiate the scan to record the measurement data.
5. Multiply the values recorded in the resulting scan by the known spectral reflectance factor values of the diffuse reflectance standard to determine the actual reflectance of the sample.
6. Repeat this entire procedure for each new sample.

This procedure eliminates single-beam substitution error (see Appendix A).

Diffuse Samples - Substitution Method

While the following substitution method may be used to obtain quick results, substitution error may be introduced, if the sample to be measured drastically changes the reflectance properties of the sphere. The substitution method is recommended only if the reflectance properties of the samples to be measured closely approximate those of the reference material. If not, errors up to 4% may be expected.

1. Place the calibrated SRS-99-010 Spectralon standard or other diffuse reflectance standard in the reflectance sample port. Place the uncalibrated USRS-99-010 standard in the dummy port.
2. Perform a blank scan.
3. Replace the calibrated Spectralon standard in the reflectance sample port with the sample to be measured.
4. Initiate the scan to record the measurement data.
5. Multiply the values recorded in the resulting scan by the known spectral reflectance factor values of the reflectance standard to determine the actual reflectance of the sample.
6. The sample just measured can be replaced with a different sample to obtain further measurements using the same instrument baseline/blank scan.

Specular Samples

The procedure for specular samples is identical to that described above, except that a specular reference standard, such as a calibrated first-surface aluminum mirror, is substituted for the diffuse reflectance standard called for above.

Samples of 'Mixed', or Non-Ideal Reflectance

Many reflectance samples display geometric reflectance distributions which do not conform closely to either the lambertian distribution of an ideal diffuser or the perfect specular reflectance of an ideal mirror. For a discussion of approaches to measuring such samples, see Appendix A.

D. Hemispherical/0° (Specular Excluded) Reflectance Factor Measurements

The integrating sphere of the accessory has two sample holders for the reflectance sample port. The standard sample holder is designed to position the sample at an 8° angle. The second sample holder is designed for normal (0°) incidence. With the 0° sample holder in place, any specular component of reflection from the sample is excluded, since this component is directed out of the sphere through the transmittance sample port. Therefore, the 0° sample holder may be used to make Hemispherical/0° reflectance factor measurements. The procedure for such measurements is as follows:

1. Remove the 8° sample holder from the reflectance sample port. Install the 0° sample holder.
2. Follow the measurement procedures in the previous section to determine the diffuse reflectance of the sample.

NOTE: Measurements performed using the specular excluded geometry should always be interpreted with caution. A port of finite size is not always effective for the complete exclusion of a broadened specular peak.

E. Transmittance Measurement Procedure

Non-Scattering Samples - Comparison Method:

1. Place the uncalibrated SRS-99-010 Spectralon standard in the reflectance port and the sample to be measured in the dummy port, if possible. If a liquid is to be measured, fit the cuvette and holder in front of the transmittance port. Otherwise, leave the port empty.
2. Darken the room.
3. Scan the blank.
4. Place the sample to be measured in the transmittance sample holder. Leave the dummy port open.
5. Initiate the scan and record the measurement data.
6. The value displayed by the instrument is the actual sample transmittance.

Non-Scattering Samples - Substitution Method:

1. Place uncalibrated USRS-99-010 Spectralon reflectance standards in both the reflectance (sample) and dummy ports. If a liquid is to be measured, fit the cuvette and holder in front of the transmittance port. Otherwise, leave the port empty.
2. Scan the blank.
3. Place the sample to be measured into the transmittance sample holder.
4. Initiate the scan to record the measurement data.
5. The value displayed by the instrument is the actual sample transmittance.

Scattering Samples:

For transmittance samples exhibiting a high degree of scattering, the procedure described above does not serve as a measurement of total transmittance. Instead, the result displayed by the instrument will be an instrument-specific value which is known as regular transmittance factor. (See definitions, Appendix A.) For this accessory, the regular transmittance factor of a scattering sample closely approximates the total transmittance of the sample. For further discussion of the handling of scattering transmittance samples, see Appendix A.

V. OPERATING SUGGESTIONS

A. Instrument Parameters

Since any integrating sphere accessory acts as an attenuator of signal, run scans at a longer integration time than in ordinary transmittance work.

B. Zeroline Correction

An integrating sphere is sensitive to small-angle scatter from the sample beam coupling optics. The scattered radiation strikes the wall of the integrating sphere near the reflectance-sample port, creating a "halo" surrounding the port. This "halo-effect" gives rise to a small error in measurement of reflectance factor which is most significant when measuring samples of very low reflectance. Fortunately, this error is relatively easy to characterize and correct.

The following procedure may be used to correct reflectance factor measurements of diffuse or specular samples, in either the Hemispherical /8° or Hemispherical /0° geometries:

1. Perform a reflectance-factor measurement of a given sample as described in the Hemispherical/8 Degree Reflectance Factor Measurements section.
2. Replace the sample with a light trap (reflectance approximately 0%), and scan to record measurement data.
3. Compute the corrected reflectance factor for the sample, ρ_s , as follows:

$$r_s = \frac{(R - Z)r_r}{(100\% - Z)}$$

where:

ρ_r = the reflectance value of the reference standard,

R = the sample data displayed on the instrument and

Z = the data from the light trap measurement.

Note: This zero-correction procedure can also be used in conjunction with a mask at the reflectance sample port to accurately measure the reflectance factor of samples smaller than the accessory's reflectance sample port.

VI. MAINTENANCE

A. General Information

When not in use, the accessory should be stored in a controlled environment. Dust and moisture may adversely affect its performance.

PRECAUTIONS

- o Do not handle mirrors or lenses.
- o Do not allow foreign objects to enter the integrating sphere.
- o Clean dust and particulate debris out of the sphere using a gentle stream of clean air.

B. Mirror Cleaning Procedure

The mirrors of the transfer optics should never be touched or handled. However, the mirrors do have a protective magnesium fluoride overcoat to allow cleaning if necessary. The following procedure is recommended:

1. Spray the mirror with Fantastic Spray Cleaner.
2. Rinse the mirror with distilled water.
3. Repeat until the water sheets off the mirror.
4. Blow dry with a stream of clean nitrogen.

C. Spectralon Care

In order to maintain the unique optical and reflectance properties of Spectralon standards, keep the surfaces of the material very clean. Should Spectralon become soiled, particles may be blown off with a jet of clean air. If this is insufficient, sand the piece with 220-240 grit emery cloth under a stream of running water. Sand the surface until it is totally hydrophobic (water beads and runs off immediately) and then shake off water droplets, or blow them off with a stream of clean air.

Note: Ordinary compressed-air systems typically carry microscopic oil droplets which may contaminate Spectralon.

VII. TROUBLE CHECKS

A. Electronic Theory of Operation

The RSA-HP-84 Reflectance Spectroscopy Accessory uses the internal lamp of the HP-8452 instrument to activate a separate shutter mechanism. This shutter is used to allow the sample image to be seen by the detector array within the instrument. The shutter within the accessory is activated by opening the shutter within the instrument by use of a silicon detector and a driver board.

The shutter driver board works in combination with the shutter in the HP-8452 instrument. When the shutter in the instrument opens, the light from the instrument is seen by the silicon detector mounted to the side of the RSA-HP-84 accessory. The signal from the silicon detector is passed back through a high gain transimpedance amplifier circuit. From here the resulting voltage is passed through a driver circuit. The driver circuit is comprised of a voltage detector and a MOSFET driver amplifier. The voltage detector measures for voltage within a specific range and creates either a HI or LOW depending on the signal. This is passed along to a drive amplifier which drives a low power MOSFET. The MOSFET is used as a switch. When a HI is past along to the driver it creates a resulting HI on the gate of the MOSFET. The MOSFET is part of the load line out to a solenoid that drives the shutter mechanism. When the MOSFET is turned on, it creates a current path that allows current to flow to the solenoid. This current engages the solenoid which in turns activates the shutter in the accessory.

There is a single +12 volt DC power supply used to generate the various bias supplies for the accessory. The +12 volts is fed to an LM309K +5 volt voltage regulator. The output voltage from it (+5v) is fed to the lamp connector mounted at the rear of the main compartment. The lamp is then plugged into it.

B. Quick Troubleshooting Chart

Symptom	Preparation	Result
Shutter in RSA-HP-84 does not operate.	Remove the accessory from the HP-8452. Turn the accessory on while it is on the bench top. Use a flashlight to get light to the detector on the side of the accessory.	If the shutter activates, then the lamp in the HP-8452 is low. If it does not activate there is an internal problem in the accessory. Contact Labsphere for service.
Accessory lamp does not come on.	Remove the lamp fixture from the top of the sphere by loosening the two recessed set screws within the base of the fixture where it attaches to the sphere.	Visually inspect the lamp for any breaks either in the glass or the filament. While wearing protective gloves, push in and turn bulb counter-clockwise to replace bulb, if necessary. To obtain the correct replacement bulb, (or if no faults are found in the lamp) contact Labsphere.
Shutter appears to stick.	Remove the accessory from the HP-8452. Turn the accessory on while it is on the bench top. Use a flashlight to get light to the detector on the side of the accessory.	If the shutter sticks under test, gently press against it to reseal it. If the problem persists contact Labsphere for service.

VIII. CUSTOMER SERVICE

Customer Service is one of Labsphere's most important products! Every Labsphere product includes the finest support and service to assist you in making the most of your state-of-the-art equipment. If you have any questions or comments regarding any of Labsphere's products, please call:

(603) 927-4266

and ask for the Customer Service Department.

APPENDIX A

Technical Notes

Substitution Error

The substitution error, sometimes referred to as the single beam sample absorption error, is a systematic, predictable and non-random error inherent in single beam integrating spheres measuring reflectance or transmittance. The error is caused by the difference between the throughput of the sphere when the reference makes up a portion of the wall and the throughput when the sample is substituted for the reference. For reflectance measurements the throughput is usually lower when the sample is present since a reference material of high (nearly 100%) reflectance is usually used. For transmittance measurements the throughput is usually higher when the sample is present since an open port (zero reflectance when viewed from inside the sphere) is usually used as a reference.

Methods of Correction

When the sample and reference are of similar reflectances the substitution error is negligible. Even when the sample and reference are not of similar reflectances, at worst, the substitution error may reach only three or four percent and in many cases it is simply ignored. In quality control applications where a threshold value is used, the substitution error can be incorporated into this threshold. For chemical analysis where the locations of absorption peaks, not necessarily their exact absorbance value, are important the error can also be ignored. Where correction of the substitution error is necessary, the following techniques are available.

Double Beam

Instruments which chop between the sample beam and the reference beam have both beams present in the sphere at essentially the same time. In this case, since the sample beam and reference beam each "see" the same sphere, there is no substitution error.

Dummy Port

When room permits, single beam spheres can be designed with an additional "dummy" port. The background correction is performed with the sample to be measured in the dummy port and the physical reference in the sample port. A sample scan is then run with the positions of the two switched. In this comparison method the average reflectance (hence the throughput) of the sphere remains unchanged from the reference scan to the sample scan. This technique requires that a separate reference scan be run for each sample (unless the samples are all very similar and the residual substitution error caused by sample-to-sample differences can be ignored).

Matched Sample

If a reference is used which is very close in reflectance to the sample, the substitution error is negligible.

Low Reflectance or Transmittance

For samples with very low reflectance or transmittance the substitution error will be very small. For a sample with zero reflectance or transmittance, there is no substitution error at all. For low reflectance and transmittance samples, the substitution error is so small that it probably falls within the random noise of the instrument.

Calibrated Standards

For single beam spheres without dummy ports in applications where substitution error is a concern, the spheres can be calibrated with a set of standards such as one of Labsphere's Reflectance Standards Sets, which has been measured on a sphere without substitution error. With these standards, a table of measured vs. actual readings can be generated and used to correct for substitution error.

The RSA-HP-84 is equipped with a dummy port for correcting for substitution error when desired.

Reflectance Measurement of Non-Ideal Samples

Two simple ideal geometric reflectance distributions may be described: perfect specular reflection, and perfect diffuse reflection. Perfect specular reflection is simply reflection according to Fresnel's law, without scattering (or diffusion). Perfect diffuse reflection is reflection according to Lambert's law, and is therefore described as "lambertian." (See definitions below.) For the purposes of integrating-sphere-based reflectometry, the Spectralon standards provided with this accessory serve as an excellent approximation to the ideal of perfect lambertian reflection, while a clean, flat, first-surface mirror provides an excellent approximation to the ideal of perfect specular reflection.

If the sample to be measured closely approximates either of these ideal reflectance distributions, it is not difficult to select an appropriate reference standard. Many samples, however, do not conform to this requirement. Samples which are neither nearly specular nor nearly lambertian should, if possible, be measured relative to a standard with similar geometrical scattering properties. If calibrated standards which meet this criterion are not available - as is often the case - another approach must be taken.

The first such alternate approach is simply to measure the sample as if it were either nearly lambertian or nearly specular, and accept that such a measurement involves a higher-than ordinary degree of systematic uncertainty. Such an approach is more than adequate for many applications, especially those in which the emphasis is on repeatability, rather than absolute accuracy of measurement.

A more ambitious approach involves modeling the geometric reflectance distribution of the sample in question as an ideal "mixed" distribution, composed of two components, one of which is perfectly specular, and the other, perfectly lambertian. The sample is measured in both total hemispherical (specular included) and diffuse (specular excluded) geometries, and the relative magnitude of the two components is estimated from the results. Systematic errors involved in measuring a partly-specular sample against a diffuse reference, or a partly-diffuse sample against a specular reference, can then be corrected in accordance with this model. An example of such an approach is presented below.

Such an approach is most suitable for flat, polished samples with a distinct specular component. Many samples, however, exhibit a non-ideal “gloss,” i.e., a broadened specular peak, which is not completely captured by the specular-exclusion light trap. Many other samples exhibit a diffuse component which does not conform closely to the lambertian ideal. These deviations from the ideal reduce the accuracy of the correction prescribed by the ideal model. The accuracy of such correction also depends upon the efficiency with which the instrument removes the specular component of reflection when in the specular-excluded configuration.

Example

A first-surface mirror is measured with the instrument in the standard hemispherical configuration, first using a specular reference standard (M), then using a diffuse reference standard (M'). The geometric correction factor, γ , is calculated as follows:

1. $\gamma = M/M'$

Next, the test sample is measured, using a diffuse reference, in both the standard hemispherical ('specular included') and diffuse ('specular excluded') configurations. The results are given as R_{SIN} and R_{SEX} , respectively.

Given the assumption that the geometric reflectance distribution of the test sample can be separated into two ideal components, one specular (S) and one perfectly lambertian (D), and further assuming that the reference sample approximates a perfect lambertian distribution, so that its reflectance factor (ρ_o) is the same for both specular-included and specular-excluded geometries, the following relations hold:

2. $R_{SEX} = D/\rho_o$

3. $R_{SIN} = (S' + D)/\rho_o$

4. $D = R_{SEX} * \rho_o$

5. $S' = (R_{SIN} - R_{SEX})\rho_o$

where S' represents the contribution of the specular component of the test sample to the apparent hemispherical reflectance factor of the sample. This quantity is analogous to the value M' given in equation (1), and its relation to the actual specular component of hemispherical reflectance (S) is described in equation (6):

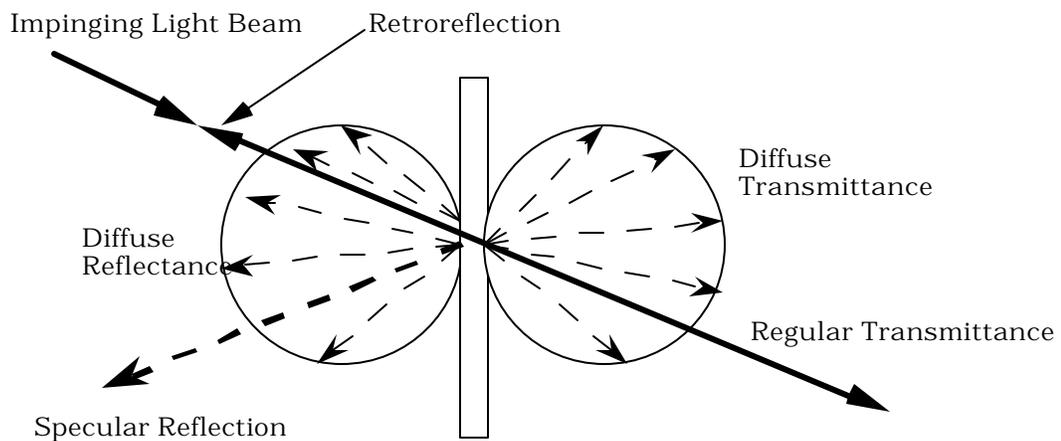
$$6. S = \gamma S'$$

$$7. S = \gamma(R_{SIN} - R_{SEX})\rho_o$$

On this analysis, the best estimate for the hemispherical/8° reflectance factor of the test sample (R_T), is given as follows:

$$8. R_T = S + D = (\gamma R_{SIN} + (1-\gamma)R_{SEX})\rho_o$$

Note that this is not intended as a rigorous treatment of the problems associated with the measurement of samples of mixed reflectance, but only as an illustration of the sort of analysis which may be used to reduce some of the systematic errors associated with such measurements. For a more comprehensive treatment of errors associated with reflectance-factor measurement, see the references listed at the end of this manual.



Idealization of Reflectance and Transmittance Phenomena Showing Components*

Measurement of all of the above components except retroreflection may be made using the Labsphere RSA accessory. A general technique for each is described below.

* From ASTM E 179-91a

Transmittance Measurement of Scattering Samples

For scattering samples, there are three primary types of transmittance measurement: These are measurements of *total transmittance*, *diffuse transmittance*, and *regular transmittance*. All measurements are taken after a background correction (blank) scan of the instrument has been taken. The background correction calculates the amount of incident light entering the sphere; it should be taken with Spectralon standards in both the sample and dummy ports and with a sample cuvette (if used) fitted in front of the transmittance port. (See DEFINITIONS below.)

Measurement of Total Transmittance (T_t):

To measure the total transmittance of a sample, the sample scan is performed with the sample to be measured fitted in front of the transmittance port, and Spectralon standards are placed in both the sample and dummy ports.

Measurement of Diffuse Transmittance (T_d):

Diffuse transmittance, which measures the amount of light that is scattered after passing through a sample, can be determined by performing the sample scan with the sample to be measured in the transmittance port, a Spectralon standard in the dummy port, and a light trap* in the sample port. The light trap is located across from the transmittance/entrance port so that the directly transmitted portion of the sample spectrum is eliminated from the results.

Determination of Regular Transmittance (T_r):

Regular transmittance can be calculated using the numerical results of the first two scans. Regular transmittance is the part of the incident light that is transmitted without scattering. The total transmittance minus the diffuse transmittance equals the regular transmittance component of the sample spectrum.

$$T_r = T_t - T_d$$

*Instead of using a light trap during diffuse transmittance scans on the RSA-HP-84 accessories, the sample port can be left open (empty). The open sample port will effectively serve as a light trap, with transmittance only slightly greater than zero. If an open port scan is performed, the room must be darkened so that external light does not enter the sphere and interfere with the transmittance measurements.

Note: The *regular transmittance factor*, as defined by the ASTM, should not be confused with *regular transmittance*. (See DEFINITIONS below.) *Regular transmittance* is a real property of the sample, which may be more or less accurately measured by different instruments.

Regular transmittance factor, on the other hand, is an instrument-specific quantity, not merely in practice, but by definition.

Definitions

The following definitions are given in ASTM Standard E-284-90:

diffusion: change of the angular distribution of a beam of radiant flux by a transmitting material or a reflecting surface such that flux incident in one direction is continuously distributed in many directions, the process not conforming (on a macroscopic scale) to the laws of Fresnel (regular) reflection and refraction and there being no change in frequency (wavelength) of the monochromatic components of the flux.

Lambertian diffuser: ideal surface that reflects or transmits radiation completely in accord with Lambert's cosine law. When illuminated from any direction, its radiance is the same for every direction of view.

Lambert's law: the intensity (flux per unit solid angle) emitted in any direction from a surface varies as the cosine of the angle between the normal to the surface and the direction of the emitted flux (also called Lambert's cosine law).

Reflectance

reflectance: ratio of the reflected radiant or luminous flux to the incident flux in the given conditions.

Discussion - The term *reflectance* is often used in a general sense or as an abbreviation for *reflectance factor*. Such usage may be assumed unless the above definition is specifically required by the context.

reflectance factor: ratio of the flux reflected from the specimen to the flux reflected from the perfect reflecting diffuser under the same geometric and spectral conditions of measurement.

diffuse reflection: reflection in which flux is scattered in many directions by diffusion at or below the surface.

specular reflection: reflection without diffusion, in accordance with the laws of optical reflection, as in a mirror.

mixed reflection: partly specular and partly diffuse reflection.

perfect reflecting diffuser: ideal reflecting surface that neither absorbs nor transmits light, but reflects diffusely, with the radiance of the reflecting surface being the same for all reflecting angles, regardless of the angular distribution of the incident light.

Transmittance

transmittance: the ratio of transmitted flux to incident flux, under specified geometric and spectral conditions.

transmittance factor : ratio of the flux transmitted by the specimen to the flux transmitted by the perfect transmitting diffuser under the same geometric and spectral conditions of measurement.

regular transmittance : ratio of undiffused transmitted flux to incident flux.

diffuse transmittance: ratio of the flux transmitted by a specimen to the incident flux, the transmitted flux being measured for all forward angles except the regular transmittance angle.

total transmittance: the ratio of the flux transmitted at all forward angles to the incident flux.

* **regular transmittance factor:** the ratio of the flux transmitted by a specimen and evaluated by a receiver to the flux passing through the same optical system and evaluated by the receiver when the specimen is removed from the system.

Discussion-In some cases, this quantity is practically identical to the transmittance, but it may differ considerably. It exceeds unity if the system is such that the specimen causes more light to reach the receiver than would in its absence.

mixed transmission: a combination of diffuse and regular transmission.

perfect transmitting diffuser: ideal transmitting specimen that neither absorbs nor reflects light, but transmits diffusely, with the radiance of the specimen being the same for all transmitting angles, regardless of the angular distribution of the incident light.

APPENDIX B

Typical Hemispherical/8° Reflectance Factor for SRS-99-010

Wavelength (nm)	Reflectance
400	0.991
500	0.991
600	0.992
700	0.992
800	0.991
900	0.991
1000	0.992
1100	0.992

APPENDIX C

Recommended Reading

Reference Materials for Diffuse Reflectance

- Brunsting, A., R. S. Hernaiz and A. J. Dossman, "Small Area Measurements of Diffuse Reflectance from 410 nm to 700 nm", *Applied Optics*, Vol. 23, No. 23, 1984.
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