Diffuse Reflectance Accessory (external)

Note: This document is also available in PDF format for improved print quality. PDF files are stored in the "**Manuals**" folder on the Help & Videos CD-ROM.

For the Cary 4000, 5000 and 6000i

Part Numbers: External DRA 900: 00-100818-00
External DRA 1800: 00-100819-00
External DRA 2500: 00-100820-00

Last updated:

Installation category II
Pollution degree 2
Safety class 1 (EN 61010-1)
Overvoltage category I
Refer to the Safety section of the online Help for an explanation of the warnings and cautions used in this document.

Table of Contents

Introduction

The traditional use of the spectrophotometer is to measure the absorbance or transmission of a clear or translucent liquid or solid. Typically UV/VIS/NIR spectroscopy applications include reaction kinetics, quantitative analysis or the identification of the chemical constituents in a substance. The measurement of reflectance offers an added dimension to the spectroscopy capabilities of your spectrophotometer, the analysis is no longer limited to the portion of the beam that penetrates the sample. Accordingly, the analysis is no longer limited to samples that are clear or translucent. The DRA provides the means of measuring absorbance or transmission of opaque, turbid and reflecting substances.

Figure 1. The external DRA.

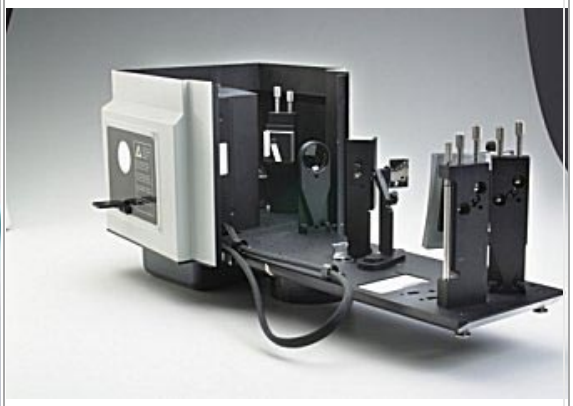


Figure 2. The external DRA viewed from the back.

There are three versions of the diffuse reflectance accessory that operate over different wavelength ranges.These are specified below. While all of the following variations are available, the configurations indicated by ticks in the table below offer optimal operation.

	(External DRA 900)	**(External DRA 1800)**	**(External DRA 2500)**
Cary 4000	☑	☒	☒
Cary 5000	☑	☑	☑
Cary 6000i	☑	☑	☒

The Varian 900, 1800, 2500 External Diffuse Reflectance accessory (DRA) consists of a 150 mm diameter integrating sphere. The sphere is easily installed in the sample compartment of the instrument and features an inbuilt high performance photomultiplier tube (PMT). The DRA also has a Lead Sulfide (PbS) or InGaAs detectors which become active Varian 2500 External DRA. The coating is Polytetrafluoroethylene (PTFE), which

exhibits NIR performance that is superior to traditional coatings[1], whilst maintaining UV-Vis performance.

Warning
If the DRA is not used in the manner specified by the manufacturer, the protection provided by the accessory may be impaired.

Theory

Reflection consists of two components: specular and diffuse. Specular reflectance is the mirror-like reflection off a sample surface. Diffuse reflectance occurs when the surface reflects light in many different directions, giving the surface a matt finish.

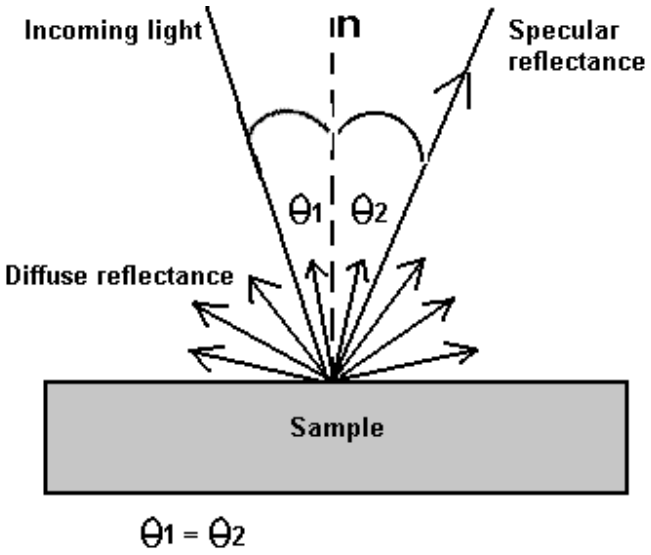


Figure 3: *The two components of reflection: specular and diffuse reflection. n represents the surface normal, an imaginary line at 90° to the sample surface.*

Traditionally, the accessory used to measure diffuse reflectance is the integrating sphere. Applications include characterizing solar materials, color measurement and characterization, and obtaining reflectance spectra of a painted surface. Integrating spheres have also proven ideal for measuring the transmission of turbid, translucent or opaque refractory materials where standard techniques proved inadequate due to loss of light resulting from the scattering effects of the sample. Samples which distort the beam of the instrument, such as a lens, can also be studied with the Diffuse Reflectance accessory.

The DRA is an integrating sphere accessory that replaces the sample compartment of the spectrophotometer instrument. An integrating sphere is a hollow optical device, either constructed from or coated internally with a white diffusing material and fitted with the same detector configuration as the host spectrophotometer. The coatings used in an

integrating sphere usually depends on the wavelength capabilities demanded by the reflectance application.

The integrating sphere configuration offers some distinct advantages over the standard sample compartment. The changes made in sample beam and reference beam geometry on your accessory do not compromise the transmission and absorption measurement capabilities already inherent to the spectrophotometer the traditional sample transmission and absorbance measurements can be performed in the same manner, with or without the DRA. Because of the geometry of the integrating sphere, it has the ability to collect most reflected or transmitted radiation, remove any directional preferences, and present an integrated signal to the detector.

The DRA accessories each include an optical bench composed of double beam transfer optics in combination with a six-inch (150 mm) diameter integrating sphere. The integrating spheres are machined from SpectralonTM a highly diffuse reflectance material. The accessory is equipped with a movable mirror in the sample beam optics to allow the beam to be focused at the transmission, reflectance, or centre-mount sample positions. This feature adds versatility for measuring various size samples. The external DRA accessories are designed to perform reflectance, transmission, or absorbance measurements of diffuse, specular, or mixed samples. The Scan application of the Cary WinUV software is usually used when operating the accessory.

Reflectance measurements

First, a baseline is recorded with the PTFE reference disk covering the reflectance port. The sample is then mounted over the port and the reflection off the sample surface is collected by the sphere. The reflectance is therefore measured relative to the PTFE disk. This is the 'substitution' method.

The total (diffuse and specular) or the diffuse-only reflectance may be measured by using either the specular plug (specular included) or the light trap (specular excluded). The specular component may be calculated from the difference of these two, or the Cary Absolute Specular Reflectance Accessory (SRA) may be used to give an absolute value of the specular component.

A variety of sample types and sizes may be used with the accessory, in conjunction with different sample holders. For powders, pastes or other material requiring a sample container, a powder cell is available for use in the DRA.

If only very small amounts of powder or paste are available, or if measurements extending beyond the range of 250–2500 nm are required, the DRA cannot be used. The 'Praying Mantis' accessory (P/N 00 100469 00) is used for these measurements.

Transmittance measurements

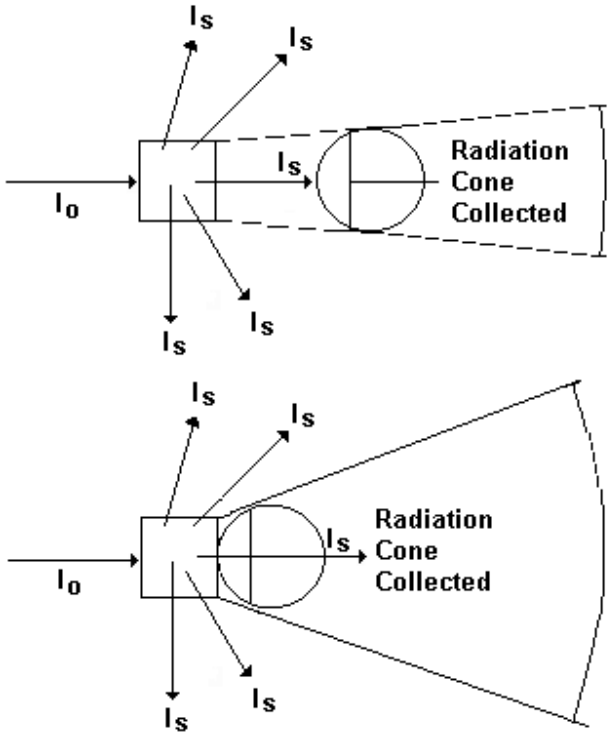


Figure 4: *Collection of scattered light by an integrating sphere. I_o = incident light, I_s = scattered light.*

In order to perform useful measurements on scattering samples, it is necessary to collect a high proportion of the scattered radiation. The integrating sphere is a highly efficient collector of scattered radiation. Because of its design, the Diffuse Reflectance accessory overcomes many of the problems associated with measuring turbid or scattering samples, which include sloping baseline, poor signal-to-noise ratio and high background absorbance.

Whilst the DRA should be used when measuring the transmission of opaque or diffusing solid samples, the accessory may also be used when measuring turbid liquids.

Because the cuvette is located so as to be part of the wall of the sphere, a greater proportion of the scattered radiation transmitted by the sample is collected by the sphere, as shown in Figure 3 above.

With the PTFE reference disk in position, a sample is placed in the cuvette, and light is passed through the sample.

Factors affecting accuracy or precision

Below are the major factors that may affect the accuracy of measurements when using the DRA.

Aperture area/total surface area ratio

Some of the reflected light escapes through the ports. This has the effect of reducing the signal to noise ratio, and thus the precision of the measurement.

The Commission Internationale de l'Eclairage (CIE) recommendation is a ratio of <10%. This figure is < 10% for the Cary External DRA.

Coating non-uniformity, ageing, or contamination

This accessory has been coated with PTFE via a unique process that ensures both a uniform coating on the inside of the sphere and the correct powder density. PTFE is durable, and does not yellow. The reflectivity of the PTFE is above 96% between 200–2500 nm, and greater than 99% between 350–1800 nm. The sphere will maintain its reflectivity indefinitely if not subjected to smoke or other contaminants. Contact with plastic materials may also contaminate the coating.

Incorrect sample placement

Theory assumes that the sample placement will coincide with the inside of the sphere wall. The sample is normally placed at a port on the outside of the sphere wall. Spacing between the sample and the sphere wall can lead to large errors due to loss of reflected light.

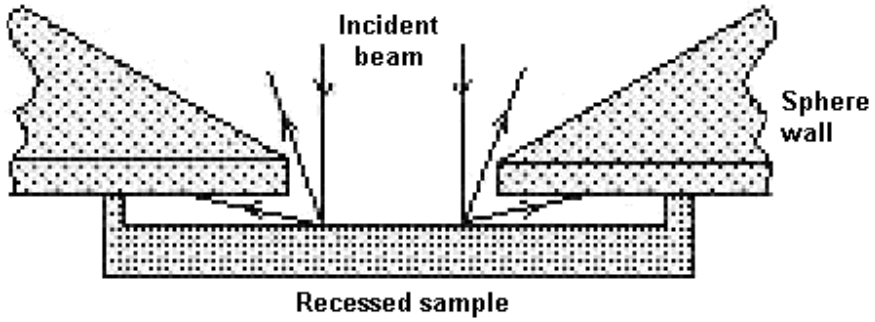


Figure 5: *Some of the wide-angle scatter is lost when there is a space between the sample and the sphere wall*

Sample recess

Theory assumes that the sample is placed coincident with the inside of the sphere wall, however the sample is placed against the outside of the sphere wall. The porthole edges have a finite thickness, and some part of the beam reflected at wide angles may be intercepted by the sphere wall.

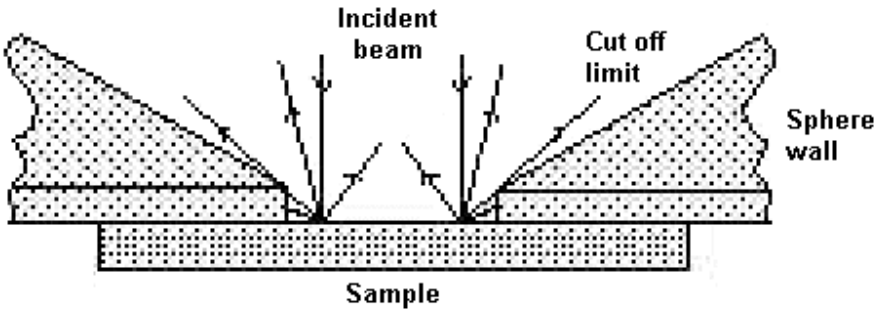


Figure 6: *Some of the wide-angle reflection is intercepted by the sphere wall*

The edges of the reflectance port are feathered to reduce this error.

Reference beam attenuation

Reference beam attenuation is most useful when the accessory or sample in the sample beam attenuates the light beam considerably. In such situations, attenuation of the reference beam will increase noise and considerably increase the dynamic range of the instrument, as the detector is not then 'seeing' two dramatically different signals. Clips are provided at the reference beam window for this purpose. Metallic mesh screens are recommended if reference beam attenuation is required (part number: 0110677500).

Stray light

If the irradiating beam overfills the reflectance port, a proportion of sphere wall reflectance is mixed with that of the sample. This gives a high reading if this is not consistent between the baseline and the sample measurement.

Gloss trap error

Gloss trap error is produced when the gloss trap is unable to completely absorb the specular component. In the measurement of diffuse-only reflectance, a 'gloss trap' is often used to absorb the specular component. Gloss traps are typically glossy black pyramidal light traps, matt black-coated cavities, or razor blade Fresnel light traps. However, the reflectance of many samples has broadened the specular peaks which cannot be excluded by standard sized light traps.

Differences between the standard and sample

It is important that the reference material be of a similar reflectivity and have similar properties to the sample. Otherwise large errors can be introduced, if for example a poorly reflecting material is measured relative to a highly reflective PTFE reference plate. The table below lists the appropriate reference materials that are recommended for use.

Surface type	Reflectivity	Reference
Matt	High	PTFE reference plate
Matt	Low	Labsphere diffuse reflectance standards
Glossy	High	PTFE reference plate
Glossy	Low	NIST SRM 2021

The Troubleshooting section contains information on the available reference surfaces.

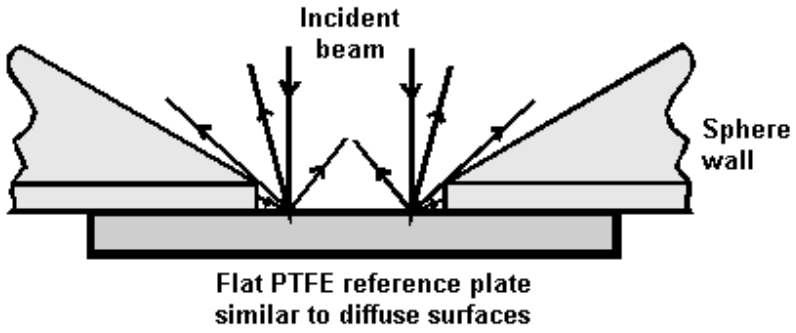


Figure 7: *Highly diffuse samples are measured against a flat PTFE reference plate*

Inaccuracy in the standard

If the standard material used to calculate a value is not accurate, this will hamper the determination of the reflectance of the sample. Great care must be taken to keep reference materials clean and unscratched.

For a detailed discussion on the factors affecting the measurement of diffuse reflectance and transmission with integrating spheres, refer to reference (2).

Description of the accessory

Accessory design

The DRA accessories are designed specifically to measure the reflectance or transmission of solids, liquids, powders, or other small objects that can fit at the transmission or sample reflectance ports. Except for the sphere detectors, the construction of each version of the DRA is identical. Basic components of the accessory include the base plate, integrating sphere, optics chamber and detector chamber. The basic components are illustrated in the figure below.

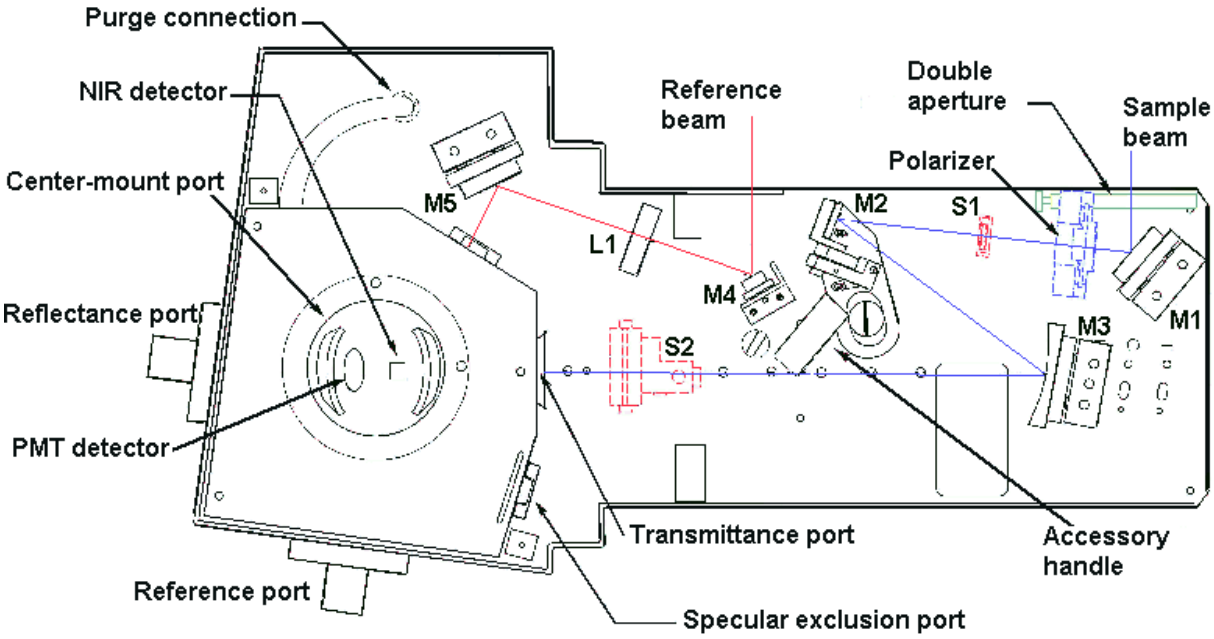


Figure 9: *A schematic view of the external DRA.*

The optics chamber contains the transfer optics that direct the spectrophotometer beams to their final destinations. The integrating sphere collects and measures the radiation transmitted or reflected from the sample surface. A removable cover fits over the DRA to provide a light-tight operating environment. The sample reflectance and reference ports each are fitted with a separate magnetic port cover. The purposes of the covers are to reduce the potential for stray light when the accessory is in use and to keep the integrating sphere and standards clean when not in use.

Beam paths within the accessory are illustrated in the next figure. Light entering the accessory is directed to one of two entrance ports on the sphere: the reference beam and sample beam entrance ports. The sample beam entrance port is usually called the transmission port.

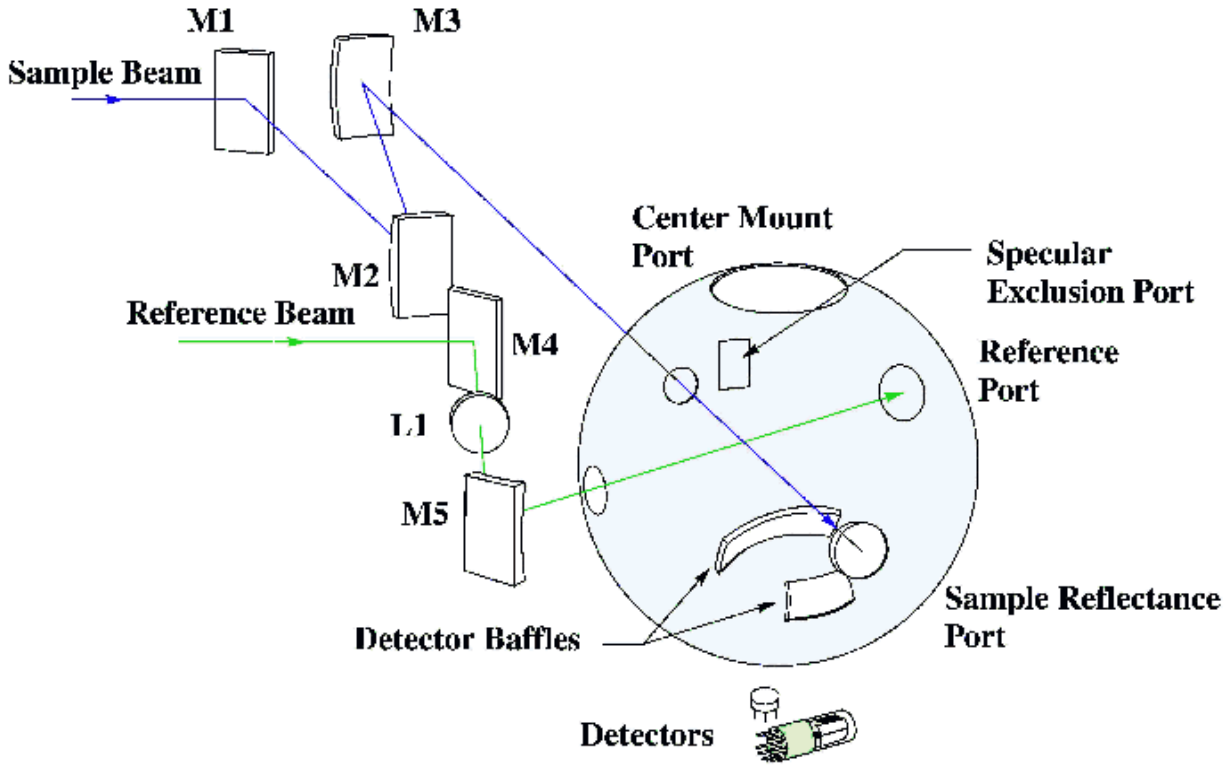


Figure 10: *The optical design of the external DRA.*

Integrating sphere

An integrating sphere is an optical device used to collect and measure electromagnetic radiation. The radiation sampled by the DRA integrating sphere is provided by the spectrophotometer reference and sample beams present inside the sample compartment. Upon entering the sphere, the light strikes the sample surface and the highly reflective walls of the cavity, undergoing many diffuse reflections. Radiation trapped inside the sphere cavity can dissipate by a combination of three ways. Most of the light eventually is absorbed into the sphere walls. Some of the light exits the cavity through one of the empty ports, and a small portion of the trapped radiation strikes the active surface of the sphere detector. A steady state radiant flux distribution is established almost instantaneously within the sphere and remains as long as the beam source is active. All integrating spheres possess one unique property with regard to a steady-state flux: the radiance all along the wall surface of the sphere is uniform and proportional to the flux of the source input. The sphere detector samples the radiance at the wall surface and relays the detector signal to the spectrophotometer for processing.

Optics

The optics chamber houses the transfer optics of the DRA accessory that direct the spectrophotometer reference and sample beams to their respective ports on the integrating sphere. The sample beam is the rear beam in the accessory. Mirrors labelled M1, M2 and M3 guide the sample beam through the sample transmission port of the integrating sphere and onto the sample reflectance port at an 8° angle of incidence. Mirrors M4 and M5 direct the reference beam through the reference beam entrance port onto the reference port.

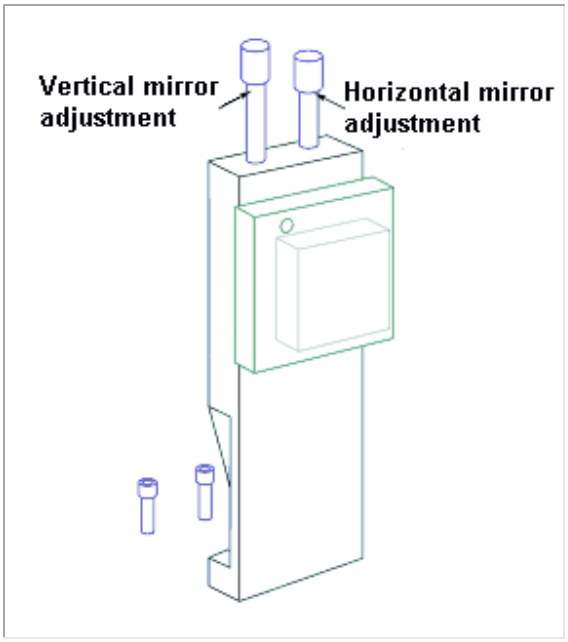


Figure 11: *Mirror M1.*

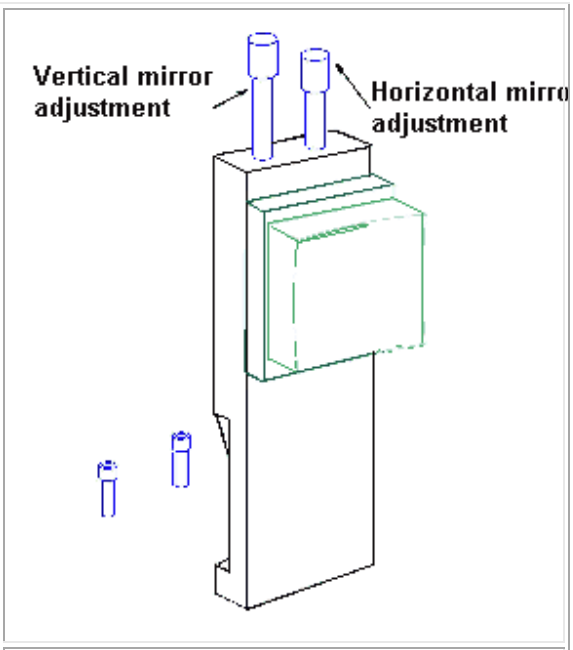


Figure 12: *Mirror M5.*

Mirrors M1 and M5 are fixed mounted mirrors that are adjustable in the vertical and horizontal planes. Mirror M1, illustrated in the figure above, holds a flat mirror that helps direct the instrument sample beam into the integrating sphere transmission port. M5 holds a concave mirror surface that reflects the reference beam into the integrating sphere. The design of mirror M5, shown in the figure below, is very similar to mirror M1. The fixed mirrors are mounted to the base plate by two M5 bolts that can be adjusted using a 4 mm hex wrench. All mirror surfaces are coated with a Al/MgF_2 thin film.

Mirror M2 is a pivoting mirror assembly that positions a flat mirror surface into the sample beam path, directing the beam onto the movable mirror, M3. The mirror mount pivots about a fixed point. To rotate the mirror assembly horizontally, loosen the two screws shown in the figure and rotate the floor adjustment with a large flathead screw driver. To adjust the mirror surface vertically, use a 3 mm allen wrench.

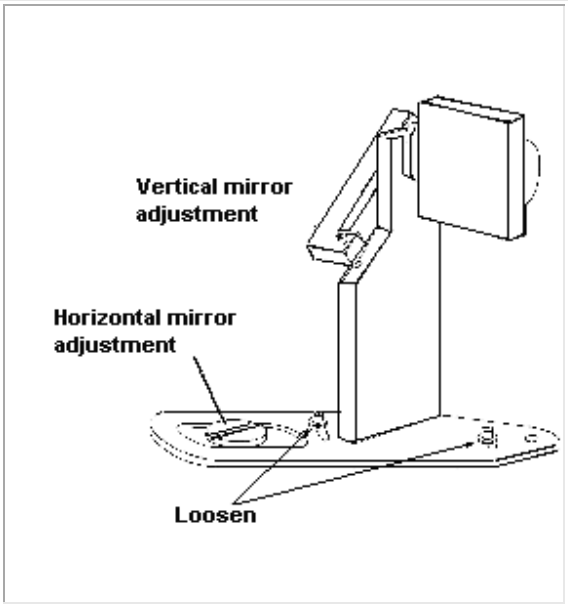


Figure 13: *Mirror M2.*

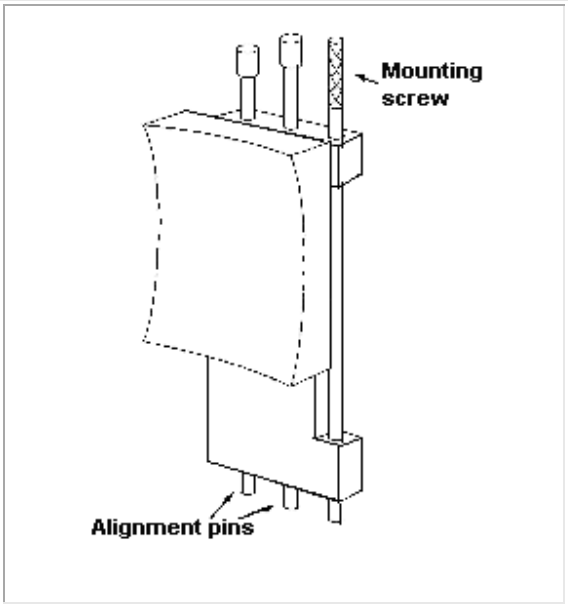


Figure 14: *Mirror M3.*

Mirror M3 is a spherical mirror that condenses the sample beam onto the target sample. The mirror can be mounted at any of three different positions on the base plate of the DRA. These positions correspond to the transmission port, centre mount sample holder and sample reflectance port, and are labelled "T", "C" and "R" respectively. M3 features a knurled mounting screw for convenient installation and removal as well as the standard vertical and horizontal adjustments. The mirror is illustrated below.

Note
If you move M3 to a different position, you will need to realign the transfer optics.

Mirror M4 directs the raw reference beam through the collimating optic and onto mirror M5. The mirror mount is constructed in a manner to prevent stray light scattered from mirror M1 from reaching the integrating sphere. The mirror has a single adjustment in the vertical plane. If beam adjustment is required in the horizontal plane, loosen the cap screws at the base of the mirror mount and rotate the device slightly in either direction.

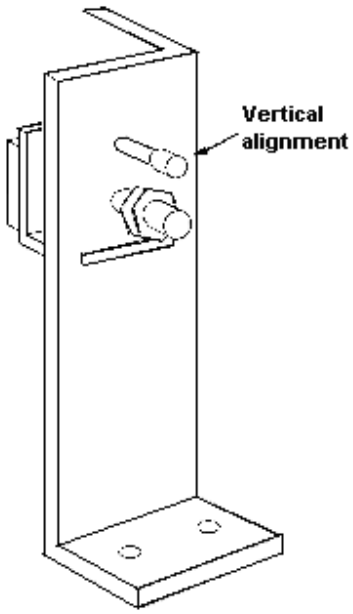


Figure 15: *Mirror M4.*

The accessory is shipped from in a protective wooden case. The accessory should be stored in the case when the DRA is not being used.

Detector

The detector chamber attaches to the bottom of the base plate and is located directly underneath the integrating sphere.

Reflectance standards

The DRA is shipped with three Spectralon reflectance standards. If a standard is calibrated, a calibration certificate that lists the reflectance data across the wavelength spectrum will be included. The typical reflectance values may be very close to the actual spectral reflectance of the standards, but the typical data is not traceable to NIST. The uncalibrated standards should be loaded at the reference and sample ports to protect the internal surfaces of the sphere when the accessory is installed but not in use.

Cleaning instructions for uncalibrated reflectance standards are provided when the standards are purchased. Calibrated standards can be returned to Labsphere for recalibration yearly, or whenever the reflective surface of the standard is soiled or damaged.

You may want to convert the reflectance values for your standards to a file compatible with Cary WinUV. This can be accomplished by copying the calibrated or uncalibrated reflectance data and pasting it into a spreadsheet such as Microsoft EXCEL. Save the

data in the comma separated variable format (.CSV) and paste the file into the Scan data directory. Consult the online help for more details.

Transmission sample holder

The external DRA accessory uses either a clasp-style or cuvette-style sample holder that fits over the dovetail mount at the transmission port of the integrating sphere. The clasp-style device, shown below, is best suited for solid samples large enough to completely fill the transmission port. The optional cuvette sample holder mounts a liquid filled cuvette at the transmission port.

Specifications

		DRA 900	DRA 1800	DRA 2500
Wavelength range	**Cary 4000**	200 nm to 900 nm *	200 nm to 900 nm	200 nm to 900 nm
	Cary 5000	200 nm to 900 nm *	200 nm to 1800 nm *	200 nm to 2500 nm *
	Cary 6000i	200 nm to 900 nm *	200 nm to 1800 nm *	200 nm to 1800 nm
Detectors				
	UV/VIS	R928 PMT	R928 PMT	R928 PMT
	NIR		TE* cooled InGaAs	TE* cooled PbS
Sphere diameter		150 mm		
Internal coating		Polytetrafluoroethylene		
Coating density		1 g/cm3		
Coating thickness		4 mm		
Port area/total surface area ratio		<10% (CIE recommendation: <10%)		
Minimum sample size[#]		~ 20 mm (3/4")		
Maximum sample size		Unlimited		
Power input		Sample compartment DRA connector 15-pin D-range connector with two high voltage pins: -1000 volts DC.		

Purge gas connections	Clean flexible tubing of 6 mm (1/4") inside diameter (Tygon PVC or equivalent).
Size packed	W x L x H, 500 mm x 840 mm x 470 mm
Size unpacked	W x L x H, 330 mm x 660 mm x 300 mm
Weight packed	35 kg
Weight unpacked	12 kg

* Thermoelectrically cooled.
[#] The optional small spot kit, with the aperture kit, permits the measurement of samples down to approximately 5mm.

The accessory is suitable for indoor use only. Suitable for Installation Category I and Pollution Degree 2. Environmental conditions are the same as for the Cary spectrophotometer.

Getting started

This section describes how to unpack, install, and align your Diffuse Reflectance accessory.

Caution
This accessory includes mirrors, lenses and other fragile parts.
Treat the packaging and accessory with care. Do not jar the accessory. Avoid finger contact with mirrors and lenses. Do not attempt to clean or repair damaged surfaces.

Caution
If the equipment is not used in a manner not specified by the manufacturer, the protection provided by the equipment may be imparied.

Unpacking

Open the shipping case and the enclosed packages with care.

Warning
Do not touch the mirrors or other transfer optics mounted on the accessory. Fingerprints will induce optical scattering and may reduce reflectance or transmission of these components at certain wavelengths.

As well as the particular external DRA that you have ordered, included in the package are the following:

Item	Description	Part number
3 x Uncalibrated PTFE reference plates	Three PTFE plates for collecting baselines and performing calibrations.	XXXX
1 x Accessory cover (installed)		XXXX
2 x Magnetic sample port covers (installed)	Magnetic covers for the reflectance and reference ports.	XXXX
1 x Light seal/guillotine	I metal plate to seal the join between the DRA and the sample compartment.	XXXX
1 x Lockdown pin	Metal pin to hold DRA into place.	XXXX
2 x Dovetail sample ledge (installed)	Metal ledges to assist in mounting of samples and holders.	XXXX
2 x Spring loaded sample holder	Round sample holder on a spring loaded base for use at the reflectance and reference ports.	XXXX
1 x Specular port plug		XXXX
1 x Specular light trap	Metal plate with PTFE cut-out to seal the specular port.	XXXX
1 x Centre port plug	Round PTFE plate to fit into the centre mount port.	XXXX
1 x Transmission port holder	Metal plate with two clips, for mounting of solid samples and the cuvette holder.	79 100479 00
1 x M3 moveable mirror	Mirror that can be mounted in the Centre, Reflectance or Transmission position depending on the type of measurement being taken.	XXXX
1 x Purge adaptor	Stainless steel adaptor for applying a nitrogen purge to the DRA.	XXXX
1 x 5/32 Hex driver		XXXX
1 x Allen key		XXXX
1 x Test results	Results of factory tests on the DRA.	Not applicable
1 x Cary WinUV Help and Video CD Rom		85 101953 00
1 x Patches CD ROM		XXXX

XXXX These part numbers were not available at the time of publishing, please refer to the Varian website, for the most up to date information. See www.varianinc.com

Inspect all parts for damage in transit. Any damage should be reported immediately. Please refer to the accessory by model and serial number.

1. Unpack M3 and screw it into the "R" position. Set the mirror alignment pins into the correct locator holes and secure the mount with the mounting screw. Refer to figure 19.

2. Remove the accessory cover.

Figure 16. Removing the accessory cover.

3. Remove the light seal/guillotine.

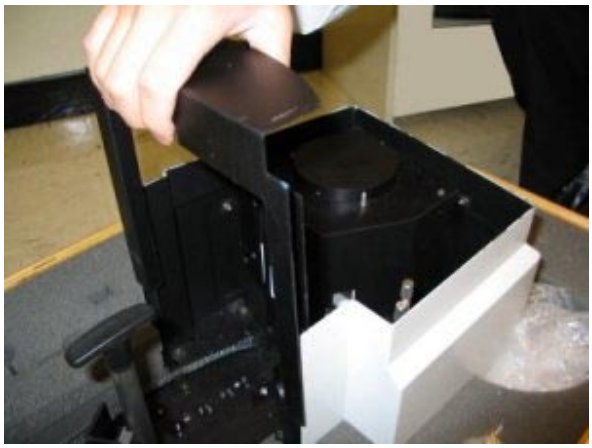


Figure 17. Removing the light seal.

4. Remove the magnetized covers from the reference and reflectance ports.

5. Install the centre mount port plug into the top of the sphere. Refer to figure 19.

6. Install the specular port plug. Refer to figure 19.

7. Use the "T" handle and the corner (marked "Lift Here") to lift the DRA out of the box. See figure 18.

Warning The accessory is heavy (32.5 kg). Safe lifting procedures should be used.

When the accessory is in its box, two people are required to do the lifting. There is a handle at each end of the box to assist in the lifting.

Figure 18. Lift the DRA from the wooden packing case as shown. One hand on the "T" handle, the other under the corner marked "Lift here".

If you have also ordered any of the specialized sample mount options you must also inspect these kits. The Standard Sample Holder kit is included as standard with each DRA.

Installation

The DRA fits into the spectrophotometer sample compartment using the lockdown mechanism.

Warning
Do not touch the mirrors or other transfer optics mounted on the accessory. Fingerprints

will induce optical scattering and may reduce reflectance or transmission of these components at certain wavelengths.

1. Turn off the spectrophotometer and remove any cell holders or other accessories from the sample compartment (the PC can be left on).

2. Turn the power to the spectrophotometer on and allow time for the instrument to warm up (about two hours).

3. Fully open the sample compartment lid and remove the front panel.

4. Make sure mirror M3 is set to the position in the DRA required for the intended application. The reflectance position "R" is the one closest to the front of the accessory; the transmission position "T" is the position furthest back. To move the mirror, loosen its mounting screw, lift the mirror off the base plate and move it to the position required. Set the mirror alignment pins into the correct locator holes and secure the mount with the mounting screw. See figure 19.

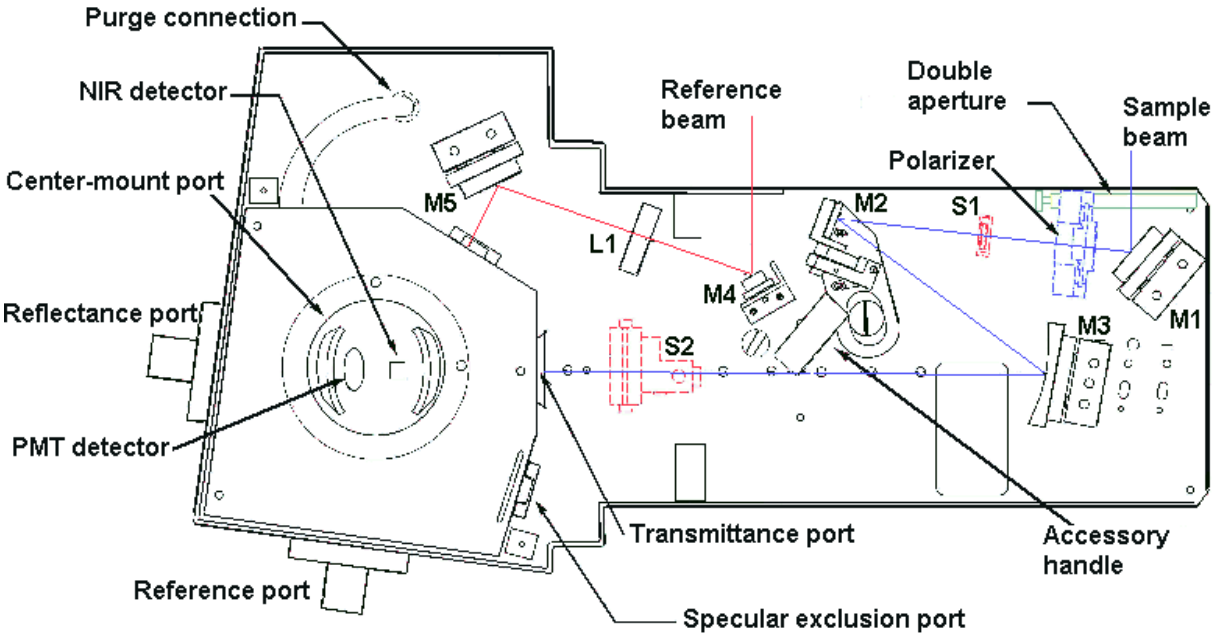


Figure 19: *The Varian 900, 1800, 2500 Diffuse Reflectance accessory with the optics cover removed. The large reference plate is clamped over the sample port at the centre of the sphere cap.*

5. If not installed already fit the centre mount port plug into the top of the sphere.

6. If not installed already install the specular port plug.

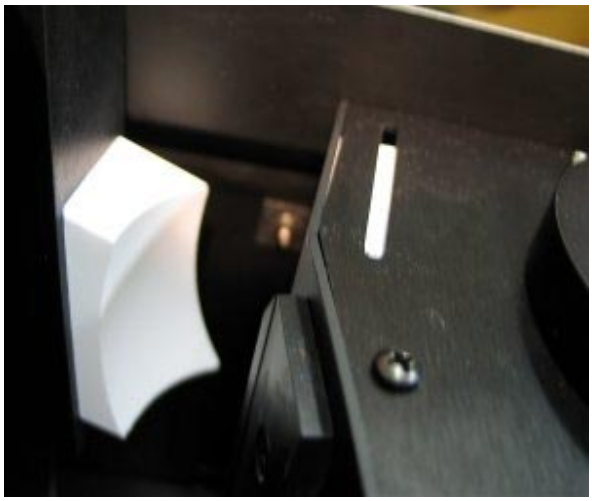
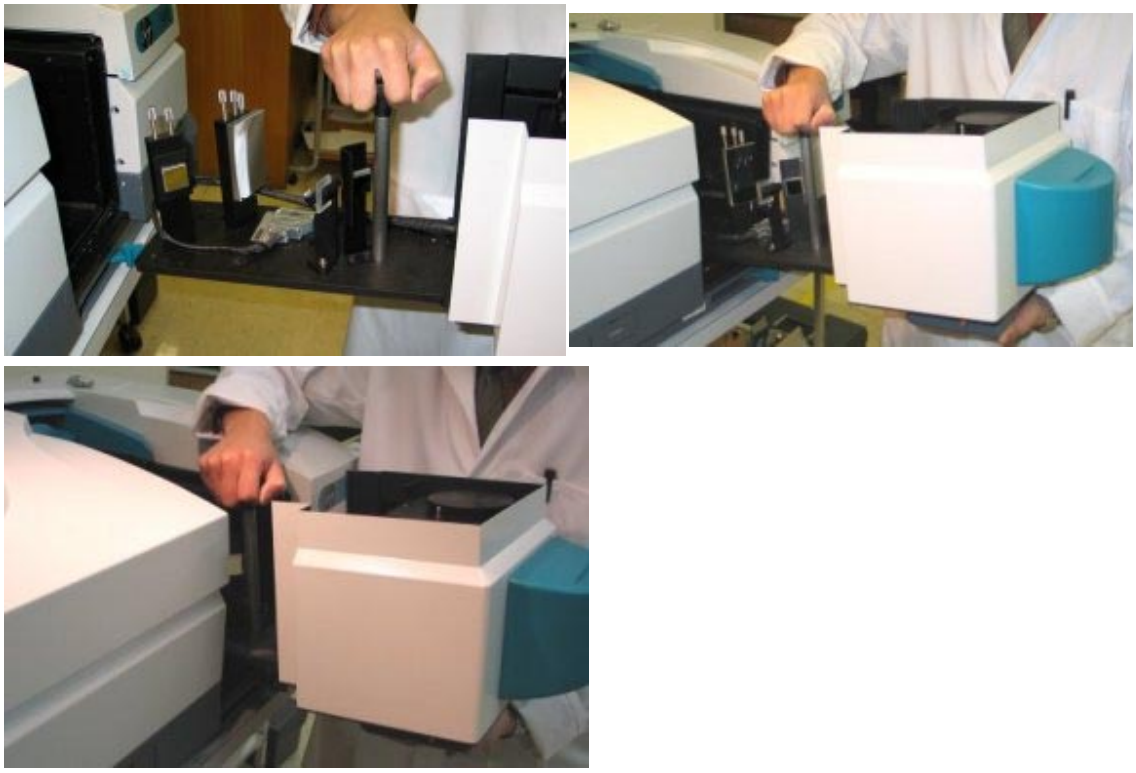


Figure 20. Install the specular port plug.

7. Remove the accessory from the wooden container by lifting the accessory by the "T" handle and the corner marked "Lift Here".

Figure 21. Lift the DRA from the wooden packing case as shown. One hand on the "T" handle, the other under the corner marked "Lift here".

8. Slide the DRA into the sample compartment at a 20° angle.



9. Take care to align the metal edges of the DRA with the guide tabs on the edge of the instrument sample compartment. This will correctly align and engage the rear lockdown pins into the the sample compartment floor. The following two photos illustrate how to line up the tabs, note inparticular the red circle indicating where the tabs should sit.

Figure 22. Align the tabs on the DRA with those on the instrument sample compartment.

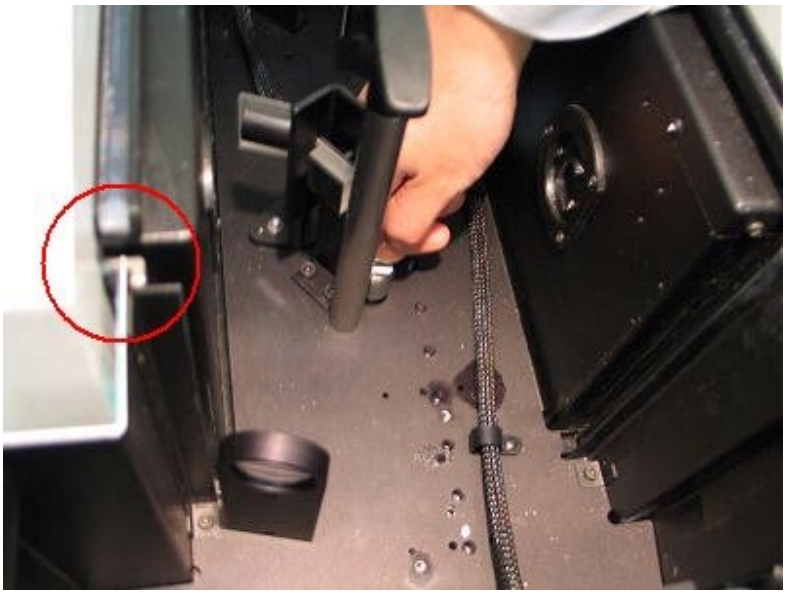


Figure 23. Place the front lock down pin into position.

10. Place the front lockdown pin into the hole in the DRA floor next to the "T" handle.

11. Gently manoeuvre the DRA until the pin falls into place.

12. Flick the lockdown lever on the front instrument panel to the left. Do **not** connect the accessory cable to the instrument.

Caution
The spectrophotometer must be turned off before plugging in the DRA, or serious damage may result.

13. Install the light seal.

14. Slide the adjustable sample ledge on to the dovetail ledges at the reflectance and reference ports.

15. Fit the spring loaded sample holders onto the ledges at the reflectance and reference ports. Load a reflectance standard at each port and install the magnetic covers at each port.

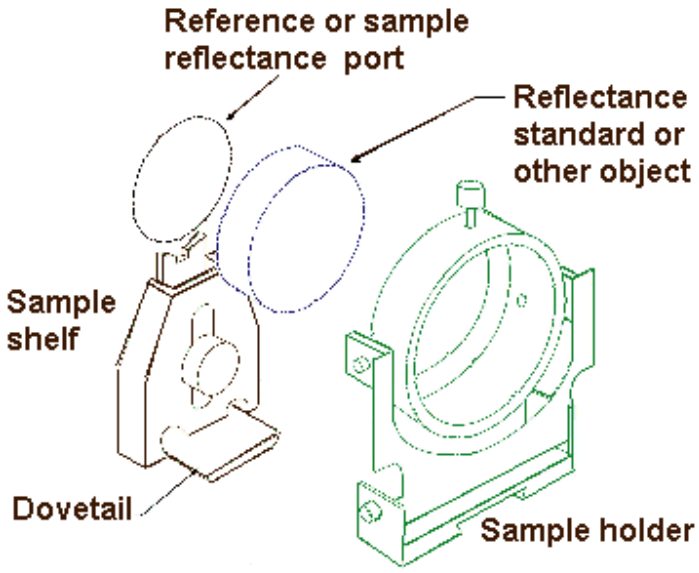


Figure 24. *The sample holder fits onto the dovetail at either the reference or reflectance port.*

16. Install the transmission sample holder at the transmission port, it should slide easily into the dovetail cut-out (just behind the sphere) with the metal clips pointing up.

17. Click the Windows Start button, then Programs, then Cary WinUV, and then Align.

18. The first time you install the DRA you must check the optical alignment of the accessory as described in the Alignment check section. If the accessory fails the alignment check, proceed to the Aligning the DRA procedure.

19. If the DRA is correctly aligned close the sample compartment lid and turn the spectrophotometer instrument **OFF**.

Caution
The spectrophotometer must be turned off before plugging in the DRA, or serious damage may result.

20. Connect the accessory detector cable to the matching connector at the rear of the sample compartment, refer to the figure below.. Make sure the detector cable does not obstruct the beam path inside the optical chamber of the accessory. Close the sample compartment lid.

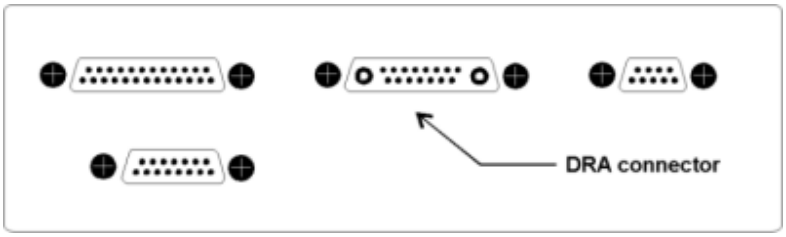


Figure 25: DRA socket

21. Click the Windows Start button, then Programs, then Cary WinUV, and then Validate. Turn on the spectrophotometer. If the instrument will not initialize, proceed to the Troubleshooting section.

22. Close the Validate application.

23. If desired, confirm the proper operation of the accessory by performing the diagnostic scans described in the Diagnostic Scans section of the online help. You may wish to perform these tests on the initial installation of the DRA.

24. Run the Calibration checks.

Warning This accessory contains electrical circuits, devices, and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury, or painful electrical shock.

To prevent electric shocks, operators and other unauthorized personnel must never remove the main cover. This must be opened only by Varian-trained, Varian-qualified, or Varian-approved service engineers.

Alignment

Warning

To prevent eye damage use care when looking into the various ports during the alignment process. Do not look into the light beams.

After installing the DRA it is necessary to check the alignment of the accessory. In some cases it will then be necessary to Align the DRA.

You will need:

- A flat blade screw-driver
- 3mm Allen key (provided)

Alignment check

This procedure can be used to check the optical alignment of the accessory without disturbing any mirror adjustments. A one inch square of translucent paper (e.g., tissue) is required.

1. Open the Align (if it is not already running) by clicking the Windows Start button, then Programs, then Cary WinUV, and then Align.

2. Darken the room and set the instrument for white light operation as follows:

Cary tab	
Beam Mode	Double
Y Mode	%R
Ave Time	0.1
SBW	2
Slit Height	Full

Select the *Zero Order* checkbox and click *Apply*.

2. Remove the cover to the accessory and remove the centre mount plug.

3. Slide the piece of translucent paper (e.g., tissue) into the reflectance port just behind the port opening. Looking through the centre mount port, examine the location of the sample beam relative to the port. Check that the beam is centred, and falls completely within the port surface area. The beam should not overfill the port.

Figure 26. Checking the beam at the reflectance port.

4. Repeat the previous step for the reference beam at the reference port.

Figure 27. Checking the beam at the reference port.

5. Mount or hold the translucent paper directly in front of the transmission port. Check that the sample beam is confined to the surface area inside the transmission port.

6. Repeat the previous step for the reference beam at the reference beam entrance port.

7. If the reference and sample beam profiles meet the criteria specified in the previous steps, the accessory optics are aligned properly and no further alignment is necessary. Return to Step 16 of the Installation procedure. If the accessory alignment does not meet the criteria, proceed to the following section.

Aligning the DRA

This procedure is used to align the transfer mirrors in the accessory optical chamber. A one inch square of translucent paper is required that clearly displays the outline of the sample or reference beam when illuminated. The following procedure should be performed using white light operation. Refer to the illustration in the figure below when performing the optics alignment.

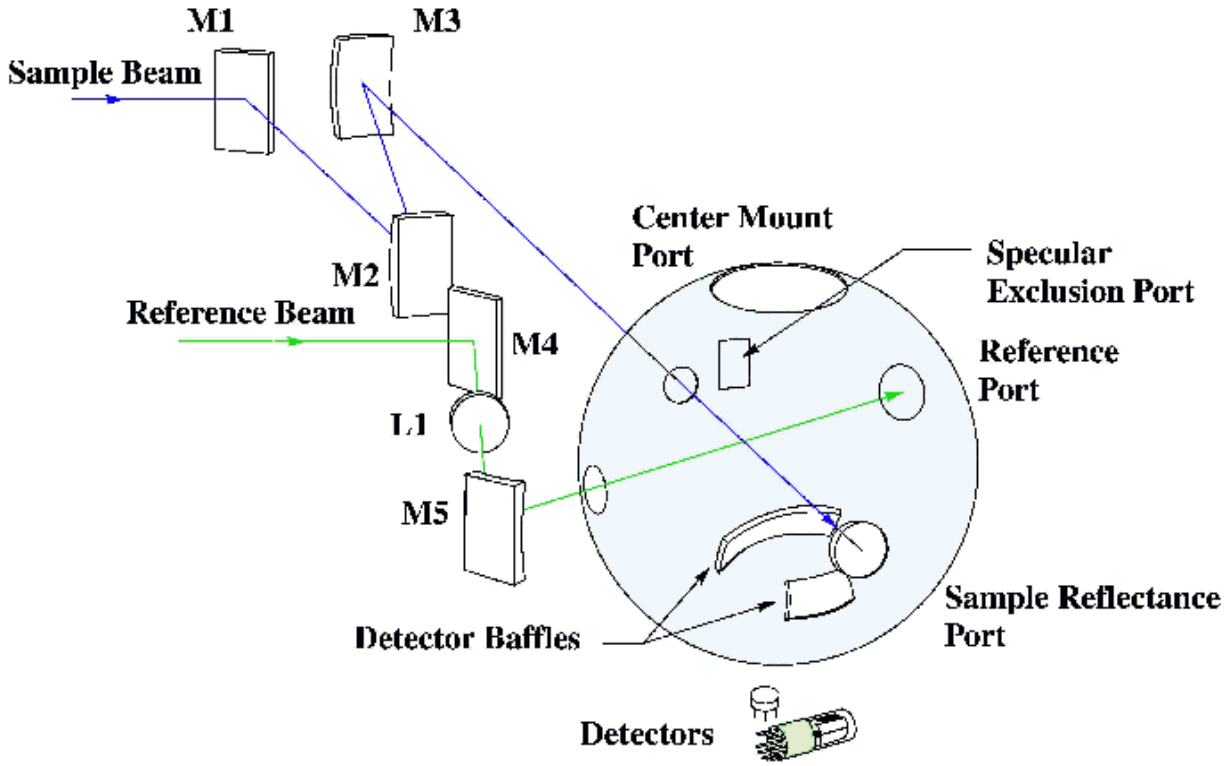


Figure 28: The optics of the DRA.

1. Click the Windows Start button, then Programs, Cary WinUV and then Align.

2. Click the *Setup* button.

3. Darken the room and set the instrument for white light operation by setting the following parameters:

Cary tab	
Beam Mode	Double
Y Mode	%R
Ave Time	0.1
SBW	2
Slit Height	Full

4. Select the *Zero Order* checkbox and click *Apply*.

Place a piece of translucent paper in the sample beam path in front of M1 to check that the beam is centreed. If the beam is not centreed:

- Check that that DRA is correctly locked down onto the instrument sample compartment floor.
- If the DRA is correctly installed, and misalignment persists, contact your local Varian service representative.

Place a piece of translucent paper in the sample beam path in front of M2. Turn the adjustment screws of mirror M1 until the beam is centred on M2.

7. Place the translucent paper in the sample beam path in front of M3. Adjust M2 until the beam is centreed on M3. Use the hex ball screw driver to loosen the locking nut on M2. Use the flat blade screw driver to adjust M2 until the beam is centreed on M3.

8. Remove the cover to the accessory.

8. Make adjustments to mirror M3 using the translucent paper and one of the following steps.

a. For reflectance measurements, slide the piece of translucent paper into the reflectance port just behind the port opening. Examine the location of the sample beam relative to the port. Check that the beam is centred, and falls completely within the port surface area. If necessary, turn the adjustment screws of mirror M3 until the beam is centred on the opening at the sample reflectance port. Hold the piece of translucent paper at the transmission port just in front of the port opening. The beam should not overfill the reflectance or clip the transmission port. The sample beam is now aligned.

b. For transmission measurements, hold the piece of translucent paper at the transmission port just in front of the port opening. Turn the adjustment screws of mirror M3 until the sample beam is centred on the transmission port. In this configuration, the sample beam may overfill the reflectance port, but it should be tightly focused at the transmission port. The sample beam is now aligned.

c. For centre mount use, load the translucent paper into one of the centre mount sample holders and load the entire assembly into the sphere. Block the reference beam with a plate and remove the reference port plug. View the centre mount holder through the reference port. Turn the adjustment screws of mirror M3 until the beam is centred on the sample area but does not clip the edges of the transmission port. When finished remove the blocking plate.

9. Place the paper in the beam path in front of M5. Turn the adjustment screws of mirror M4 until the reference beam is centred on M5.

10. Turn the adjustment screws of mirror M5 until the beam is centred on the reference port. If using a centre mount sample holder, the reference beam should pass directly in front of the centre mount device.

11. Alternately adjust mirrors M4 and M5 to centre the reference beam on both the reference beam entrance and the reference ports such that the beam is not clipped by the cente mount sample holder. The reference beam is now aligned.

12. Return to Step 7 of Installation.

Error checks and diagnostic scans

The following section describes the procedures for a series of diagnostic scans or error checking. The scans should be performed as part of the installation procedure and routinely as part of a preventive maintenance program. The successful performance of the diagnostic scans validates the operation of the accessory. The accessory optics should be aligned and the electronics calibrated before proceeding with the diagnostic scans.

Hot Tip
It may be useful to construct a method file for each of these scan procedures. The scan results should be retained for future use. If problems develop with the accessory in the future, the diagnostic scans can be repeated with copies sent to Varian for analysis. All diagnostic scans are set up and executed from the WinUV Scan application.

In the normal spectrophotometer configuration, the front beam serves as the sample beam and the rear beam is the reference beam. The DRA units all use the reverse beam configuration, where the rear beam is the sample beam and the front beam path serves as the reference beam. When a DRA is installed in the spectrophotometer, beam reversal is applied automatically during system initialization. Therefore, the *Beam mode—Double* checkbox should be selected.

Note
Parameters activated by a radio button that are not specifically mentioned in the following procedure should be set to 'Off' (e.g., the Signal-to-noise mode radio button). These parameters will not affect the procedure.

1. Click the Windows Start button, then Programs, then Cary WinUV, and then Scan.

2. Click the Setup button.

3. From the Setup menu, set the following parameters to carry out the error checking.

Cary tab	
X mode: Mode	Nanometers
X mode: Start/Stop	User-set range
Y mode: Mode	% R
Y mode: Y min	-5.00
Y mode: Y max	110.00
Scan controls: Ave time (s)	1.000
Scan controls: Data interval (nm)	0.500
Scan controls: Scan rate (nm/min)	30.000
Options tab	
SBW/Energy: Fixed SBW	ON
SBW/Energy: SBW (nm)	2.00
SBW/Energy: Beam mode	Double
SBW/Energy: Slit height	Reduced
Source: Lamps	UV-Vis
Source: Source changeover (nm)	350.0
Baseline tab	
Correction	None
Autostore tab	
File Storage: Storage	OFF

4. Click the 'Goto' button to open the 'Goto wavelength' dialog box box. Enter '500' in the wavelength field and click OK. The instrument will then drive to 500 nm.

Zero %R error check - alignment check

1. With the transmission ports uncovered, place the large reference plate over the reflectance port.

2. From the main 'Scan' dialog box box, click the *Zero* button and wait for the instrument to zero.

3. Remove the reference plate so the beam is going straight through the sphere into the sample cover. Close the sample compartment, the reading should not be greater than 0.5%R. If it is then check the alignment of the beam through the sphere (adjust M2 as necessary), check that the lens is clean and free from dust, and ensure that the beam is not clipping the reflectance port.

Zero %T error check - Electronic calibration check

1. Place the large reference plate over the reflectance port, close the sample compartment and then zero the instrument

2. Block the beam with a blanking plate (a large solid object) placed in front of the transmission port of the sphere.

3. The reading should be less than 0.01%R. If it is not, use the *Calibration* menu item in the *Validate* application. Refer to the Calibration section for further details.

Zero Abs error check - photometric noise check

1. Open the Validate application and select noise test under instrument performance tests.

2. Enter a tolerance of 0.00012 Abs for UV-Vis and 0.00016 Abs for NIR test.

3. Run the test. If the test fails, use the Calibration menu item in the Validate application. Refer to the Calibration section for further details. Also run the noise tests without the DRA installed. If necessary align the visible lamp in the instrument.

Specular component exclusion check

1. Position the specular light trap over the specular port.

2. Place the large reference plate over the reflectance port.

3. Click the *Goto* button to open the *Goto wavelength* dialog box box. Enter '500' in the Wavelength field and click OK. The instrument will then drive to 500 nm. Close the sample compartment lid.

4. Click the *Zero* button and wait for the instrument to zero.

5. Place a mirror over the reflectance port, and close the sample compartment. Note the reading. If the reading is greater than 1.5%R, then either the specularly reflected beam is not entering the light trap, dirt on the lens is scattering the beam and creating a 'halo' effect on the reflectance port, or the sample mirror does not have a good specular surface. If the reading is greater than 1.5%R, you should repeat the check for the specular component as detailed in the Checking for the specular component section.

6. Remove the specular light trap and replace the specular port plug. When the sample compartment is closed, the reading should be approximately 90%.

Attachment options

There are several different attachments options available for use with the external DRA. These greatly increase the versatility of the accessory. The attachment options are:

- Transmission port cuvette holder
- Centre-Mount Sample Holders
- Small spot kit
- Double aperture accessory
- Polarizer
- Powder cell
- Apertures

Transmission Port Cuvette Holder

The transmission port cuvette sample holder mounts a standard 1 cm cuvette directly in front of the transmission port. Transmission measurements made with this device at the transmission port will include the a portion of the scattered component of radiation in the transmission scan data. The sample holder is mounted into the clip style transmission port and is illustrated below.

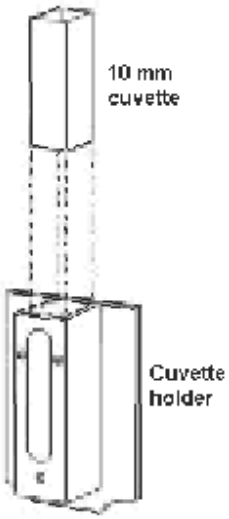


Figure 29: *The cuvette holder*

Installation and alignment

To install the cuvette holder:

Slide the cuvette holder over the dovetail so that it fits securely into the clips.

Check the sample beam alignment before proceeding with transmission measurements.

To record transmission data with the cuvette holder, refer to the 0°/d Transmission Measurements Using the Comparison Method.

Centre-Mount Sample Holders

A centre-mount sample holder offers two distinct advantages over the external sphere sample holders on the DRA. One is the ability to vary the angle of incidence for reflectance and transmission measurements. The other advantage is the ability to measure the transmission and reflectance of a sample together (transflectance).

There are several centre-mount designs, depending on the reflectance or transmission application intended.

- A variable angle sample holder mounts a solid sample at the centre of the sphere at specified angles of incidence from the sample beam. The variable angle sample holders may be clip or jaw style.
- A cuvette centre-mount sample holder positions a cuvette inside the sphere at a fixed angle normal to the sample beam.

Installation and alignment

To install a centre mount sample holder:

1. Remove the sphere cap from the top of the sphere.

2. Lower the centre mount through the opening in the sphere.

3. Fit the two posts on the centre mount onto the sphere by using the two locating holes in the top of the sphere.

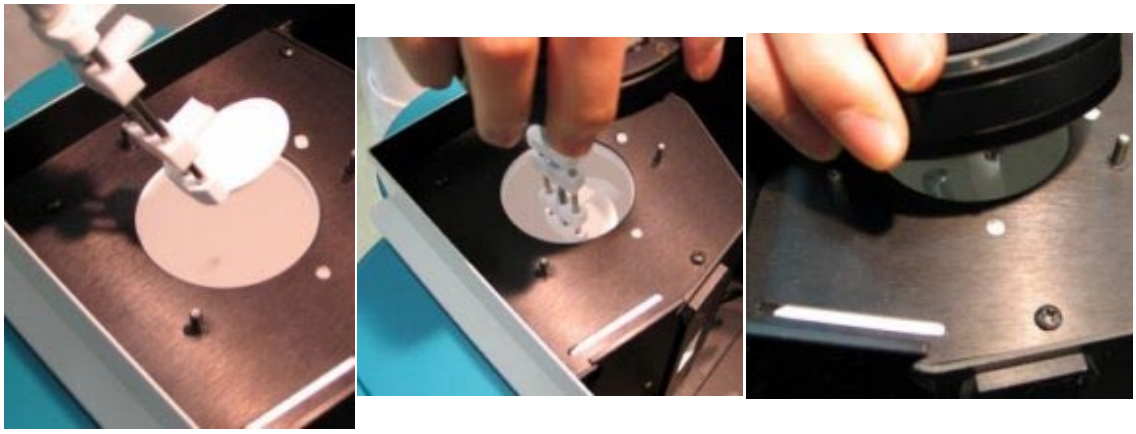


Figure 30. Lower the centre mount through the opening in the sphere, user the locating holes in the top of the sphere to secure the centre mount.

4. Align the centre mount sample holder.

Alignment

Occasionally, the requirement exists to conduct fine tune alignment of the beam inside the integrating sphere. Use white light operation and observe the beam path adjustments through the accessory cover opening.

To check white light alignment:

1. Alternatively block the beam at the reference and transmission entrance ports and view the alignment through the opposite port.

2. Click the Windows Start button, then Programs, then Cary WinUV, and then Align.

For centre mount use, load the translucent paper into one of the centre mount sample holders and load the entire assembly into the sphere. Block the reference beam with a plate and remove the reference port plug. View the centre mount holder through the reference port. Turn the adjustment screws of mirror M3 until the beam is centred on the sample area but does not clip the edges of the transmission port. When finished remove the blocking plate.

Variable Angle sample holders

A variable angle centre-mount sample holder enables the sample reflectance and absorbance to be studied at various incidence angles of the sample beam. The variable angle centre-mount sample holder uses a rotating dial to allow the user to quickly set the desired incidence angle to the nearest 1°. Depending on the sample size and the instrument beam characteristics, the user can perform measurements at beam incidence angles ranging from 0 - 60°.

Clip and Jaw style centre mount sample holders

The clip and jaw type centre-mount sample holders fix the sample at the centre of the integrating sphere at selectable incidence angles.

The clip style sample holder, shown in the figure below, uses a spring-loaded clip to hold the sample. This is useful for measuring transflectance. The sample must be in a sheet configuration and large enough to accommodate the entire sample beam.

The jaw style sample holder (shown in figure 31) is better for holding large, bulky samples. The sample must be opaque or an absorbent backing must be applied. The sample holder is designed for reflectance measurements of samples that do not transmit light.This centre mount sample holder is not designed for transflectance measurements. The jaws accommodate sample sizes up to 1.5" in length and width. A clean disc of SRM-99O optical grade Spectralon material approximately 30 mm in diameter can be used as a reflectance standard.

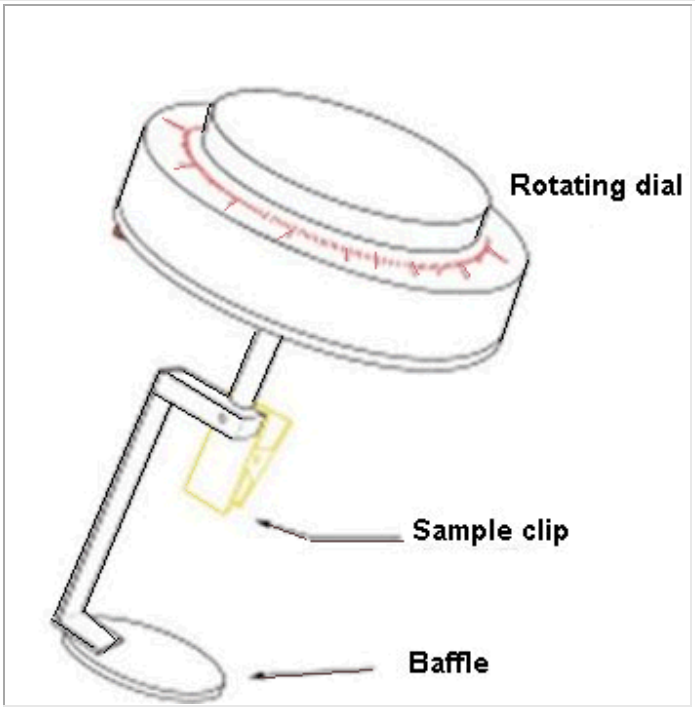


Figure 31: *The clip style variable angle sample holder.*

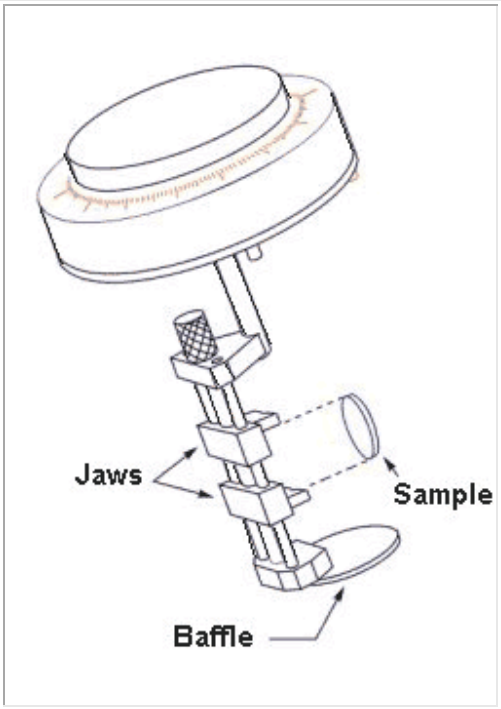


Figure 32: *The jaw style centre mount.*

The width limitation of the jaw style sample holder depends on the thickness of the sample and the maximum angle of incidence the application requires. The jaws protrude 12 mm from the frame of the sample holder. If possible, the reflectance standard and sample should be loaded so the reflecting surface is even with the front edge of the jaws. This will place the front surface at the rotational axis of the sample holder.

Centre-Mount Cuvette Sample Holder

The transmission port cuvette holder is used for measuring the transmission of a liquid sample. The centre-mount cuvette sample holder provides a liquid sample measurement capability to the DRA accessory that includes the scattered component of transmitted radiation. A centre-mounted cuvette allows the simultaneous measurement of a turbid sample diffuse transmission and reflectance, so that absorbance can be derived in one easy measurement. The device is ideal for measuring turbid samples such as sea water, proteins, and other biological solutions. Measurements performed with this device are in the 0°/d geometry. Since the cuvette is fixed at 0° angle of incidence, the specular component of reflection is directed out of the sphere through the transmission port.

Alignment

Upon first time use, the sample holder should be loaded into the centre-mount port and checked for proper angle alignment to the sample beam, the beam should strike at normal incidence when the dial is set at 0°. If it does not, rotate the knob until the angle is normal to the sample beam, loosen the four screws along the perimeter of the dial and rotate the dial so it reads 0°.

The reflecting surface of the standard should be the same angle of incidence as the intended sample. When properly aligned, the 0° dial reading on both clip style and jaw style sample holders holds the sample for normal incidence where the reflecting surface is perpendicular to the path of the sample beam.

Ideally, the beam geometry for a reflectance accessory should be optimized for the application concerned. For centre-mount applications, the ideal configuration has the sample beam focused on the front surface of the sample at the centre of the sphere. In general, the maximum dimensions of a centre-mounted sample is proportional to the size of the integrating sphere. It should be obvious that the smaller the sample, the more accurate the reflectance measurements - as long as the sample beam does not overfill the surface area of the sample. For measurements with an Edwards sphere, ASTM E 903 recommends limiting specimen size to one percent of the surface area of the sphere. Thus, for a 150 mm diameter integrating sphere, the maximum specimen size is a total surface area of approximately 700 mm^2. Sample sizes exceeding the recommended specimen dimensions may interfere with the radiance uniformity at the wall surface or clip the reference beam inside the integrating sphere.

Centre-mount sample holders inflict further restrictions on reflectance samples. The DRA accessories utilize a three position mirror to focus the sample beam at the transmission port, sample reflectance port, or centre-mount positions, depending on the sphere configuration. Mirror M3, however, is a concave mirror such that the spot produced by the sample beam depends on the position of the mirror and the angle of incidence to the sample. The spot size grows larger as the angle of incidence increases, to a point where the spot may no longer fit on the sample surface. At near-normal angles, the specular component of the reflected beam is rejected completely out the transmission port and is not collected during the scan. Finally, the horizontal dimension of the sample or reflectance standard may clip the reference beam at steep angles of incidence, thereby placing premature limitations on reflectance measurements.

Loading a sample into the jaw style centre sample holder

Tighten the jaws using the thumbscrew. Do not overtighten the jaw mechanism as this could damage the sample.

When loading a sample into the clip sample holder, the sample should be held perfectly vertical by the clip with the plane of the reflecting surface along the centre of rotation of the dial. The sample beam path will strike the sample at a point midway between the

bottom tip of the clip and the baffle. There is no need to use a diffuse reference standard with the clip style sample holder, the sphere wall serves as the reference.

After loading the sample holder into the accessory, view the sample from the reference port, making sure the reference beam is not clipped by any part of the assembly at the incidence angles required.

Centre-Mount Cuvette Sample Holder

The cuvette holder (figure 33) is designed to hold a standard 10 mm path length cuvette at the centre of the integrating sphere. A pocket in the bottom platform to the sample holder orients the cuvette at normal incidence to the sample beam. The upper platform is spring loaded to hold the sample in place. The baffle located under the sample prevents the detectors from directly viewing first strike reflections.

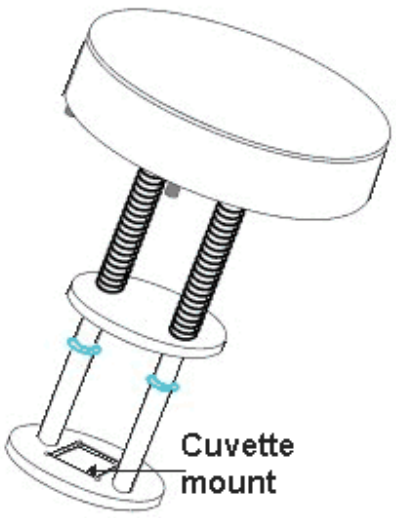


Figure 33: *The cuvette variable sample holder.*

Using the centre mount cuvette holder

1. Position M3 at the centre-mount location "C"

2. Check the alignment of the accessory.

3. Carefully place the filled cuvette into the holder and carefully lower the sample holder into position.

Caution
Exercise great care when loading the centre-mount cuvette holder into the integrating sphere, the sample holder is bulky and spilled sample solutions may damage the interior surfaces of the sphere.

Small Spot Kit

A sample loaded at the DRA reflectance port or other sample site should be the same size as the focused sample beam. If the sample is smaller than the beam spot, the accuracy of the results will be reduced. If the sample surface fills the entire port, the spectra obtained may be accurate but the signal-to-noise ratio is reduced by the absorption characteristics of the sample. The small spot kit allows the user to focus a small beam spot at the transmission port, reflectance port or sphere centre of the diffuse reflectance accessory. The kit adds versatility to the DRA in three ways. The kit can be used to reduce the sample beam size to measure small samples. It can be used to focus the sample beam to measure a small portion of a larger sample. Or, it can be used with a centre-mount sample holder to prevent the sample beam from overfilling the sample at high measurement angles. The small spot kit consists of the following standard and optional components:

Description	**Part number**
Iris Assembly	XXXX
Collimating Mirror Assembly	XXXX
Lens Holder w/ (2) Mounting Screws	XXXX
Transmission Lens	XXXX
Centre-mount Lens	XXXX
Reflectance Lens	XXXX
Wire Attenuator Screen	XXXX
Tool Kit, 3/16² hex key	XXXX
Reflectance Port Aperture Kit	XXXX

XXXX These part numbers were not available at the time of publishing, please refer to the Varian website, for the most up to date information. See www.varianinc.com

The lens holder and rail are illustrated in the figure below. Each lens is marked by a letter identifying the corresponding to the sample location and an arrow indicating the correct orientation for installation.

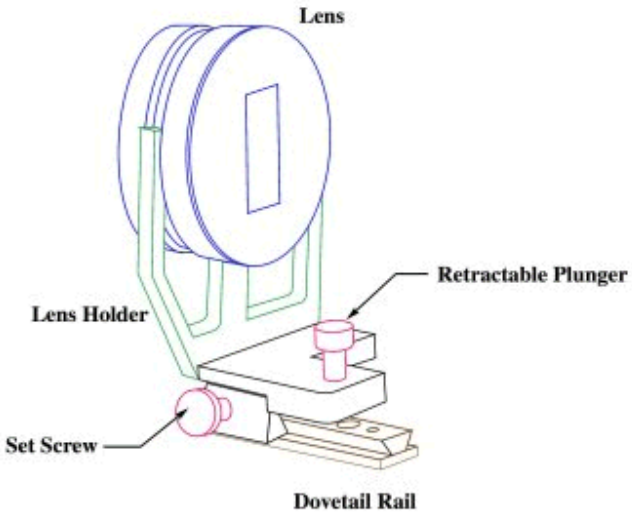


Figure 34: *The small spot kit lens holder, rail and lens.*

Note
The small spot kit replacement for mirror M3 must be used instead of the standard accessory mirror. The two mirrors look similar, but the small spot kit mirror is visibly less concave than the standard accessory M3 and is marked "M-3 SSK".

Installation and Alignment

The small spot kit requires an initial installation. Once installed, the user can refer to the Operation section for details regarding reflectance measurements. To install the small spot kit, follow these instructions:

1. Remove the accessory from the instrument sample compartment and install the replacement mirror to M3 (M-3 SSK) at the "T" position. The replacement mirror assembly must always stay at the "T" position when using the small spot kit

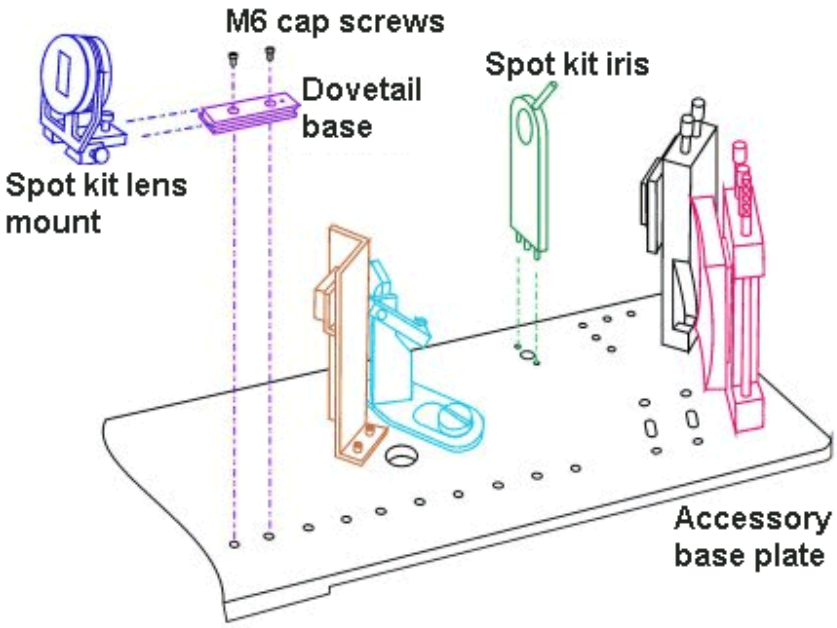


Figure 35: *Installing the small spot kit.*

2. Install the iris assembly in the position shown above. The mounting plate for the iris is magnetized to hold the iris firmly in place. Two adjustment controls are provided on the iris, one for adjusting the iris position laterally and one for controlling the aperture size. Open the iris fully.

3. Remove the lens holder from the dovetail base and mount the rail onto the accessory base plate as shown. Load reflectance standards at the reference and reflectance ports.

Figure 36. The small spot kit in the DRA.

4. Turn on the instrument on and allow it to warm up (approximately 2 hours).

5. Set the instrument to Zero order using the Align application. See Installation step 14.

6. Once the instrument is warmed up, remove the reflectance standards from the ports (if they are currently loaded). Load the reflectance lens into the small spot kit lens mount. The reflectance lens is marked "R", the transmission lens is marked "T", and the centre-mount lens is marked "C". The lens should be oriented so the arrow points toward the integrating sphere. Gently rotate the lens until it clips into place. The grooves in the outside of the lens sit on the clips on the mount.

7. Remove the transmission holder. (The transmission holder may clip the beam, reducing the energy entering the sphere.)

7. Install the DRA into the instrument. Turn the instrument ON and allow it to initialize.

8. Install a beam blocker over the reference beam entrance port.

9. Find the sample beam at mirror M2 and centre if necessary.

10. Fully close the iris. Look at the top left hand corner of M3 and observe the beam on the back of the iris. When the iris is closed to its smallest opening size, the beam should be taller than the hole but centreed in the iris opening. To realign the iris assembly in the beam path, first loosen the thumb screw that secures the two sections of the iris assembly together. Move the top portion of the iris assembly, sliding it laterally, until the beam is centreed on the iris opening. Retighten the thumb screw.

11. Fully open the iris. Dim the light in the room.

12. Adjust mirror M2 to centre the beam on the M3 SSK mirror. See Alignment for instruction on aligning the mirrors.

13. Hold the translucent paper just outside the open reflectance port. Adjust the M3 SSK to centre the sample beam within the reflectance port opening. It may be necessary to adjust the aperture size of the iris or relocate the lens mount along the dovetail rail to achieve the desired spot size and make a note of the position of the iris.

Figure 37. The beam from the small spot kit with the iris partially open.

14. Remove the beam blocker from the reference beam path.

15. Replace the reflectance plug. The small spot kit is now aligned.

16. Turn off the instrument.

17. Plug in the DRA using the connector at the back of the sample compartment.

18. Fully open the iris and replace the accessory cover.

19. Turn on the instrument and allow it to initialize.

20. Return the iris to the position noted in step 13.

Powder cell

In some cases, a reflectance measurement may deliver better diffuse reflectance spectra when the sample is ground to powder form. As shown in Figure 38 and 39, the powder cell is designed to fit inside the reflectance sample holder in the same manner as the reflectance standard. Each sample holder comes as a set: one cell with a Spectralon insert for use as a reflectance standard, and an empty cell for loading the powdered sample. The quartz window for each cell is compatible with each DRA accessory across the entire 250-2500 nm wavelength range.

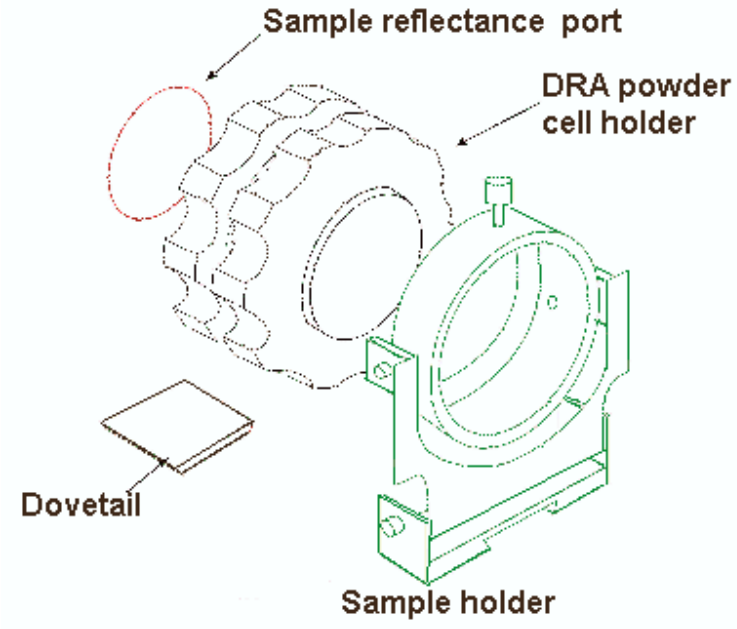


Figure 38. The powder cell fits into the sample holder at the reflectance port.

Figure 39. The powder cell in position at the reflectance port.

The design of the PCH-150 sample holder does not facilitate an accurate means of loading a powdered sample to an accurate packing density. An approximate packing density and reflectance measurement can be obtained using the following procedures. Prior to loading, the sample should be ground to the desired particle size.

Sample Preparation Using the PCH-150 Powder Cell Holder

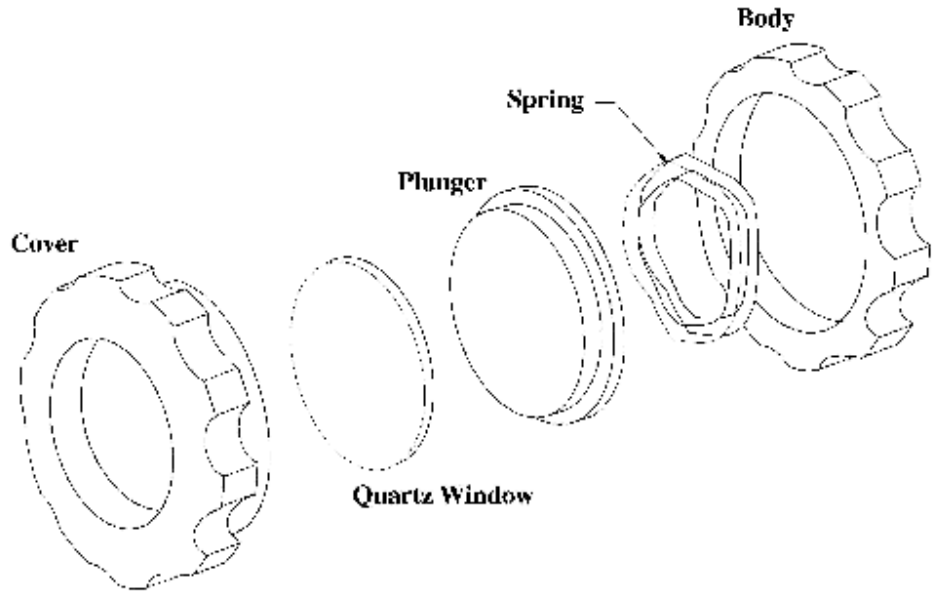


Figure 40. An exploded view of the powder cell.

1. Disassemble the powder cell holder. DO NOT disassemble the reference cell holder. Place the front cover onto the flat surface of a balance scale, face down, and load the quartz window into the cover.

2. Record the weight of the two sample holder components.

3. Load a portion of the sample into the powder cell cavity over the quartz window. Gently tap the side of the holder against the work table, or use a spatula to smooth the surface of the sample. Repeat this loading process until the desired sample depth or sample weight is achieved.

4. Record the weight on the balance scale and calculate the total weight of the powdered sample.

5. Estimate the average height of the sample by measuring the distance from the sample to the top surface of the container.

6. Load the plunger and spring over the sample and screw the body over the entire assembly. Screw the pieces together snugly, but do not apply excessive force.

7. Examine the surface around the sample holder for any loose sample material that may have spilled out of the sample holder cavity. Weigh the spilled powder and make corrections to your calculation in Step 4.

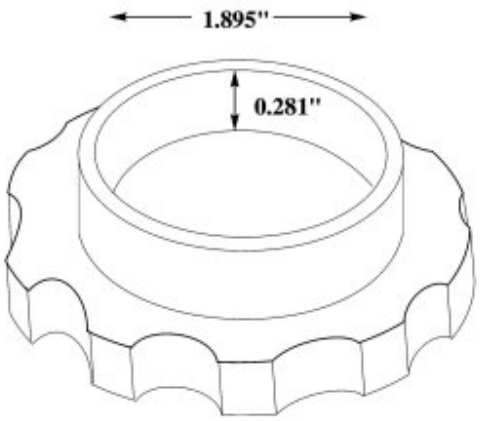


Figure 41. Calculating the packing density.

8. Calculate the packing density using the sample weight recorded in the previous steps.

Reflectance Measurements Using the Powder Cell Holder

To perform reflectance measurements using the Powder cell holder, proceed as follows.

1. Load the powder cell containing the Spectralon insert against the reflectance sample port of the sphere. There should be a Spectralon reflectance standard mounted at the reference port.

2. Record one of the baseline scans as directed previously. DRA configuration during the baseline scan should conform exactly to the configuration anticipated during the sample scan.

3. Replace the powder cell sample holder at the sample reflectance port with the powder cell containing the sample. Do not change the configuration of the integrating sphere in any other way.

4. Open the Setup Dialog and check the instrument parameter Setup. Normally, the Setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

5. Record the sample scan by selecting the **Start** button in Cary WinUV software.

6. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected without the standard option, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard.

To obtain the best results follow these guidelines when using the Powder cell holder.

- The sample reflectance depends upon sample handling factors, such as the packing density, surface uniformity, and characteristics of the sample. Finely ground powder samples will give different reflectance results than the same sample when measured in its coarse, granular, or pressed powder form. The spectrum shape of the sample reflectance does not change with packing density, that is, the peak location does not change. Only the relative reflectance value changes with changes in the sample packing density.
- If you wish to compare subsequent sample measurements, take care to ensure that sample presentation to the beam and packing density are identical between scans.

Double aperture accessory

The double aperture accessory is used to determine the absolute photometric accuracy of the DRA in the UV-VIS and NIR spectral range. The accessory mounts directly in the sample beam path on the base plate.

Components accompanying the double aperture apparatus include the following:

- Base mount
- Aperture plate
- Masking Wheel
- Assorted fasteners

A detailed explanation of the double aperture method is provided in the Validate section of the Help.

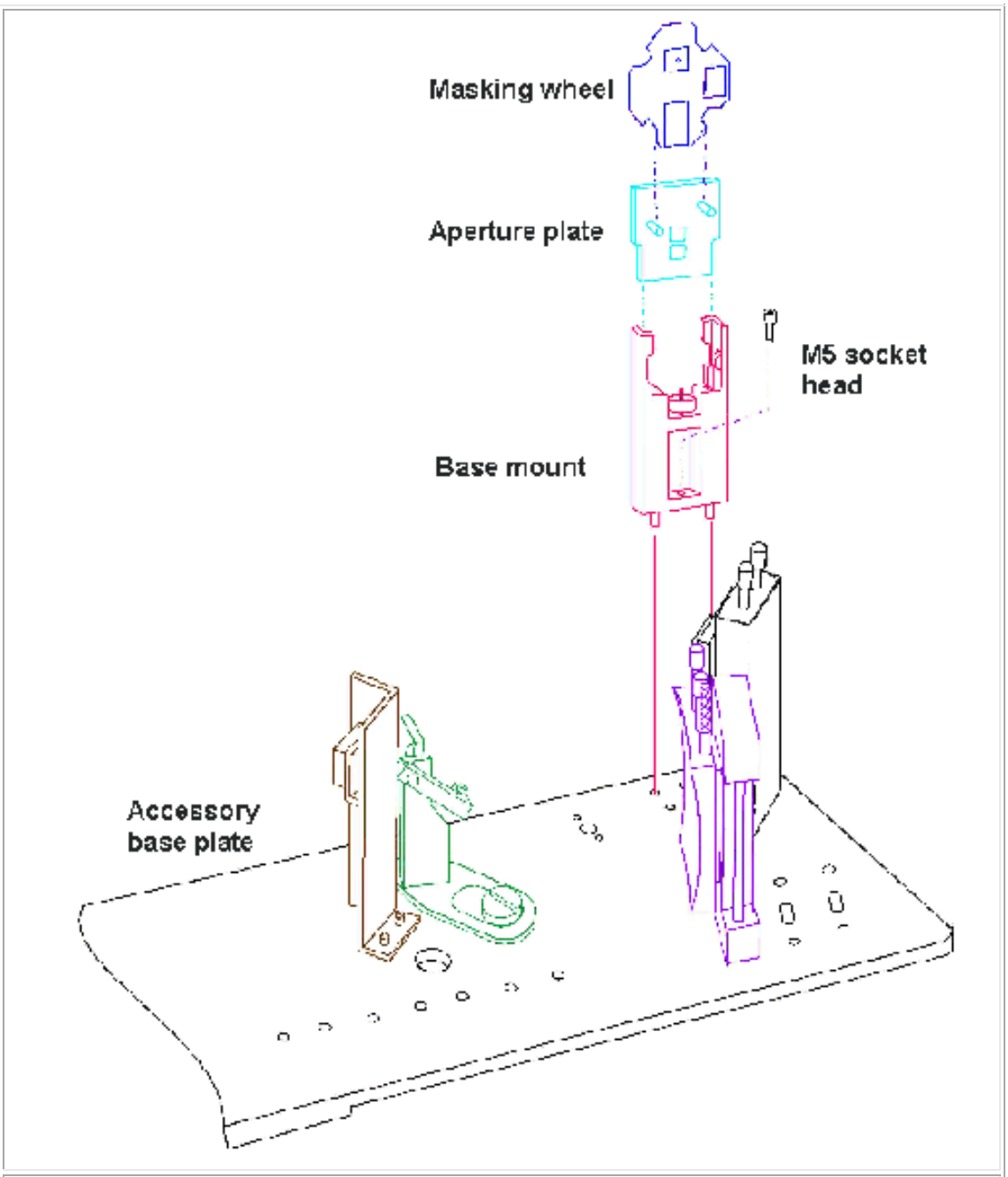


Figure 42: *The optics of the double aperture accessory.*

Figure 43. The double aperture attachment on the polarizer mount.

The accessory optics should be aligned according to the Installation instructions before installing the double aperture. The base mount for the double aperture apparatus is the same base used for the polarizer. Install the double aperture accessory as follows:

1. Remove the DRA from the instrument sample compartment. Locate mirror M3 at the "R" position.

2. Insert the base mount into the locator holes in the DRA base plate as shown in the figure above. The orientation of the mount is not important. Fasten the device to the accessory using the M5 socket head screw provided and a 4 mm hex wrench.

3. Re-install the DRA into the sample compartment.

4. Click the Windows Start button, then Programs, then Cary WinUV, and then Align.

5. Turn on the instrument and allow the instrument time (about 2 hours) to warm up.

6. Remove the reflectance standards at each port.

7. Open the sample compartment lid and slide the aperture plate into the base mount slot as shown in the figure. The protruding alignment pins should point into the accessory.

8. Block the reference and sample beam entrance ports to the integrating sphere and configure the sample beam for white light operation.

9. Observe the beam profile as it strikes the aperture plate, adjust the height of the plate so the rectangular apertures are centreed on the sample beam.

10. Remove the beam blocker from the sample beam path. Observe the reflectance port from the open centre-mount port, mount a piece of translucent paper in the sample holder against the reflectance port. Check that the images of the double apertures lie within the perimeter of the sample reflectance port. Make slight adjustments to mirror M3 as necessary. The double aperture device is aligned.

There are four aperture positions on the masking wheel each spaced at 90° intervals along the perimeter. A dimple is drilled at the centre of the wheel to match the tiny centre pin on the aperture plate. Both the aperture plate and masking wheel should be removed from the accessory during reflectance and transmission measurements.

Note
If reflectance or transmission applications require use of the optional polarizer, the base mount must be relocated to the polarizer position.

Polarizer/Depolarizer

A polarizer optic can be provided as an optional assembly to either of the DRA accessories for conditioning the polarization of the sample beam. The polarizer is a Glan-Taylor constructed device. Specifications of the optic is provided on the Help and Videos CDROM in the Cary WinUV software package. The light reflecting off the Spectralon walls is completely diffuse, so a depolarizing optic is never required for reflectance measurements. It may be useful for transmission measurements on polarizing samples. The following components accompany the polarizer attachment:

Base mount with attachment screws Lens holder Polarizer lens

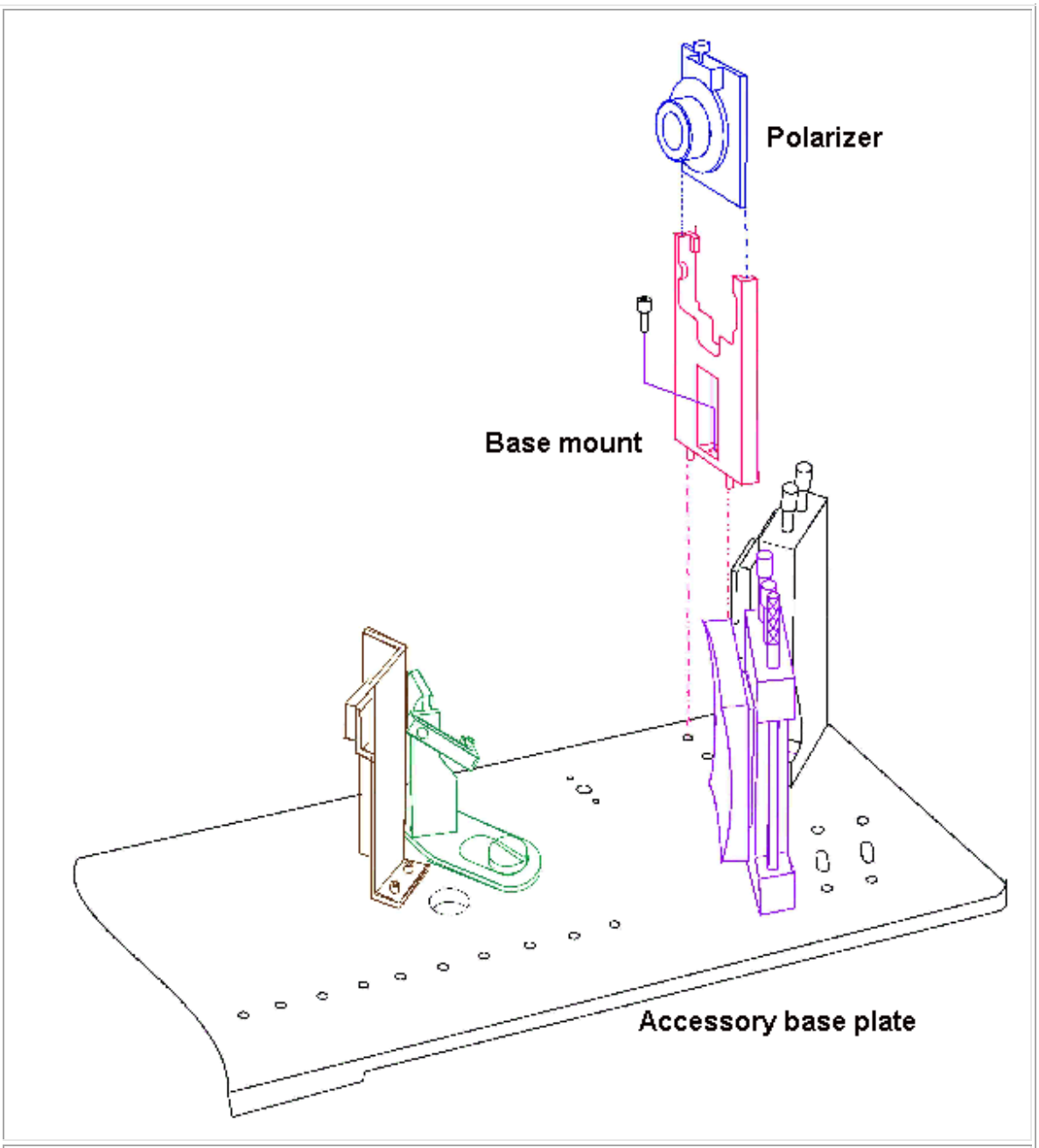


Figure 44. The polarizer attachment

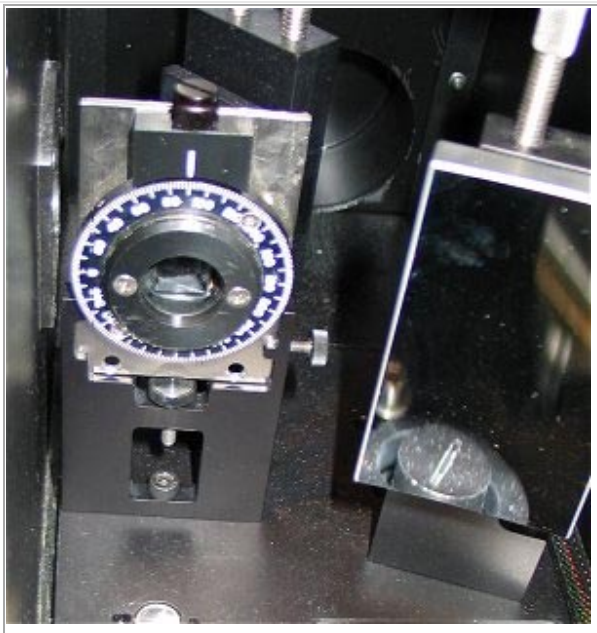


Figure 45. The polarizer attachment in the DRA.

The polarizer assembly uses the same base mount as the double aperture apparatus. Install the polarizer assembly into the accessory as follows:

1. Insert the base mount for the polarizer into the locator holes in the accessory base plate as shown in the figure below. Fasten the device to the accessory base plate using the M5 socket head screw provided and a 4 mm hex wrench.

2. Click the Windows Start button, then Programs, then Cary WinUV, and then Align. Turn power to the spectrophotometer on. Install a beam blocker at the reference beam and sample beam entrance ports to the sphere and configure the sample beam for white light operation.

3. Remove reflectance standards from the reference and reflectance ports. Remove the centre-mount port plug.

4. Slide the polarizer optic into the base mount slot. The optic should be oriented in the direction shown in the figure above.

5. Observing the sample beam profile as it strikes the entrance aperture to the polarizer, adjust the height of the device so the sample beam penetrates the optic unclipped.

6. Remove the blocking device from the sample beam. Check the sample beam alignment at the reflectance port using a piece of translucent paper. Make any adjustments to mirror M3 as necessary.

7. Suspend white light operations and remove the blocking device from the reference beam. The accessory is aligned for polarizer operations.

The base mount can remain installed in the accessory when the polarizer is not being used. If adjustment to the sample beam alignment is required, an alignment check should be performed whenever the polarizer is removed.

Note
The polarizer is an attenuation device and may reduce significantly the signal-to-noise ratio of the accessory during use.

Operation

Samples can be mounted at the transmission or reflectance ports. At the reference port, attenuators may be mounted.

Routine DRA installation

The accessory must be installed in the sample compartment and the DRA cable connected before turning the instrument power switch on. During initialization, the spectrophotometer will adjust instrument radiation levels appropriate to the high attenuation characteristics of the DRA, making the necessary gain adjustments to the detector circuitry. Follow these instruction to install and configure for routine DRA operation:

1. Turn the instrument off. Remove any cell holders or other accessories from the instrument sample compartment.

Caution
The spectrophotometer must be turned OFF before plugging in the DRA, or serious damage may result.

2. Slide back the sample compartment lid and remove the compartment front panel.

3. Remove the accessory from the wooden container by lifting the accessory by the "T" handle and the corner marked "Lift Here".

4. Slide the DRA into the sample compartment at a 20° angle. Take care to align the metal edges of the DRA with the guide tabs on the edge of the instrument sample compartment. This will correctly align and engage the rear lockdown pins into the the sample compartment floor.

5. Place the front lockdown pin into the hole in the DRA floor next to the "T" handle.

6. Gently maneuver the DRA until the pin falls into place.

7. Flick the lockdown lever on the front instrument panel to the left.

8. Install the guillotine/light seal.

9. Load a reflectance standard at the reference and sample reflectance ports. Remove any centre-mount sample holders installed in the sphere and install the centre port plug.

10. Connect the accessory detector cable to the connector at the rear of the sample compartment. Make sure the detector cable does not obstruct the beam path inside the optical chamber of the accessory. Install the accessory cover.

11. Click the Windows Start button, then Programs, then Cary WinUV, and then Scan.

12. Close the sample compartment lid and turn power to the instrument ON.

13. Allow the instrument and accessory to warm up for at least 30 minutes before proceeding with measurements.

Removing the DRA from the instrument

Use the following procedure to disconnect and remove the DRA from the instrument:

1. Turn the spectrophotometer off.

2. Open the sample compartment lid and disconnect the accessory cable.

3. Remove the accessory cover.

Figure 46. Removing the accessory cover.

4. Remove the guillotine/light seal.

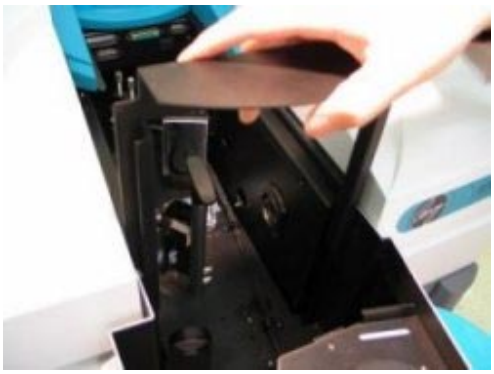


Figure 47. *Removing the light seal.*

Note
The lockdown lever is accessible just underneath the DRA base plate.

5. Slide the lever on the instrument front panel to the right.

6. Remove the front lockdown pin.

7. Tilt the front end of the accessory. Using the accessory handle, lift the DRA up and out of the sample compartment and store it in the accompanying wooden box.

8. Replace the sample compartment front panel.

Collecting baseline scans

When taking reflectance measurements using a reflectance accessory, three types of baseline corrections can be applied to the scan data. A simple correction is used in the diagnostic scans that is quick and does not require zeroline or reflectance standard data. A second and third method uses a zero baseline correction that can be applied by one of two methods: either the sample beam can be blocked off from the integrating sphere during the 0%R baseline scan or a light trap can be installed over the sample reflectance port.

Note
The blocked beam method is discouraged by ASTM E 903 for **reflectance** measurements because the effect of scattered light incident on the sphere wall is not included in the data collection.

During transmission measurements, either the standard baseline or the blocked beam method should be used. The spectrophotometer and accessory must be warmed up for 30 minutes prior to recording any of these baseline scans.

An integrating sphere is sensitive to small-angle scatter from the sample beam coupling optics. Sometimes, the scattered radiation strikes the wall of the integrating sphere near the reflectance sample port, creating a "halo" surrounding the port. This halo-effect causes a small error in the measurement of the reflectance factor that is most significant when measuring samples of very low reflectance. This error is easily corrected by using the light trap zero baseline correction method discussed above.

Simple Baseline Scan for Reflectance or Transmission Measurements

Baseline scan data is recorded from the Cary WinUV Scan application. Prior to recording the scan, make sure that mirror M3 is located in the correct position. To collect a baseline scan:

1. Click the *Setup* button and set up instrument parameters as required by the intended application. Select the %R or %T ordinate scale, depending on the type measurements anticipated. The *Beam mode* should be double.

2. Select the Baseline radio button on the Baseline tab.

3. Load a Spectralon standard at the reference port and a calibrated reflectance standard, if available, at the sample reflectance port. If a calibrated standard is unavailable for reflectance measurements, an uncalibrated Spectralon standard can be substituted. Install the magnetic port covers over their respective ports.

4. Click the Baseline button.

The recorded baseline correction will be effective until replaced by a follow-up baseline scan.

Zero Baseline Scan for Reflectance or Transmission Measurements

Prior to recording the scan, make sure that mirror M3 is located in the correct position. To record a zero baseline scan:

1. Click the *Setup* button and set up instrument parameters as required by the application. Select the %R or %T ordinate scale, depending on the type measurements anticipated. The *Beam mode* should be double.

2. For reflectance measurements, select the *Zero/baseline correction* or *Zero x std ref correction* radio button on the Baseline tab. For transmission measurements, *Zero/ baseline correction* should be selected.

3. Load a Spectralon standard at the reference port and a calibrated reflectance standard, if available, at the sample reflectance port. If a calibrated standard is unavailable for reflectance measurements, an uncalibrated Spectralon reflectance standard can be substituted. Install the magnetic port covers over their respective ports.

4. Click the Baseline button.

5. At the 100%T scan prompt, select OK. The standard at the reflectance port serves as the 100%R reference.

6. At the 0%T scan prompt when running reflectance scans, either block the sample beam at the transmission port or replace the reflectance standard at the sample port with a light trap. When running transmission scans, the sample beam should be blocked for the 0%T scan.

The recorded baseline correction will be effective until replaced by a follow-up baseline scan.

Note
If a light trap is unavailable, the baseline scan can be run with the sample reflectance port open and the outside magnetic cover to the reflectance port installed.

8°/h Reflectance Factor Measurements (Comparison Method)

For reflectance measurements, make sure that mirror M3 is placed in the reflectance position, closest to the sphere. Repeat the alignment procedure if the mirror has been moved. This mirror configuration provides a focused spot size of approximately 6.5 mm wide x 15 mm high. If a large reflectance spot is desired, such as for measurement of fabrics or other non-homogenous samples, place mirror M3 in the transmission mirror position. This mirror configuration provides an unfocused spot size of approximately 20 mm wide x 25 mm high. Place the mirror in the centre-mount position if an intermediate beam size is desired.

The reflectance port is constructed at an 8° angle from the incident sample beam to facilitate 8°/h measurements. Additionally, the DRA has a specular exclusion port - the diffuse and specular reflectance components can be separated by removing the specular port plug. During 8°/h reflectance measurements, the specular port plug should be installed. The following procedure should be used to measure the hemispherical reflectance factor of a sample:

1. Collect a Baseline scan.

2. Replace the reflectance standard at the sample reflectance port with the sample. Do not change the configuration of the integrating sphere in any other way.

3. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

4. Click the *Start* button.

5. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard.

8°/d Reflectance Factor Measurements (Comparison Method)

The DRA integrating sphere is fitted with a specular exclusion port. When the exclusion port plug is removed, the specular component of reflection from the sample is directed out of the sphere and is not sampled by the detector. The diffuse component of the main beam reflection remains inside the sphere and is sensed by the sphere detector. The procedure for diffuse reflectance factor measurement is as follows:

1. Collect a Baseline scan.

2. Replace the reflectance standard at the sample reflectance port with the sample.

3. Replace the specular port plug with the specular port light trap. Do not change the configuration of the integrating sphere in any other way.

4. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline..

5. Click the *Start* button.

6. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard.

8°/h Reflectance Factor Measurements (Single Beam Mode)

Although the DRA is a double beam accessory, single beam operation is still possible. When operating the DRA in this mode, a substitution error will be present in the reflectance measurement data. The reference beam entrance port can be blocked off using a Spectralon surface during the baseline and sample scans. The following procedure can be used to measure the hemispherical reflectance of a sample.

1. Collect a Baseline scan, making sure the instrument is configured for single rear beam mode.

2. Replace the reflectance standard at the sample reflectance port with the sample. Do not change the configuration of the integrating sphere in any other way.

3. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

4. Click the *Start* button.

5. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard.

0°/h Transmission Measurements (Comparison Method)

For transmission measurements, make sure that mirror M3 is placed in the transmission position, marked "T" on the base plate, located farthest from the sphere. If this mirror is moved or if the cuvette holder is installed over the transmission port, the transfer optics must be realigned. Repeat the alignment procedure if M3 has been moved. The "T" position mirror configuration provides a condensed spot size of approximately 5 mm wide x 9.5 mm high. If a large transmission spot size is desired, such as for the measurement of fabrics or other homogeneous samples, place mirror M3 in the reflectance "R" position. This mirror configuration provides a spot size of approximately 11 mm wide x 28 mm high. If an intermediate spot size is required, place the mirror in the centre-mount "C" position.

1. Collect a Baseline scan, following the procedures specific to transmission measurements. If the sample resides on a substrate, load an untreated substrate blank at the transmission port for the baseline scan recordings. If the transmission sample is suspended in solution, load a blank cuvette at the transmission port for the baseline.

2. Load the sample at the transmission port. Do not change the configuration of the integrating sphere in any other way.

3. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

4. Click the *Start* button.

5. The value displayed by the instrument is the sample transmission. No data correction is required.

0°/d Transmission Measurements (Comparison Method)

The approximate diffuse transmission of a given test sample can be measured using the DRA as follows:

1. Collect a Baseline scan, following the procedures specific to transmission measurements. If the sample resides on a substrate, load an untreated substrate blank at the transmission port for the baseline scan. If the transmission sample is suspended in solution, load a blank cuvette at the transmission port for the baseline.

2. Load the sample at the transmission port and replace the reflectance standard at the reflectance port with a light trap. Do not change the configuration of the integrating sphere in any other way.

3. Click the *Setup* button and check the parameters. Normally, the Setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

4. Click the *Start* button.

5. The value displayed by the instrument is the sample transmission. No data correction is required.

Loading samples at the reference or reflectance ports

When taking a sample at either the sample or reflectance port requires the use of the sample holder as shown below.

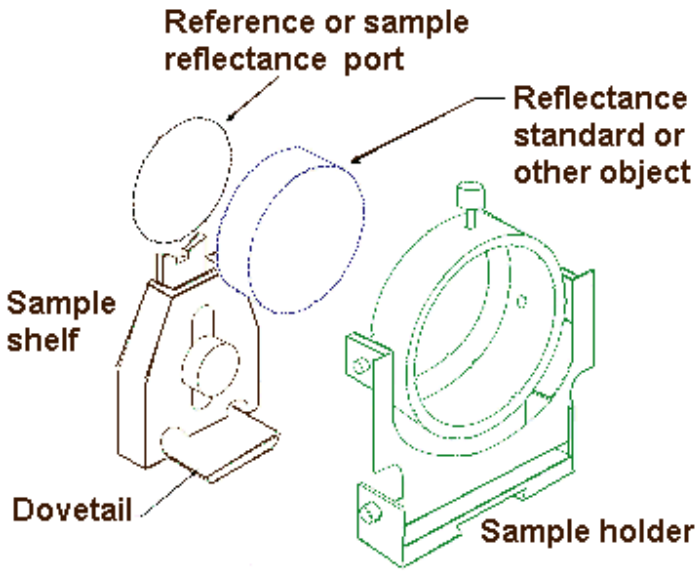


Figure 48. The sample is mounted at the reflectance or reference port using the sample holder.

Variable Angle Reflectance Measurements (Centre-Mount Sample Holder)

The DRA can collect variable angle of incidence reflectance data when a centre-mount sample holder is installed inside the integrating sphere. Shift mirror M3 to the centre-mount position and align the transfer optics before proceeding with either of the following procedures. The centre-mount sample holder should be employed only when operating the instrument in double beam mode. Photometric accuracy of the variable angle data may be reduced due to absorption characteristics of the sample holder surfaces.

When using centre-mount devices, correct beam alignment is essential. Occasionally, the requirement exists to conduct fine tune alignment of the beam inside the integrating sphere. Rather than subject the sphere detectors to white light radiative flux, the operator can drive the instrument to 500 nm and observe the beam path adjustments through the accessory cover opening.

Note
Not all transfer optics are accessible through the sample compartment trap door, and white light operation may be necessary.

To drive the instrument to 500 nm, select *Goto* from the Commands menu, enter 500 nm and click *OK*. The sample compartment lid must remain shut in this mode of operation. To establish white light operation temporarily, load beam blocks at the transmission and reference beam entrance ports and select *Align* from the Commands menu.

Clip Style Sample Holder Measurements

1. Darken the room and drive the instrument to 500 nm.

2. Mount the sample into the sample holder, making sure it is properly centreed and secured to the holding clip. Load the assembly into the integrating sphere and set the anticipated angle of incidence using the attached dial.

3. While viewing the sample surface through the reference port, check that the sample beam strikes the centre and does not overfill the sample at the angle selected. This step may require use of a piece of translucent paper and will be more difficult at greater angles of incidence. If the beam is off centre or will not fit on the sample, either adjust the sample mounting in the sample holder or readjust the sample beam optics to achieve the correct alignment. If any adjustments are made to the optics, make sure the sample beam does not clip the transmission port.

4. Examine the path of the reference beam inside the integrating sphere. The beam should strike the reference port without clipping any part of the sample or sample holder.

5. Remove the sample from the sample holder clip and load the empty sample holder into the centre-mount port.

6. Load Spectralon reflectance standards at the reference and sample reflectance ports. Install the magnetic covers over each respective port.

7. Collect a Baseline scan.

8. Mount the sample into the centre-mount sample holder and load the assembly into the integrating sphere.

9. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

10. Click the *Start* button.

The value displayed by the instrument is the absolute sample reflectance. No data correction is required.

Jaw Style Sample Holder Measurements

1. Darken the room and drive the instrument to 500 nm.

2. Mount the sample onto the sample holder, making sure it is properly centreed and secured to the holding jaws. Load the assembly into the integrating sphere and set the anticipated angle of incidence using the attached dial.

3. While viewing the sample surface through the reference port, check that the sample beam strikes the centre and does not overfill the sample at the angle selected. This step may require use of a piece of translucent paper and will be more difficult at greater angles of incidence. If the beam is off centre or will not fit on the sample, either adjust the sample mounting in the sample holder or readjust the sample beam optics to achieve the correct alignment. If any adjustments are made to the optics, make sure the sample beam does not clip the transmission port.

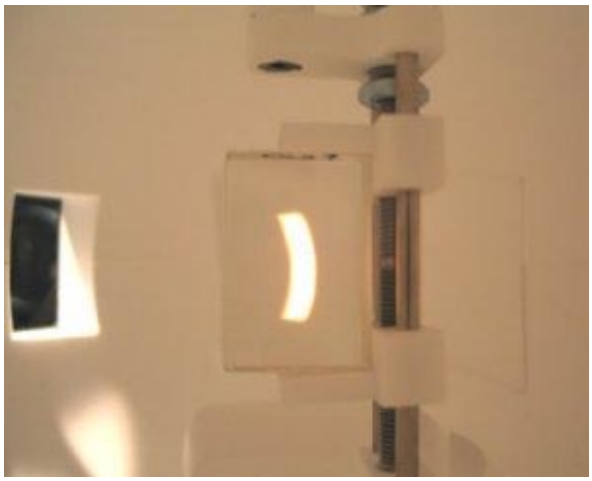


Figure 49. The beam falling on the reference mounted in the jaw style centre mount.

4. Examine the path of the reference beam inside the integrating sphere. The beam should strike the reference port without clipping any part of the sample or sample holder.

5. Remove the sample holder from the sphere and replace the sample with a calibrated diffuse reflectance standard. If a calibrated standard is not available, use an uncalibrated Spectralon standard. Load the sample holder assembly into the integrating sphere and install the accessory cover.

6. Collect a Baseline scan.

7. Mount the sample into the centre-mount sample holder. For the jaw style device, the transmitted component from the sample beam must be eliminated so it is not collected by the sphere. This can be accomplished by backing the sample with a piece of black felt cloth. Load the sample holder assembly back into the sphere.

8. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

9. Click the *Start* button.

10. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard or the typical data in Appendix B. Reflectance standards at Labsphere are calibrated using 8°/h geometry

Absorbance Measurements (Cuvette Centre-Mount Sample Holder)

The following procedure can be used for obtaining absorbance measurements with a centre-mounted cuvette. In the centre-mount configuration, normally reflected light exits the sphere through the transmission port and is not counted.

1. Darken the room and drive the instrument to 500 nm.

2. Fill a clean cuvette with the pure solvent used as the sample preparation.

Note
If the intended sample is turbid, use an empty cuvette for the baseline scan. Load the blank cuvette into the holder and load the sample holder assembly into the centre-mount port. Position the alignment pins on the centre-mount device to match the holes in the sphere.

3. While viewing the front cuvette surface through the reference port, check that the sample beam strikes the centre of the cuvette. This step may require use of a piece of translucent paper. If the beam is off centre or will not fit on the sample, re-adjust the sample beam optics using M3 to achieve the correct alignment. If any adjustments are made to the optics, make sure the sample beam does not clip the transmission port.

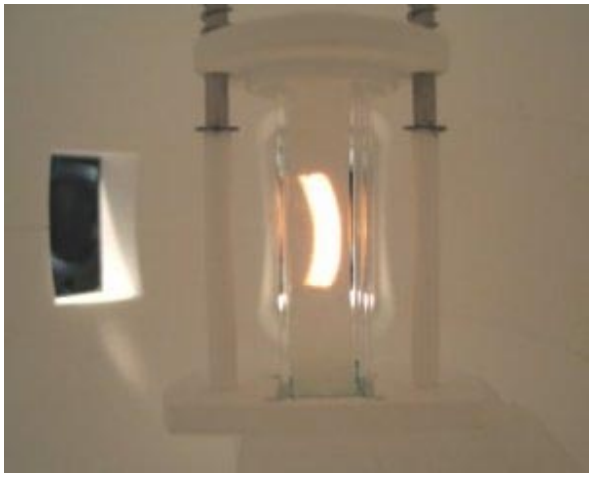


Figure 50. The beam falling on the cuvette centre mount.

4. Examine the path of the reference beam inside the integrating sphere. The beam should strike the reference port without clipping any part of the cuvette or sample holder.

5. Collect a Baseline scan, or use a zeroline baseline, using the blocked beam method.

6. Remove the sample holder and replace the blank cuvette with the sample cuvette. Load the sample holder assembly back into the sphere.

7. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

8. Click the *Start* button.

The value displayed by the instrument is the combined transmission. No data correction is required.

Using the Small Spot Kit

The DRA small spot kit is composed of three lens options that focus the beam to a small diameter spot at the selected sphere location. The top of each lens is marked as either: "R" for reflectance port, "T" for transmission port, or "C" for centre-mount location. An arrow appears next to each lens marking. The direction of the arrow indicates the side of the lens which should face the sphere. After the desired lens is installed into the lens holder assembly, it can be moved along its rail to focus the sample beam. When the transmission lens is installed, the overall beam spot size will change as the lens holder is adjusted along the dovetail rail. The spot size can be reduced down to 2.5 - 3 mm diameter at the transmission port. For controlling beam size at the centre-mount or sample reflectance port locations, the iris should be used in conjunction with the corresponding lens. For the reflectance beam spot, the iris assembly can be closed to reduce the spot size from approximately 8 mm tall by 2 mm wide to approximately 3.5 mm tall by 1.5 mm wide. Similar results are achieved with the centre-mount lens and iris combination. A mask should be fitted over the transmission or reflectance ports when collecting small sample measurements.

When operating in small spot mode, the sample beam undergoes considerable attenuation before it reaches the integrating sphere. Increased energy and an expanded slit width may be required for collecting accurate spectra. When measuring highly absorbing samples, it may be necessary to attenuate the reference beam. A wire attenuation screen is provided for this purpose. The screen is designed to fit at the entrance port for the reference beam. The reference beam energy should be attenuated so that it matches the sample beam energy in its most reduced state. Consult the Cary instruction manual for more information on instrument requirements regarding sample and reference beam energy balance.

Note
A reflectance port aperture kit, Labsphere Part No. AS-02021-000, is available through your Varian representative to reduce the DRA reflectance port diameter to better suit small samples. The kit consists of five different aperture plates which mount on the reflectance port dovetail rail. These plates effectively change the reflectance port diameter to 1”, 0.5”, 0.375”, 0.25”, or 0.15” (25 mm to 4 mm diameter). A special sample restraining clamp is provided, and it can be fitted with one of five suitably sized spacers to adapt to any sample size.

Transmission Measurements (Small Spot Kit)

At the transmission port location, the DRA small spot kit can be used to measure a small test sample, or to measure a small portion of a larger sample. Prior to entering this procedure, the small spot kit should be installed and aligned. Mirror M3 must always be installed at the "T" position for small spot kit measurements.

1. Install the transmission lens, marked “T”, into the small spot kit lens holder. The arrow marked on the lens should be pointing towards the sphere.

2. Darken the room and drive the instrument to 500 nm.

3. If the intended sample size is smaller than the transmission port opening, secure a mask over the transmission port. The mask should be attached to the transmission port or holder, and it must remain in place for both the baseline and sample scans. If the sample is larger than the port size, place a piece of translucent paper into the clips of the transmission sample holder and install the holder at the transmission port.

4. Adjust the position of the small spot kit lens assembly on its dovetail rail until the desired spot size is obtained at the transmission port. The spot should be adjusted to fit just within the sample surface. In general, the transmission spot size will be smallest when the lens is moved away from the sphere. To optimize the signal reaching the sphere detectors, the iris should remain fully open for small spot kit transmission scans.

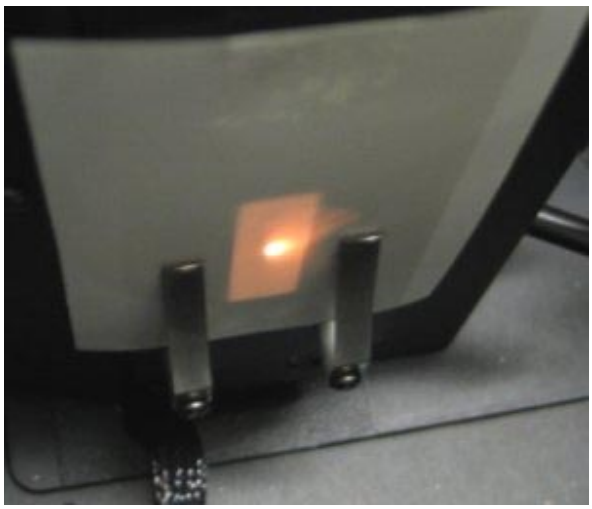


Figure 51. The beam at the transmission port

5. Collect a Baseline scan, following the procedures specific to transmission measurements. If the sample resides on a substrate, load an untreated substrate blank at the transmission port for the baseline scan. If the transmission sample is suspended in solution, load a blank cuvette at the transmission port for the baseline.

6. Load the sample at the transmission port. Do not change the configuration of the integrating sphere in any other way.

7. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

8. Click the *Start* button.

9. The value displayed by the instrument is the sample transmission. No data correction is required.

Reflectance Measurements at the Centre-Mount Location (Small Spot Kit)

For samples loaded at the centre-mount location, the DRA small spot kit can be used to measure a particularly small test sample, a portion of a larger sample, or to increase the variable angle measurement capabilities of the accessory. Prior to entering this procedure, the small spot kit should be installed and aligned. Mirror M3 must always be installed at the "T" position for small spot kit measurements.

1. Install the centre-mount lens, marked “C”, into the small spot kit lens holder. The arrow marked on the lens should be pointing towards the sphere.

2. Darken the room and drive the instrument to 500 nm.

3. Remove the transmission sample holder from the dovetail at the integrating sphere transmission port.

4. Mount the sample onto the centre-mount sample holder, making sure it is properly centreed and secured. Load the assembly into the integrating sphere and set the anticipated angle of incidence using the attached dial.

5. Adjust the iris aperture to full open.

6. Adjust the position of the small spot kit lens assembly on its dovetail rail, changing the distance between the “C” lens assembly and the sphere until the beam spot obtained on the centre-mount sample is crisp and well-focused. Make sure that the beam does not overfill the edges of the sample. In general, the centre-mount spot size and focus will be optimized when the lens is moved away from the sphere. If it is necessary to reduce the beam size to eliminate sample overfilling, adjust the iris diaphragm until the spot on the sample fills approximately 75% of the entire sample surface.

7. While viewing the sample surface through the reference port, check that the sample beam strikes the centre and does not overfill the sample at the angle selected. This step may require use of a piece of translucent paper and will be more difficult at greater angles

of incidence. If the beam is off centre or will not fit on the sample, either adjust the sample mounting in the sample holder or re-adjust the sample beam optics using M3 to achieve the correct alignment. If any adjustments are made to the optics, make sure the sample beam still fits within the small spot lens aperture.

Figure 52. The small spot kit beam falling on a sample using one of the centre mount options.

8. Examine the path of the reference beam inside the integrating sphere. The beam should strike the reference port without clipping any part of the sample or sample holder.

9. Once the sample beam spot is set, remove the sample from the centre-mount holder, noting the correct orientation and position. In the case of the jaw style sample holder, mount a reflectance standard or, in the case of the clip style sample holder, leave the sample holder empty. Load the sample holder assembly back into the sphere.

10. Collect a Baseline scan. If using a jaw style centre-mount sample holder, the zeroline standard reference correction may be used.

11. Mount the sample into the centre-mount sample holder. For the jaw style device, the transmitted component from the sample beam must be eliminated so it is not collected by the sphere. This can be accomplished by backing the sample with a piece of black felt cloth. Load the sample holder assembly into the sphere.

12. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

13. Click the *Start* button.

14. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected and the jaw style sample holder was employed, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard or the data in Appendix B. If using the clip style centre-mount sample holder, no correction is required.

Reflectance Measurements at the Reflectance Port (Small Spot Kit)

For samples loaded at the sample reflectance port, the DRA small spot kit can be used to measure a particularly small test sample or a portion of a larger sample. Prior to entering this procedure, the small spot kit should be installed and aligned. Mirror M3 must always be installed at the "T" position for small spot kit measurements.

1. Install the centre-mount lens, marked "R", into the small spot kit lens holder. The arrow marked on the lens should be pointing towards the sphere.

2. Darken the room and drive the instrument to 500 nm.

3. Remove the transmission sample holder from the dovetail at the integrating sphere transmission port.

4. If the intended sample size is smaller than the reflectance port opening, secure a mask over the port. The mask should be attached to the reflectance port, and it must remain in place for both the baseline and sample scans. If available, the optional reflectance port aperture kit, Labsphere Part No. AS-02021-000 can be used. If the sample is larger than the port size, place a piece of translucent paper over the reflectance port sample holder and install the holder at the port.

5. Adjust the iris aperture to full open.

6. Adjust the position of the small spot kit lens assembly on its dovetail rail, changing the distance between the "R" lens assembly and the sphere until the beam spot obtained at the reflectance port is crisp and well-focused. Make sure that the beam does not overfill the edges of the sample. In general, the spot size and focus will be optimized when the lens is moved closer to the sphere. If it is necessary to reduce the beam size to eliminate sample overfilling, adjust the iris diaphragm until the spot on the sample fills approximately 75% of the entire sample surface. Once the beam size is set, remove any paper at the reflectance port of the sphere, so that the port or mask is open.

7. Load a calibrated diffuse reflectance standard against the reflectance port of the sphere. The standard should be placed behind the sample port mask or aperture plate, if one is used. Record one of the baseline scans as directed previously in this chapter.

8. Replace the reflectance standard at the sample reflectance port with the sample. Do not change the configuration of the integrating sphere in any other way.

9. Click the *Setup* button and check the parameters. Normally, the setup parameters for the application should match those used for the baseline scan. If using a previously defined baseline correction, retrieve the correct baseline.

10. Click the *Start* button.

11. If the standard reference option was used for the baseline scan, the reflectance spectra is generated automatically. If the simple or zero baseline correction was selected, multiply the sample scan data by the spectral reflectance factors for the calibrated reflectance standard.

Small Sample Masking Techniques

Small samples that do not completely fill the sample reflectance or transmission port should be measured using masking techniques. Masking in this case may be necessary if only to mount the sample at the appropriate port. When using the small spot kit to measure the reflectance or transmission of samples smaller than the port diameter, both the reference and the sample can be masked such that the port is filled completely.

Generally a black mask is used to prevent reflectance from the mask adding to the reflectance of the sample. Another reason for a black mask is consistency - a black mask is always the same and easy to make. The reflection off the black mask is very small and does not contribute to the reflectance properties of the sample. Simple and effective masks can be prepared from a piece of cardboard. A manila file is excellent because it is thin and easily manipulated. The mask must be constructed large enough to fill the port. The internal surface of the mask should be painted black with flat black Krylon™ paint or its equivalent. The illustrations in the figures above and below demonstrate the use of a mask at the transmission and reflectance ports, respectively. The following guidelines will help when constructing masks:

- The mask surface area should cover the entire transmission or reflectance port.
- The mask aperture should be in the same profile of the sample surface, but slightly smaller in size.
- The sample beam should overfill the mask aperture.
- The baseline scan should be collected during reflectance measurements with the reflectance standard in the same mask geometry. For transmission measurements, the masked port constitutes the 100% transmission standard.

Note
There are two negatives of using a mask in your analyses. Firstly, using a mask at either the transmission port or the sample reflectance port reduces the throughput of the accessory and energy seen by the detector. Secondly, using a mask at the sample reflectance port upsets the balance between the sample and reference beams because the reference beam strikes the high reflectance inner surface of the sphere. If the spectrophotometer is operating in single beam mode, of course, this is not a

problem. Impact of these factors can be minimized if you can localize the beam to the target area of the sample using the small spot kit so the beam just barely fits over the edges of the mask.

Large sample reflectance measurements

When measuring large samples that cannot be accommodated within the sample reflectance port cover, you will need to follow the steps below:

Prior to recording the scan, make sure that mirror M3 is located in the correct position. To measure a sample::

1. Click the *Setup* button and set up instrument parameters as required by the application. Select the %R ordinate scale. The *Beam mode* should be double.

2. Select the *Zero/baseline correction* or *Zero x std ref correction* radio button on the Baseline tab.

3. Load a Spectralon standard at the reference port and a calibrated reflectance standard, if available, at the sample reflectance port. If a calibrated standard is unavailable for reflectance measurements, an uncalibrated Spectralon reflectance standard can be substituted. Install the port covers over their respective ports.

4. Click the Baseline button.

5. At the 100%T scan prompt, select OK. The standard at the reflectance port serves as the 100%R reference.

6. At the 0%T scan prompt block the sample beam at the transmission port or remove the reflectance standard and use the reflectance port cover as a light trap.

7. Remove the reflectance port cover, the spring loaded sample holder, the sample shelf and unscrew the dovetail using a 3mm hex driver.

8. Position and secure your sample up against the reflectance port and start you reflectance measurement.

The recorded baseline correction will be effective until replaced by a follow-up baseline scan.

Note
If you have adequate control over the room lighting, it is recommended that you run your 0%T scan without the reflectance port cover in place. In this configuration the room itself becomes the light trap.

Applying a Nitrogen Purge

The Cary 4000/5000/6000i instruments are fitted with connection points for purging the optical system with nitrogen to enhance the performance of each instrument at extremes of its range. More details are provided in the Cary Hardware operation manual (part number 8510197200), supplied with the instrument.

Nitrogen supplies are not available from Varian but may be obtained from commercial suppliers. Liquid nitrogen (in conjunction with a heat exchanger) is recommended because it is generally less costly than compressed nitrogen and is of better quality. Where compressed nitrogen must be used, the gas must be dry, oil–free and uncontaminated. Do not use compressed nitrogen from a supplier who uses oil or water in the compression process (these methods leave fine particles of oil or water suspended in the nitrogen that may be deposited on the instrument optics). Only use nitrogen from a supplier who fills containers from immersion pumps lubricated with liquid nitrogen.

Note
The instrument warranty will be void if damage is caused by the use of contaminated nitrogen.

Operating pressure at the pressure regulator (# 7 in figure 53) for the nitrogen purging system is 83 to 172 kPa (12 to 25 psi). Use a suitable regulator and gauge assembly to ensure that the nitrogen supply is maintained at the correct pressure.

Nitrogen supply tubing should be clean, flexible plastic tubing 6 mm (1/4“) inside diameter (Tygon PVC or equivalent). Do not use rubber tubing as this is usually treated internally with talc which will be carried into and contaminate the instrument optics.

The nitrogen system should include a manifold assembly with inlet from the supply and two outlets for connection to the instrument. Manifold outlets should each be fitted with a stop valve and flowmeter for control of gas flow to the instrument. Flow meters should be adjustable for flow rates of 0 to 30 litres per minute (0 to 64 cubic feet per hour). Refer to the figure below for more details.

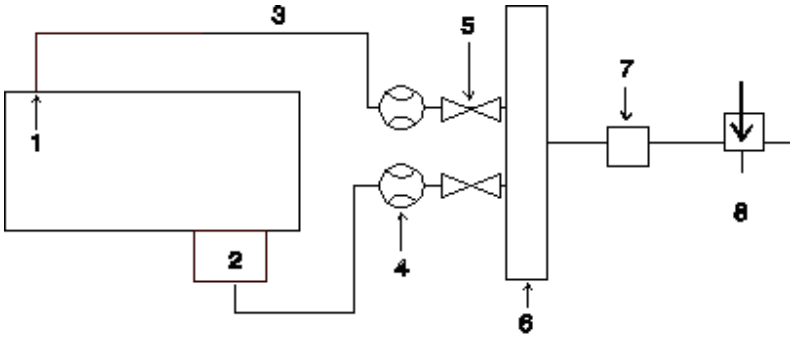


Figure 53. The position of flowmeters when purging with nitrogen:

1. Instrument *	2. External DRA
3. Tubing	4. Flow meters
5. Shut-off valves	6. Manifold
7. Pressure regulator	8. Nitrogen control valve

**Refers to the purge inlet labelled "Instrument".*

The DRA accessories each have an inert gas purge capability for reducing water vapour absorption inside the integrating sphere. The gas nozzle is located underneath on the left-hand side of the overhanging accessory. A nitrogen purge can be applied as follows:

1. Install the accessory into the instrument sample compartment.

2. Attach a suitable gas regulator to the gas outlet of a nitrogen dewar or N2 cylinder.

3. Connect a suitable hose between the regulator outlet and the nitrogen connector on the accessory.

Figure 54. Attach the connector on the hose to the connector on the under side of the integrating sphere.

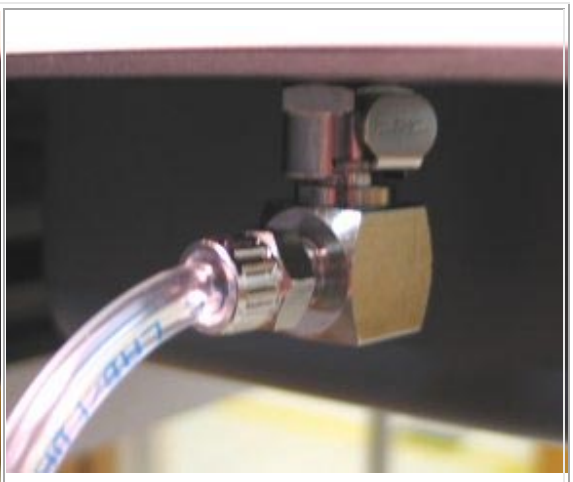


Figure 55. The connectors snap together.

4. Crack open the shut off valve until the flow meter indicates the flow of nitrogen. Wait approximately five minutes before proceeding with reflectance or transmission measurements.

NIR reflectance measurements (not for Cary 4000)

Note
Parameters activated by a radio button that are not specifically mentioned in the following procedure should be set to 'Off' (e.g. the Signal-to-noise mode radio button). These parameters will not affect the procedure.

1. Click the Windows Start button, then Programs, then Cary WinUV, and then Align.

2. Click the *Setup* button.

3. Set the following parameters:

Cary tab	
X mode: Mode	Nanometers
X mode: Start	2000
X mode: Stop	300
Y mode: Mode	% R
Y mode: Y min	0.00
Y mode: Y max	100.00
Show status display	ON
Options tab	
SBW/Energy: Beam mode	Double
SBW/Energy: Slit height	Reduced
Source/Detector: Lamps	UV-Vis
Source: Source changeover (nm)	350.0
Source: Detector changeover (nm)	800.0
Independent tab	
Independent control	ON
Measurement mode	Auto
UV-Vis controls	
Ave time (s)	0.1

Data interval (nm)	1.000
Scan rate (nm/min)	600.00
SBW (nm)	2.00
NIR controls	
Ave time (s)	1.000
Data interval (nm)	2.000
Scan rate (nm/min)	120.00
Energy level	3.00
Baseline tab	
Correction	Zero correction
Autostore tab	
File Storage: Storage	Storage on (prompt at start)

Note
The NIR detector is noisier than the UV-Vis detector, so a higher *Signal Averaging Time (Ave time)* may need to be set in the NIR. To obtain a constant signal-to-noise ratio over the entire wavelength range, set a value in the *Signal-to-noise field*. If you require a faster scan rate, increase the *Data interval* or lower the *Ave Time*.

4. Place the appropriate PTFE reference plate over the sample port.

4. Place the appropriate PTFE reference plate over the sample port.

5. Press the 'Goto' button to open the 'Goto wavelength' dialog. Enter '801' in the wavelength field and click OK. The instrument will then drive to 801 nm.

Note
Note the SBW reading in the *Status Display* dialog.

6. The SBW reading needs to be at approximately 18. This can be adjusted by changing the *NIR Energy level* in the *Setup* page, under the *Independent* tab. If the reading is greater than 18, raise the value of the *Energy level* field. If the SBW is under 18, lower the energy level. Once the Energy level has been adjusted, you will need select OK in order to note the change to the SBW reading. Then press the *Goto* button and again drive the instrument to 801 nm by entering '801' in the wavelength field and pressing OK. This procedure may need to be repeated in order to reach the required setting. Please note that each time you adjust the Energy level reading, you must also drive the instrument to 801 nm.

7. Select the *Baseline* button from the *Scan* dialog. Follow the on-screen prompts to perform a 100%T baseline scan and a 0%T baseline scan.

Hot Tip
When performing reflectance measurements, remove the PTFE reference disk from the sample port and allow the light to be trapped by the magnetic port covers.

8. Once the baseline correction is complete, the *Ordinate status display* in the top left corner of the dialog will show 'Zero baseline' in red text.

9. Clamp the sample over the sample port. The sample should be homogeneous, substantially flat, and large enough to completely cover the image.

10. Press the *Start* button. The *Save As* dialog will appear, allowing the method to be saved as either a data file or a batch file. If the file is saved as a batch file, all of the method parameters will be stored with the scan.

Specular-only reflectance measurements

Note
Parameters activated by a radio button that are not specifically mentioned in the following procedure should be set to 'Off' (e.g., the Signal-to-noise mode radio button). These parameters will not affect the procedure.

1. From the Setup menu, set the following parameters:

Cary tab	
X mode: Mode	Nanometers
X mode: Start	800
X mode: Stop	300
Y mode: Mode	% R
Y mode: Y min	0.00
Y mode: Y max	100.00
Scan controls: Ave time (s)	0.100
Scan controls: Data interval (nm)	1.000
Scan controls: Scan rate (nm/min)	600.00

Options tab	
SBW/Energy: Fixed SBW	ON
SBW/Energy: SBW (nm)	2.00
SBW/Energy: Beam mode	Double
SBW/Energy: Slit height	Reduced
Source: Lamps	UV-Vis
Source: Source changeover (nm)	350.0
Baseline tab	
Correction	Zero baseline/correction
Autostore tab	
File Storage: Storage	Storage on (prompt at start)

2. Place the specular port plug into position.

3. Place the appropriate PTFE reference plate over the sample port.

4. Select the *Baseline* button from the Scan dialog. Follow the on-screen prompts to perform a 100%T baseline scan and a 0%T baseline scan.

Hot Tip
When performing specular measurements, block the sample beam in the transmission position with a black masking.

5. Once the baseline correction is complete, the *Ordinate status display* in the top left corner of the dialog will show 'Zero baseline' in red text.

6. Clamp the sample over the sample port. The sample should be homogeneous, substantially flat, and large enough to completely cover the image.

7. Press the *Start* button to perform the scan of the total (specular and diffuse) reflectance of the sample.

8. Remove the specular port plug and install the light trap in its place and click the *Start* button to collect the diffuse-only data.

9. From the main Scan window, click on the Calculator icon  to open the open the Maths dialog.

10. On the graph displayed, highlight the first scan (of the total reflectance), by clicking on it. When the scan is selected, it will be highlighted in red.

11. From the *Maths* dialog choose *Selected Trace* and then the '-' (minus) sign.

12. On the graph displayed, highlight the second scan (of the diffuse-only reflectance), by clicking on it. When the scan is selected, it will be highlighted in red.

13. From the *Maths* dialog choose *Selected Trace* and then the '=' (equal) sign.

14. The results of this specular data equation will be displayed in a new graph.

Measurement calibration and corrections

Calibration

The detector calibration functions store separate sets of correction factors for the instrument detectors and for the DRA detectors, so both require calibrations. The instrument will recognize when an external DRA has been connected to the instrument and will restore any previous calibrations stored in the external DRA.

Calibrations are performed using the Validate application, under the Calibration menu.

Caution
Both instrument and DRA are calibrated at the factory and should be recalibrated only when tests indicate the need for it.

When should I perform a calibration?

Below are some common reasons that may indicate the necessity for calibration.

Calibrate PGA (Programmable Gain Amplifier)

PGA calibration should be executed each time a DRA is installed in a Cary instrument for the first time, and after each time it is removed. At other times, the need for it may be seen on an uncorrected baseline as steps or plateaus or on a corrected baseline as small spikes-either positive or negative.

UV/VIS 0%T Correction

This calibrates the photomultipliers for 0%T (electronic zero) effects. This is executed every time the instrument is switched on except if a DRA is installed (where the DRA correction factors stored in the DRA EEPROM are used).

Instrument check: If high precision is needed at high absorbance, measure the 0%T error at 500 nm in the UV-VIS (blocked sample beam). The error should be less than 0.01 %T. Calibrate with the instrument fully warmed up. It may be desirable to calibrate Calibrate PGA prior to this. Click here for the instrument parameters.

NIR 0%T Correction

This is the NIR equivalent for 0%T error.

Instrument check: After instrument has fully warmed up, go to wavelength 1200 nm with grating change at 800 nm and block the sample beam with a black metal mask. If the error exceeds 0.1 %T calibration may be required.

Calibration using Cary Win UV Software

The recommended method for performing calibration is by using the Auto Calibrate function in the Validate application.

Caution
Both instrument and DRA are calibrated at the factory and should be recalibrated only when tests indicate the need for it. Failure to complete or the use of incorrect filters may render the instrument unusable.

Signal processing

This causes the Cary system to calibrate the programmable gain amplifiers on the photomultiplier tube. This test is performed during initialization of the Cary 4000/5000/6000i instruments.

UV/Vis 0 %T correction

This causes the Cary system to close the shutter and calibrate the UV/Vis detector for 0 %T errors. If a DRA is connected a mask must be placed in front of the transmission port (rear instrument beam) during the calibration process. The software will prompt you for this.

Note
This calibration is part of the normal initialization procedure, however calibration is advised when working at high absorbance where a repeat calibration after the

instrument is fully warmed up would be beneficial.

NIR 0%T correction

This closes the shutter and calibrates the NIR detector for 0 %T errors. If a DRA is connected a mask must be placed in front of the transmission port (rear instrument beam) during the calibration process. The software will prompt you for this.

Auto calibrate

Will perform all the calibrations relevant to the instrument and DRA combination being used. User interaction is required if a DRA is in place.

Wavelength status

This allows the user to view the Wavelength offsets stored in the EEPROM.

Performing a correction to ASTM E903-C-OS/2

If you are using a NIST Standard Reference material (SRM) (e.g. White ceramic tile) as the reference disk then you should enter the calibrated reflectance values as a continuum in the Cary system. To do this, create an ASCII file and enter the list of X and Y data pairs (separated by commas). Save this ASCII file in the '.CSV' format.

If you are using the PTFE reference disk supplied with the DRA, then the following values may be used. These values are approximate only. The PTFE reference plate has not been calibrated. The reflectance values are representative of the PTFE reference plates supplied with the DRA(3).

1. Click the Windows Start button, then Programs, then Cary WinUV, and then Scan.

2. Click the *Setup* button.

3. Set the following parameters:

Cary tab	
X mode: Mode	Nanometers
X mode: Start	300
X mode: Stop	2000
Y mode: Mode	% R
Y mode: Y min	-5.00
Y mode: Y max	110.00

Show status display	ON
Options tab	
SBW/Energy: Beam mode	Double
SBW/Energy: Slit height	Reduced
Source/Detector: Lamps	UV-Vis
Source: Source changeover (nm)	350.0
Source: Detector changeover (nm)	800.0
Independent tab	
Independent control	ON
Measurement mode	Auto
UV-Vis controls	
Ave time (s)	0.1
Data interval (nm)	1.000
Scan rate (nm/min)	600.00
SBW (nm)	2.00
NIR controls	
Ave time (s)	1.000
Data interval (nm)	2.000
Scan rate (nm/min)	120.00
Energy level	3.00
Baseline tab	
Correction	Zero x std ref correction
Autostore tab	
File Storage: Storage	Storage on (prompt at start)

Place the reference disk or SRM in the DRA.

Run a baseline as follows:

1. Select the *Baseline* button in the Scan dialog box. Follow the on-screen prompts to perform a 100%T baseline scan and a 0%T baseline scan.

Hot Tip
When collecting the 0%T baseline scan do not block the beam instead, remove the PTFE reference disk from the reflectance port and allow the light to be trapped by the magnetic port covers.

2. Once the baseline correction is complete, the Ordinate status display in the top left corner of the dialog box will show 'Zero baseline' in red text.

3. Clamp the sample over the reflectance port. The sample should be homogeneous, substantially flat, and large enough to completely cover the image.

4. Click the *Start* button. The Save As dialog box will appear, allowing the method to be saved as either a data file or a batch file. If the file is saved as a batch file, all of the method parameters will be stored with the scan. The sample will be auto-corrected for 100%T, 0%T and Standard Reference.

5. Place your sample in the DRA and click Start.

The Cary will scan your sample and automatically perform the ASTM correction.

Maintenance

The performance of the integrating sphere is dependent on the efficiency of the coating on the sphere and the reference plates, and the cleanliness of the optical components of the accessory.

Keep the reference plates clean; if they become dirty, new plates are available from Varian. The part number for the flat PTFE reference plate is 04 1014439 00, and for the protruding PTFE reference plate 04 101988 00.

For technical support please contact Varian on:

Varian Analytical Instruments, Melbourne
679 Springvale Road
MULGRAVE, VIC 3170

Cleaning and Inspection

You may gently flush the accessory with pure nitrogen to remove water.

Perform this maintenance procedure weekly when the accessory is in use. Record the results in the accessory or instrument maintenance log.

1. Remove the accessory from the wood container and set it on a flat surface.

2. Check that each mirror mount in the optics chamber is fastened securely to the base plate.

3. Blow clean air or nitrogen over the transfer optics to remove any dust lying on the mirror surfaces.

4. Examine the surface of each mirror using a flashlight. Look for dust particles or film damage that might scatter the incident radiation.

5. Remove the reflectance standards and centre mount plug from the integrating sphere or box. Inspect the reflective surfaces of the standards for damage or dirt. Use a magnifying glass, if necessary.

6. Inspect the reflecting surface of the centre mount plug.

7. Illuminate the sphere interior with a flashlight and inspect the surfaces for soiling. Blow out any debris using clean dry air or nitrogen.

8. Re-install the centre mount plug and reflectance standards to their respective sphere ports or store the entire accessory in the wooden container.

Mirror Cleaning Procedure

Hot Tip
Holding the mirror under a bright light will show up any marks on the surface of the mirror.

The transfer optics mirrors should not be touched or handled with bare fingers. Never clean the mirrors with abrasive cleaners or tissue paper. The mirrors have a protective magnesium fluoride coating to allow cleaning if necessary. The following cleaning procedure is recommended:

1. Remove the mirror fixture.

2. Spray the mirror with Fantastic Spray Cleaner.

3. Rinse the mirror with distilled water.

4. Repeat until the water sheets off the mirror.

5. Blow dry the mirror surface with a stream of clean nitrogen.

6. Reinstall the mirror fixture and check the alignment of the optics.

Standards

Reference surfaces for reflectance and transmission in the solar wavelengths are available from (among others) Labsphere in the USA, the National Institute of Standards and Testing (NIST) in the USA, and from the National Physics Laboratory (NPL) in the UK.

Labsphere provide reference surfaces with reflectances varying from 2%R to 99%R. Labsphere may be contacted at:

P.O. Box 70
Shaker St,
North Sutton, N.H. 03260

Tel: 603 927 4266
Fax: 603 927 4694

NIST provide a range of transmission filters and solutions, and also provide the following materials for reflectance:

- First surface, Aluminium on glass
- First surface, Gold on glass
- Second surface, Aluminium on fused quartz
- Second surface, Aluminium on fused quartz, with wedge.

For more information:

Office of Standard Reference Materials
Room 205, Building 202
National Institute of Standards and Technology
Gaithersburg, MD 20899

Reference Materials available from NPL for reflectance include:

- Russian opal
- White ceramic tile
- Black ceramic tile
- Colored tiles
- Aluminium mirrors
- Absorbing glass.

NPL also have transmission standards of neutral glass filters with transmission values of 0.92 to 0.001.

Contact:

Division of Electrical Science
National Physical Laboratory
Teddington, Middlesex
TW11 OLW
UK.

Troubleshooting

Warning This instrument contains electrical circuits, devices, and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury, or painful electrical shock.

To avoid electrical shock operators and other unauthorized personnel must never remove the main cover. This must be opened only by Varian-trained, Varian-qualified, or Varian-approved service engineers.

Problem	**Solution**
Measurements on standard materials are not producing certified values.	Ensure that you are referring to the hemispherical reflectance values of the SRM, not the directional and hemispherical values. Ensure that your PTFE disks are clean and in good condition. If you are calibrating the PTFE disk, you need only apply the necessary correction to your measured values.
The signal-to-noise ratio is low.	This can be caused by degraded sphere coating, poor detector performance, the lamps requiring replacement, unclean mirrors or lens, or bad alignment. Check the sample port image, and re-align the accessory if necessary. Check that the lens and mirrors are clean, and carry out a Zero %R error check.
A *Failure to initialize instrument* error message occurs. Error number: 9:9303SEQ:E9303	Check that the: The DRA is plugged in. The beams are not blocked. The DRA is not out of alignment.
A *Cary Exception* error occurs.	An ADL command is necessary to clear this error. Click here to view/print a PDF instructing you how to use this ADL.

References

1. Weidner V.R., Hsia J.J: J.Opt.Soc.Am. 71/7 (1981)
2. Zwinkels J., Dodd C.X.: Workshop on Optical Property Measurement Techniques, Commission of the European Communities. (1988).
3. Weidner V.R., Hsia J.J., Adams B.:Applied Optics 24/14 (1985)

Alignment targets

l (nm)	R	l (nm)	R	l (nm)	R	l (nm)	R
250	0.973	700	0.994	1900	0.985	2220	0.978
260	0.976	750	0.994	1950	0.984	2230	0.978
270	0.978	800	0.994	2000	0.981	2240	0.977
275	0.979	850	0.994	2010	0.979	2250	0.977
280	0.980	900	0.994	2020	0.978	2260	0.976
290	0.982	950	0.994	2030	0.976	2270	0.976
300	0.984	1000	0.994	2040	0.975	2280	0.975
310	0.985	1050	0.994	2050	0.973	2290	0.974
320	0.987	1100	0.994	2060	0.972	2300	0.972
325	0.988	1150	0.994	2070	0.971	2310	0.971
330	0.988	1200	0.993	2080	0.970	2320	0.970
340	0.989	1250	0.993	2090	0.969	2330	0.968
350	0.990	1300	0.992	2100	0.968	2340	0.966
360	0.990	1350	0.991	2110	0.967	2350	0.965
370	0.991	1400	0.991	2120	0.966	2360	0.964
375	0.991	1450	0.992	2130	0.964	2370	0.963
380	0.991	1500	0.992	2140	0.964	2380	0.963
390	0.992	1550	0.992	2150	0.965	2390	0.962
400	0.993	1600	0.992	2160	0.967	2400	0.962
450	0.993	1650	0.991	2170	0.970	2450	0.961
500	0.994	1700	0.990	2180	0.973	2500	0.960
550	0.994	1750	0.990	2190	0.975		
600	0.994	1800	0.990	2200	0.977		
650	0.994	1850	0.986	2210	0.977		

Sample Mounting Kits

The following mounting options are available:

Item	Description	Part number
Double aperture	Mount and mask for checking the photometric accuracy of the instrument and accessory.	79 100473 00
Centre mount sample holder (jaw)	Jaw style, variable angle, centre mount sample holder. Fits into the top of the sphere and can be located at difference angles. Samples are clamped into place.	79 100475 00
Centre mount sample holder	Clip style, variable angle, centre mount sample holder. Fits into the top of the sphere and can be located at	79 100476 00

(clip)	difference angles. Samples are clipped into place.	
Centre mount cuvette holder	Centre mount holder for cuvettes. Fits into the top of the sphere.	79 100387 00
Transmission cuvette holder	Holds standard 10 mm quartz cuvettes in %T port of DRA. Mounts to the transmission port holder.	79 100383 00
Solid sample holder		79 100478 00
Aperture kit	Kit containing 3 x apertures (1 mm, 2 mm and 5 mm), 4 small spacers and 1 large spacer. Select an aperture plate slightly smaller in diameter than the sample surface and a spacer small enough to clamp the sample against the outer aperture plate surface.	79 100381 00

Small spot kit (with apertures)

Samples must be greater than 3mm in width and length. The kit includes the following:

Item	Description	Part number
Small spot kit	Kit containing, iris and lens for focusing the sample beam for taking measurements of small samples at the reflectance port.	79 100472 00

Powder cell kit

Item	Description	Part number
1 x Powder cell kit	Fits inside the reflectance sample holder in the same manner as the reflectance standard. Each sample holder comes as a set: one cell with a Spectralon insert for use as a reflectance standard, and an empty cell for loading the powdered sample	79 100477 00