

# Diffuse Reflectance Accessory (Internal)

 **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

[Click here for Cary 4, 400, 5, 500 and 500i](#)

Part Numbers: Internal DRA 900: 0010080900  
Internal DRA 1800: 0010081000  
Internal DRA 2500: 0010081100

Installation category I

Pollution degree 2

Safety class 1 (EN 61010-1)

Refer to the Safety section of the online Help for an explanation of the warnings and cautions used in this document.

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## Introduction

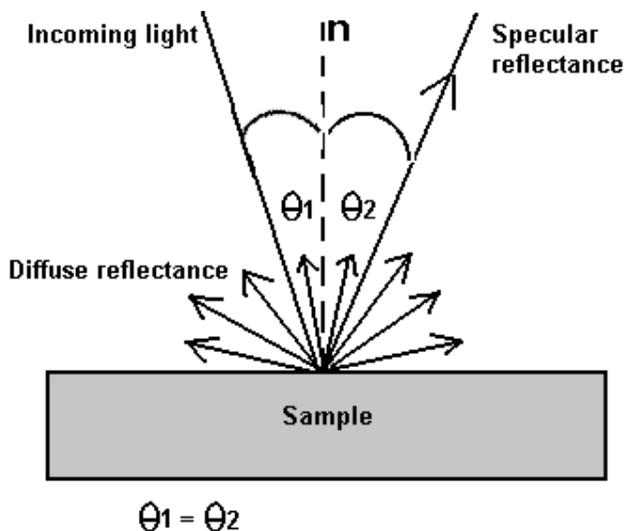
The three different internal DRAs are compatible with each of the Cary 4000, 5000 and 6000i instruments. While all of the following variations are available the configurations indicated by ticks in the table below offer optimal operation.

	(Internal DRA 900)	(Internal DRA 1800)	(Internal DRA 2500)
Cary 4000	✓	✗	✗
Cary 5000	✓	✓	✓
Cary 6000i	✓	✓	✗

The Varian internal DRA provides different mounting options:

- standard sample holder kit;
- liquid sample holder;
- small sample holder;
- powder cell;
- small sample mount:
- polarizer;
- double aperture attachment.

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 appearance.



**Figure 1:** The two components of reflection: specular and diffuse reflection.  $n$  represents the surface normal, an imaginary line at  $90^\circ$  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 Cary 4000, 5000 and 6000i Internal Diffuse Reflectance accessory (DRA) consists of a 110 mm diameter integrating sphere. The coating is Polytetrafluoroethylene (PTFE), which exhibits NIR performance that is superior to traditional coatings<sup>(1)</sup>, whilst maintaining UV-Vis performance. The sphere is easily installed by the lock down™ mechanism in the sample compartment of the instrument. The three different versions of the DRA have different detectors that determine the wavelength range at which measurements can be taken. These are detailed in the [Specifications](#) section.

Reflectance measurement of a sample is made relative to a reference material (either a PTFE or a BaSO<sub>4</sub> plate) or a standard reference material. The reference material is used to establish a baseline, and when the standard reference material is being used, the absolute reflectivity of a sample may be calculated from that of the reference. For further details, refer to the [Reflectance measurements](#) section.

The DRA requires the Extended Sample Compartment, part number 00 00100798 00 (Cary 4000, 5000, 6000i and DeepUV).

## Theory

Integrating spheres measure reflectance and transmittance factors, commonly called reflectance and transmittance. The relative spectral reflectance (transmittance) is defined as the ratio of the flux reflected (transmitted) by the specimen to that of a standard surface under identical geometrical and spectral conditions. For transmittance the standard surface is air, and for reflectance the standard surface is a secondary white standard calibrated relative to the perfectly reflecting diffuser.

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.

### **Reflectance measurements**

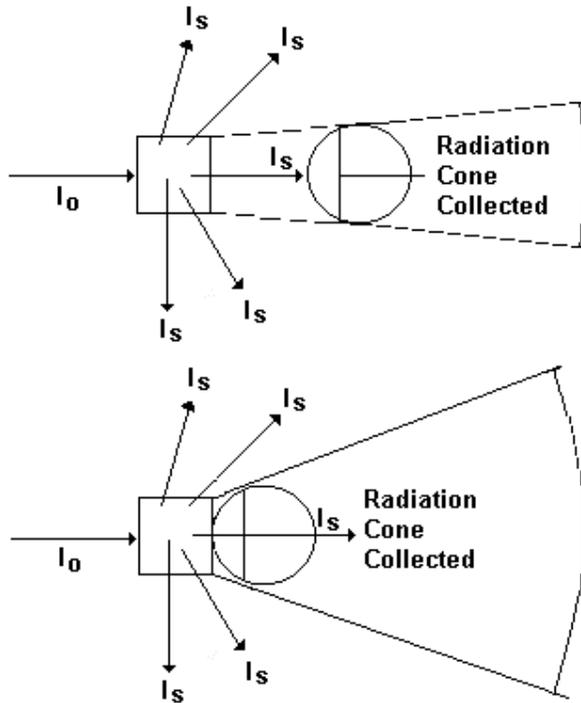
First, a baseline is recorded with the PTFE reference disk covering the reflectance port. The sample is then mounted over the port and the reflection off the sample surface is collected by the sphere. The reflectance is therefore measured relative to the PTFE disk. This is the 'substitution' method.

The total (diffuse and specular) or the diffuse-only reflectance may be measured by mounting the sample against the sphere port in two different configurations. The specular component may be calculated from the difference of these two, or the Cary Absolute Specular Reflectance Accessory (SRA P/N 00 100438 00) 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. Alternatively, you can use the [powder cell](#) in conjunction with the small sample holder.

### **Transmittance measurements**



**Figure 2:** 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 transmittance 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 2 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.

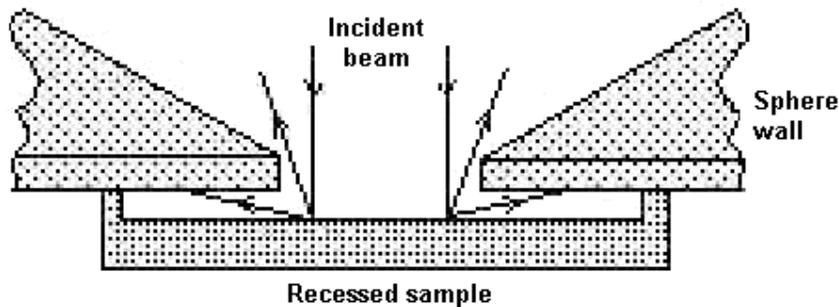
The Commission Internationale de l' Eclairage (CIE) recommendation is a ratio of <10%. This figure is only 3% for the Cary internal DRA.

### **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**

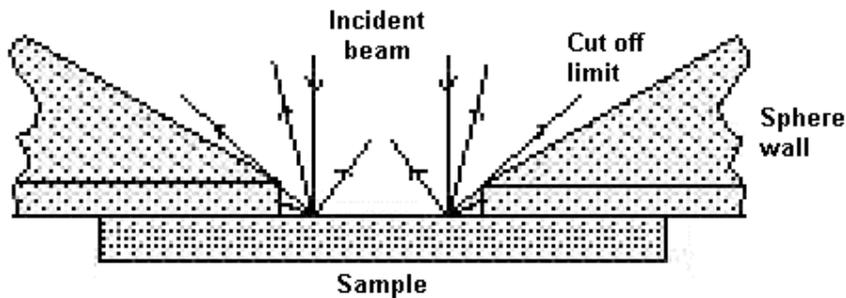
Theory assumes that the sample placement will coincide with the inside of the sphere wall. The sample is normally placed at a port on the outside of the sphere wall. Spacing between the sample and the sphere wall can lead to large errors due to loss of reflected light.



*Figure 3: 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 4:** Some of the wide-angle reflection is intercepted by the sphere wall

The edges of the reflectance port are feathered to reduce this error. A protruding PTFE reference plate is provided with the accessory to more accurately represent the perfect diffuser.

### **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.

### **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**

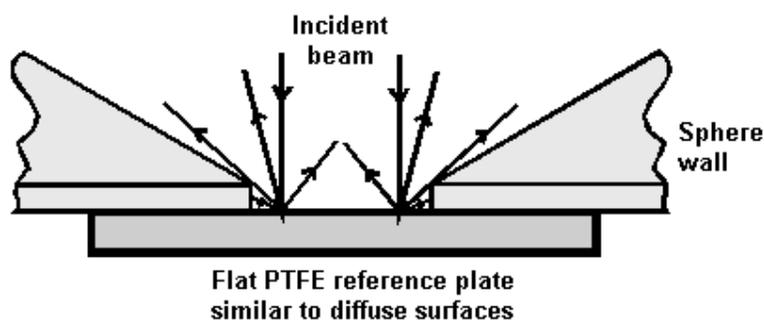
Gloss trap error is produced when the gloss trap is unable to completely absorb the specular component. In the measurement of diffuse-only reflectance, a 'gloss trap' is often used to absorb the specular component. Gloss traps are typically glossy black pyramidal light traps, matt black-coated cavities, 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. The Cary DRA eliminates this source of error entirely by reflecting the specular component out of the transmission port.

### **Differences between the standard and sample**

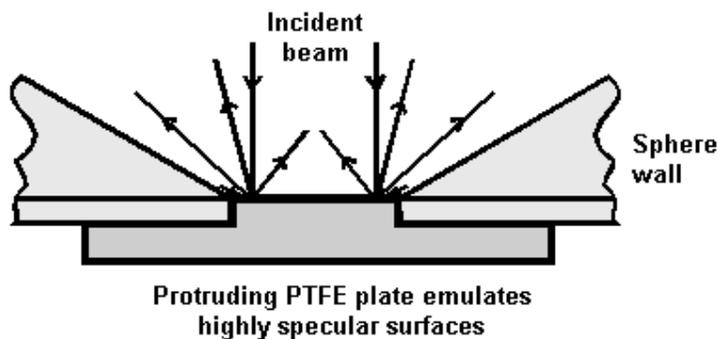
It is important that the reference material be of a similar reflectivity and have similar properties to the sample. Otherwise large errors can be introduced, if for example a poorly reflecting material is measured relative to a highly reflective PTFE reference plate. The table below lists the appropriate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

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

The [Troubleshooting](#) section contains information on the available reference surfaces.



*Figure 5: Highly diffuse samples are measured against a flat PTFE reference plate*



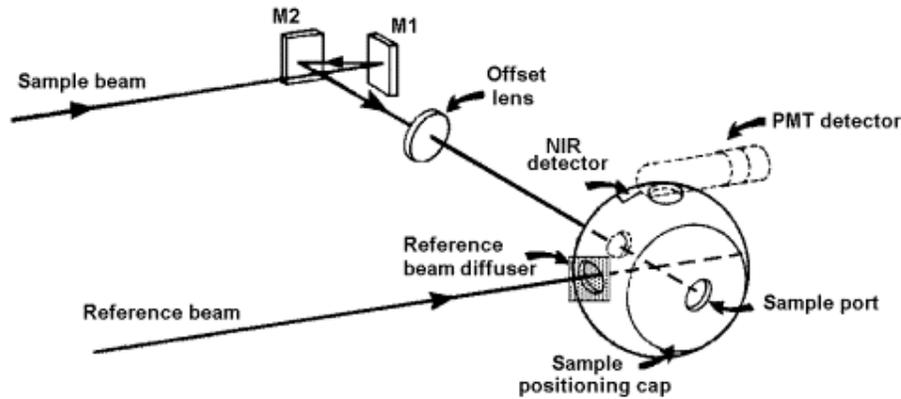
*Figure 6: The protruding PTFE reference plate more accurately emulates highly specular samples*

### **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.

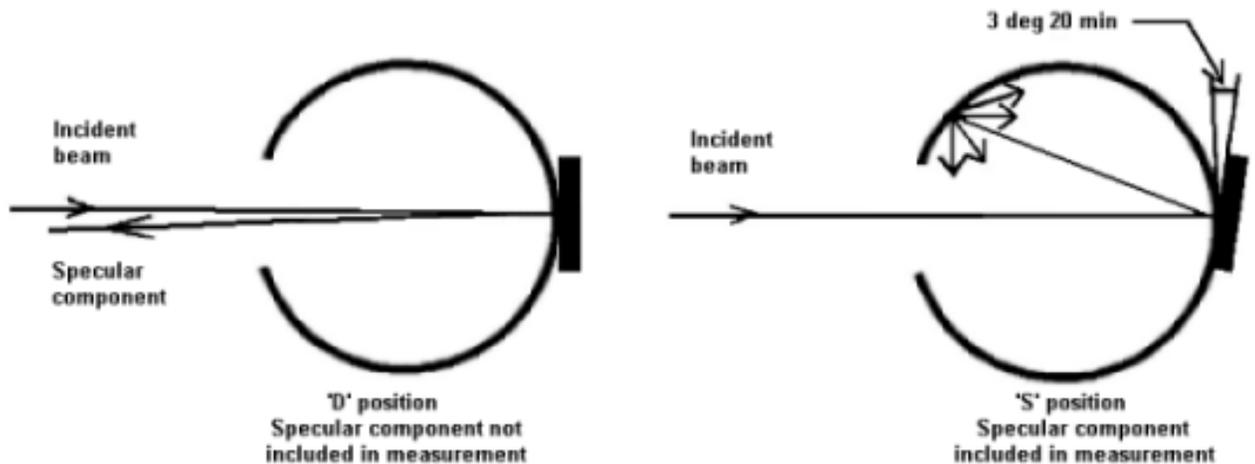
For a detailed discussion on the factors affecting the measurement of diffuse reflectance and transmittance with integrating spheres, refer to reference (2).

## Optical design



*Figure 7: The optical design of the DRA*

1. The sample beam hits mirror M1 and is then reflected to M2.
2. The beam travels through the lens and is focused into the transmission port, and onto the reflectance port.
3. The reflected beam is diffused throughout the sphere before being measured by the detector.
4. The reference beam enters the sphere directly through the reference port and is dispersed.
5. The removable end cap has two positions:
  - In the 'D' position, the angle of the beam from the normal to the sample plane is approximately 0 degrees. Any specular component of the reflection is reflected back through the transmission port and is deflected by the angled lens, preventing re-entry into the sphere.
  - With the cap in the 'S' position, the angle of incidence is  $3^{\circ} 20 \text{ min}$ . The specular component will hit the sphere wall and be diffused within the sphere. The total reflectance will then be measured.



**Figure 8:** Position 'D' measures diffuse reflectance only. Position 'S' measures total reflectance by including the specular component.

The sphere has five ports:

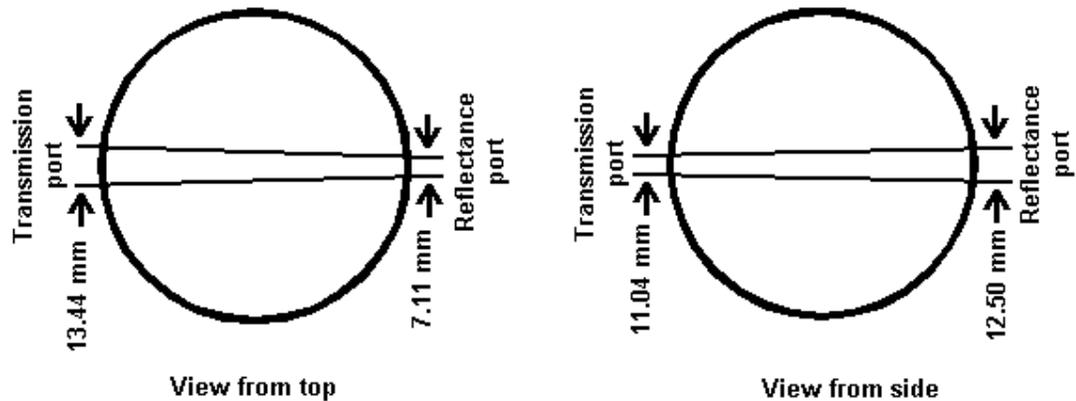
1. Sample beam transmission port (19 x 17 mm)
2. Sample measurement port (16 mm diameter)
3. Reference beam port (11 x 13 mm)
4. NIR detector port (11 mm diameter)
5. PMT port (oval, 30 mm long x 14 mm wide)

The PMT, PbS and InGaAs detectors are mounted on top of the sphere, which is alternately illuminated by sample and reference beams.

Beam image dimensions:

transmission port 11.04 mm (h) x 13.44 mm (w)

reflectance port 12.50 mm (h) x 7.11 mm (w)



**Figure 9:** The beam through the accessory

The reference disks supplied with the accessory are 6 mm deep and the powder is added to a density of 1 g/cm<sup>3</sup>. This is the optimum thickness and density of PTFE, to ensure maximum reflectivity(3) and therefore the best absolute accuracy.

## Specifications

		<b>DRA 900</b>	<b>DRA 1800</b>	<b>DRA 2500</b>
+ 85 volts DC (Cary 4000/5000/6000i)				
Wavelength range	<b>Cary 4000</b>	200 nm to 900 nm *	200 nm to 900 nm	200 nm to 900 nm
	<b>Cary 5000</b>	200 nm to 900 nm *	200 nm to 1800 nm *	200 nm to 2500 nm *
	<b>Cary 6000i</b>	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 <sup>#</sup> cooled InGaAs	TE <sup>#</sup> cooled PbS
Sphere diameter	110 mm			
Internal coating	Polytetrafluoroethylene (PTFE)			
Coating density	1 g/cm <sup>3</sup>			
Coating thickness	4 mm			
Sample plane normal deviation	0° (diffuse reflectance only) 3° 20 min (total reflection) (CIE recommendation: <10°)			
Port area/total surface area ratio	3% (CIE recommendation: <10%)			
reflectance port size	16 mm diameter			
Minimum sample size	Approx 8 mm (w) x 12 mm (h)			
Maximum sample size	100 mm (w) x 200 mm (h)			
Power input	Sample compartment DRA connector 15-pin D-range connector with two high voltage pins: -1000 volts DC,			
Size packed	180 mm x 390 mm x 270 mm (W x L x H)			
Size unpacked	140 mm x 340 mm x 200 mm (W x L x H)			
Weight packed	8 kg			
Weight unpacked	5 kg			

\* The blue colouring indicates the recommended configurations of instruments and DRAs. While other configurations will still work the performance will not be optimal.

<sup>#</sup>Thermoelectrically cooled

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.



**Figure 10.** The internal DRA. Note the clips on the left hand side of the accessory. These are for attaching metal screens during [reference beam attenuation](#).

**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 impaired.

**Unpacking**

Open the shipping case and the enclosed packages with care.

Included in the package are the following:

Item	Description	Part number
1 x Mirror	Alignment target	20 100625 00
1 x Ball driver	M4 3mm hexagonal ball driver for horizontal mirror alignment	72 100208 00
1 x 5.5 mm AF nut driver	For vertical mirror alignment	72 100243 00
1 x Polarizer mount	(already installed)	
1 x Large PTFE reference plate	1 ¾" diameter	04 101439 00
1 x Protruding PTFE reference plate		04 101988 00
1 x Alignment target	Translucent polyester film	24 180044 00

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

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.

The Standard Sample Holder kit includes the following:

Item	Description	Part number
1 x Sample clamp	Spring clip sample clamp	01 103359 00
1 x Paddle clamp	White plastic sample clamp	01 106122 00
1 x Small clamp	Black plastic sample clamp	08 180146 00
6 x Round poles	Permits mounting of 1" and 2" ledges, paddles and clamps in %T and %R modes	08 101699 00
2 x Poles	Round poles with one flat side (1 already installed)	15 180540 00

1 Sample mount ledge	Permits mounting of 1" and 2" samples in %T and %R modes	08 101697 00
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The Standard Sample Holder Kit will accommodate mounting of reflectance standards, specifically:

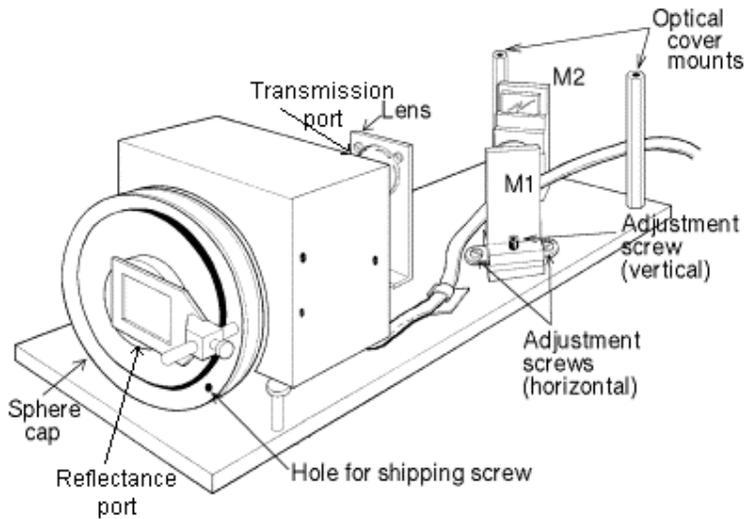
Item	Description
Protruding PTFE reference plate	
Large PTFE reference plate	1 3/4" diameter
2" Reflectance standards	Reflectance standards (Spectralon) 2", for use with Diffuse reflectance accessories
1" color standards	Calibrated diffuse color standards set for DRA. Set includes four 1" D diffuse color standards. Red, green, blue and yellow

[Click here](#) for details of what is included in each of the other sample mounting kits.

## Installation

You will need:

- A flat blade screw-driver (is included in the instrument tool kit)
  - A 3 mm hexagonal ball driver
  - The Extended Sample Compartment
1. Turn off the spectrophotometer and remove any cell holders or other accessories from the sample compartment. The PC can be left on.
  2. Close the sample compartment, turn the spectrophotometer on and allow it to initialize.
  3. Open the accessory case and remove the metal clamp and alignment target.
  4. Use the hex ball driver to remove the shipping hex screw from the the end cap of the sphere.
  5. Remove the accessory cable from the white cable clamp.
  6. Remove the cover over the mirrors by loosening the two screws on top of the cover and place to one side.



**Figure 11:** The Cary 4000, 5000 and 6000i Internal Diffuse Reflectance accessory with the optics cover removed. The large reference plate is clamped over the reflectance port at the centre of the sphere cap.

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

7. Position the accessory so that the two rear lock-down feet are situated at the back of the sample compartment and gently lower the accessory so that the front pin on the base plate fits into the post clamp on the sample compartment floor. Ensure that the base of the accessory is parallel to the sample compartment floor.
8. Slide the lever at the front of the sample compartment to the left, to lock the base plate into position.

**Caution**

Do **not** plug the DRA into the spectrophotometer when the latter is turned on, or electronic damage may occur.

9. Take the clamp post from under the foam in the wooden shipping case and fasten it in the hole in the accessory cap. Place the sample clamp on the clamp post.

**Alignment**

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



1. Click the Windows Start button, then Programs, then Cary WinUV, and then Align.
2. Click the *Cary* tab.
3. Set the following parameters:

Double beam:	Normal
Y mode:	%R
Ave time:	0.1
SBW:	2
Slit height:	reduced

4. Select the Zero order check box and click OK.

### **Caution**

The DRA should not be plugged in and operated with the instrument set to 0 nm. If it is, the detectors will be flooded with white light, which will decrease the sensitivity of the accessory and increase noise. If this should happen, place the DRA in a dark place for 24 hours to allow it to recover.

5. Remove the front cap from the accessory and observe the diffuser at the reference port. The image should be centered on the diffuser.  
If it is not:
  - 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.
6. Replace the sphere cap with the 'S' mark upwards.
7. Tape a card over the diffuser on the reference port to exclude light. Make sure you do not contaminate the surface of the diffuser.
8. Check the adjustment of the Beam on to mirror M2. If necessary adjust the horizontal alignment of the beam by loosening the screws on either side of mirror M1 and rotating it slightly.
9. Fasten the screws.

10. Adjust the vertical alignment of the beam on mirror M2 by adjusting the hexagonal screw at the front of mirror M1.
11. Check that the light is not clipping the lens, the transmission port to the sphere (at the back of the sphere), or the reflectance port on the sphere (at the front of the sphere).
12. If necessary, adjust M2 in the same way as M1.

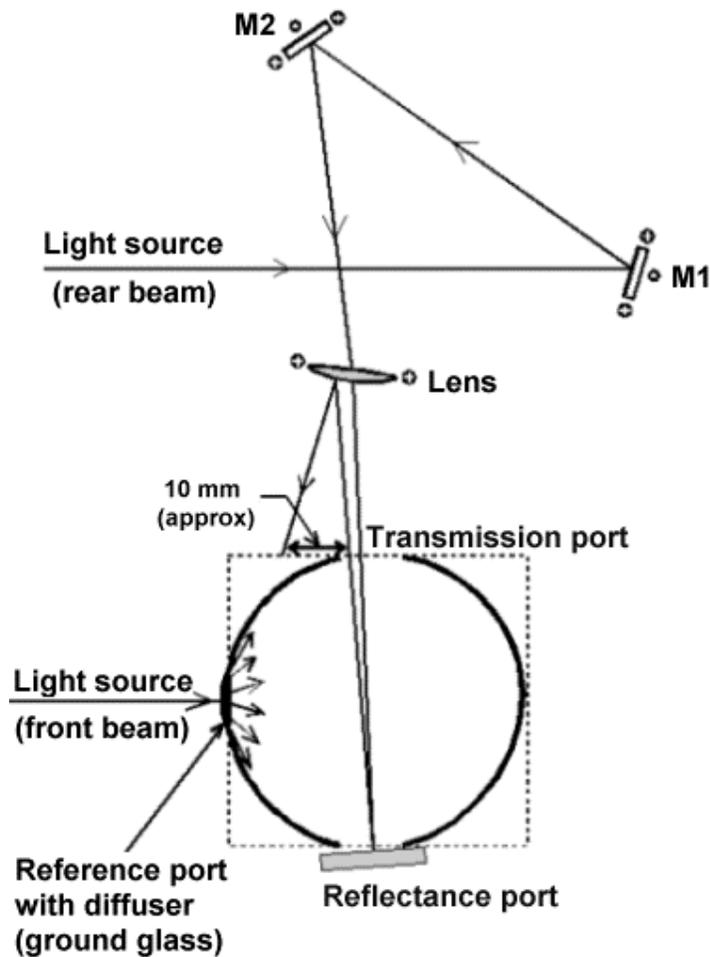


**Hot Tip**

You will have to remove the accessory from the sample compartment to adjust the vertical position of the beam with the hexagonal screw at the back of M2.

**Checking for the specular component**

1. Place the accessory cap in position with the 'D' mark upwards.
2. Clamp the mirror over the reflectance port. The reflective surface of the mirror should face the reflectance port.
3. The beam reflected from the lens should strike the back of the sphere housing approximately 10 mm from the edge of the transmission port.
4. Adjust the beam by loosening the screws on either side of the lens and then rotating the lens to move the beam position.



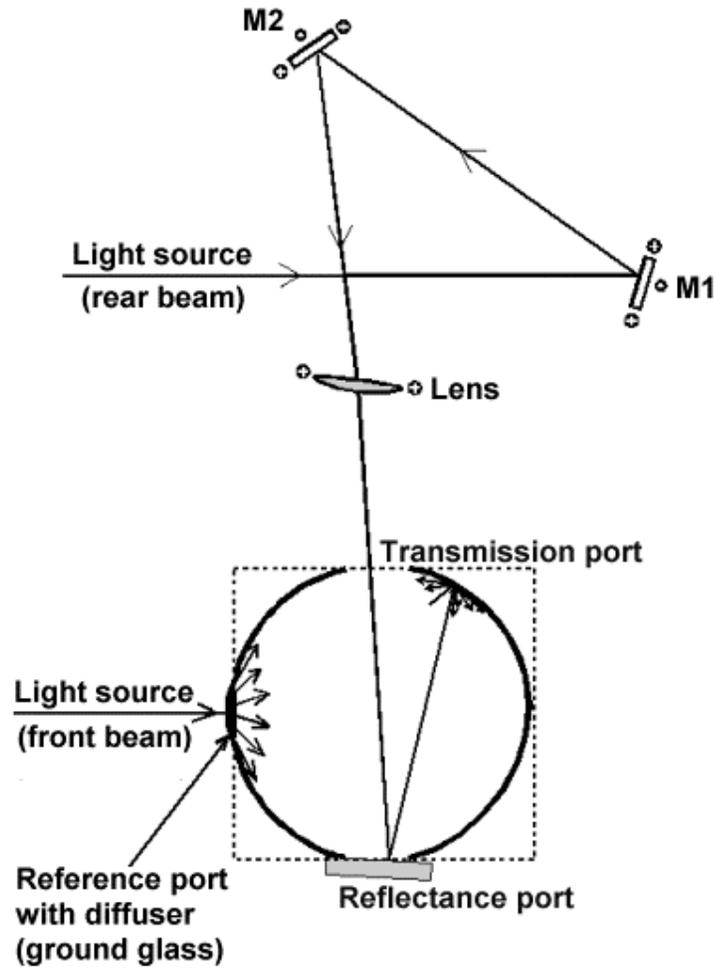
*Figure 12: Simplified beam diagram of the accessory with the sphere cap in the 'D' position. The specular component is reflected back out of the sphere onto the lens. The reflection from the lens should be about 10 mm from the transmission port.*

5. Re-tighten the screws and re-check the alignment, to see if any lens adjustment is necessary.
6. Position the sphere cap so that the 'S' mark is facing up. If any part of the reflected beam exits the transmission port, repeat the alignment and specular component checks.



#### **Hot Tip**

Place the alignment target at the edge of the transmission port (without blocking the beam from the lens) and rock the mirror over the reflectance port to identify the reflected beam. When the mirror is placed over the reflectance port, no part of the beam should be observed exiting the transmission port.

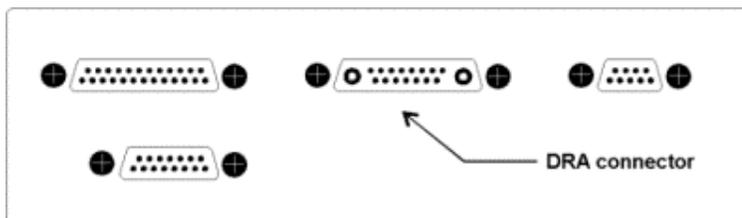


**Figure 13:** Simplified beam diagram of the accessory with the sphere cap in the 'S' position

7. Carefully remove the mask from the diffuser (see [step 7](#)). Turn off the instrument and remove the DRA from the sample compartment.
8. Replace the optics cover on the DRA.
9. With the spectrophotometer turned **off**, place the DRA in the sample compartment and plug it into the corresponding socket at the top of the sample compartment.

**Caution**

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



**Figure 14: DRA socket**

10. Place the PTFE reference disc over the reflectance port.
11. Install the Extended Sample Compartment according to its instruction manual.
12. Close the sample compartment, turn on the instrument and wait for correct initialization to be completed.



**Note**

Improper alignment of the DRA may prevent the Cary from finding the 656.1 nm wavelength calibration line.

**Error checks**



**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.

<b>Cary tab</b>	
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
<b>Options tab</b>	
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
<b>Baseline tab</b>	

Correction	None
<b>Autostore tab</b>	
File Storage: Storage	OFF

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

### Zero %R error check

1. With the transmission ports uncovered, place the large reference plate over the reflectance port.
2. From the main 'Scan' dialog 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 Extended Sample Compartment. 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.

### 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 0.5%R. If it is not, use the *Calibration* menu item in the *Validate* application. Refer to the [Calibration](#) section for further details.

### Specular component exclusion check

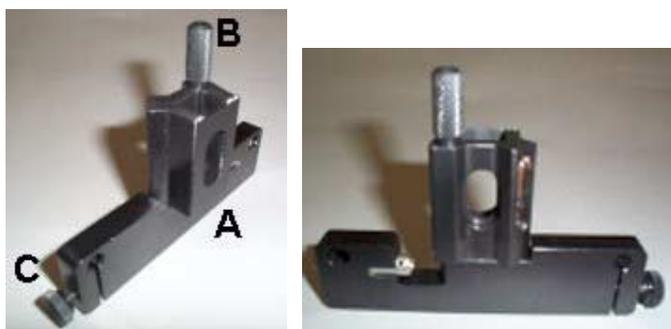
1. Position the sphere cap so the 'D' is facing up.
2. Place the large reference plate over the reflectance port.
3. Click the *Goto* button to open the *Goto wavelength* dialog box. Enter '500' in the Wavelength field and click OK. The instrument will then drive to 500 nm. Close the sample compartment lid.
4. Click the *Zero* button and wait for the instrument to zero.
5. Place the supplied 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 exiting the sphere at the transmission port, 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. Rotate the sphere cap 180° so that the 'S' mark is facing up. When the sample compartment is closed, the reading should be approximately 90%.

## Installing attachments

Attachments can be installed into the DRA to increase its flexibility and versatility. The following are optional attachments:

- Liquid sample holder
- Small sample holder;
- Powder cell
- Small sample mount
- Polarizer/Depolarizer
- Double Aperture

### Installing the liquid sample holder



**Figure 17.** The liquid cell holder. "A" the flat side that must sit against the DRA, "B" the raiser screw and "C" the screw for securing the attachment to the posts on the DRA.

1. Screw the posts into position at the transmission port of the sphere.
2. Slide the liquid sample holder onto the posts. The flat side must sit against the DRA.
3. Check the alignment of the cell holder using the target supplied. Ensure that the beam is not clipping the edge of the sample holder and is centered on the sample.



#### Hot Tip

To assist in preventing the beam from being clipped by the edge of any of the attachments a SBW of 2 nm is recommended.

4. If necessary [recheck the alignment of the DRA.](#)
5. Load a cuvette into the sample holder so that transmission measurements of a liquid sample can be taken.

### Using the liquid sample holder to perform transmission measurements

You must first install the [liquid sample holder](#).

1. Place the protruding reference plate in position in the reflectance port, and position the cuvette at the transmission port.
2. Set the instrument as detailed in the [transmission measurements](#) section.



#### 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.

3. Fill the cuvette with solvent. If necessary, run a baseline as follows:
4. Click the *Start* button, then *Programs*, then *Cary WinUV* then click on *Scan*.
5. Click the *Baseline* button. Follow the on-screen prompts to perform a baseline scan.

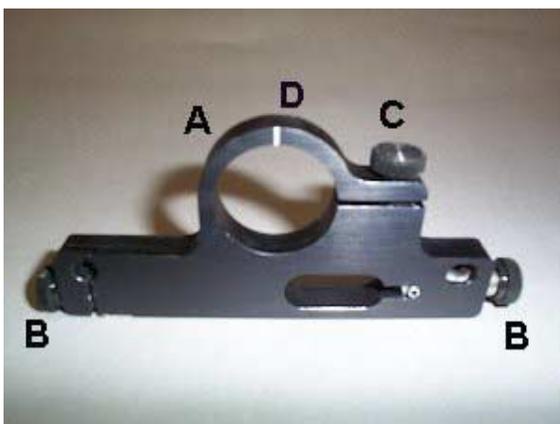


#### Hot Tip

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

6. 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.
7. Fill the cuvette with the sample and place cuvette in the holder in the same orientation that was used to collect the baseline.
8. Click the *Start* button to perform the scan. 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.

### Installing the small sample holder



**Figure 18.** The small sample holder. A = where either the small sample mount or powder cell are mounted. B = screws for securing the small sample holder to the DRA. C = screw for holding the small sample mount or powder cell into position. D = groove for the correct positioning of the powder cell and small spot mount.

The small sample holder is used to measure reflectance of small samples. The powder cell can be mounted in the small sample holder to enable the reflectance of powder samples to be measured. The small sample holder is also used to hold the small sample mount when measuring the reflectance of very small samples.

1. [Install the DRA](#) into the instrument.
2. Screw the posts into position at either the reflectance or transmission port.
3. Slide the small sample holder onto the posts.
4. Secure the sample holder onto with the screws at either side of the attachment.
5. You can now install either the [small sample mount](#) or the [powder cell](#) into the small sample holder.



#### Note

If you are going to use the powder cell, the small sample holder must be installed at the reflectance port.

6. You must now [check the alignment of the attachment](#).

### Installing the powder cell



*Figure 19. The powder cell. A = lid, B = sample cup, C = funnel.*

The powder cell enables you to determine the reflectance of a powder sample.

1. [Install the DRA](#) into the instrument.
2. Install the [Small Sample Holder](#) onto the DRA at the reflectance port.
3. Fit the funnel onto the sample cup.
4. Load your sample into the sample cup.
5. Remove the funnel.
6. Screw on the lid.
7. Insert the powder cell into the small sample holder.



**Figure 20.** Insert the powder cell into the small sample holder.

8. Tighten the screw on the small sample holder to secure the powder cell.



**Figure 21.** The powder cell fitted into the small sample holder, note that the textured edge of the powder cell sits flush with the edge of the small sample holder. The screw to the right of the powder cell is used to secure the powder cell.

8. You must now [check the alignment of the attachment](#).
9. You can use the powder cell to perform [reflectance measurements](#). However follow this procedure to collect a baseline.
  - a) Place a PTFE standard into the powder cell.
  - b) Click the Zero/Baseline button in the Scan application and collect a 100% T baseline.
  - c) Remove the powder cell (containing the PTFE reference), leaving the reflectance port open.
  - d) Collect a 0% T baseline. When the 0% baseline is completed the ordinate status display in the top left corner of the Scan application will show "Zero baseline" in red text.

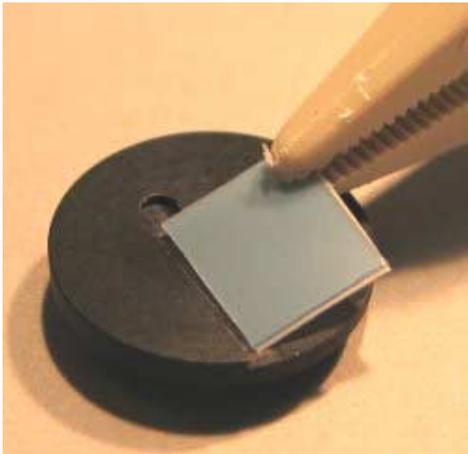
## Installing the small sample mount

The small sample mount enables you to take reflectance readings of very small solid samples, for example gemstones. The kit features five different mounts and three different apertures. The sizes of the apertures are 1mm, 3mm and 5mm. A PTFE reflectance standard is also included.



**Figure 21.** The Small Sample mount. "A" sample mounts, "B" apertures, "C" sample cup and "D" PTFE standard.

1. [Install the DRA](#) into the instrument.
2. Install the [Small Sample Holder](#) onto the DRA.
3. Place the sample onto the sample mount, you may need to use tweezers to correctly position the sample.



**Figure 22.** Placing the sample onto the mask.

4. Place the mount (the size mount used is determined by the size of your sample) onto the sample cup.



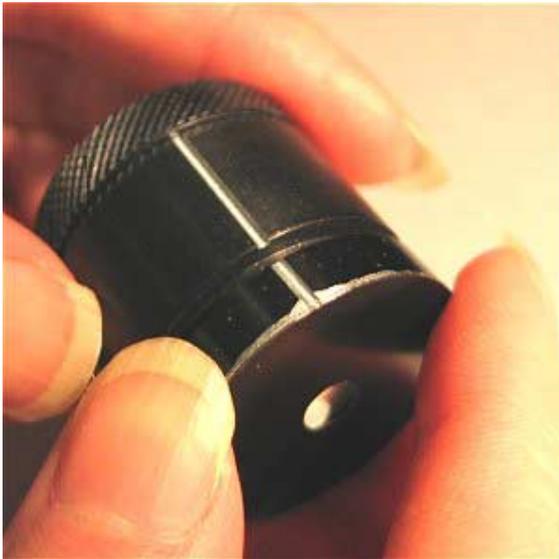
**Figure 23.** The mask and sample are placed on the sample cup.



**Note**

The sample mounts have V-shaped cutouts that enable small samples to assist in the mounting of samples.

5. Fit the aperture (the aperture size used is determined by the size of your sample) onto the sample cup, lining up the marking on the lid and the sample cup.



**Figure 24.** Align the markings on the sample cup and the lid before fitting them together.

6. The small sample mount is loaded into the small sample holder using the grooves on each to correctly position the small sample mount into the small sample holder.



**Figure 24.** The small sample mount fits into the small sample holder. Note that in this picture the small sample holder and small sample mount are in the correct position for performing transmission measurements.

7. Tighten the screw on the small sample holder to secure the small sample mount.



**Note**

Ensure that the small sample mount is sitting forward in the small sample holder, so that the thumb screw does not prevent the lid of the small sample mount from sitting up against the DRA. The white lines on the small sample holder and the powder cell should be lined up with each other.

8. You must now [check the alignment of the attachment](#).
9. You can use the small sample mount to perform [reflectance](#) or [transmission](#) measurements. But use the following procedure to collect the baseline. The following procedure applies to collecting a baseline for reflectance measurements. If you are going to be performing a transmission measurement, follow the same procedure but position the sample cup at the transmission port.
  - a) Place the PTFE small spot standard into the sample cup. (%R measurements only).



**Figure 25.** The PTFE standard fits into the sample cup.

- b) Fit the aperture onto the sample cup.
- c) Place the sample cup over the reflectance or transmission port.



**Note**

It is recommended that you use the same aperture size to run the baseline as the one you are going to use for the sample.

- d) Click the Zero/Baseline button in the Scan application to collect the 100% T baseline.
- e) If you are performing a reflectance measurement, remove the PTFE standard from the sample cup and replace the sample cup at the reflectance port with the aperture attached. If you are performing a transmission measurement, block the transmission port with a blocking plate.
- f) Click the Zero/Baseline button to collect the 0% T baseline.
- g) Once the baseline is complete the ordinate status reading will display 'Zero Baseline' in red text.

**Installing the polarizer**



**Figure 26.** The polarizer installed on the polarizer mount.

You can install the Polarizer/Depolarizer into the DRA to control the plane polarization of the light beam in a spectrophotometer. For example: if the sample being measured is sensitive to plane polarized light e.g. liquid crystals, the transmission of the sample will change as the plane of polarization changes. It is thus important to control the plane of polarization of the incident beam.

To install the polarizer/depolarizer:

1. [Install the DRA](#) into the instrument
2. Remove the optics cover.
3. Slide the Polarizer/Depolarizer onto the polarizer mount. The dial should face away from the optical area.
4. Tighten the screw to hold the polarizer in place.
5. If necessary [re-align the DRA](#).

You must now [check the alignment of the attachment](#).

### **Installing the double aperture attachment**

This attachment is used in conjunction with the Validate application to check that the detector in the DRA is performing correctly. The advantage of using the Double Aperture attachment to determine photometric accuracy is that there is no limitation on temperature, wavelength or SBW. Refer to the [online Help](#) for instructions on how to use a double aperture attachment. Click [here](#) for the theory behind the use of a [double aperture accessory](#).



#### **Note**

The default tolerance values in the Validate application are applicable to the instrument only. An internal or external DRA accessory should not be expected to perform to the same level of photometric accuracy.



**Figure 27.** The Double Aperture attachment. A indicates the locating pin at the bottom of the attachment.

1. Turn on the instrument and allow it to warm up for at least two hours.
2. Remove the optics cover.
3. Mount the double aperture base onto the polarizer mount.



**Figure 28.** Install the double aperture base onto the polarizer mount.

4. [Install the DRA](#) into the instrument.
5. You must now align the attachment as follows.
6. Place the mask onto the double aperture base so that both apertures are open.



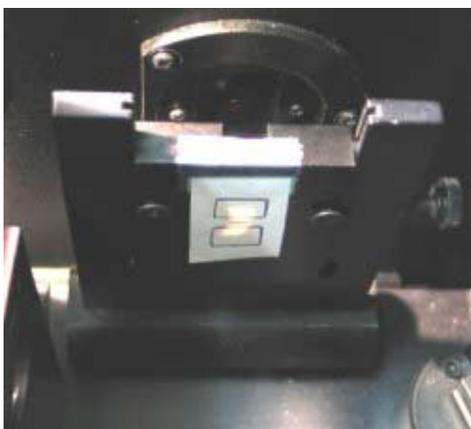
**Figure 29.** The mask fits onto the double aperture base. This figure shows only one aperture is open.

7. Click the Windows Start button, then Programs, then Cary WinUV, and then Align.
8. Set the SBW to 2.00.
9. Close the sample compartment lid.
10. Tick the Zero Order check box, click the Apply button to drive the instrument to zero.
11. Open the sample compartment lid and adjust the height adjustment screw on the side of the polarizer mount until the image is centred over the apertures.



**Hot tip**

Holding a piece of white paper in front of the apertures will make the beam image easier to see. You can use the aperture mask to draw the apertures on the paper before mounting the mask.



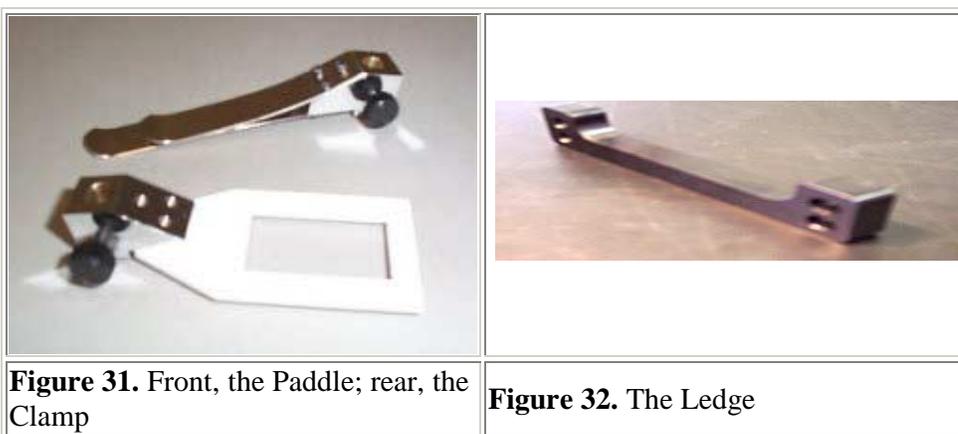
**Figure 30.** The image should be centred over the apertures.

12. Close the sample compartment lid and go to the wavelength that you are going to use for the measurement. Take note of the % transmission reading.
13. Open the sample compartment lid and rotate the aperture mask so that only the lower aperture is open.

14. Adjust the height adjustment screw on the side of the polarizer mount until the transmission reading is half (+/- 1%) of the reading noted in step 12.
15. Open the sample compartment lid and rotate the aperture mask so that only the upper aperture is open. Repeat the measurement until the reading for the upper aperture is half (+/- 1%) of the reading when both apertures are open.
16. Use the Validate application to check the photometric accuracy of the DRA with the double aperture attachment. Refer to the [Validate](#) section of the help.

## Operation

Samples can be mounted at the transmission or reflectance ports. At the reference port, attenuators may be mounted. Paddles, clamps and a sample ledge are provided you to assist with mounting your samples.



The Varian internal DRA provides different mounting options:

- standard sample holder kit;
- liquid sample holder;
- small sample holder;
- powder cell;
- small sample mount;
- polarizer;
- double aperture attachment

The following table illustrates which type of readings can be taken with which attachments.

Attachment	%T	%R
Standard	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Liquid sample holder	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Small sample mount	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Powder cell	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>

### Caution

When performing measurements and calibrations with the DRA some steps require

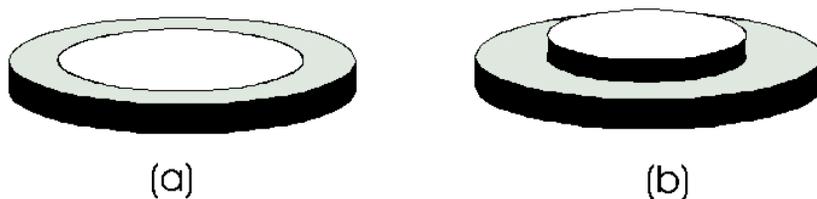
that the instrument to be turned on and off at various times. It is important to take care that you follow these steps as failure to do so can cause damage to your DRA.

### Reflectance measurements (%R)

The sample should be homogeneous, substantially flat, and large enough to completely cover the image. Finely powdered samples may be studied using a [powder cell](#). There is also a mounting option available for [small samples](#).

When measuring diffuse only reflectance, position the sphere cap with the 'D' mark up. Position the 'S' mark up to measure the specular+diffuse (total) reflectance.

When performing measurements on glossy materials (high specular component), use the protruding reference plate. The reference surface protrudes into the sphere and behaves more nearly like a perfect diffuser. As discussed in the [Differences between the standard and sample](#) section, you should use reference materials with similar reflectivity and properties to the sample.



**Figure 15:** Reference disks supplied with the DRA. The large reference plate (a), and the protruding reference plate (b).



#### 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. After you have carried out the Alignment procedures, turn off the instrument.
2. Plug the DRA into the instrument using the communications plug, it fits into the green plug at the back of the instrument's sample compartment.
3. Cover the reflectance port with a PTFE reference disc.

#### Caution

Before turning on your instrument you **must** cover the reflectance port with a PTFE reference disc to prevent damage to your DRA.

4. Install the [extended sample compartment](#).
5. Close the sample compartment lid.
6. Turn on the instrument, allow the instrument to initialize.
7. Click the Windows *Start* button, then Programs, Cary WinUV and then Scan.

8. Click the Setup button.
9. From the Setup menu, set the following parameters:

<b>Cary tab</b>	
X mode: Mode	Nanometers
X mode: Start	700
X mode: Stop	400
Y mode: Mode	% R
Y mode: Y min	-5.00
Y mode: Y max	110.00
Scan controls: Ave time (s)	0.100
Scan controls: Data interval (nm)	1.000
Scan controls: Scan rate (nm/min)	600.00
<b>Options tab</b>	
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
<b>Baseline tab</b>	
Correction	Zero/baseline correction
<b>Autostore tab</b>	
File Storage: Storage	Storage on (Prompt at start)

10. Select the 'Baseline' button from 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 Extended sample compartment.

11. 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.
12. Clamp the sample over the reflectance port. The sample should be homogeneous, substantially flat, and large enough to completely cover the image.



#### **Hot Tip**

Use the sample mount ledge for added control over the positioning of your sample. To use the ledge (see figure 16 ) slide it onto the posts fitted at either the reflectance or transmission ports.



**Figure 16.** *The sample mount ledge.*

13. 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.

This procedure will measure the % reflectance relative to the PTFE reference disk.



**Note**

To determine the absolute diffuse reflectance of a sample, you need to purchase an NBS standard and calibrate your PTFE disks. The NBS standards SRM 2021 (a black porcelain enamel tile) or SRM 2019c (a white ceramic tile) are suitable for this purpose.

**To determine position and size of the image**

To determine the position and size of the image so that samples can be mounted correctly, follow this procedure:



**Note**

Initialize the Cary instrument before placing the DRA into the sample compartment. After initialization, place the DRA into the compartment. Do **not** turn the accessory on (i.e., do not plug the DRA into the instrument).

1. Click the *Goto* button in the *Scan* application.
2. Select the *Zero order* check box and click OK. The spectrophotometer will drive to 0 nm and select the Vis lamp.
3. Place a piece of white paper in front of the sample clamp. The image size should now be visible.

**NIR reflectance measurements (Cary 5000 and 6000i)**



**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.

[Follow steps 1 to 6](#) of the transmission measurements procedure then:

1. Click the Windows Start button, then Programs then Cary WinUV and then Scan.
2. Click the Setup button.
3. Set the following parameters:

<b>Cary tab</b>	
X mode: Mode	Nanometers
X mode: Start	1800
X mode: Stop	300
Y mode: Mode	% R
Y mode: Y min	-5.00
Y mode: Y max	110.00
Show status display	ON
<b>Options tab</b>	
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
<b>Independent tab</b>	
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
<b>Baseline tab</b>	
Correction	Zero correction
<b>Autostore tab</b>	
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.

- Click the *Goto* button to open the 'Goto wavelength' dialog box. 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 box.

- 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 to click *OK* in order to note the change to the SBW reading. Then click 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.
- Click 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 Extended sample compartment.

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

**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.

[Follow steps 1 to 6](#) of the transmission measurements procedure then:

- From the Setup menu, set the following parameters:

<b>Cary tab</b>	
X mode: Mode	Nanometers
X mode: Start	800

X mode: Stop	300
Y mode: Mode	% R
Y mode: Y min	-5.00
Y mode: Y max	110.00
Scan controls: Ave time (s)	0.100
Scan controls: Data interval (nm)	1.000
Scan controls: Scan rate (nm/min)	600.00
<b>Options tab</b>	
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
<b>Baseline tab</b>	
Correction	Zero baseline/correction
<b>Autostore tab</b>	
File Storage: Storage	Storage on (prompt at start)

2. Position the sphere cap with the 'S' mark up.
3. Click 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 performing specular zero (%T) collect, remove the reference PTFE disc and allow the light to be trapped by the external sample compartment.

4. 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.
5. Clamp the sample over the reflectance port. The sample should be homogeneous, substantially flat, and large enough to completely cover the image.
6. Click the *Start* button to perform the scan of the total (specular and diffuse) reflectance of the sample.
7. Turn the sphere cap so the 'D' mark is up, and click the *Start* button to collect the diffuse-only data.
8. From the main Scan window, click the Calculator icon  to open the open the Maths dialog box.
9. 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.
10. From the *Maths* dialog box choose *Selected Trace* and then the '-' (minus) sign.
11. 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.

12. From the *Maths* dialog box choose *Selected Trace* and then the '=' (equals) sign.
13. The results of this specular data equation will be displayed in a new graph.

## Transmission measurements (%T)



### 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. Place the accessory cap in the 'S' position, and place the protruding reference plate in position over the reflectance port. Use the metal clamp to hold the reference cap in position.
2. Set the following parameters:

<b>Cary tab</b>	
X mode: Mode	Nanometers
X mode: Start	700
X mode: Stop	400
Y mode: Mode	%T
Y mode: Y min	-5.00
Y mode: Y max	110.00
Scan controls: Ave time (s)	0.100
Scan controls: Data interval (nm)	1.000
Scan controls: Scan rate (nm/min)	600.00
<b>Options tab</b>	
SBW/Energy: Fixed SBW	ON
SBW/Energy: SBW (nm)	2.00 (See note below)
SBW/Energy: Beam mode	Double
SBW/Energy: Slit height	Reduced
Source: Lamps	UV-Vis
Source: Source changeover (nm)	350.0
<b>Baseline tab</b>	
Correction	None
<b>Autostore tab</b>	
File Storage: Storage	Storage on (prompt at start)



### Note

If you are performing a transmission measurement using the small spot mount, [click here](#), for the procedure for collecting a baseline.



### Note

When operating in the visible range, the maximum SBW value you should use for a standard 10 mm square cuvette is 1.5 nm.

3. Click 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 performing the 0%T baseline collect, block the sample beam in the transmission position with a blanking plate (a large solid object).

4. 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.
5. Use the plastic clamp to hold the sample over the sphere transmission port.

**Hot Tip**

Use the sample mount ledge for added control over the positioning of your sample. To use the ledge (see figure 16 ) slide it onto the posts fitted at either the reflectance or transmission ports.



**Figure 16.** The sample mount ledge.

6. Click the *Start* button to perform the scan. 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.

## 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 internal DRA has been connected to the instrument and will restore any previous calibrations stored for the internal 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 0.0003 %T. Calibrate with the instrument fully warmed up. It may be desirable to calibrate Calibrate PGA prior to this.

#### **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 0.003 %T calibration may be required.

#### **Calibrate UV/VIS wavelength range**

Execute this if wavelength accuracy fails using certified standards.

This option causes the Cary to use the deuterium lamp to calibrate the instrument at 656.1, 486.0 and 0.00 nm.



#### **Note**

When performing a wavelength calibration you must firstly remove the DRA, perform the calibration, then return the DRA to the instrument. Refer to the [Wavelength Accuracy](#) help in the Validate application.

#### **Calibrate NIR wavelength range**

Execute this if wavelength accuracy fails using certified standards.

This option causes the Cary system to use the deuterium lamp to calibrate the instrument at 2624.4, 1312.2 and 0.00 nm.



**Note**

When performing a wavelength calibration you must firstly remove the DRA, perform the calibration, then return the DRA to the instrument. Refer to the [Wavelength Accuracy](#) help in the Validate application.

## 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:

<b>Cary tab</b>	
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
<b>Options tab</b>	
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
<b>Independent tab</b>	
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

<b>Baseline tab</b>	
Correction	Zero x std ref correction
<b>Autostore tab</b>	
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 Extended sample compartment.

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 and cleaning

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.

The mirrors are over-coated with quartz so the aluminium layer is protected. To clean the mirrors either place them in an ultrasonic bath or gently flush ethanol across the surface of the mirror, using a cotton wool bud to gently wipe marks from the surface of the mirror.



### Hot Tip

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

Never clean the mirrors with abrasive cleaners or tissue paper.

You may gently flush the accessory with pure nitrogen to remove water by using the N<sub>2</sub> connector lines at the back of the instrument.

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

## Standards

Reference surfaces for reflectance and transmittance 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 transmittance 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 transmittance standards of neutral glass filters with transmittance values of 0.92 to 0.001.

Contact:

Division of Electrical Science  
 National Physical Laboratory  
 Teddington, Middlesex  
 TW11 0LW  
 UK

## Troubleshooting

**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.



Operators and other unauthorized personnel must never remove the 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 value	<p>Ensure that you are referring to the hemispherical reflectance values of the SRM, not the directional and hemispherical values.</p> <p>Ensure that your PTFE disks are clean and in good condition.</p> <p>If you are calibrating the PTFE disk, you need only apply the necessary correction to your measured values.</p>
The signal-to-noise ratio is low	<p>This can be caused by degraded sphere coating, poor detector performance, the lamps requiring replacement, unclean mirrors or lens or bad alignment.</p> <p>Check the reflectance port image, and re-align the accessory if necessary.</p> <p>Check that the lens and mirrors are clean, and carry out a <a href="#">Zero %R error check</a>.</p>
Failure to initialize	<p>Ensure that that PTFE reference disc is in the reflectance port.</p>

Cannot select the required wavelength	Check that the wavelength ranges of instrument and DRA are compatible. Refer to the <a href="#">Specifications</a> table.
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## Spare parts

The following spare parts are available from Varian for the Diffuse Reflectance accessory. Only Varian-supplied parts should be used, unless specified otherwise:

Item	Description	Part number
1 x Mirror	Alignment target	2010062590
1 x Ball driver	M4 3mm hexagonal ball driver for horizontal mirror alignment	7210020800
1 x 5.5 mm AF nut driver	For vertical mirror alignment	7210024300
1 x Polarizer mount	(already installed)	
1 x Large PTFE reference plate	1 3/4" diameter	0410143990
1 x Protruding PTFE reference plate		0410198890
1 x Alignment target	Translucent mylar film	2418004400
1 x Sample clamp	Spring clip sample clamp	0110335990
1 x Paddle clamp	White plastic sample clamp	0110612200
1 x Small clamp	Black plastic sample clamp	0818014600
2" Reflectance standards	Reflectance standards (Spectralon) 2", for use with Diffuse reflectance accessories	9910080900, 9910081000, 9910081100
1" Color standards	Calibrated diffuse color standards set for DRA. Set includes four 1" D diffuse color standards. Red, green, blue and yellow	9910084300

## Alignment targets

l (nm)	R	l (nm)	R	l (nm)	R
250	0.973	700	0.994	1900	0.985
260	0.976	750	0.994	1950	0.984
270	0.978	800	0.994	2000	0.981
275	0.979	850	0.994	2010	0.979
280	0.980	900	0.994	2020	0.978
290	0.982	950	0.994	2030	0.976
300	0.984	1000	0.994	2040	0.975
310	0.985	1050	0.994	2050	0.973
320	0.987	1100	0.994	2060	0.972
325	0.988	1150	0.994	2070	0.971
330	0.988	1200	0.993	2080	0.970
340	0.989	1250	0.993	2090	0.969
350	0.990	1300	0.992	2100	0.968
360	0.990	1350	0.991	2110	0.967
370	0.991	1400	0.991	2120	0.966

375	0.991	1450	0.992	2130	0.964	↕
380	0.991	1500	0.992	2140	0.964	↕
390	0.992	1550	0.992	2150	0.965	↕
400	0.993	1600	0.992	2160	0.967	↕
450	0.993	1650	0.991	2170	0.970	↕
500	0.994	1700	0.990	2180	0.973	↕
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

### Liquid Cell %T Holder

Only standard 10 mm path length cells can be used.

Item	Description
1 x Cuvette Holder	Holds standard 10 mm quartz cuvettes in %T port of DRA. Mounts on standard poles.

### Powder Cell

Powder mounting – a powder mount is required to enable powder samples to be examined. The following are included:

Item	Description
1 x Fixing Plate	Holder for aperture can.
1 x Powder can	22 mm diameter window. Volume - maximum 1 cm <sup>3</sup> sample.
1 x funnel	For loading samples into the sample can/cup.
1 x reference	A 22 mm diameter window for collecting baseline reference scans.

### Small sample mount

Powder mounting – a powder mount is required to enable powder samples to be examined. The following are included:

Item	Description
4 x Sample mounts	4 mounts of different size to accommodate different sized samples.
3 x Apertures	3 apertures of different sizes.
1 x Sample cup	1 cup for holding the sample mount.

## 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)