Extended performance infrared directional reflectometer for the measurement of total, diffuse and specular reflectance

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ABSTRACT

This paper presents a description of a fully automated, computer-controlled Hemispherical Directional Reflectometer (HDR). It fills the need in many fields of research and development for a device with HDR measurement capabilities which is state-of-the-art in wavelength coverage to 25.0 µm and higher, angular polarization resolved coverage 20 to 80°, partition of reflected radiation into specular and scattered components, and scattered transmittance.

This performance is made possible using an 18" major axis electroformed gold-plated specular hemiellipsoid with a 1.8" foci separation. The radiance throughput to the FTIR of this design exceeds by a factor of more than 200 that of the usual diffuse gold integrating spheres.

Derived data, based on reflectance and using provided software, includes the IR component of solar absorptance, the index of refraction *n* and *k* for dielectrics and conductors for Fresnel materials, and both directional and hemispherical emittance.

1.0 INTRODUCTION

There are numerous and expanding requirements for Directional Reflectance (DR) data in many fields including defense, space exploration, observables control, tailored coatings design, quality control in manufacturing, etc. Parallel with the data requirements, a variety of reflectometers have been marketed which provides directional reflectance as a function of wavelength. These reflectometers, which vary widely in basic design, vary even more widely in performance parameters including accuracy, adequate wavelength coverage, elevated temperature measurements, polarized data, and measurement beam high angles. They all share one basic common feature - manual operation.

This paper describes a directional reflectometer which is computer-controlled with all time consuming operations automated and which provides extended capability including all the performance features (far infrared coverage, etc.) as noted in the above paragraph, and provides the measured data in graphical and tabular format in a matter of minutes.

2.0 DIRECTIONAL REFLECTANCE DEFINITIONS

Reflectance of infrared radiation from surfaces is widely measured for both scientific, industrial, and defense applications. These measurements fall into two general categories: Directional and Bidirectional Reflectance (DR & BDR), see Figures 2-1(a) and 2-1 (b). They are related in that the integration of bidirectional reflectance over a hemisphere is equal to the directional reflectance.

Applications of the data to problems tends to divide the practitioners of reflectance measurements into groups. As a result identical or very similar measurements have acquired different data formats and different common names. Furthermore, the measurement instrumentation is sometimes rather different having been modified to fit a specialized requirement. An example is the Total Integrated Scatter (TIS) instrumentation which is configured to measure "unwanted" (*i.e.,* less is better) scattering with high precision. To this end, the specular beam is not recorded.

Directional Reflectance by definition <u>does</u> include the specular reflectance.

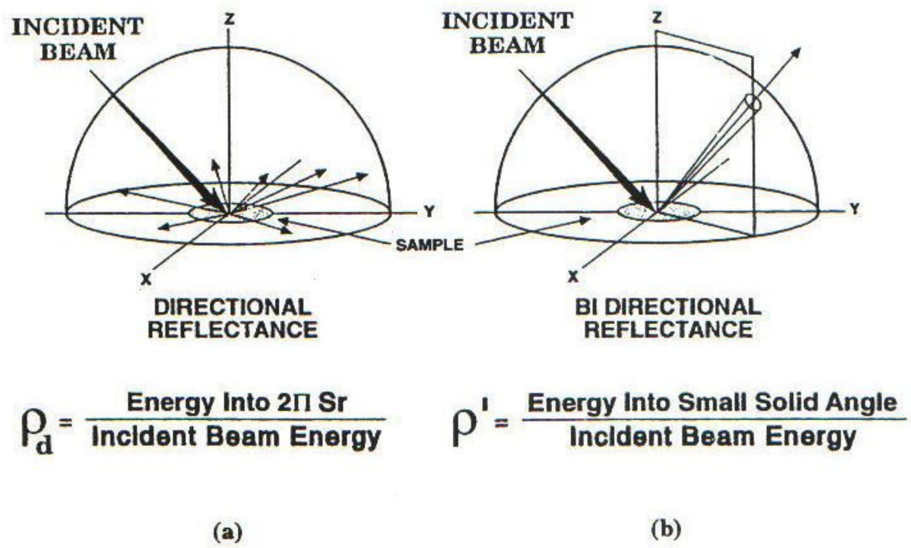


Figure 2-1. Directional and bidirectional reflectance angles definition.

For many applications, the directional reflectance can be measured with and without the specular lobe as is the case with the instrument described in this paper. This measurement allows determination of the total reflected radiation, the scattered portion, and the specular lobe portion. For applications in which the data is used to calculate the "signature" (*i.e.*, observed spectral radiance) of an object, it is particularly useful in determining whether scattered radiation is sufficiently large to require a BDR measurement for determining the solar reflectance component.

<u>3.0 TYPES OF EXPERIMENTAL APPARATUS</u>

The current commonly used reflectometers which measure IR directional reflectance fall into two general categories:

1. Integrating spheres, usually diffuse gold; and
2. Specular mirror devices.

The latter includes two parabola mirror devices, hemispheres and hemiellipsoids. These devices operate in the infrared region, measure directional reflectance as a function of wavelength, and generally use grating or FTIR monochrometers. These devices may be operated in either of two modes as schematically illustrated in Figures 3-1 (a) and 3-1 (b).

In one case (Figure 3-1 (a)), the sample is illuminated with a collimated beam and the reflected radiation into the hemisphere over the sample is collected and measured. The measurement is called directional hemispherical reflectance or simply the direct method. In the reciprocal method (Figure 3-1 (b)), the sample is uniformly illuminated over a hemisphere and a collimated beam from the sample is detected. The measurements have been shown to be equivalent.[1] In theory, but not necessarily in practice, one instrument could be used to make both direct and reciprocal measurements by simply interchange of the source and the detector.

Angular definitions are the same for both methods, as shown in Figure 3-2, whereby the "directional beam" points in one direction or the other, depending on whether the mode is direct or reciprocal.

For both methods, when using spheres, hemiellipsoids, and two parabolas for experimentally measuring directional reflectance, they suffer one common and unavoidable source of error, "the incomplete hemisphere error".

Illumination or detection over the full 2π Sr hemisphere is not physically possible since introducing the directional source beam (direct mode) or detecting the directionally reflected beam (reciprocal mode) blocks or eliminates some part of 2π Sr hemisphere. The item may be:

- the hole in the hemisphere or hemiellipsoid for the sample beam;
- the overhead mirror and support brackets for directing the sample beam;
- blockages.

In all cases some finite solid angle of the hemisphere over the sample is not available to fully perform its function. Good design can minimize this error by minimizing the offending solid angle, but cannot eliminate it.

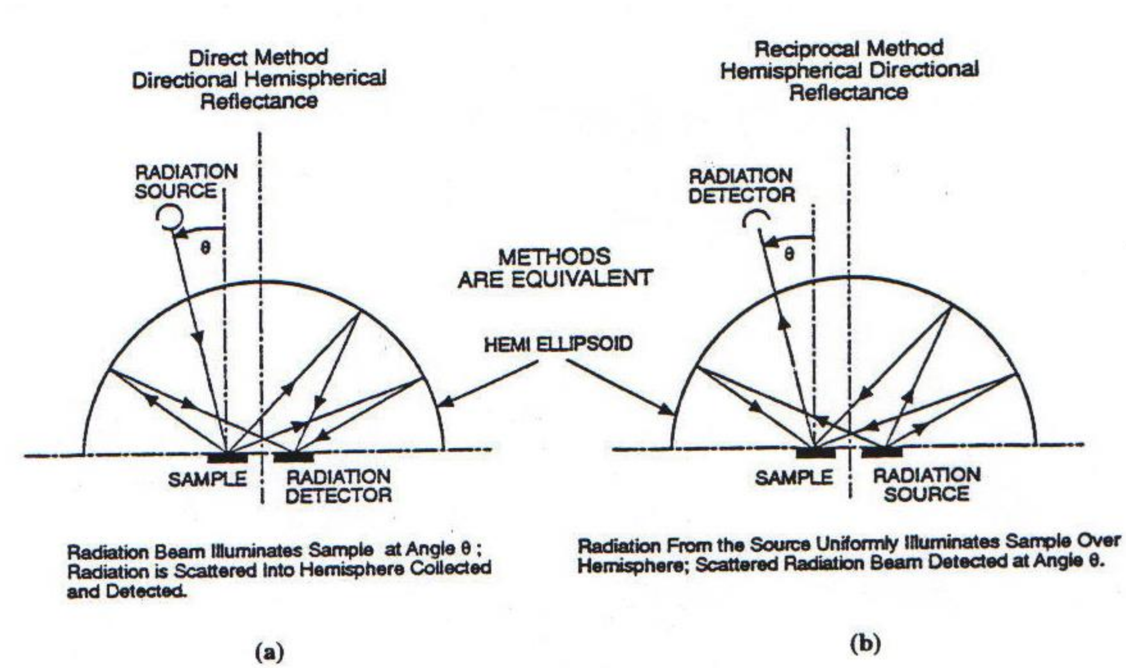


Figure 3-1. Direct and reciprocal DR measurement methods.

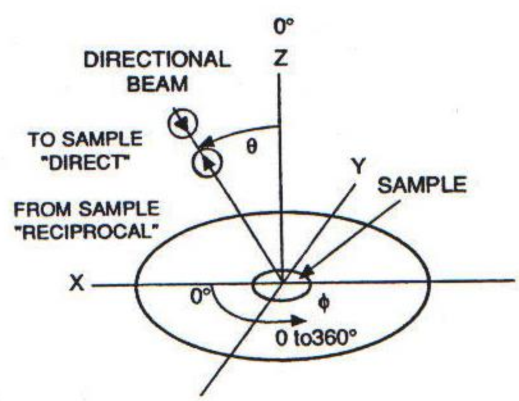


Figure 3-2. Angle definitions, Directional Reflectance.

4.0 SELECTION OF RECIPROCAL MODE

A reflectometer design employing a hemiellipsoid in the reciprocal mode has been selected as the most suitable design for an automated extended capability directional reflectometer. A detailed discussion of all the pros and cons of design goes beyond the scope of this paper. But the importance of the sample-source relationship is explained since it bears on the mode selection and is not immediately obvious.

In practice, both modes require attention to uniform hemispherical radiation. The direct mode detector must respond uniformly to radiation in the hemisphere, irrespective of the angle the rays strike or enter the detector. Similarly the source in the reciprocal mode must generate radiation uniformly into a hemisphere to illuminate the sample.

In a hemiellipsoid device a point at one focus is imaged at a point on the other focus; a finite image is necessarily distorted, based on focusing properties of a hemiellipsoid (see for instance Brandenberg[2]). The image area measured on a sample when reimaged at the source (reciprocal mode) or the detector (direct mode) requires a significantly larger area than the image on the sample, for proper functioning,[3] see Figure 4-1.

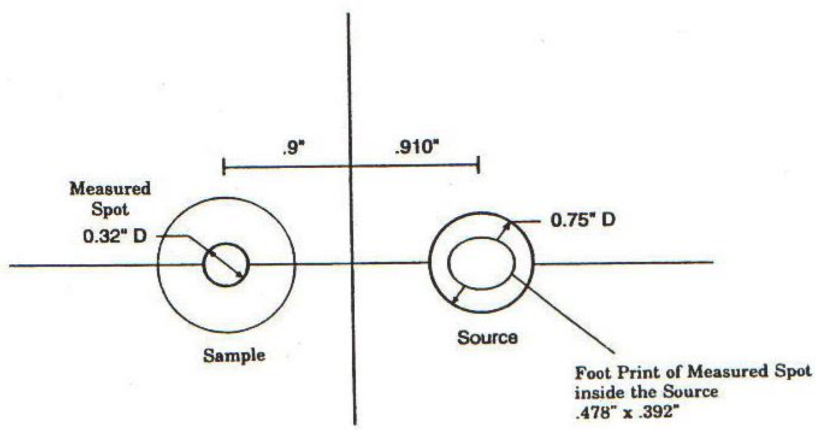


Figure 4-1. SOC-100 sample-source relation.

As an example, consider the reflectometer which is the subject of this paper. For a "measured spot" on the sample (.32"D) for the SOC-100, a foot print source area of .478 x .392 is required to provide uniform sample illumination, Figure 4-1. Actually, a 0.75"D blackbody cavity opening is provided to allow for mirror ellipse imperfections and alignment. Thus by providing a 0.75"D uniform radiance source which is large enough to encompass each and all theoretical pencils of radiation from the "measured spot", uniform illumination of the measured spot is assured based on considerations of reciprocity.[3]

Generally, the direct method is preferred, however, the critical item required, a large aperture (0.75"D) detector with uniform hemispherical response and high sensitivity from 1.5 to 45 µm, is simply not available. Accordingly, the reciprocal mode was selected.

5.0 OUTLINE OF DESIGN FEATURES

The subject reflectometer may be considered as consisting of three parts: the Directional Reflectance Unit (or Measurement Cell), the FT-IR Spectrometer, and the Computer with Software.

5.1 Directional Reflectance Unit

Figure 5-1 shows an isometric drawing of the directional reflectance unit, with the hemiellipsoidal reflector mirror in dashed lines. It is 18" in diameter (major axis) electroformed gold plated specular reflector. The radiation source is centered on one focus, the sample on the other. These two foci are 1.8" apart, and lie in a plane which contains the major and minor axes. For reasons illustrated in Figure 4-1, it is desirable to minimize the ratio of the distances between the foci as related to the major axis (in this case 1.8/18 = 0.1).

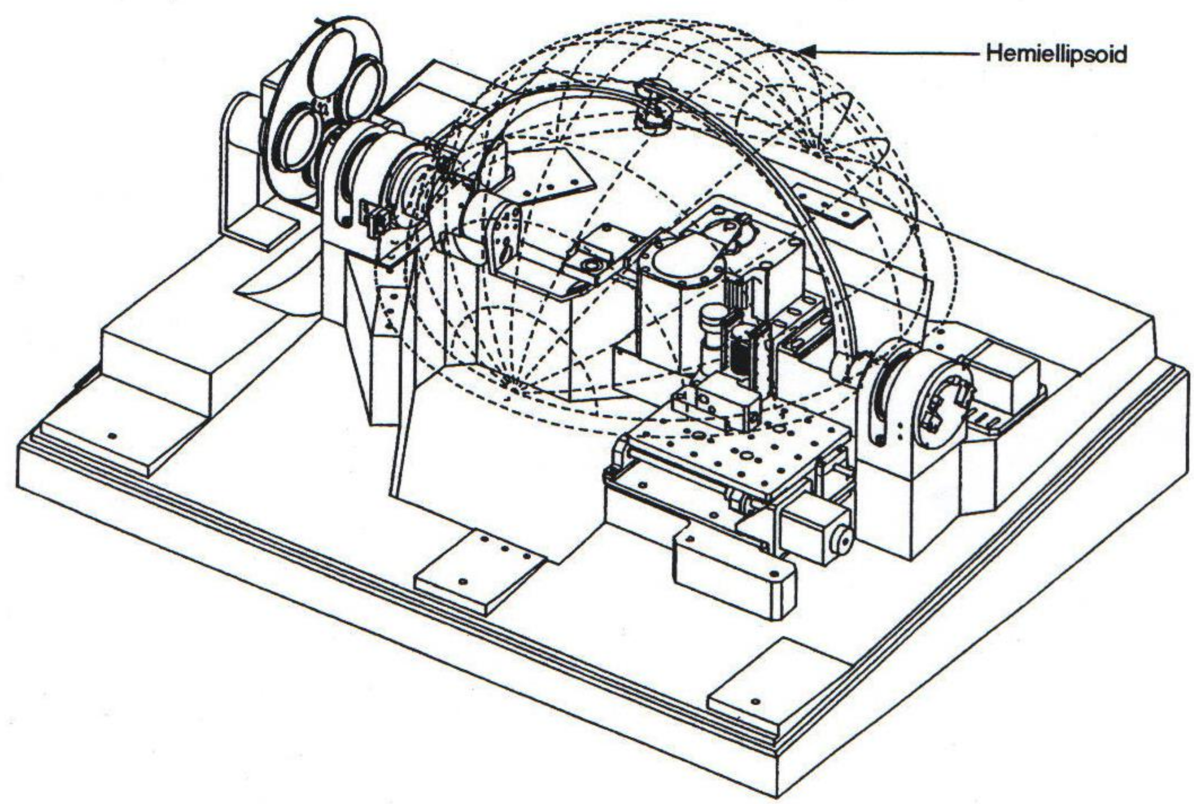
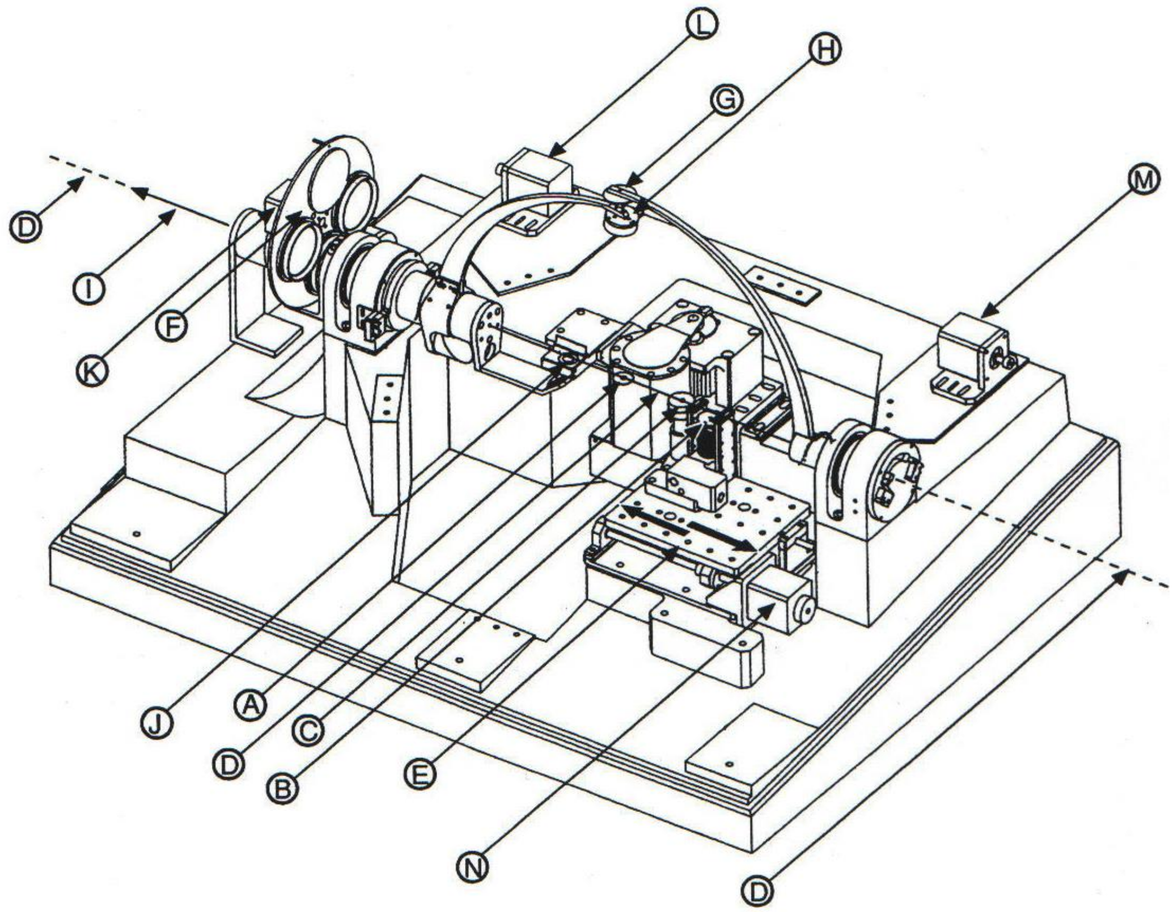


Figure 5-1. Directional Reflectance unit.

Figure 5-2 shows the reflectance unit with the components labeled and the hemiellipse removed.



- (A) Position For Sample Measurement
- (B) Sample Holder
- (C) Standard
- (D) Rotational Axis
- (E) Sample Translation Stage
- (F) Polarization Mechanism
- (G) Beam Blocker
- (H) Overhead Mirror
- (I) Beam To FTR
- (J) Aperture
- (K) Polarization Stepper Motor
- (L) Overhead Mirror Stepper Motor
- (M) Blocker Stepper Motor
- (N) Sample Stage Stepper Motor

Figure 5-2. Reflectance unit with major components (hemiellipse removed).

5.1.1 Optical Train

The elliptical overhead mirror receives radiation from a 0.32"D spot on the sample and focuses it on a 0.32"D aperture in the ellipse plane. Past this aperture, the expanding beam is directed by two flats to a 1½"D 90° off-axis parabola which renders the beam parallel.

This portion of the optical train rotates around the measurement beam rotational axis through the sample face as shown schematically in Figure 5-3 (a), allowing the overhead mirror to view the sample from directly overhead (0° beam angle) to directly below the sample position (180°). This allows angular recording of HDR 20 to 80° and measurement of scattered transmittance of semitransparent samples to about θ = 50°.

The parallel beam from this rotating section is directed to the FTIR by two flat mirrors.

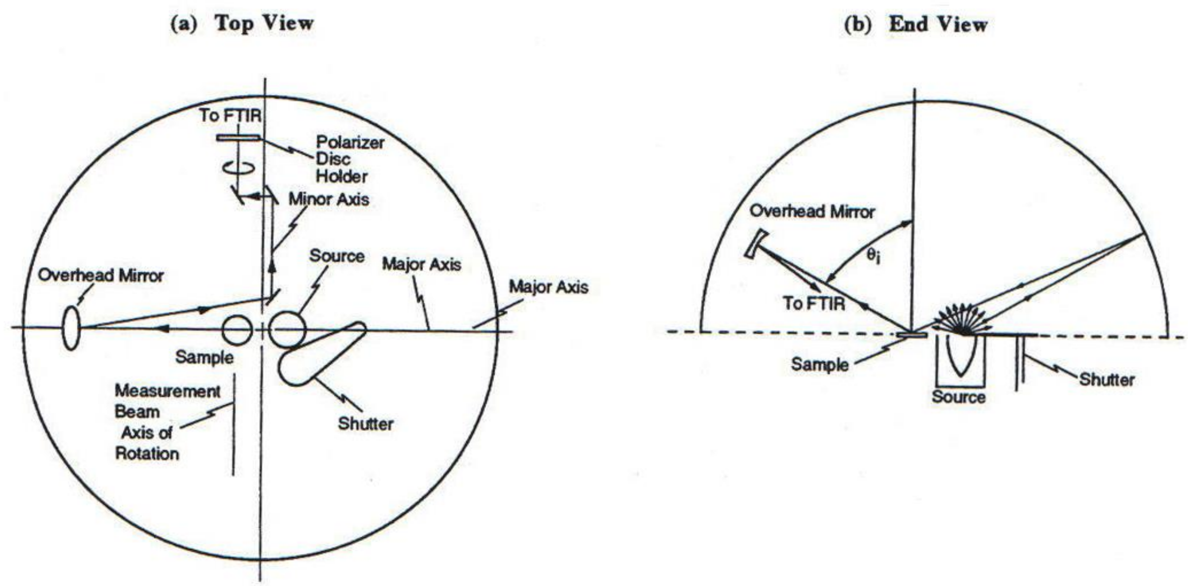


Figure 5-3. HDR measurement configuration.

5.1.2 Source

The heated cavity is sized to 0.75"D so that all possible hypothetical rays traced from the sample measurement spot to the hemiellipsoid and reflected to the source unit can enter the cavity openings, see Figure 4-1. The source is a hohlraum with a special gold plated conical collar which assures uniform illumination to very high angles.

5.1.3 Source Radiation Shutter

Precise switching of the source radiation from open to closed is provided by a two-position (open and closed) shutter, stepper motor controlled and computer activated by a signal from the FTIR. The transition is accomplished in less than 30 milliseconds; the dwell time closed and open is variable, depending on selected FTIR resolution.

5.1.4 Sample Position

The measurement position may be occupied by a reflectance sample, a reflectance standard, a transmittance sample, or left unoccupied. Interchange between these automated options is computer controlled.

5.1.5 Energy Partition

The reflectometer is equipped with a beam blocker to allow approximate partition of the directional reflectance into a scattered component and a "specular lobe".

Figures 5-4 (a) and 5-4 (b) show a schematic of the reflectometer in the 60° angle of beam positions with and without the energy partition blocker in place. Without the blocker, the sample is irradiated over a hemisphere and the reflected radiation viewed by the overhead mirror is directed to the spectrometer where it is recorded. In this configuration, comparison of the sample and the reference standard is used to determine the "total" directional reflectance made up of the specularly and diffusely scattered radiation. When the independently adjustable blocker is moved into position as shown in Figure 5-4 (a), the specular reflectance lobe radiation is blocked; only the scattered radiation component is measured. Subtraction of the scattered component from the total directional reflectance provides the specular lobe reflectance.

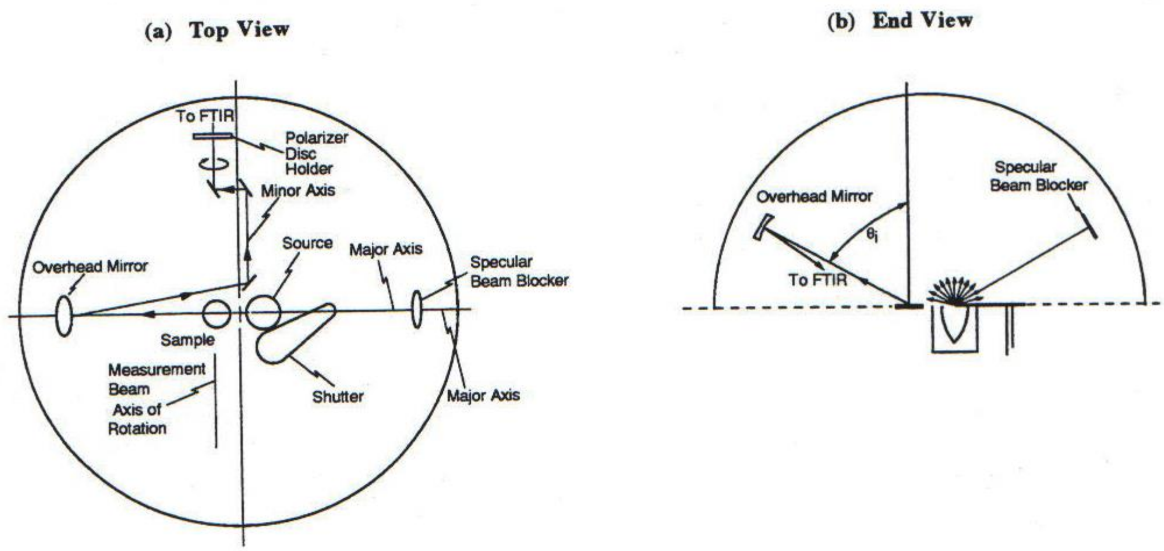


Figure 5-4. Specular blocker in position.

The blocker position is adjusted by rotation about the ellipse's measurement beam rotational axis. It is designed to intercept radiation at polar incidence angles 20, 30, 40, 50, 60, 70, 75, and 80°, and is rotated below the instrument horizon when not in use.

The determination of the specular-diffuse ratio in the range from Θ_i = 20 to 80° offers the opportunity to screen materials for specularity as a function of wavelength. If a sample is seen to be essentially a specular reflector, it may be so treated in signature calculations, thus eliminating the need for extensive and relatively expensive BDR measurements.

5.1.6 Polarization

Applications for data often require measurements done as a function of beam angle Θ. It is possible to make DR measurements from near-normal (about 20°), to about Θ = 80°. When the reflected energy is significantly polarized, it is necessary to employ experimental procedures which eliminate the effects of polarization bias (i.e., $\parallel$ and $\perp$ transmission mismatch) in the measurement optical train. Prism and grating spectrometers, and to a much lesser degree

FTIR's, have a polarization bias which varies as a function of wavelength. Making two measurements of the HDR, one parallel and one perpendicular and averaging the two measurements, allows determination of the true reflectance for unpolarized light.

An automated disc with two polarizer plate positions and one open position is positioned in the collection beam as shown in Figure 5-2 and 5-4 (a). A CsI polarizer provides coverage from 2.0 to 35.0 µm. An optional additional unit is available with coverage from 20 to 200 µm. Instrument wavelength coverage is dependent on FTIR optical components, see ¶ 5.2 and Table 5-1.

Table 5-1
FTIR

FTIR	Nicolet 550 or 750			
WAVELENGTH COVERAGE	Depends on IR optics			
	SPECTRAL RANGE	SPLITTER	DETECTOR	DETECTOR WINDOW
	1.5 to 25.0 µm	KBr	DTGS	KBr
	1.8 to 45.0 µm	CsI	DTGS	CsI
	14.0 to 200 µm	CsI	DTGS	Polyethylene
RESOLUTION	Variable, 16 cm^{-1} and better			

5.1.7 Heated Samples

Emittance data up to 500°K is ordinarily derived from reflectance data measured at room temperature, since reflectance vs. wavelength is invariant with temperature in the absence of chemical or physical changes in the sample. If the samples do change chemically or physically upon heating, reflectance of a heated sample is required. In this case, measurements may be made on samples maintained at elevated temperatures to 500°C using an optional heated sample holder. The measurement rejects the sample emission signal through the use of "between the source - and sample chopping".

5.2 FTIR Monochrometer

The FTIR unit provided is a Nicolet 550 with KBr optics and DTGS detector. Optional upgrades may be used as shown in Table 5-1. No special alteration of either the 550 or the 750 is required for functioning of the SOC-100.

5.3 Computer and Software

Basic start-up tasks - turning on power, water, mounting samples and standard - are manually accomplished. Desired measurements and data output are then entered into the computer from an extensive prompting menu. Upon measurement activation, the computer then directs all the data acquisition tasks including:

- Directing the FTIR.
- Directing and sequencing stepper motors in the reflectance unit.
- Providing real-time graphical display during data recording.
- Processing and compiling the data to provide tabular and graphical plots.
- Printing of tabular and graphical data.

- Inquiring if user wishes to measure another sample.

5.3.1 Computer

An IBM compatible computer is used as the control processing unit for the instrument. The Intel 486 based system hosts SOC's HDR software package to perform automated system control, data acquisition, and data reduction. The system is configured with a Super Video Graphics Adapter (VGA) color monitor for real-time data display as well as a laser printer for hard copy output.

5.3.2 Computer Controlled Rotational Axes

Standard:

- Source radiation "Open - Closed".
- Positioning items in sample position (samples, standards, etc.).
- Beam sample angle rotation.
- Automated beam blocker for energy partition.
- Automated rotation of the beam polarizing filter holder.

6.0 PERFORMANCE CAPABILITIES

An extended capability directional reflectometer allows collection of a wide variety of important quantities as tabulated below:

Table 6-1
Quantities Measured Directly

1.	Hemispherical Directional Reflectance.
2.	Scattered Transmittance of semi-transparent materials
3.	Partition of total radiation reflected from a diffuse reflector into the specular lobe and scattered radiation.

Table 6-2
Derived Quantities

1.	Directional and Hemispherical Emittance as *f* of T
2.	Solar absorptance, endo and exo
3.	Total integrated scatter
4.	Index of refraction, *n* and *k*

A partial listing of the range of variables measurable are listed for direct measurements in Table 6-3 and derived quantities in Table 6-4.

Table 6-3
Direct Measurements

HDR DATA AS A FUNCTION OF	RANGE OF MEASUREMENT
a. Directional Angles θ: 20, 30, 40, 50, 60, 70, 75, 80°	Near-normal, 20 to 80°
b. Azimuthal Angles φ	0 to 360°
c. Wavelength	2.0 to 25.0 μm, 2.0 to 45.0 μm,* 2.0 to 200 μm*
d. Beam Polarization	Parallel and Perpendicular
e. Sample Temperature	Room temperature to 500°C w/heated sample holder

* Requires available alternative FTIR optical components, see Table 5-1, and subject to certain S/N limitations.

Table 6-4
Derived Quantities

QUANTITY	DATA AS A FUNCTION OF	METHOD OF DERIVATION
Emittance derived from ambient temperature measurement of DR of stable materials which do not undergo physical or chemical changes upon heating 100 to 500°K. • Total hemispherical emittance. • Directional emittance	Bandwidth, θ = 20 to 80°, T°K, 100 to 500°K λ = 2.0 to 25.0 μm*, θ = 20 to 80°, T°K, 100 to 500°	Planck and Kirchhoff equations Kirchhoff's equations
Emittance derived from DR measurement of sample at controlled temperature. Method used for materials that may change physically or chemically with temperature. • Total hemispherical emittance • Directional emittance	Bandwidth, θ = 20 to 80°, T°K, 100 to 500°K λ = 2.0 to 25.0 μm*, θ = 20 to 80°, T°K, 100 to 500°	Planck and Kirchhoff equations Kirchhoff's equations
Partition of reflected radiance into specular lobe and scattered components using the specular beam blocker	λ= 2.0 to 25.0 μm*, θ = 20 to 80°	Measurement using blocker, computer algorithm
Endo and Exo Solar Absorptance*	Directional Angle θ = 20 to 80°	Application of measured DR data to NASA published solar data

QUANTITY	DATA AS A FUNCTION OF	METHOD OF DERIVATION
Index of Refraction - *n* and *k* for Dielectrics and Conductors for "Fresnel" materials	λ= 2.0 to 25.0 µm*	At a given wavelength, Fresnel's equation is used to calculate *n* and *k* and the Brewster angle using ‖ and ⊥ polarized DR as a function of angle

* Optional coverage to 45.0 µm or 200 µm, see Table 5-1.

REFERENCES

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2. W.M. Brandenberg, "Focusing Properties of Hemispherical and Ellipsoidal Mirror Reflectometers", J. Opt. Soc. Am. 54, 1235 (1964).

3. D.K. Edwards, Applied Optics, Vol. 5, pg. 175 (1966).