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NEW INSTRUMENTATION FOR MEASURING SPECTRAL GONIOMETRIC REFLECTANCE

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March 1989

Reprinted from 1989 SPIE Orlando Proceedings

New Instrumentation for Measuring Spectral Goniometric Reflectance

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ABSTRACT

The development of a computer-controlled system which enables testing both spectrally and goniometrically will be discussed. Built around a multi-purpose spectroradiometer system, the exit optics attachment allows users to test samples by varying both incident and measurement angles. The general spectroradiometer system and the individual attachment will be highlighted. Originally developed for measuring the reflectance of specular samples from 200nm-30μm, this instrument may also be used for other types of spectral measurements, including diffuse reflectance and transmittance. The above measurement areas will be described, incorporating test sample graphs and system performance data.

1. INTRODUCTION

The gonio-spectroradiometric instrument was designed to enable users to make both specular (mirror) and diffuse reflectance measurements over a wide range of wavelengths from the UV to IR (200nm-30μm) by using a new variable angle reflectance attachment with a research grade spectroradiometer. While primarily designed for specular measurements, the instrument is also well adapted to various other measurement configurations by the addition of easy to install accessories. These options allow the user to make measurements of diffuse reflectance, as well as diffuse and rectilinear transmittance.

2. GENERAL INSTRUMENTATION

The basic computer controlled spectroradiometer system uses a versatile lock-in radiometer with a single or double monochromator[1,2]. The overall system is illustrated in Figure 1. The system is comprised of modules which can be reconfigured for a wide variety of measurement applications, including detector measurement, radiance and irradiance calibration, and reflectance and transmittance measurements, all using the same basic instrumentation with the addition of various accessories. Each optical component is carefully designed to accurately mount to the monochromator thereby producing a stable, rigid unit. A brief description of each component is given, following from right to left in Figure 1. The 740-75M reflectance attachment is described separately.

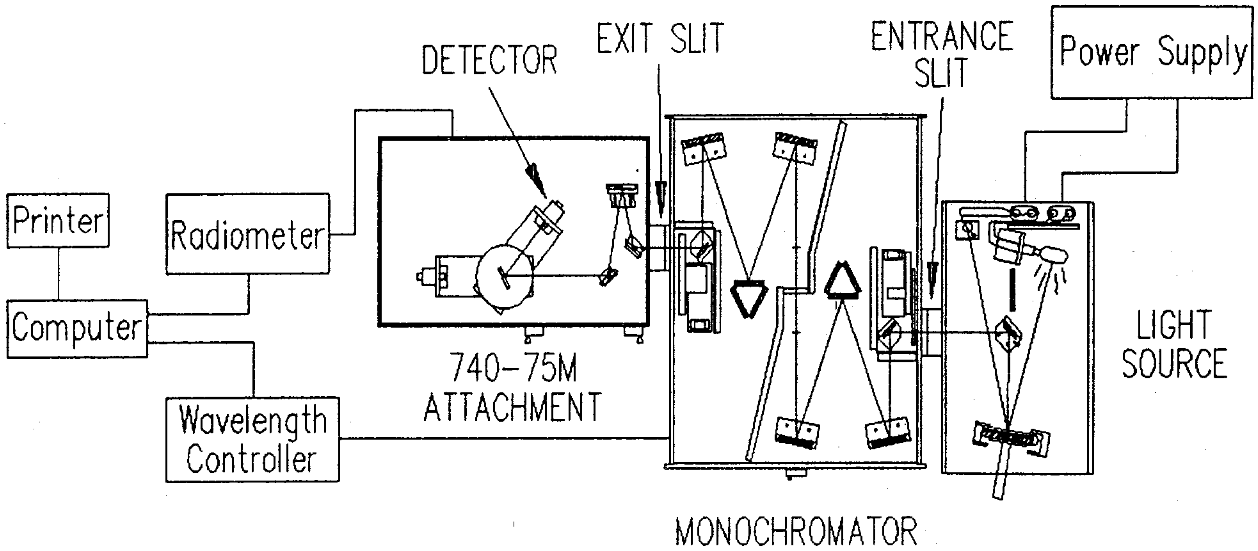

Figure 1. Spectroradiometer System

Illumination is provided by a dual high intensity source attachment. Depending on the wavelength range required, one can choose from a Deuterium arc lamp for the ultraviolet (200-400nm), a DZE Tungsten-Halogen lamp for work in the visible and near infrared (0.3-3μm), and a ceramic Nernst glower for the far infrared (1-30μm) wavelength ranges. A beam switching mirror focuses the flux from either source onto the entrance slit of the monochromator. A digitally selectable power supply provides constant current to the sources.

The source energy is dispersed through a Czerny-Turner type grating monochromator. Either a single or a double design may be used. A double monochromator increases the spectral resolution of the system while significantly reducing potential stray light. Interchangeable slits are provided to allow selection of bandpass and flux intensity. A servo controlled, quartz locked optical chopper mounted at the entrance to the monochromator modulates the incoming beam. This allows the use of various infrared detectors as well as greatly reducing interference from ambient light. Tri-grating turrets with precision indexing bases are employed to allow quick and accurate grating selection, thus producing a very broad coverage of wavelength. A stepper motor driven order sorting filter wheel effectively blocks second order effects from the diffraction gratings.

Interchangeable detectors, which all have standardized housing dimensions, are used with a versatile multimode preamp and lock-in amplifier. Available detector types include photomultiplier tubes (PMT), silicon (Si), germanium (Ge), lead sulfide (PbS), lead selenide (PbSe), indium antimonide (InSb), mercury cadmium telluride (HgCdTe), and pyroelectric.

An autoranging lock-in amplifier displays the detector signal in volts or amps. A switch on the front panel allows for the selection of detector type, thus providing the appropriate preamplification and phasing for that particular detector. The lock-in also controls the optical chopper in the monochromator.

A wavelength drive unit controls the monochromator's wavelength drive circuitry as well as the motorized order sorting filter wheel.

An IBM compatible computer (with printer) serves as the automated control mechanism. BCD outputs from the amplifier and wavelength controller enable computer controlled scans to be made. Factory developed application software performs the otherwise tedious chores of datalogging and calculation, in addition to wavelength drive, signal reading, and filter wheel control.

System measurement capabilities from the UV to IR (200nm - 30μm) are made possible by the variety of detectors, gratings, and sources available. The equipment can be provided with nitrogen purge fittings which, when used, can assist in difficult wavelength regions such as atmospheric absorption bands. Table 1 lists some of the configurations and wavelength ranges covered.

Table 1. Sample Spectral Range Configurations

Wavelength Range (μm)	Grating Blaze (μm)	Source	Detectors
0.28 - 1.1	0.5	Quartz-Halogen	Silicon
0.28 - 2.5	0.5/1.6	Quartz-Halogen	Silicon TE Cooled PbS
1.0 - 3.2	2.0	Quartz-Halogen	TE Cooled PbS
1.0 - 5.0	1.6/4.0	Quartz-Halogen	TE Cooled PbSe
1.0 - 18.0	1.6/4.0/10.0	Quartz-Halogen Nernst Glower	Thermal Detector

3. MODEL 740-75M REFLECTANCE ATTACHMENT

This attachment was originally designed to make specular reflectance measurements as a function of wavelength and angle of incidence. It can easily be adapted to make other types of measurements such as transmittance and diffuse reflectance (especially for highly reflective materials). The housing bolts directly to the exit port of the monochromator forming a rigid, stable unit. Due to its all mirror optical design, measurements can be made over a very wide wavelength range (200nm - 30μm). Another major design feature is the ability to directly measure the incident beam flux. Many instruments of this type only permit measurements to be made with reference to a known standard. This instrument allows the user the additional option of measuring absolute reflectance or transmittance by direct comparison to the incident beam. The detector arm is capable of rotating 180 degrees around the sample which allows both the 100% and test position to be measured (see Figure 2). The mirror optics collect the monochromatic flux and form a 3X magnified image of the exit slit at the detector position. For example, with the use of the 1.5mm diameter circular exit slit, a 4.5mm spot is formed at the detector plane. In order to reduce the focused spot size for the smaller area infrared detectors, a removable lens holder containing a field lens is installed in front of the detector on the rotatable arm. A removable baffle tube shields the detector from extraneous scattered light when measuring diffuse reflectance. The beam is converging as it strikes the sample plane with an approximately ± 2 degree angular field and an 8 X 12mm cross section.

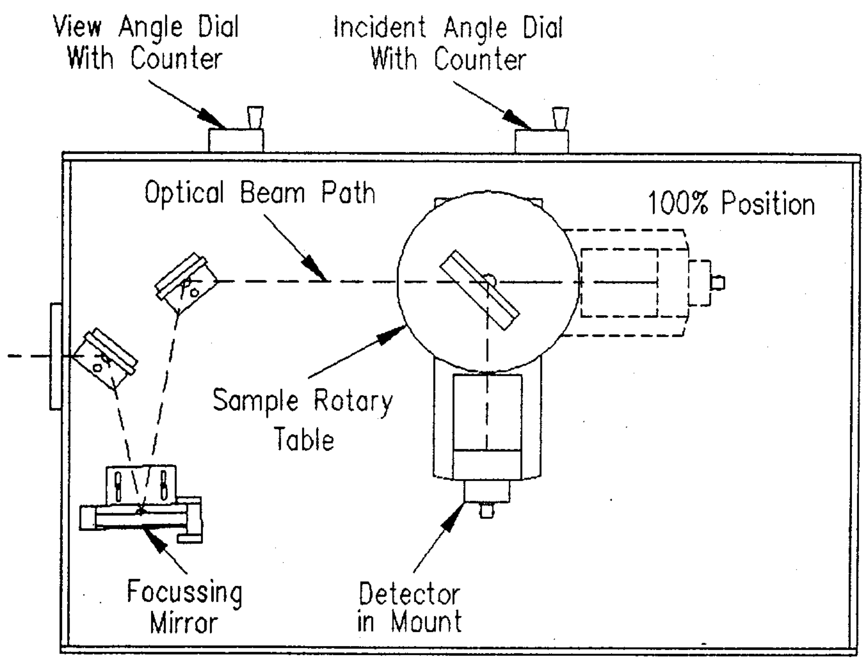

Figure 2. Specular Reflectance Attachment
(Top View)

The flexible receiver design allows the angle of incidence to be selected from 10 - 60 degrees with an accuracy of ± 0.1 degree. The angle of incidence and the detector viewing angle are independently displayed on digital readout counters by means of a sophisticated mechanical drive mechanism. The unique differential type gearing allows the digital counters to maintain the proper angular display at all times. The first counter displays the position of the sample table in degrees relative to the incident beam. The second counter displays the detector arm position in degrees relative to the sample holder. A dual concentric worm gear drive rotary table enables both the sample holder and detector arm to be rotated about the same axis. Independent hand cranks allow precise positioning of both rotations. A motorized option allows computer controlled positioning of both the sample rotary table (incident beam angle) and detector arm (reflected beam angle).

Another available option provides polarization of the incident beam, thus allowing polarization sensitive materials to be measured in their respective maximum and minimum orientations. Specular samples are, of course, very sensitive to polarization at angles greater than 10 - 15 degrees as shown by Maxwell's equations. For large angles of incidence, reflectance should be measured in terms of perpendicular and parallel polarization components.

Samples are held precisely at the center of rotation on a 6 inch diameter rotary table. The minimum sample size depends on the angle of incidence and should be considerably larger than the beam. The supplied sample holder can accommodate circular samples ranging from 2 inches to 4 inches in diameter or rectangular samples from 2 inches square to 4 x 6 inches. Sample thickness can vary from 1/16 to 1/2 inch.

4. SPECULAR REFLECTANCE MEASUREMENTS

Measurement of specular reflectance is accomplished by comparing the signal level of the incident beam to the signal level of the reflected beam off the sample. The technique is relatively straightforward. Remove the test sample and rotate the sample table to allow the incident beam to pass unobstructed through the sample holder. Position the beam on the detector surface by placing the detector arm in the 100% position. Record the signal levels over the desired wavelength range and interval. Insert the specular test sample and rotate the sample table to the desired incident angle. Rotate the detector arm to intercept the reflected beam. Record the signal levels over the same wavelength range and interval as before. Calculate the percent reflectance of the sample at each corresponding wavelength by dividing the test signal by the 100% signal, then multiplying by 100.

All of these calculations are performed automatically and printed out in hard copy at each wavelength in the scan by the computer program. Final values of reflectance for the sample can then be stored on a mass storage device for subsequent examination.

The instrument's performance was tested by measuring the reflectance of a NIST (NBS) calibrated standard mirror over the wavelength range of 300-1000nm at a 12 degree angle of incidence with a monochromator bandpass of 8nm. The agreement between values reported by NIST and the experimental data taken with this system was within 0.6% or less (see Table 2). Over the course of 3 hours, the system drift in the 100% readings was less than ±0.5%.

Table 2. Comparison to NIST Standard Mirror

Wavelength(nm)	NIST Value(%)	Measured(%)	% Difference
300	89.5	89.4	-0.1
350	89.6	89.6	0.0
400	89.5	89.7	-0.2
450	89.2	89.2	0.0
500	88.7	88.7	0.0
550	88.2	88.1	-0.1
600	87.5	87.5	0.0
650	86.6	86.7	+0.1
700	85.4	85.0	-0.4
750	83.5	83.0	-0.5
800	80.8	80.3	-0.5
850	80.1	79.9	-0.2
900	84.9	84.3	-0.6
1000	91.2	91.2	0.0

Of considerable importance to several users are measurements of polished silicon wafers with special coatings (see Figure 3). Additional tests have been made of commercial mirrors coated with aluminum and silver vacuum deposited films (see Figures 4 & 5).

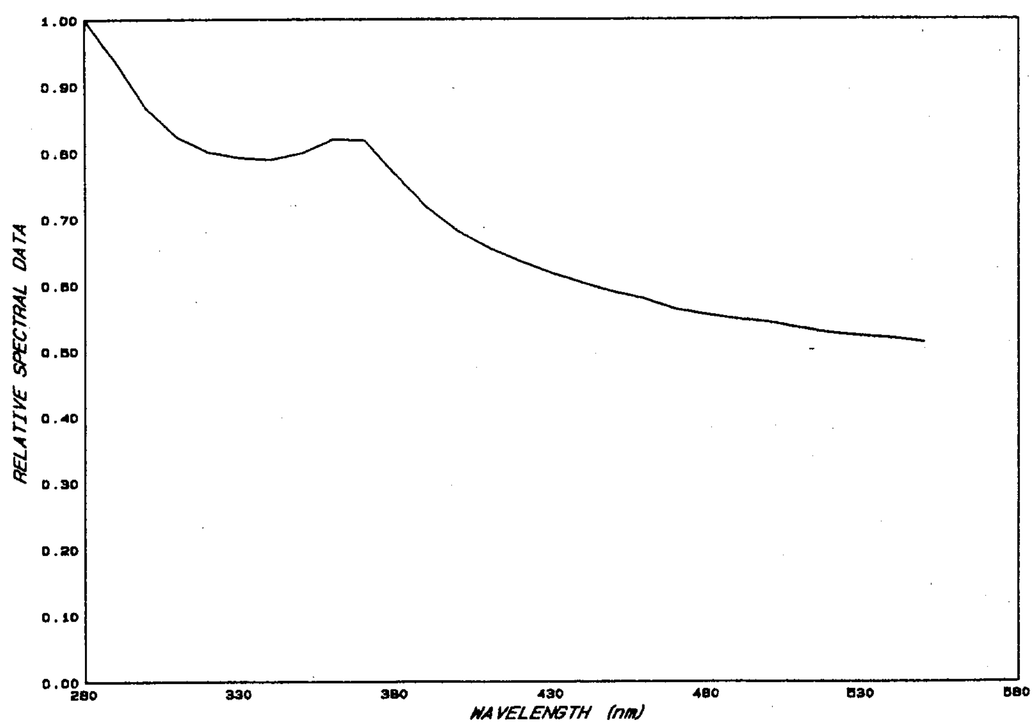

Figure 3. Silicon Wafer Reflectance at 10 degrees

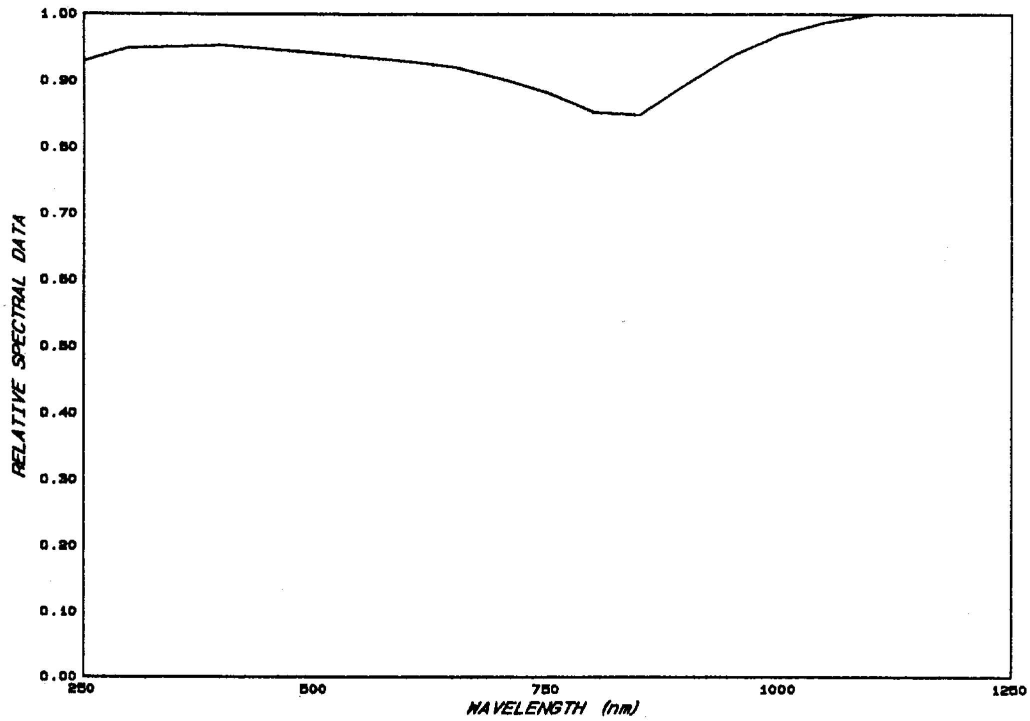

Figure 4. Reflectance of Aluminum Mirror at 10 degrees

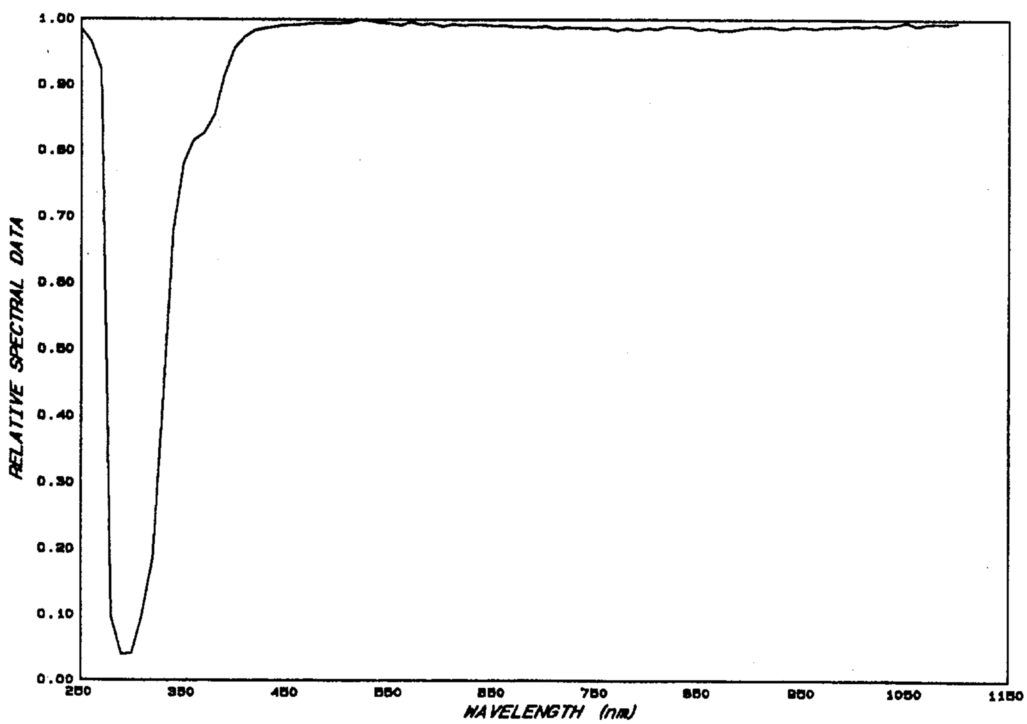

Figure 5. Reflectance of Silver Mirror at 45 degrees

Samples that have little or no scatter as they transmit (i.e. rectilinear) may also be measured. The incident beam flux is first measured with the detector arm in the 100% position. The sample is then inserted into the sample holder and the incident angle chosen. The detector arm is left in the 100% position. A potential application of this type of measurement are filter testing, including testing angle of incidence versus bandpass in interference filters. Figure 6 depicts a user application for measuring the transmittance and reflectance of a thin film metallized window glass. Note the sharp ringing phenomena in the curves, and also how the peaks and valleys between the two curves coincide.

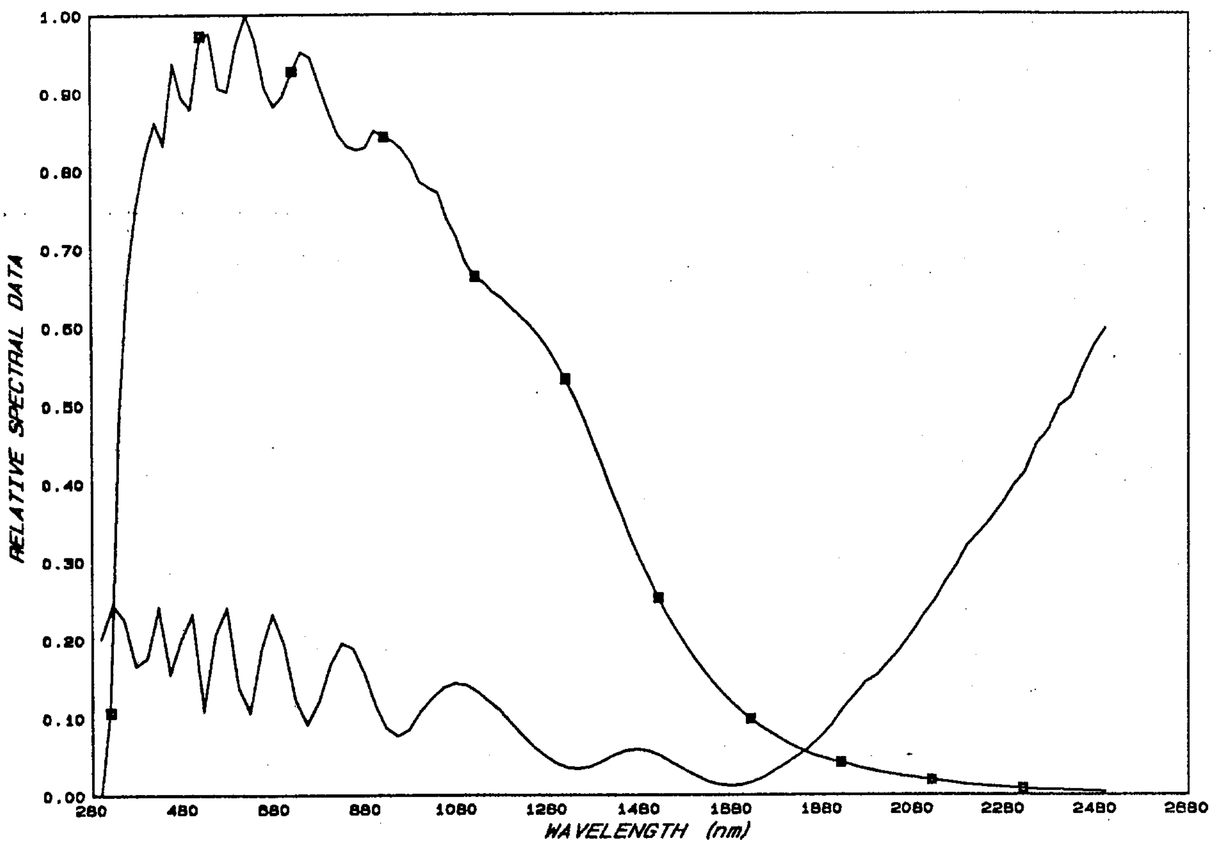

Figure 6. Transmittance and Reflectance of Metallized Glass

5. DIFFUSE REFLECTANCE MEASUREMENTS

One of the more complex measurements is the assessment of spectral diffuse reflectance. The method of making such measurements is not as straightforward as in specular reflectance. Indeed, in comparing the two types of measurements, several differences should be noted. When the incident beam strikes the diffuse surface, the reflection is scattered (or, distributed) over a wide angle, as opposed to specular measurements where the angle of reflectance equals the angle of incidence. The detector size and distance from the sample determines the amount of flux collected, thus making the reflectance value a function of instrument design rather than a property of the sample. A method of correcting for the instrument geometry which is generally used by researchers is to calculate the reflected radiance divided by the incident beam's irradiance. This quantity is strictly a function of the sample. Since the distribution of reflected energy from the sample can vary with the particular incident and viewing angle, these conditions must be specified in the reporting of data. Thus, the term "Bidirectional Reflectance Distribution Function (BRDF)" is used to describe these measurements. The radiance is determined from the measurement of the reflected flux divided by the detector solid angle and the projected area. Several references are available which explain this nomenclature and the geometrical considerations involved in greater detail[3,4].

Measurements of diffuse reflectance can be made relative to the incident beam when possible, or referenced to a stable diffuser of known reflectance. PTFE or $BaSO_4$ are commonly used due to such desireable characteristics such as their lambertian (i.e. uniform) scattering, high absolute reflectance over a relatively wide wavelength range, good temperature characteristics, and freedom from fluorescence[5]. The reflectance properties of these materials have been previously documented in the literature[6,7].

Many users will find it adequate to perform absolute measurements only occasionally in order to make a working reference standard for the materials under test. When comparison is made to a known reference standard, the reflectance of the test material is more accurately termed a reflectance factor. In a reflectance factor measurement, a ratio is made between the reflected flux from the standard and the test sample (which are measured under the same conditions). The resultant ratio is then multiplied by the standard's previously known absolute reflectance. This method can provide some cancellation of measurement error if the two surfaces are similar in reflectance[8]. Of course, since the incident angle and viewing angle must also be specified in order to yield meaningful results, the correct terminology for this type of measurement is a "Bidirectional Reflectance Distribution Factor".

An optional tilt table allows measurements to be made in a third dimension (see Figure 7). This type of three dimensional data can be integrated to calculate the sample's directional hemispherical reflectance. Intercomparison measurements on $BaSO_4$ samples from 350 to

1100nm made with an integrating sphere instrument, which measures directional hemispherical reflectance, show good correlation with the 740-75M data.

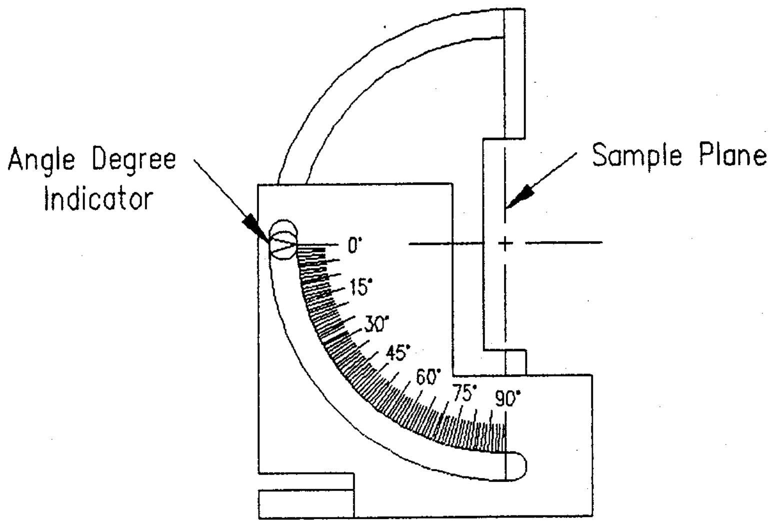

Figure 7. Optional Sample Tilt Mount
(Side View)

ACKNOWLEDGEMENTS

The authors wish to express their appreciation to the following individuals for their contribution: David Goebel for his instrumentation design and application expertise, many years of outstanding service to the optical community, and assistance in reviewing/editing this paper. William E. Schneider for additional editorial suggestions. Dennis Hull for his clever mechanical engineering and providing the AutoCad drawings. Theresa Schehr for her word processing efforts, and to the rest of the staff for their assistance.

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