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Optical Measurement Techniques
Applied to Solar Selective Coatings

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OPTICAL MEASUREMENT TECHNIQUES APPLIED TO
SOLAR SELECTIVE COATINGS

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ABSTRACT

Optical measurement techniques have been developed for determining the solar absorptance, α_s , and emittance, ϵ , of solar coatings. The optical equipment includes a Beckman DK-2 Spectroreflectometer and a Gier Dunkle Solar Reflectometer (Model MS-251) for α_s measurements and a Gier Dunkle Infrared Reflectometer (Model DB-100) for ϵ measurements. Because all this equipment is designed to accommodate flat samples, special calibration and measurement procedures were developed for the determination of the optical properties of coatings deposited on cylindrical surfaces. In addition, a small zero offset associated with the MS-251 is discussed, and its incorporation in the measurement procedure for this instrument is presented. The accuracy of the MS-251 for measurements of the α_s properties of black chrome coatings was determined to be better than ± 0.03 absorptance units. Although the Gier Dunkle Infrared Reflectometer is designed to measure the room temperature emittance, two modifications of the instrument's capability are discussed. The first modification involves removal of an internally mounted polyethylene filter so that the measurement spectrum shifts to approximately a 100°C blackbody. The accuracy of the emittance values determined in this mode of operation is better than ± 0.02 emittance units for the black chrome coating. The second modification replaces the polyethylene filter with a sapphire filter. In this operating condition, and when properly calibrated, the reflectometer can be used to determine the 300°C emittance of the black chrome coating.

Introduction

In support of solar energy materials programs, several optical measurement procedures and techniques for the determination of the solar absorptance, α_s , and emittance, ϵ , properties of solar coatings have been developed. The equipment used for these measurements includes commercial laboratory as well as portable instruments. Although some of the techniques have been developed specifically for electrodeposited black chrome solar coatings,¹ they can be used with other solar coatings if properly calibrated and applied.

Because optical instrumentation used to characterize solar coatings is usually designed to accommodate flat samples, special measurement procedures have been developed so that the optical properties of coatings applied to cylindrical samples (receiver tubes) can be determined. These measurements are important both to provide property data as well as to check uniformity of coatings and processing information for tubular geometries relative to flat plate geometries. The techniques have been used to characterize the optical properties of coated receiver tubes 41.3 mm (1.625") in diameter that are used in Sandia's Solar Total Energy Test Facility.²

An instrument that is commonly used for solar absorptance measurements is a Gier Dunkle Solar Reflectometer. A small but significant zero offset associated with this instrument, if not properly accounted for, can introduce a substantial error in the measured solar absorptance values, especially for highly absorbing materials. A determination of this zero offset and its incorporation in the measurement procedure are presented. This source of error may also be present in other similar optical equipment. The accuracy of black chrome absorption measurements is also discussed.

An instrument that is used for room temperature emittance measurements is a Gier Dunkle Infrared Reflectometer. In addition to comparing the emittance values obtained with this instrument for a variety of materials with independent total hemispherical emittance measurements, two modifications of the measurement capability of this instrument are discussed. The first modification involves shifting the measurement spectrum from a room temperature blackbody to a 100°C blackbody. The second shifts the measurement spectrum so that the 300°C emittance properties of the black chrome

coating can be measured. The calibration and measurement spectrum of the reflectometer for both modifications are presented.

Spectroreflectometer

Adapter for Cylindrical Sample Measurements:

A spectroreflectometer, such as a Beckman DK-2 with an integrating sphere reflectance attachment,³ measures the hemispherical reflectance over the solar spectral range for approximately normally incident radiation. By averaging these data over a solar spectral distribution, the normal solar absorptance is obtained. This instrument is designed to measure flat samples which are mounted over a measurement port, approximately 20 mm in diameter, which is located in the integrating sphere wall. Therefore, solar coatings applied to flat plates or strips represent the most convenient sample geometry for these measurements. However, in some focused solar collector systems, the absorbing coating is deposited on a relatively small diameter (12 to 50 mm) receiver tube.²

It would be very desirable to perform spectral absorptance measurements on actual coated collector tubes. This information, besides providing absolute values for the solar absorptance, could also be used to check processing, plating or evaporation techniques developed for tubular geometries as compared with flat plate geometries. In addition, one could obtain information on the uniformity of the coatings relative to the deposition geometry or the position in the solar collector.

In response to this need, an adapter for a Beckman spectroreflectometer that will accommodate collector tubes of various diameter and lengths has been built and tested. The adapter positions the collector tube in front of the usual sample measurement port at a location external to the integrating sphere as shown in Figure 1. Measurements are then performed in the usual manner but with a black cloth draped around the complete assembly in order to exclude background radiation from the optical system.

Clearly, the most serious problem associated with this measurement arrangement for a curved surface is that some reflected radiation will not be collected by the integrating sphere but will fall outside the sample port and thereby be lost. Thus, reflectance values measured from a curved

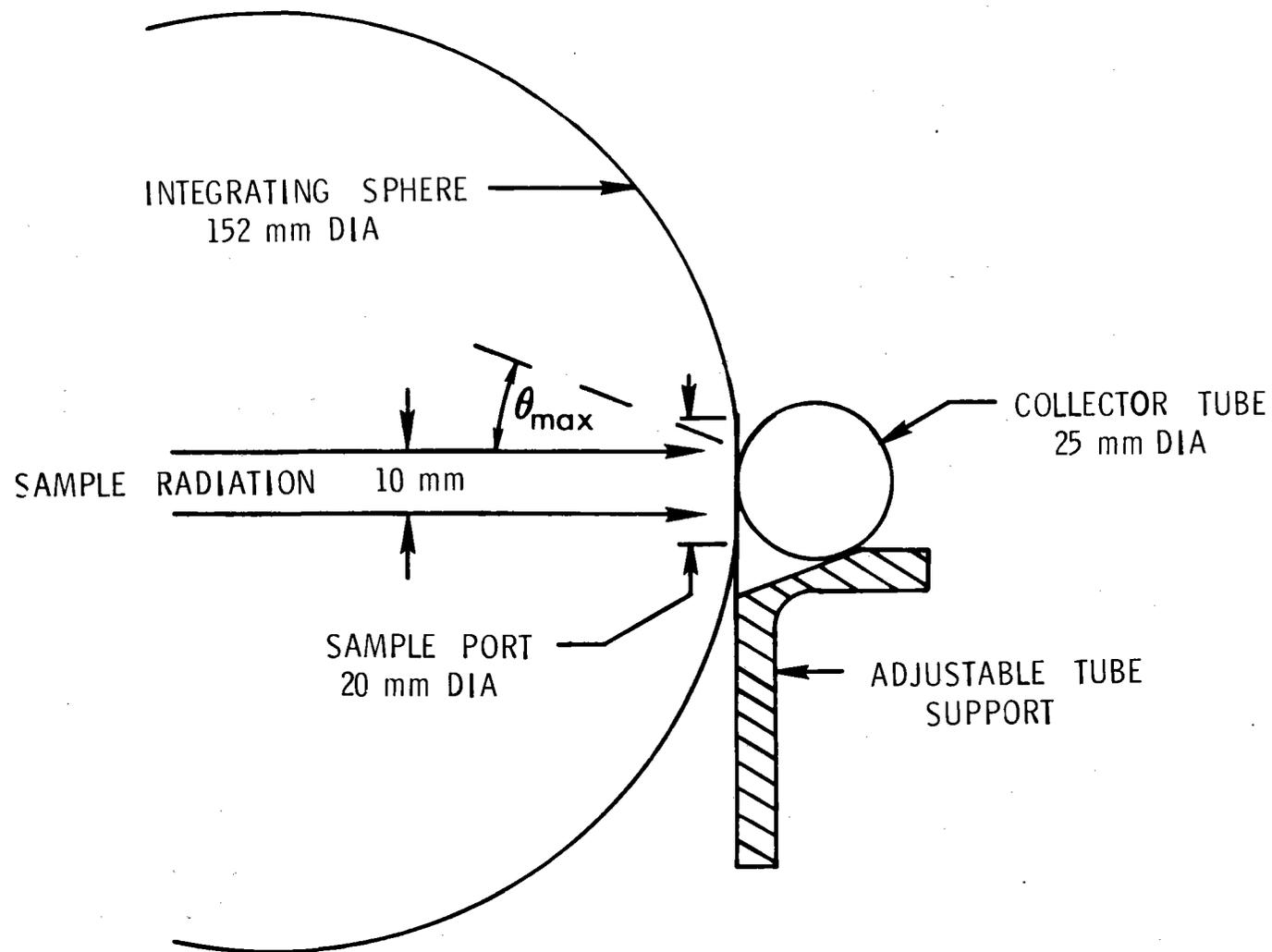


Figure 1. Hemispherical reflectance arrangement showing the relationship of the integrating sphere to the collector tube. The angle θ_{max} is defined as the maximum incident angle for radiation on the collector tube.

sample should be lower than reflectance values measured from an identical flat sample. However, the amount of radiation that is lost will depend upon the incident beam size, the pipe diameter, the sample port diameter, as well as the angular distribution of the reflected radiation from the sample (which can vary from specular to diffuse). Secondary effects, such as reflectance changes with incident angle, direct illumination of the detector with reflected radiation, intensity variations across the incident beam and positioning of the sample relative to the incident beam and sample port must also be considered.

In order to provide some information of these measurement problems, an analysis of reflectance (absorptance) measurements on 25 mm diameter collector pipes was completed. The analysis consisted of measuring the reflectance of several thin, opaque calibration materials both flat and wrapped around the collector pipe. Figure 2 shows the reflectance results for a piece of aluminized film, which represents a specularly reflecting sample. The two curves virtually overlap, indicating that all reflected radiation is collected by the integrating sphere. This result agrees with a geometrical calculation for the system as shown in Figure 1. Indeed, from this calculation, the specular component will be collected by the sphere for the present system until the pipe diameter becomes smaller than 16 mm. In addition, for the 25 mm diameter pipe, the maximum incident angle for radiation on the pipe, θ_{\max} , (see Figure 1) is $\sim 24^\circ$ from normal, while for a 16 mm diameter pipe, θ_{\max} is $\sim 36^\circ$ from normal.

The reflectance results for a diffusely reflecting sample are shown in Figure 2. The sample used for this measurement was a piece of non-glossy white paper, whose reflectance profile for normally incident radiation corresponds very closely to a perfectly diffuse reflector (i.e., follows the $\cos \theta$ law). Note that the reflectance from the curved sample is now approximately 2 to 3 percent lower than the reflectance from the flat sample and that this difference is approximately wavelength independent. Thus, the reflectance measured from a diffusely reflecting coating on a 25 mm diameter pipe using the present instrument will be 2-3 percent lower than the reflectance measured from an identical flat sample. Clearly, the most accurate results will be obtained when the reflectance

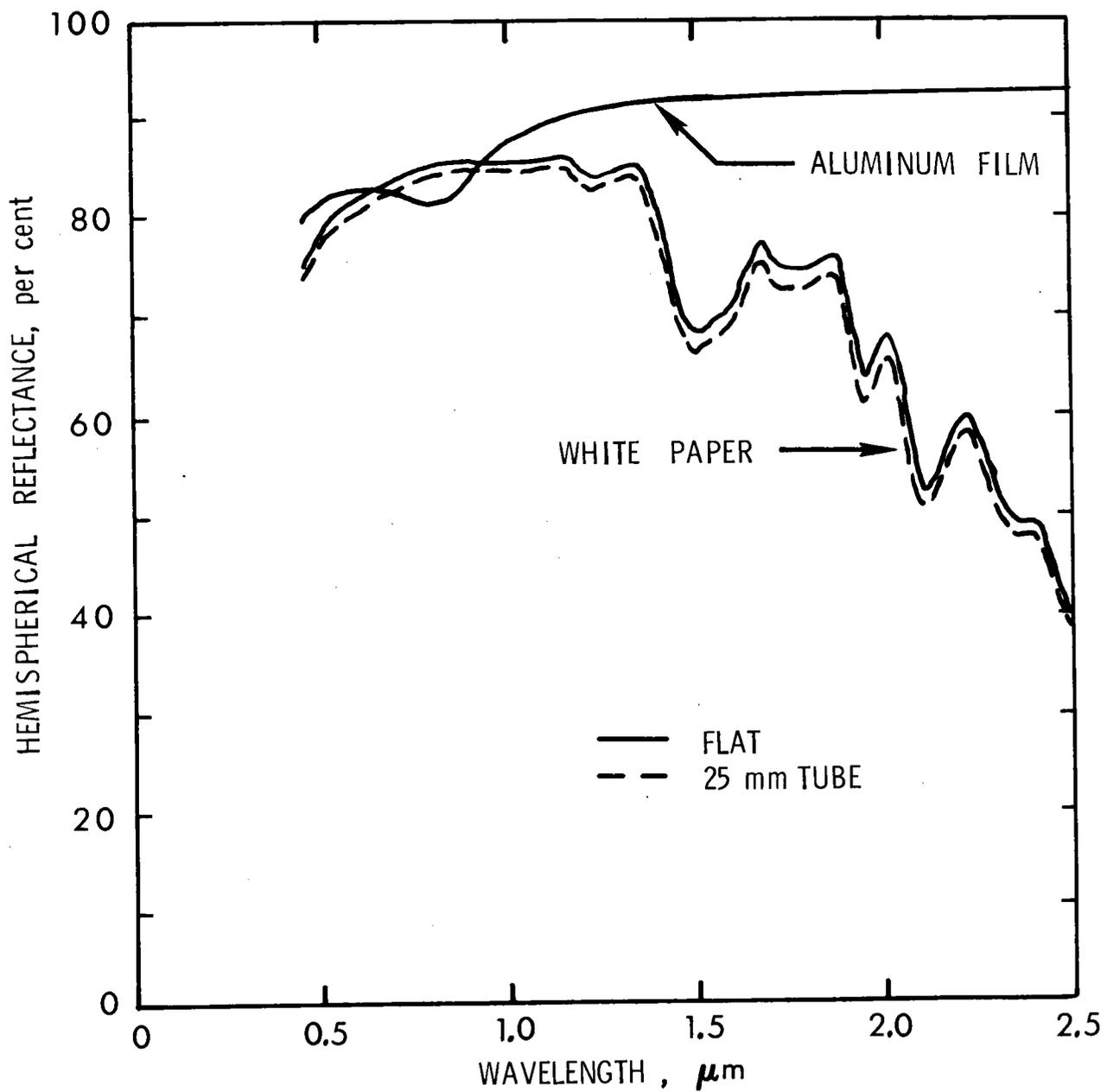


Figure 2. Hemispherical reflectance of aluminized film and white paper both as flat samples and as wrapped around a 25 mm diameter tube. Data overlap for aluminized film.

properties of the calibration material and the surface to be measured have the same scattering distribution.

With the arrangement described above, measurements of larger diameter pipes will result in less than 3% reflectance loss for diffusely reflecting samples. For smaller diameter pipes, the height of the incident beam can be decreased in order to decrease the amount of lost reflected radiation. Alternatively, a highly reflecting "light pipe" that redirects reflected radiation scattered outside the integrating sphere back into the sphere could be used to decrease the amount of lost radiation. However, in this case, any remaining reflectance loss might be wavelength dependent due to any wavelength dependence of the optical properties of the "light pipe."

Gier Dunkle Solar Reflectometer

Operation and Accuracy

The Gier Dunkle Solar Reflectometer (Model MS-251) is a portable instrument that measures the normal solar reflectance (or absorptance) of flat, opaque materials. This is accomplished by measuring the hemispherical reflectance averaged over the spectral distribution of a filtered Xenon lamp source. The Xenon lamp irradiance spectrum approximates an extraterrestrial solar spectrum⁴ so that the measured reflectance corresponds to an air mass zero⁵ (AM ϕ) solar average reflectance.

The instrument incorporates a measurement head which contains the Xenon lamp and its associated optical components, an integrating sphere and a thermocouple detector. The Xenon lamp measurement beam is directed into the sphere through an aperture in the sphere wall. With a sample placed over the measurement aperture, the measurement sequence consists of first obtaining a zero reading, V_0 , by blocking the beam before it enters the sphere. Next the 100% reading, V_{100} , is obtained by directing the beam onto the sphere wall. Finally the sample reading, V_s , is obtained with the beam directed onto the sample. The reflectance of the sample, ρ_s , is calculated from the formula $\rho_s = (V_s - V_0) / (V_{100} - V_0)$.

The spectral irradiance of the measurement beam at the sample measurement port of the instrument was measured over the wavelength range 350 nm to 1200 nm using an EG&G spectroradiometer (the accuracy is $\pm 20\%$) and is shown in Figure 3 together with a normalized AM ϕ solar spectrum. The spectral irradiance determined in this way includes the spectral distribution of the Xenon lamp together with the effects of the reflectance and transmittance properties of the optics. However, it does not include the spectral efficiency of the integrating sphere or the spectral response of the detector. The measured curve has been extrapolated outside the measured wavelength region using previously measured spectra for a Xenon lamp. ⁴

The spectrum shows the characteristic Xenon intensity peaks in the infrared region between 800 nm and 1000 nm which are not present in the AM ϕ solar spectrum. This can lead to some errors in the measured solar reflectance values, especially if the reflectance spectrum of the sample is selective. An indication of the magnitude of this discrepancy can be calculated by assuming a sample has the idealized step-function reflectance spectrum shown in the inset in Figure 4. The absorptance of this step function reflector is calculated for the Gier-Dunkle and compared to the AM ϕ solar average absorptance in Figure 4. A maximum deviation of 0.17 absorptance units occurs at $\alpha_s(\text{AM}\phi) = 0.60$ which corresponds to the reflectance spectrum step occurring at $\lambda = 830$ nm. However, for $\alpha_s(\text{AM}\phi) \geq 0.80$, the error is less than 0.05 absorptance units, while for $\alpha_s(\text{AM}\phi) \geq 0.90$, the error is less than 0.03 absorptance units. Approximately the same results apply for an AM1 solar spectrum. Therefore, as pointed out in the instruction manual, for spectrally selective samples the use of the various cutoff filters included with the instrument is advised.

The accuracy of the Gier Dunkle Solar Reflectometer was determined for measurements of the solar absorptance of electrodeposited black chrome coatings. The AM ϕ solar absorptance for each coating was calculated from spectral hemispherical reflectance data measured with a Beckman DK-2A spectroreflectometer over the wavelength range 350 nm to 2500 nm. The accuracy of these calculated values is ± 0.01 absorptance units. The black chrome coating was applied to two different nickel substrates:¹ (1) Zodiac nickel which has a bright, shiny surface and

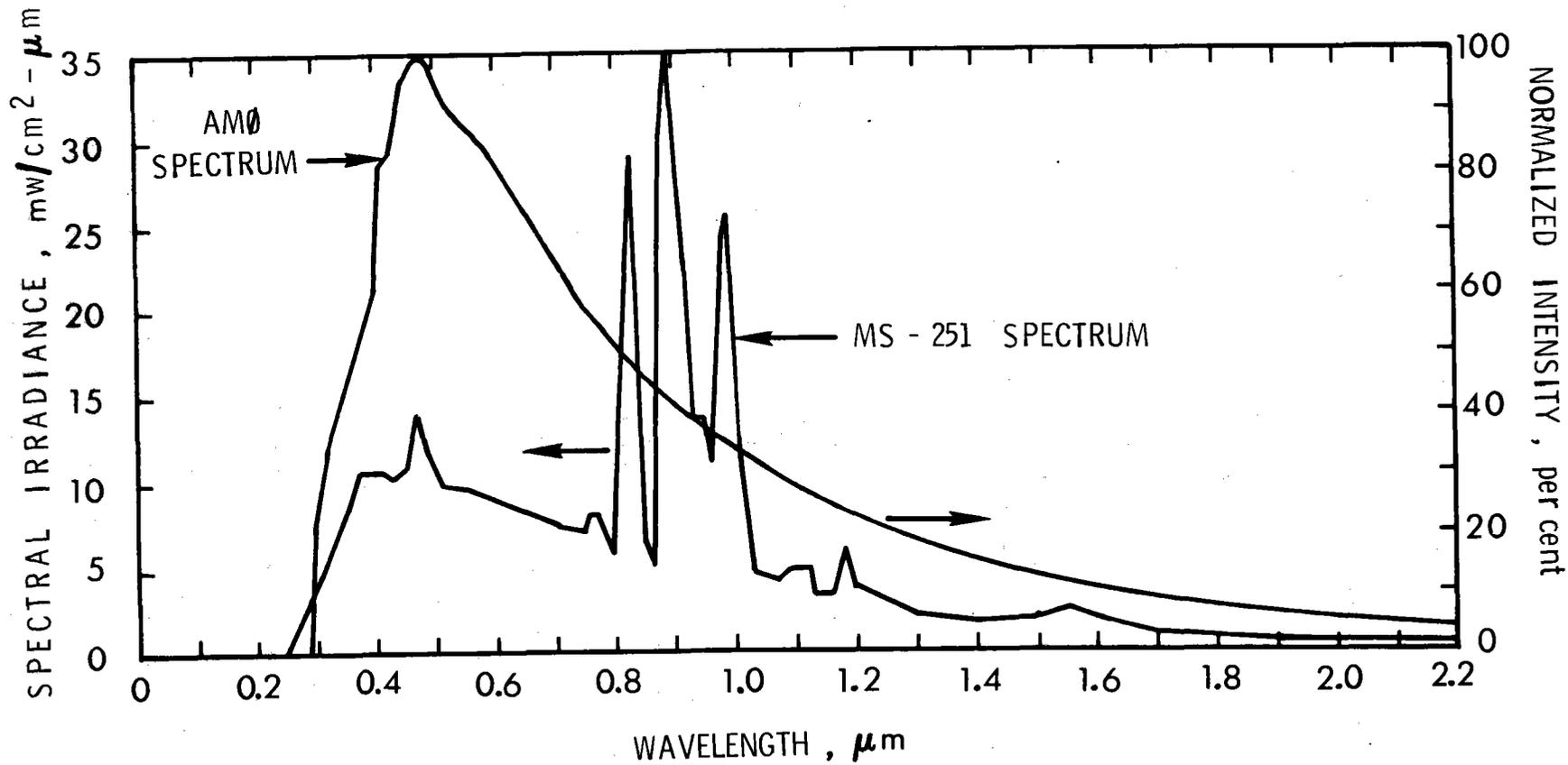


Figure 3. The spectral irradiance of measurement beam of the Gier Dunkle Solar Reflectometer, Model MS-251, together with a normalized air mass zero (AM0) solar spectrum.

(2) Perflow nickel which has a rough, dull surface. For the samples studied, the solar absorptance values ranged from 0.85 to 0.96 (see Figure 5). The average deviation of the absorptance values determined with the Solar Reflectometer was ± 0.013 absorptance units, which is within the measurement accuracy of the data. The maximum deviation from the AM \emptyset value was 0.03 absorptance units. Samples more than 0.015 absorptance units in error showed a rapid change in their reflectance spectrum near 900 nm. This is exactly the region where the Xenon lamp spectral peaks are located and where the maximum deviation in absorptance occurred for the idealized reflectance spectrum as shown in Figure 4. Therefore, solar absorptance values determined for samples which have a reflectance change near 900 nm may be in error by more than ± 0.01 absorptance units. However, for the samples with $\alpha_s(\text{MS-251}) \geq 0.94$, the true solar absorptance was always ≥ 0.93 .

Zero Offset:

As currently designed, there is a small, but significant, zero offset associated with the Gier Dunkle Solar Reflectometer. As a result, the reflectometer can indicate a solar reflectance as large as $\sim 10\%$ even for a perfectly absorbing sample (i.e., a sample whose solar reflectance is 0%). This zero offset results from a small amount of scattered radiation in the measurement beam as it enters the integrating sphere in the measurement head. With the measurement beam directed at the sample, this scattered radiation lies outside the sample measurement port and it is collected by the sphere and detected. The amount of scattered radiation is not included in the zero adjustment procedure since the entire beam (i.e., scattered plus central beam) is blocked from the sphere during this adjustment. The scattered radiation is also detected during the 100% adjustment procedure. A method for determining the amount of scattered radiation and incorporating its value in the measurement procedure is outlined below. Because the scattered radiation probably results from dust accumulation on the optical components which direct the beam into the sphere, the zero offset will most likely increase slowly with time. However, it may also show sudden changes depending upon abrupt dirt accumulation or removal as a result of handling the measurement

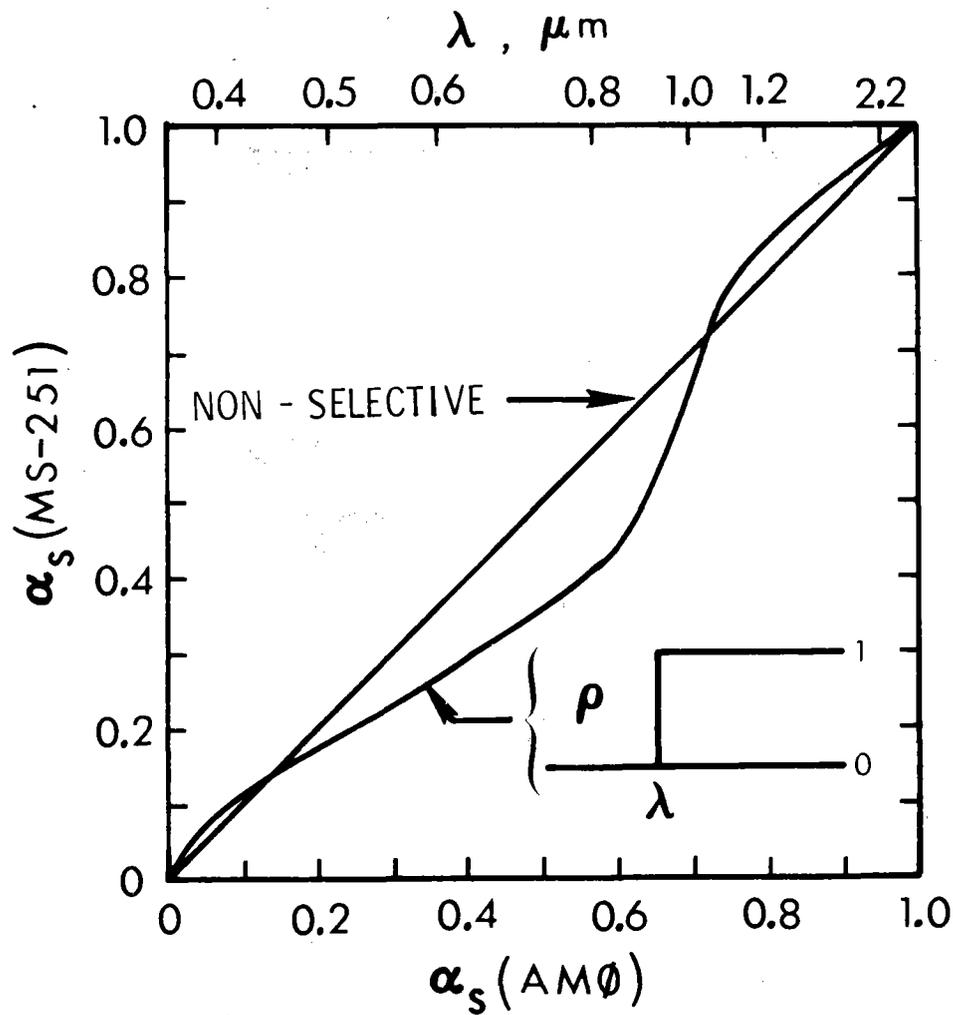


Figure 4. Comparison of AM0 solar absorptance with the solar absorptance measured with the MS-251 for an idealized step-function reflectance spectrum shown in the insert, and for a non-selective absorber.

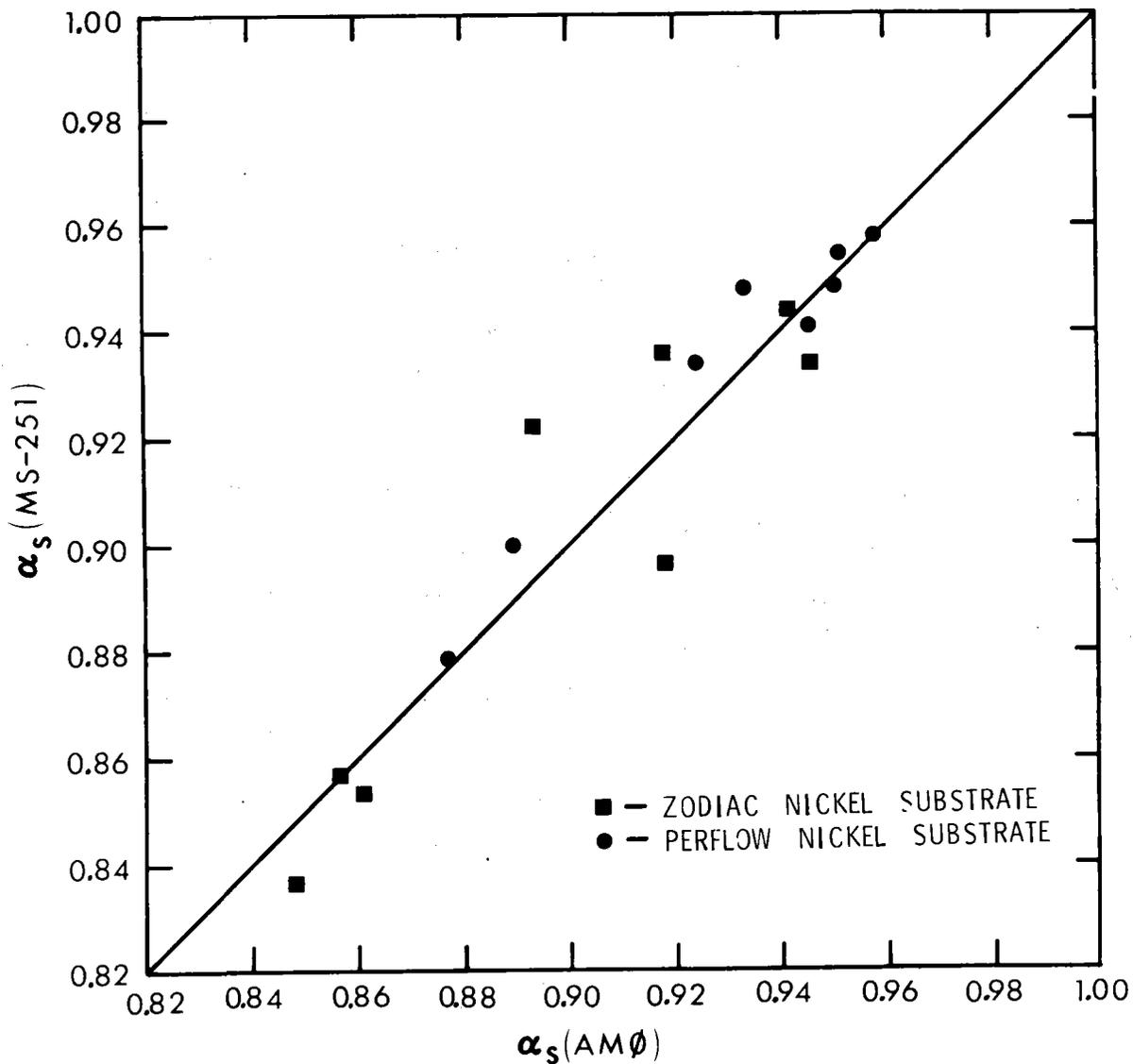


Figure 5. Comparison of AM ϕ solar absorptance with the solar absorptance measured with the MS-251 for various electrodeposited black chrome coatings on both Zodiac and Perflow nickel substrates.

head. Therefore, it is recommended that the zero offset be checked before each measurement or series of measurements. The existence of this zero offset is not presently described in the instruction manual supplied with the instrument.

The simplest method for determining the zero offset is accomplished by leaving the sample measurement port open (i.e., letting the measurement beam illuminate the room) and proceeding with the usual measurement sequence. We have found that the room lights in a normal laboratory have no effect on the offset measurement. [Alternatively, a "light trap" fabricated from a black material (i.e., black over the spectral range of the measurement) can be used as a 0% reflectance sample.] The reading obtained in this way represents the residual zero offset of the instrument. In order to incorporate the zero offset in subsequent sample measurements, the offset should be added to the average zero readings which are obtained during the normal sample measurement sequence (see instruction manual). Thus, if V_o is the average sample zero readings, V_{100} the average sample 100% readings, V_s the sample reading and V_{off} the previously determined zero offset, the corrected sample reflectance, ρ_s , is calculated from the formula

$$\rho_s = \left[\frac{V_s - (V_o + V_{off})}{V_{100} - (V_o + V_{off})} \right] \quad (1)$$

This correction has been applied to all data reported here.

Pipe Measurements:

The Gier Dunkle Solar Reflectometer is designed to measure the reflectance of flat samples larger than ~15 mm in diameter. Special procedures must be employed in order to measure the solar reflectance of cylindrical or otherwise curved samples. As with the Beckman Spectro-reflectometer, the main source of error is the radiation reflected by the sample that is not collected by the integrating sphere located in the measurement head. Any lost radiation will be a function of the sample curvature, the sample's reflectance distribution function and the distance between the sample and the measurement port.

The use of a mounting fixture to position the sample over the sample measurement port is important. The main requirements of the fixture are to position samples as close to the measurement port as possible in order to eliminate lost reflected radiation and to position samples in a reproducible manner so that any correction factor can be consistently applied.

The calibration procedure is similar to the procedure used to calibrate the Beckman Spectroreflectometer. First, the reflectance of a flexible, opaque calibration sample is measured both with the sample flat, R_f , and with the sample wrapped around the curved surface to be measured, R_c . The correction factor, C , is then calculated from the ratio:

$$C = \frac{R_f}{R_c} \quad (2)$$

Note that C should always be greater than 1.0. The reflectance determined for the unknown sample is then multiplied by C in order to obtain the corrected reflectance value.

Because the mounting fixture may reflect radiation back into the sphere, a zero offset should be determined in the usual manner with the mounting fixture in place. The reflectance distribution function of the calibrating sample can influence the correction factor. For example, with a 25 mm diameter cylindrical sample, a specularly reflecting calibration sample (e.g., aluminized plastic film) has a correction factor of 1.00 while a diffusely reflecting calibration sample (e.g., white paper) has a correction factor of 1.04. Spacing the 25 mm diameter sample 1.25 mm away from the measurement port changes the correction factor to 1.15 for the specular calibration sample and 1.22 for the diffuse calibration sample.

Gier Dunkle Infrared Reflectometer

Operation:

A Gier Dunkle Infrared Reflectometer is a portable instrument that measures the normal infrared reflectance (or emittance) of flat, opaque materials.⁶ The measurement head incorporates a rotating cavity mounted below the 20 mm diameter sample measurement port. The inside of the

cavity is divided into two semicylindrical chambers, which are painted black. The temperature of one chamber is raised to approximately $T_h = 45^\circ\text{C}$ (113°F) while the other chamber is unheated but reaches a steady state temperature of approximately $T_c = 33^\circ\text{C}$ (91°F). As the cylinder rotates, a sample placed over the measurement port is alternately irradiated with a black body radiation spectrum corresponding to the temperature of each cavity. The optical detector system is positioned so that it views the sample in a near-normal direction from the bottom of the rotating cylinder. Thus the thermocouple detector receives both infrared radiation emitted by the sample together with the infrared radiation reflected by the sample from each semicylindrical cavity. By amplifying only the alternating component from the detector output, a signal, S , is generated which is given by

$$S = \int \rho_\lambda(T_s) L_\lambda(T_h) d\lambda - \int \rho_\lambda(T_s) L_\lambda(T_c) d\lambda \quad (3)$$

where $\rho_\lambda(T_s)$ is the spectral hemispherical reflectance of the sample at a temperature, T_s , near room temperature, and $L_\lambda(T)$ is the spectral irradiance of a blackbody at a temperature T . $L_\lambda(T)$ is given by

$$L_\lambda(T) = \frac{C_1}{\pi \lambda^5} \left[\exp\left(\frac{C_2}{\lambda T}\right) - 1 \right]^{-1} \quad (4)$$

where

$$C_1 = 3.7415 \times 10^{-16} \text{ W} \cdot \text{m}^2 \quad (5)$$

$$C_2 = 1.4388 \times 10^{-2} \text{ m} \cdot \text{K}$$

The integration region in equation (3) is over the wavelength range of the optical detection system which is approximately $0.25 \mu\text{m}$ to $30 \mu\text{m}$.

Equation (3) can be rewritten so that

$$S = \int_{0.25 \mu\text{m}}^{30. \mu\text{m}} \rho_\lambda(T_s) \left[L_\lambda(T_h) - L_\lambda(T_c) \right] d\lambda \quad (6)$$

Thus the sample reflectance is averaged over a spectral distribution which is given by the difference between blackbody spectral irradiance curves

at the two cavity temperatures. This distribution, appropriately normalized, very closely approximates a 100°C blackbody spectrum as shown in Figure 6. From the figure, it can be seen that both curves have a maximum at $\sim 7.7 \mu\text{m}$ although the relative intensity of the 100°C blackbody spectrum is slightly higher at wavelengths away from the maximum. The effect of this difference for non-grey samples can be estimated by considering the idealized step-function reflectance spectra inset in Figure 7. A comparison of the true emittance at 100°C to the emittance measured with the Gier Dunkle Infrared Reflectometer for these idealized reflectance spectra show a maximum deviation of only 0.044 emittance units. Note that the maximum deviation occurs at $\lambda_1 = \lambda_2 = 13 \mu\text{m}$. The accuracy of the instrument for a variety of samples will be discussed in a later section.

As normally manufactured, the instrument includes a polyethylene filter in the optical path between the sample and the detector. The purpose of this filter is to shift the approximately 100°C blackbody measurement spectrum toward a lower temperature blackbody spectrum. The transmittance of this filter from 2.5 to 15 μm together with the resulting measurement spectrum and a 25°C (approximately room temperature) blackbody spectrum are shown in Figure 8. The transmittance of the filter varies approximately linearly from 15% transmittance at 2.5 microns to 55% transmittance at 15 microns, except for the characteristic polyethylene absorption bands at 6.9, 7.4, 9.3 and 14 μm . Although the measurement spectrum has been shifted to longer wavelengths, the agreement with a 25°C blackbody spectrum is only approximate. This can result in larger errors for non-grey samples than those calculated for the instrument without this filter. Therefore, more accurate results will be obtained for non-gray samples if the instrument is operated without the polyethylene filter. In addition, the 100°C blackbody measurement spectrum obtained without this filter corresponds to the operating temperature of many flat plate collectors.

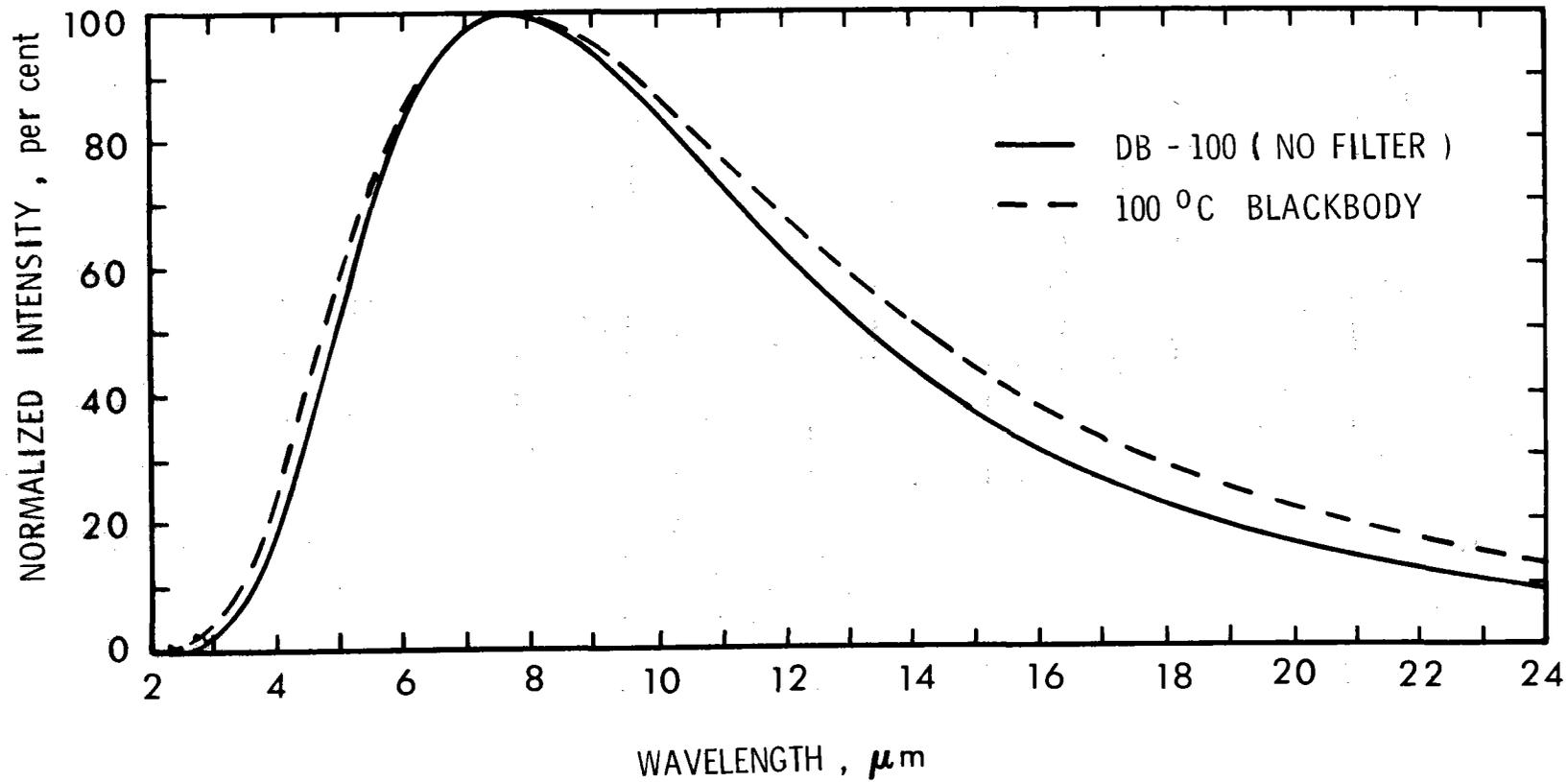


Figure 6. The measurement spectrum of the Gier Dunkle Infrared Reflectometer, Model DB-100, (no filters) together with a normalized 100°C blackbody spectrum.

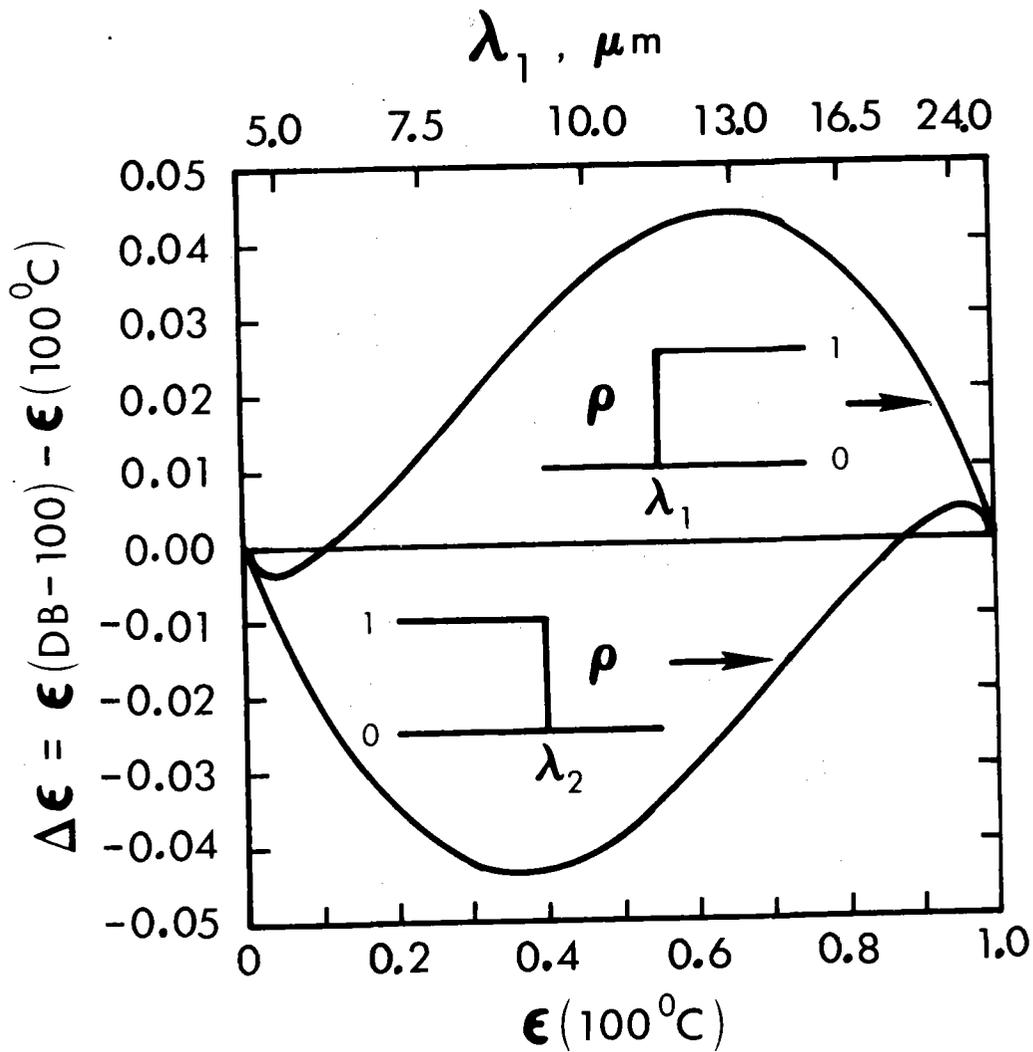


Figure 7. Comparison of true emittance with the emittance measured with the DB-100 (no filters) for the idealized step-function reflectance spectrum shown in the insert.

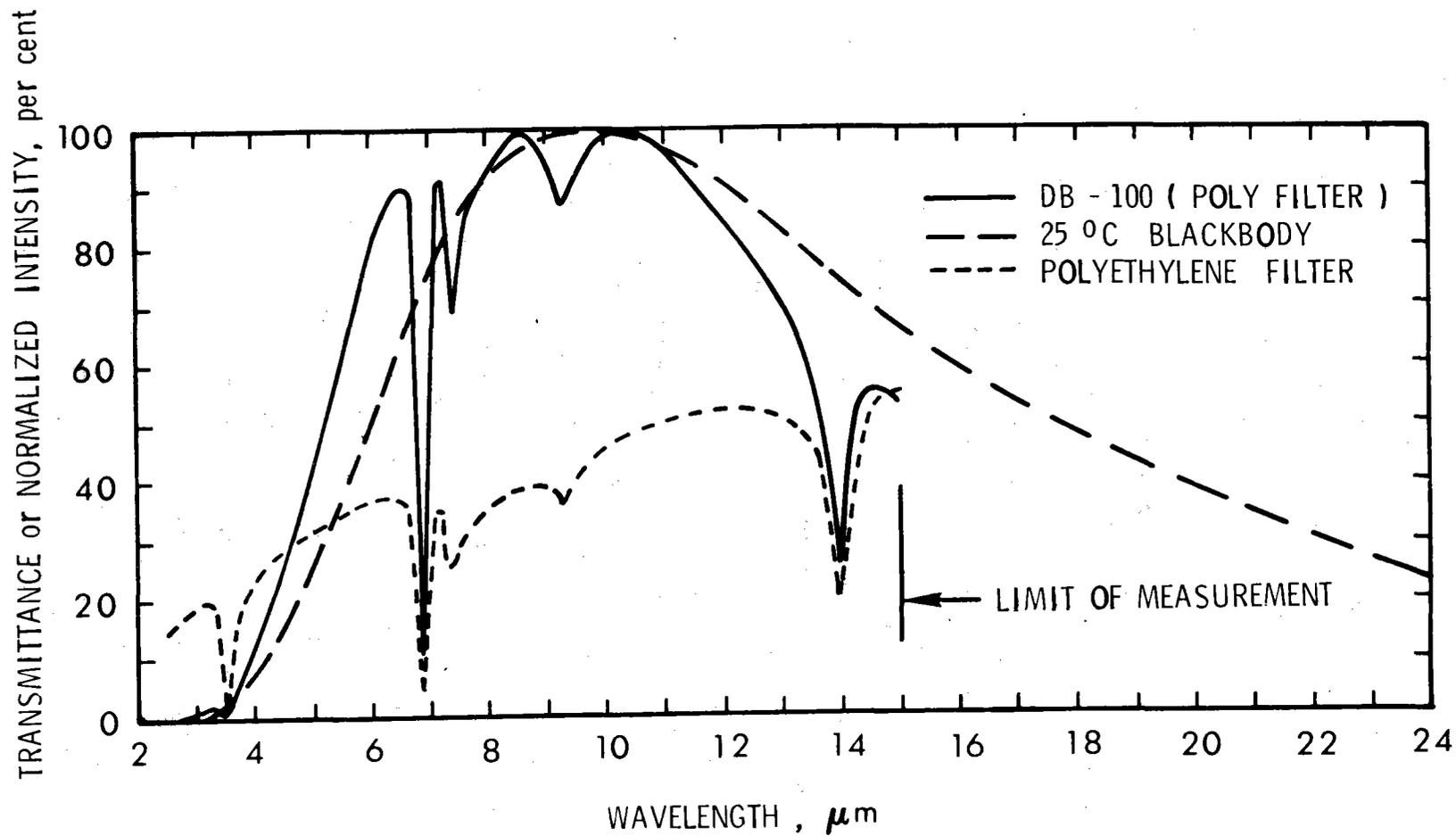


Figure 8. DB-100 measurement spectrum with the polyethylene filter in the optical system, the transmittance properties of the filter and a normalized 25°C blackbody spectrum as a function of wavelength.

Sapphire Filter:

Because the operating temperature of many solar collectors, including Sandia's cylindrical-parabolic collector,² is much greater than 100°C, it is important to be able to measure the emittance of coated receiver surfaces at their operating temperature. Although an emittance value can be obtained at 25°C or 100°C using a Gier Dunkle Infrared Reflectometer, small errors in these values can lead to larger errors in the projected emittance value at 300°C, for example. This is particularly true for the electrodeposited black chrome solar coating. As previously shown,¹ the total hemispherical emittance of this coating is a linear function of the temperature, within experimental error, between 20°C and 320°C. However, the slope of the linear relationship increases as the plating time or coating thickness increases. Thus, for black chrome deposited on sulfamate nickel, the difference between emittance values for 1 minute and 5 minute platings is 0.03 emittance units at 20°C, while at 300°C the difference is 0.09 emittance units. Thus an error of 0.01 emittance units in the 20°C emittance value can result in a 0.03 emittance error in the projected 300°C emittance value for black chrome.

In order to obtain an indication of the 300°C emittance using the Gier Dunkle Infrared Reflectometer, a filter which shifts the measurement spectrum to shorter wavelength can be used. There are several methods that can be employed. First, a filter whose transmittance decreases approximately linearly with increasing wavelengths will shift the measurement spectrum to lower wavelengths (opposite to the polyethylene filter). Properly chosen, the resulting spectrum maximum could be made to correspond to the maximum of a 300°C blackbody ($\sim 5 \mu\text{m}$). A second technique would use a cutoff filter which excludes the long wavelengths from the measurement spectrum. Because this spectrum would not correspond to a 300°C blackbody, the instrument would have to be calibrated and tested for each coating system studied. In addition, one must be careful that there is ample signal for the measurement.

The filter found to be most effective for the black chrome coating is a polished sapphire window approximately 1 mm thick. The

transmittance of the sapphire is 85% out to $\sim 8 \mu\text{m}$ where the transmittance drops to 0%. With the sapphire mounted in the optical path between the sample and detector, the normalized measurement spectrum has a peak at $\sim 5.8 \mu\text{m}$ and a full width at half maximum (FWHM) of $\sim 2.4 \mu\text{m}$ (see Figure 9). Note that the peak of this spectrum is very close to the $5.0 \mu\text{m}$ peak of 300°C blackbody.

The infrared reflectance of several black chrome plated sulfamate nickel substrates were measured with the sapphire filter mounted in the instrument. The total hemispherical emittance at 300°C , $\epsilon_{T,H}(300^\circ\text{C})$, of each sample was determined using a calorimetric heat-balance technique as previously described.⁷ The maximum error in $\epsilon_{T,H}(300^\circ\text{C})$ for these samples is less than ± 0.015 emittance units. In addition, the reproducibility of the DB-100 measurements is ± 0.01 emittance units. The relationship between the two emittance values is approximately linear over a wide range of $\epsilon_{T,H}(300^\circ\text{C})$ values, as shown in Figure 10. The two values agree at approximately $\epsilon_{T,H}(300^\circ\text{C}) = \epsilon_{T,N}(\text{DB-100}) = 0.18$; above this value $\epsilon_{T,N}(\text{DB-100}) > \epsilon_{T,H}(300^\circ\text{C})$, while below this value $\epsilon_{T,H}(300^\circ\text{C}) > \epsilon_{T,N}(\text{DB-100})$. Assuming a linear relationship, the equation relating the two emittance values is given by

$$\epsilon_{T,H}(300^\circ\text{C}) = 0.768 \epsilon_{T,N}(\text{DB-100}) + 0.042 \quad (7)$$

over the range studied: $0.05 \leq \epsilon_{T,N}(\text{DB-100}) \leq 0.44$.

The DB-100 modified with the sapphire filter has been used to provide both absolute 300°C emittance values and uniformity checks on the black chrome plated receiver tubes used in Sandia's Solar Total Energy Test Facility.²

It should be pointed out that the infrared reflectance measured in this way for a different solar selective coating will undoubtedly have a different relationship to $\epsilon_{T,H}$ at the temperature of interest. Indeed, the relationship may not be linear as in the present case for black chrome. There are several other filter materials that are readily available for use as a cutoff filter. These materials are listed in Table I together with the measurement spectrum parameters when used with the DB-100. Note

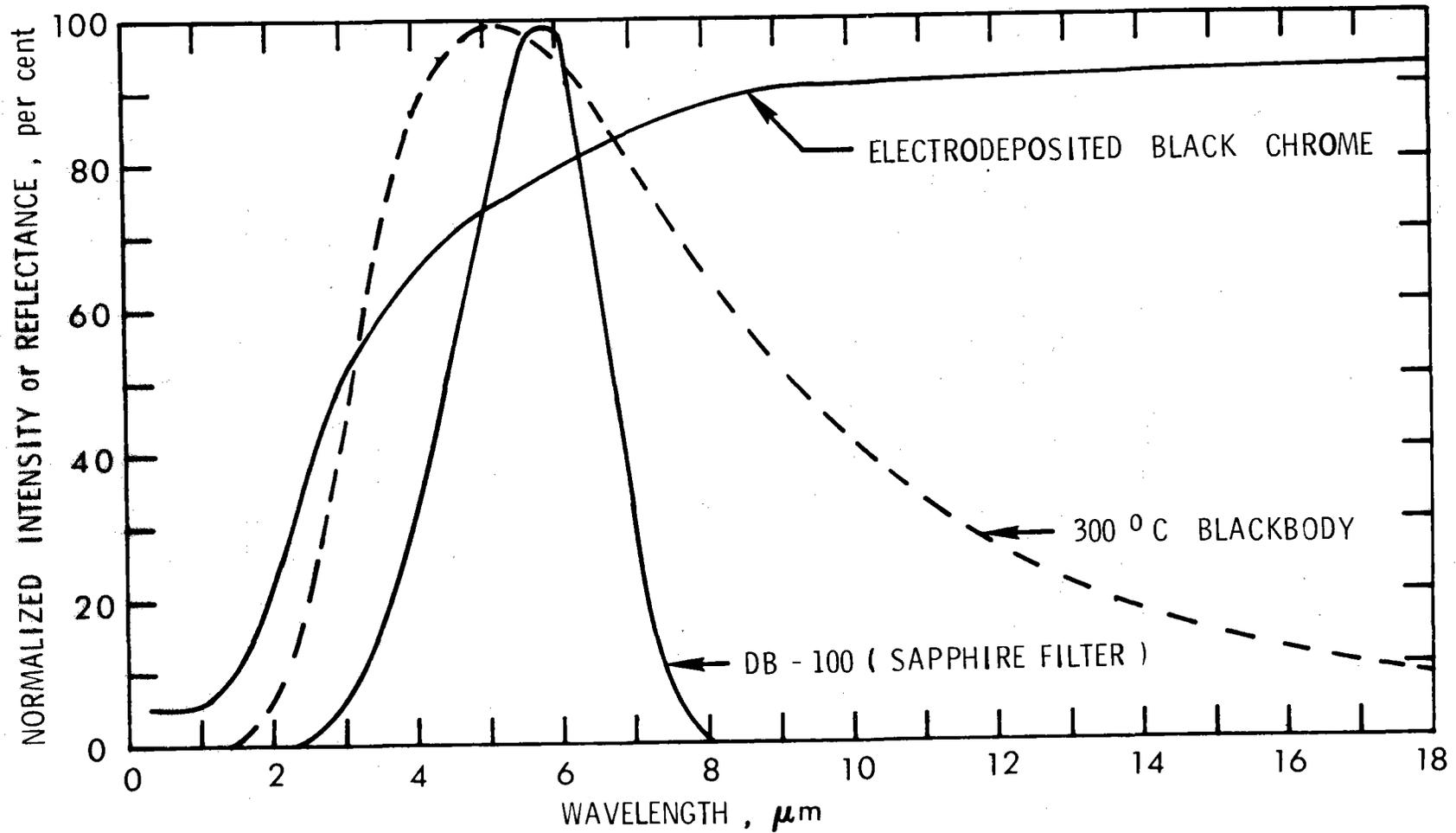


Figure 9. DB-100 measurement spectrum with a sapphire filter in the optical system together with a normalized 300°C blackbody spectrum and the reflectance spectrum for electrodeposited black chrome on nickel.¹⁰

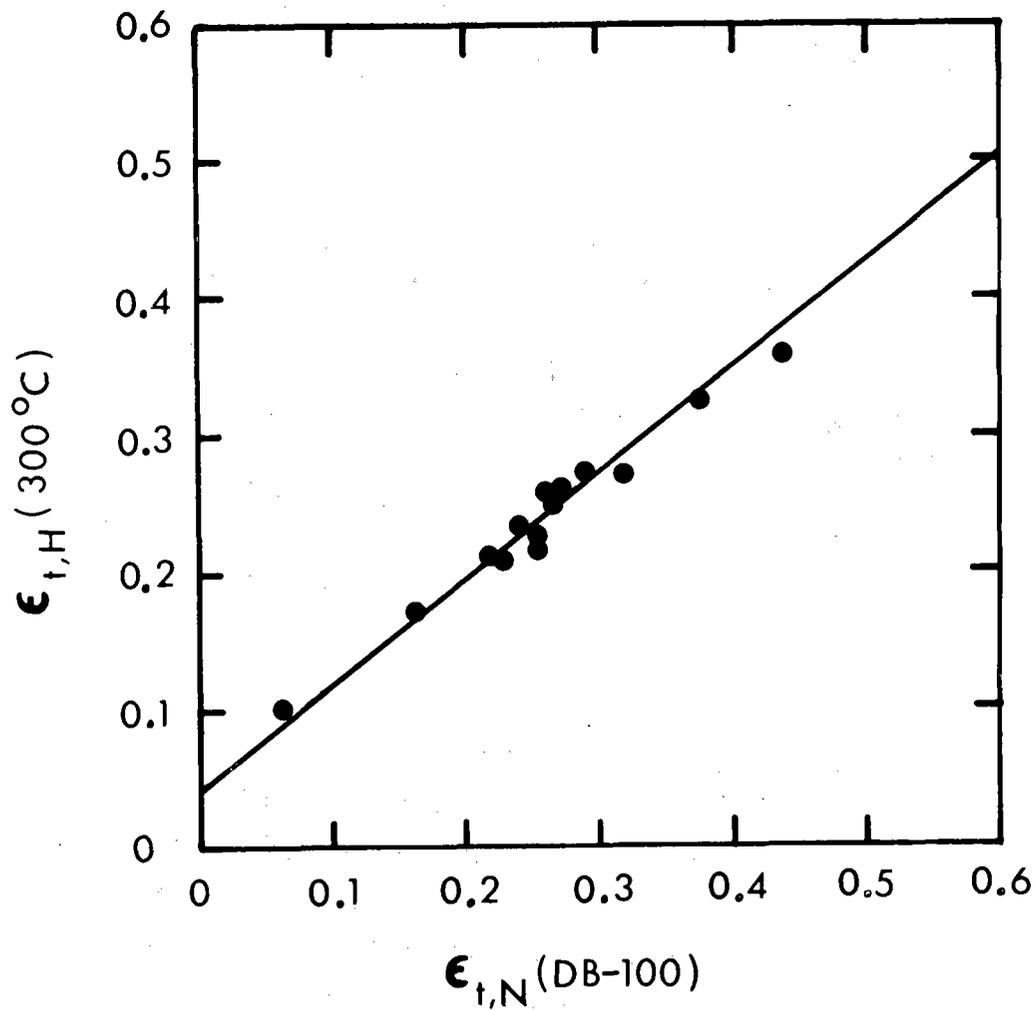


Figure 10. Comparison of $\epsilon_{t,H}(300^\circ C)$ with the emittance measured with the sapphire filter mounted in the optical system of the DB-100 for several black chrome plated sulfamate nickel samples.

that both the peak wavelength and the bandwidth of the measurement spectra are larger than the parameters obtained with the sapphire filter. A comparison between the $\epsilon_{T,H}$ (300°C) and the reflectance values determined with the DB-100 with each of these filters for the black chrome coating have not been determined.

Table I.
Measurement Spectrum Parameters for Several Materials
When Used with a Gier-Dunkle Infrared Reflectometer,
Model DB-100

<u>Material</u>	<u>Wavelength at Maximum (μm)</u>	<u>Bandwidth FWHM (μm)</u>
Sapphire	5.8	2.4
Irtran #1	6.8	3.1
Irtran #3	7.7	5.6
Irtran #5	6.6	4.3

Pipe Measurements:

Because the Gier Dunkle Infrared Reflectometer is designed to accommodate flat samples larger than 20 mm in diameter, special procedures are used in order to measure the reflectance properties of cylindrical or otherwise curved surfaces. Because the instrument is calibrated through the use of both a high and low reflectance standard, the main requirement for measuring curved surfaces is calibration standards with the same curvature as the sample. This is easily accomplished by mounting a thin, flexible material, whose reflectance has been previously measured with the instrument, on the surface to be measured and using it as a secondary calibration standard. A convenient high reflectance secondary standard is an aluminum or gold coated plastic film; a low reflectance secondary standard is a plastic film coated with 3M-101 C10 black velvet paint. As with other calibrations of this type, the highest accuracy is obtained if the reflectance distribution properties of the secondary standard are similar to the surface to be measured. For a 38 mm diameter pipe, we have found that the decrease in the reflected radiation for the pipe geometry is only $\sim 5\%$ which is easily compensated for using the flexible secondary standards.

Accuracy of 100°C Measurements:

The accuracy of the Gier Dunkle DB-100 operating without a filter in the optical system, so that the measurement corresponds closely to a 100°C blackbody spectrum, has been checked for a variety of materials. The measured emittance values were compared to total hemispherical emittance values, $\epsilon_{T,H}$, determined from calorimetric measurements at 100°C.⁷ The results are shown in Figure 11; the error bars represent the error in the measured $\epsilon_{T,H}$ values. The stated accuracy of the DB-100 is ± 0.01 emittance units for a non-selective sample (see manufacturers instruction manual). Because the DB-100 measures the total, normal emittance, $\epsilon_{T,N}$, the theoretical relationship between $\epsilon_{T,N}$ and $\epsilon_{T,H}$ for both metallic and insulating surfaces⁸ is also shown in Figure 11. Thus, for metallic surfaces $\epsilon_{T,H} > \epsilon_{T,N}$, while for an insulating surface $\epsilon_{T,N} > \epsilon_{T,H}$ for emittance values above 0.60. Some of the materials and their emittance values are shown in Table II.

Note that the results for all the materials are within the measurement accuracy of the two techniques. In addition, the results follow the trends of the theoretical predictions for the metallic and insulating substrates. If we restrict ourselves to only the electrodeposited black chrome coating deposited on electroplated nickel substrates, then the agreement is better than ± 0.02 emittance units for fifteen samples over the range $0.08 \leq \epsilon_{T,N} \leq 0.39$. A more thorough discussion of emittance measurement techniques and their associated errors has been previously reported.⁹

Conclusions

Special calibration and measurement procedures, developed for the various optical equipment, allow for the determination of the optical properties of coatings applied to cylindrical surfaces. The measurement spectrum presented here for both the Gier Dunkle Solar Reflectometer and Infrared Reflectometer can be used to indicate possible measurement errors for spectrally selective materials. The zero offset associated with the Solar Reflectometer is easily determined and incorporated in the measurement procedure. In addition, the Infrared Reflectometer can be modified so that the 300°C emittance properties of the black chrome coating can be determined.

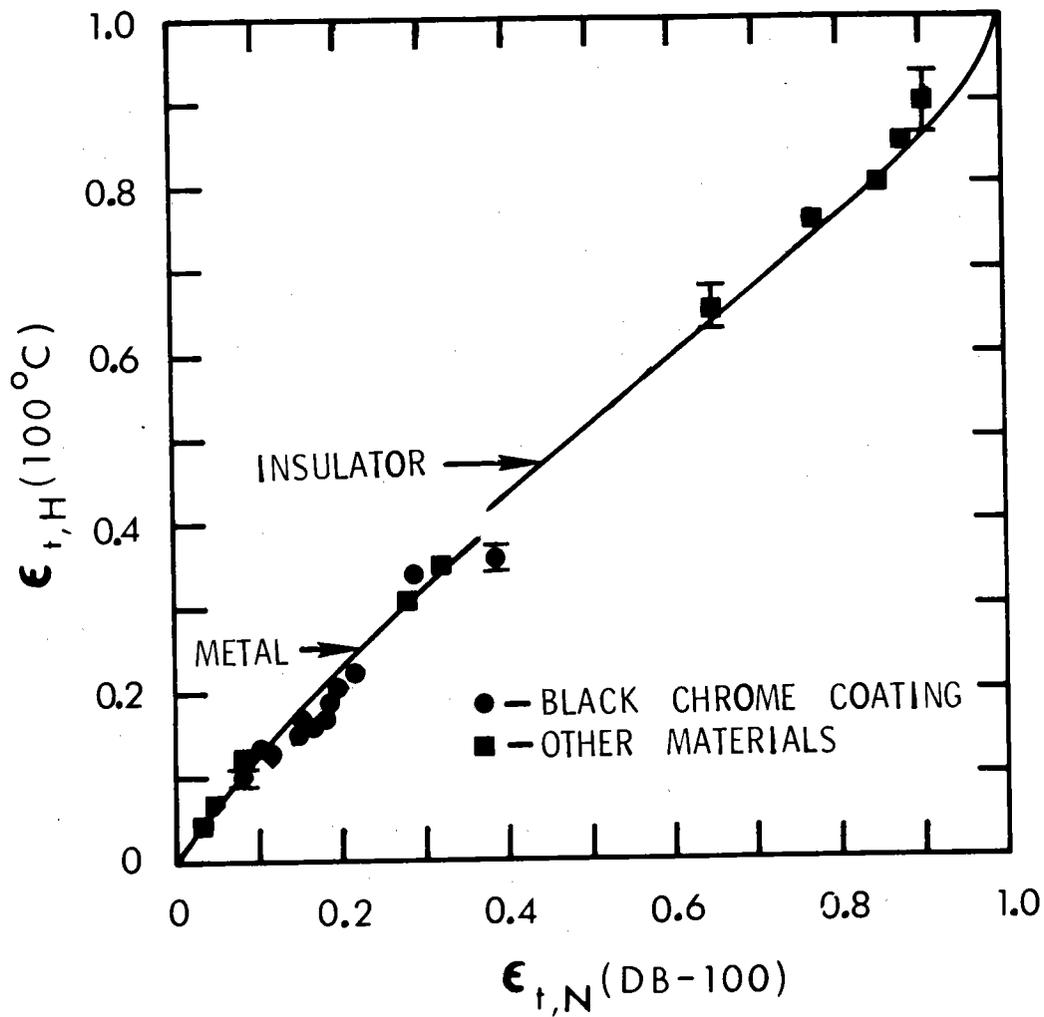


Figure 11. Comparison of $\epsilon_{T,H}(100^\circ\text{C})$ with the emittance measured by the DB-100 (no filter) for several materials, together with the theoretical relationship between $\epsilon_{T,H}$ and $\epsilon_{T,N}$ for metallic and insulating surfaces.

Table II. Emittance Values for Selected Materials

<u>Material</u>	<u>$\epsilon_{T,H}$ (100°C)</u>	<u>$\epsilon_{T,N}$, DB-100</u>
1) Electrodeposited Gold	0.03 ± .01	0.03
2) Electrodeposited Nickel	0.06 ± .01	0.05
3) OCLI Solar Selective Coating	0.12 ± .01	0.07
4) Electrodeposited Black Chrome on Nickel (several samples)	0.10 ± .01 0.15 ± .01 0.22 ± .01 0.36 ± .015	0.08 0.17 0.21 0.39
5) Ebanol "C" Treatment of Copper	0.65 ± .025	0.65
6) Ge Pigmented DC-805 Silicon Binder on Steel ¹	0.76 ± .03	0.77
7) Black Porcelain Enamel on Steel	0.80 ± .03	0.85
8) Dow Corning Thermal Control Coating 92-007	0.85 ± .035	0.88
9) 3M-401 Black Velvet Paint	0.90 ± .035	0.91

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