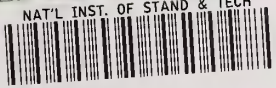


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NBS SPECIAL PUBLICATION 260-38

Standard Reference Materials:

PREPARATION AND CALIBRATION OF STANDARDS OF SPECTRAL SPECULAR REFLECTANCE

U.S.
DEPARTMENT
OF
COMMERCE

National
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² Part of the Center for Radiation Research.

³ Located at Boulder, Colorado 80302.

Addendum to NBS Special Publication 260-38
Preparation and Calibration of Standards of
Spectral Specular Reflectance

Polarization effects were believed to be negligible, and hence were ignored in the calibration of the standards of specular spectral reflectance. This belief was based on (1) the small angle of incidence on the test sample, about 9° from normal, and (2) the use of an averaging sphere in front of the entrance slit of the monochromator, which eliminated errors due to polarization effects in the monochromator.

It has since been brought to our attention^{1/} that, with gold, polarization errors are not negligible in the ultraviolet at angles of 15° or more from the normal, such as are used in many reflectometers, and may not be negligible even at the incidence angle of 9° . Computations by Luedke^{1/} using the published values of $n = 1.40$ and $K = 1.88$ for gold at $0.45 \mu\text{m}$, showed that for a plane polarized incident beam at 10° from normal $R_s = 0.4038$, $R_p = 0.3915$ and $\bar{R} = 0.3976$

The angle of incidence on mirror M1 (figure 3), which was of aluminum, was about 10° , and that on mirror M6, which was also of aluminum, was about 40° from normal. The mercury arc source used for these measurements should be free from any significant polarization introduced by mirrors M1, M3, or M4 and M6 would be in the direction normal to the plane of incidence, and hence would tend to give values that are slightly higher than would be obtained with a completely unpolarized incident beam.

Luedke^{1/} found the discrepancies to exist only at wavelengths shorter than $0.5 \mu\text{m}$, where the reflectance of gold falls off rapidly to about 0.35. Good checks were obtained at longer wavelengths, which suggests that the polarization errors are absent at the longer wavelengths.

^{1/}Personal communications from E. E. Luedke, Head, Thermophysics Section, Systems Group of TRW, Inc.

Similar discrepancies were noted by Champetier^{2/}, who ascribed the errors to scattering. Scattering becomes much more pronounced at short wavelengths than at longer wavelengths.

Because of the discrepancies noted above, anyone using the spectral specular reflectance standards under conditions other than those used in the calibration is cautioned to be aware that polarization effects and scattering can cause significant errors, particularly in the ultraviolet portion of the spectrum.

^{2/}Personal communication from R. J. Champetier, The Aerospace Corp.



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Standard Reference Materials:

Preparation and Calibration of Standards of Spectral Specular Reflectance

J. C. Richmond and J. J. Hsia

Institute for Basic Standards
U.S. National Bureau of Standards
Washington, D.C. 20234

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PREFACE

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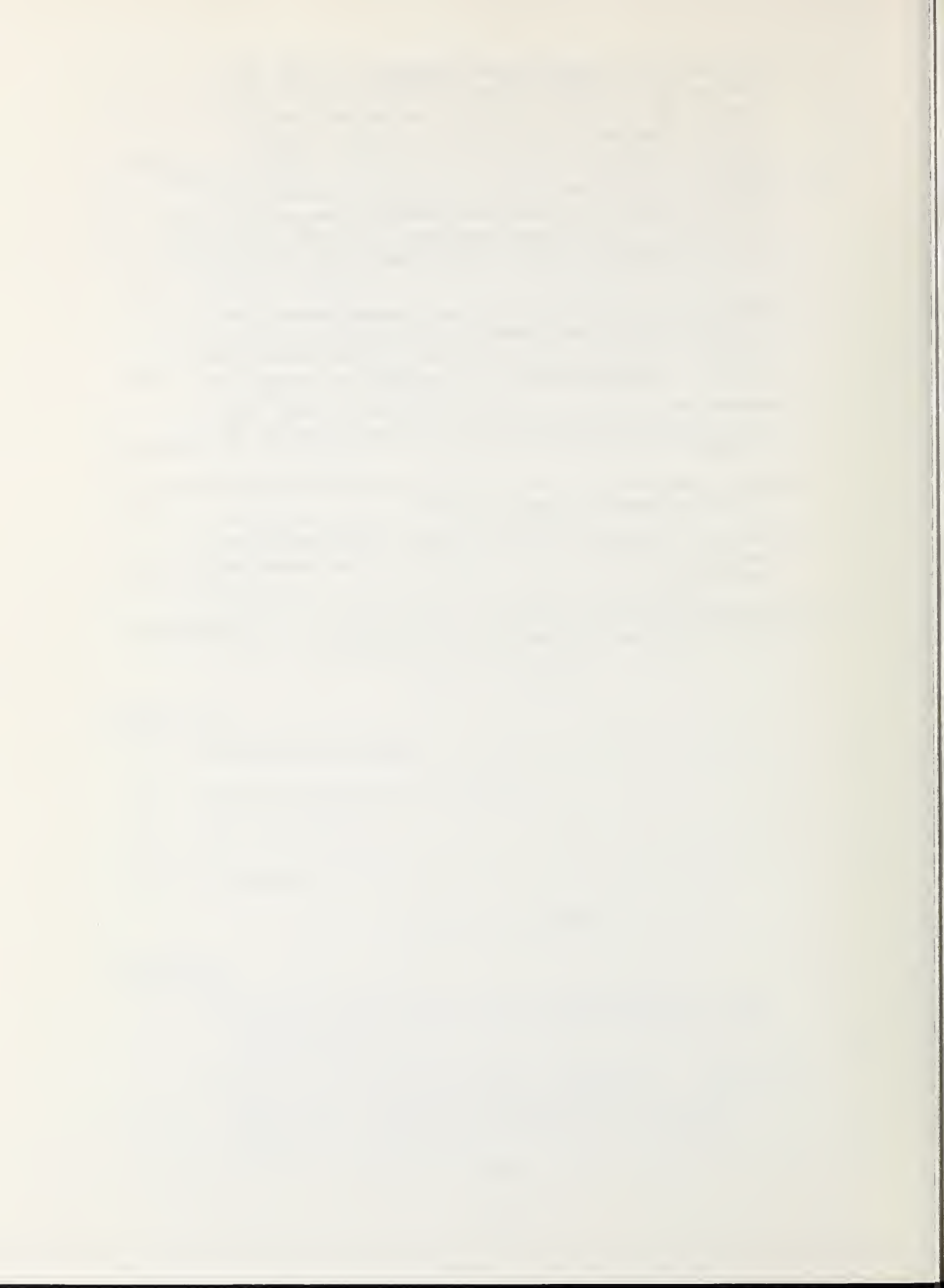
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PREPARATION AND CALIBRATION OF STANDARDS OF
SPECTRAL SPECULAR REFLECTANCE

J. C. Richmond and J. J. Hsia

Institute for Basic Standards
National Bureau of Standards
Washington, D. C. 20234

This report describes in detail the preparation and calibration of Standard Reference Materials 2001 through 2008. These standards of spectral specular reflectance were prepared commercially by vacuum depositing opaque coatings of gold and aluminum, respectively, onto polished substrates of fine-annealed borosilicate glass. The near-normal (9° from normal) spectral specular reflectance was measured by the multiple-reflection technique. Four different combinations of source, spectrometer, and detector were used to cover the wavelength range from about $0.25 \mu\text{m}$ to $30 \mu\text{m}$ and beyond.

Key words: Aluminum; gold; infrared reflectance; interferometer spectrometer; mirrors; reflectance; reflectance standards; spectral reflectance; spectral specular reflectance; specular reflectance; standards; ultraviolet reflectance.

1. INTRODUCTION

There has been a greatly increased interest in absolute reflectance measurements since the advent of Sputnik I. Much of the interest has been in the aerospace industry, where absolute spectral reflectances are required for radiant heat transfer analysis in computing heat transfer rates to and from satellites and space vehicles in space, where radiation is the only mode of heat transfer that does not also involve mass transfer. Such computations are an essential part of the thermal design of such vehicles. The wavelength range of interest extends from the lower limit of the solar range (about $0.25 \mu\text{m}$ for heat transfer purposes) out to at least $30 \mu\text{m}$. Other applications include remote sensing, where the wavelengths involved are primarily in the infrared

out to about 15 μm , solar energy conversion, industrial radiant heat transfer and appearance measurement. Standards are required primarily for calibration of reflectometers.

Absolute spectral reflectance is defined as the ratio of all the radiant flux reflected by a sample to that incident upon it, the incident flux being limited to a narrow wavelength band about a known wavelength. This definition at least implies that the incident and reflected radiant fluxes are separately evaluated. The incident radiant flux is rarely evaluated directly, particularly with commercial reflectometers.

The absolute reflectance of a good mirror is comparatively easy to measure. In this case nearly all of the reflected radiant flux is confined to directions near the direction of specular reflection, and only a small solid angle of collection need be used, hence flux losses from the reflected beam are largely eliminated. The classical method of measuring reflectance of good mirrors is the multiple reflection technique described by Strong [1] and later perfected by Bennett and Koehler [2]. A modification of this method was used in calibrating the standards of spectral specular reflectance.

All of the above discussion has been in terms of radiant fluxes. The response of a detector may or may not be proportional to the flux incident upon it. All of the detectors used in this study were checked for linearity by use of calibrated filters and rotating disc attenuators, and no detectable departures from linearity were found.

2. PREPARATION OF STANDARDS

The standards were prepared by a commercial optical company. The blanks were of fine-annealed borosilicate glass, in nominal sizes of 76 x 102 x 16 mm (3 x 4 x 5/8 in), 38 x 38 x 12.7 mm (1 1/2 x 1 1/2 x 1/2 in), and discs 28.6 mm (1 1/8 in) diameter by 9.6 mm (3/8 in) thick and 23.7 mm (7/8 in) diameter by 6.4 mm (1/4 in) thick. The surface to be coated was polished to a nominal finish of 0.05 wavelength (about 27 nm or 1.0 μ in).

The coating, of pure gold or aluminum, was applied to the center 51 x 76 mm (2 x 3 in) area of the 76 x 102 mm blanks, and the center 25.4 x 25.4 (1 x 1 in) of the 38 x 38 mm blanks, and to the entire face of the discs. Leaving the edges of the larger blanks uncoated reduces the danger of contamination of the edges from fingerprints. The smaller disc samples are easier to handle, and hence are less likely to be contaminated.

The purchase specifications called for metallic coatings to be applied at a thickness sufficient to be opaque, by fast (1 or 2 second) vacuum deposition at a pressure not to exceed 1×10^{-6} torr. These conditions have been shown [3], [4], [5] to produce coatings of relatively high reflectances in the ultraviolet.

Unfortunately, the samples supplied were evidently prepared under conditions that were not optimum. This was not discovered until actual measurements were made of the spectral specular reflectance in the ultraviolet. The reduced reflectance of aluminum films in the ultraviolet has been shown [5], [6], [7], to be due to an oxide layer on the surface of the aluminum. It has also been shown [7] that the films with lower initial reflectance show a greater reduction in reflectance with time. The reduction in reflectance in the ultraviolet is logarithmic and reaches essentially a steady condition after about one year of ageing [4].

The primary requirements of a standard of specular spectral reflectance are that its reflectance (1) be accurately known, and (2) be stable with time. There is no significant advantage to having the highest possible reflectance, provided the other criteria are met.

The samples were received during the transfer of the NBS laboratories from Washington to Gaithersburg, and this factor, combined with the press of other work, resulted in a delay of about two and one-half years between receipt of the samples and the first calibration measurements. This was ample time for the reflectance of the aluminum coatings to stabilize.

The range of values obtained in the ultraviolet and visible on samples of the different sizes is shown in figure 1.

3. DESCRIPTION OF APPARATUS

The complete apparatus used in calibration of the standards of spectral specular reflectance consisted of a source, a sample mount, focusing optics, a monochromator, a detector and an amplifier-recorder system. A sketch of the sample mount assembly is shown in figure 2, and schematic diagrams of the equipment used in each wavelength range, together with block diagrams of the source power supply and detector-recorder-amplifier system, in figures 3, 4, 5, 6, and 7. The sample mount and focusing optics were used in all wavelength ranges, and will be described first. Different combinations of source, monochromator and detector-amplifier-recorder systems were used in the different ranges.

1. Sample Mount

In order to more easily describe the optical system, which comprised primarily the sample mount and focusing optics, the position of various components will be described relative to the axis of the system which is defined as a horizontal line normal to the plane of the front surface of the sample and passing through the center of the two reference mirrors.

The sample mount assembly formed an important feature of the optical system. It was designed^{1/} and built at the National Bureau of Standards. The assembly consisted of a sample holder, two reference mirror mounts, a base plate and a mounting plate. Two reference mirrors having identical specular spectral reflectance over the wavelength range of measurements, or whose reflectance ratio is precisely known over this wavelength range, are required in this method. This point is discussed further in Part 2 of the Test Procedure. (See page 23).

The sample holder permitted one large or two small samples to be rigidly mounted so that their front surfaces were parallel to, and at a fixed distance from, the plane of the front surface of the holder. The holder fitted into a key slot in the base plate, with the plane of its front surface normal to the axis. The sample holder could be moved horizontally in the slot into or out of the optical path. A movable stop in the slot could be adjusted so that the sample was centered laterally on the axis when the holder was in contact with the stop, or if two samples were being measured, they were centered in the respective beams. Several front plates for the holder were provided to accommodate the samples of different sizes. A sketch of the sample mount assembly is shown in figure 2.

The base plate rested on two horizontal slides or ways, which were attached to the mounting plate. These slides were parallel to the axis, and permitted the base plate to be moved in a direction parallel to the axis. The top of the base plate was 8 cm below the axis. A locking device, attached to the mounting plate, was provided to hold the base plate in any desired position. Leveling screws were provided near the four corners of the mounting plate.

^{1/}By Lawrence J. Nivert

Two mounts held the reference mirrors. Each mount consisted of a base, a rod, and a mirror holder. The reference mirrors were 7 x 20 mm in size. The mirror holder held the mirror firmly in position, but was pivoted about a horizontal line normal to and intersecting the axis, and its degree of tilt could be controlled by means of adjusting screws. The mount was attached at the top of a vertical rod that fitted into a hole in the base, and could be rotated. It was held in position by a set screw. There were two dowel pins in the base of each mount, centered 25.4 mm apart, that fitted into holes in the base plate. The mount was held in place by a screw through the base that fitted into a tapped hole in the base plate. A set of mounting holes in the base plate consisted of two holes for the dowels, centered 12.7 mm on either side of a vertical plane through the axis, and a tapped hole centered in that plane and 9.5 mm farther from the key slot than the dowel holes. There were 33 sets of such mounting holes on each side of the key slot, spaced 6.35 mm apart, with the dowel holes of the set nearest the key slot 46.04 mm from the plane of the front surface of the sample, so that the plane of the reference mirror when mounted in these holes was 38.1 mm from the plane of the sample mirror. Both reference mirrors were coated with vacuum-deposited aluminum.

2. Focusing Optics

The focusing optics included (1) two spherical mirrors, 16 cm in diameter, with a 71 cm radius of curvature, (2) two plane mirrors, (3) circular apertures 10, 7.5 and 5.0 cm in diameter, cut in cardboard covered with black velvet. The position of these components will be described in the section on alignment.

3. Monochromators^{2/}

Three different monochromators were used in different spectral ranges: (1) a Leiss quartz double-prism monochromator, used in the spectral range 0.25 to 0.58 μm , (2) a Perkin-Elmer model 98 monochromator with a lithium fluoride prism for use in the spectral ranges of 0.58 to 1.2 μm and 1.2 to 4.0 μm , and (3) a Block model 200 interferometer spectrometer for use in the spectral range of 5.0 to 30 μm .

All detectors vary in response over their sensitive areas. A major source of error in the multiple reflection method of measuring specular reflectance is due to slight differences in the position of the reflected beam on the detector or on the entrance slit of a monochromator when the sample is moved into or out of the beam. Such shifts may result in a change in the fraction of the energy in the beam reaching the detector when the detector or entrance slit is overfilled, or in change in apparent gain of the detector when the slit or detector is underfilled. Such errors can be avoided in either of two ways: (a) by use of a very elaborate optical system to eliminate such shifts of the reflected beam, as was reported in reference [2], or (b) by use of an optical averaging device in the system to eliminate the effects of slight shifts of the beam on detector output. The latter approach was used in this work.

An averaging sphere [8] was used to uniformly irradiate the entrance slit of the prism monochromators. With an averaging sphere having an entrance aperture that is large compared to the cross-sectional area of the beam,

^{2/}The manufacturer and model number of certain equipment is given for identification only, and does not constitute an endorsement by the National Bureau of Standards, and does not imply that such equipment was necessarily the best available for the purpose.

the detector output will remain constant as the beam is moved provided only that the entire beam is always contained within the aperture. For the Leiss monochromator used in the range 0.25 to 0.58 μm , the averaging sphere was 76 mm in diameter and had a circular entrance port 19 mm in diameter and a rectangular exit port 19 by 6.3 mm. The ports were centered 135° apart on a plane through the center of the sphere. The long dimension of the exit port was normal to this plane. The inside of the sphere was coated with smoked magnesium oxide. The averaging sphere was placed with its exit port immediately in front of the entrance slit of the monochromator so that the slit was always completely filled, regardless of its width.

The same MgO coated sphere was also used with the Perkin-Elmer model 98 monochromator in the wavelength range of 0.58 to 1.2 μm . In this case the averaging sphere was located with its exit port in the position normally occupied by the source for the monochromator. The source optics, consisting of a short focal length spherical mirror and a small plane mirror, formed an image of the exit port of the sphere on the entrance slit of the monochromator. When the averaging sphere was placed immediately in front of the entrance slit, the off-axis paraboloid mirror in the monochromator, used to collimate the beam and reflect it onto the prism and then to refocus the dispersed beam onto the exit slit, was overfilled, and some of the undispersed radiant energy reached the detector, causing a significant error. With the image of the exit port of the sphere focused onto the entrance slit, the paraboloid mirror was slightly underfilled, and the scattered light error was eliminated. The customary checks for errors due to stray light, using auxiliary filters, showed in these measurements that the remaining errors were negligible. Errors due to overfilling the off-axis paraboloid may be expected with single-dispersion monochromators, but

should be absent with double-dispersion systems because the stray light scattered within the monochromator is blocked by the center slit of a double prism instrument, or is not chopped and hence not detected in a double-pass instrument.

A second averaging sphere, 50.8 mm in diameter, with ports of the same sizes and in the same relative positions as those of the 76 mm sphere, was used in a similar position for use in the wavelength range of 1.2 to 4.0 μm . This sphere was coated with sprayed sodium chloride [9], which has much higher reflectance than MgO in the infrared beyond about 2 μm .

In the wavelength range of 5 to 30 μm , where the interferometer spectrometer was used to disperse the reflected radiant energy, the reflected beam was focused onto an aperture 3 mm wide by 12 mm high, placed approximately 25 cm in front of the entrance window of the spectrometer. The radiant energy passing through the aperture irradiated uniformly an area with a diameter appreciably larger than that of the entrance window of the spectrometer. The image on the aperture plate was approximately 4.5 mm wide by 6 mm high, hence could be moved over a range of at least 1 mm horizontally and still cover the entire width of the aperture, and at least 4 mm vertically and still all be within the aperture. No movement of the beam was observed as the test mirror was moved into or out of the optical path.

4. Sources

Three different sources were used in the different spectral ranges. An Osram mercury arc lamp model Hg3, with accompanying power supply, was used in the wavelength range 0.25 to 0.58 μm . This lamp is a limited-pressure arc, and as such is much more stable than other types. The arc is confined to a sealed quartz tube with inside dimensions of 2 mm diameter by 20 mm long. The outer glass envelope of the lamp has an opening through which the ultraviolet radiant energy passes. An aperture 4 mm wide by 6.35 mm high was

placed immediately in front of the lamp. Selected lines of the mercury spectrum from this source were used in the calibration, as indicated in Table I. A tungsten lamp with a strip filament 3 x 15 mm in size was used in the 0.58 to 1.2 μ wavelength range. The filament was viewed by the optical system through a plane quartz window. The lamp was enclosed in a shield having a circular aperture 17.5 mm in diameter, directly in front of the quartz window. The power to the lamp was regulated by a regulated d.c. power supply. A Globar source, nominally 6.35 mm in diameter and 51 mm long, was used at wavelengths beyond 1.2 μ m. The power to the Globar was regulated by a controller. The source was enclosed in a water-cooled mount, to reduce convection, and a water-cooled aperture, 4.5 mm wide and 6 mm high, was placed immediately in front of the aperture in the enclosure.

5. Detector-Amplifier-Recorder Systems

In the 0.24 to 0.58 μ m wavelength range the incident beam was chopped near the source at 33 Hz. The detector was an EMI type 9558 QA photomultiplier tube, used at a potential of 450V. The signal from the detector was amplified by a preamplifier, then fed to a synchronous amplifier where it was further amplified and rectified. The output of the synchronous amplifier was a d.c. potential, which was fed to a digital voltmeter. The signal from the voltmeter was read by a TWX coupler, converted into ASCII code and typed in printed form and on punched paper tape by a Teletype machine.

The same system was used in the 0.58 to 1.2 μ m range, except that an EMI photomultiplier tube, type 9684B with a S-1 cathode was used as the detector, at a potential of 760V.

In the 1.2 to 4.0 μm range a similar system was used, but in this case the chopper frequency was 11.3 Hz, and a liquid-nitrogen-cooled lead sulfide detector, at a bias potential of 180V, was used. The preamplifier and amplifier were different units of the same model number as before, but peaked at the lower chopper frequency.

The detector-amplifier-recorder system used with the interferometer spectrometer was quite complex, and will be described only in general terms. The interferometer recorded the interferograms for the wavelength range of 4 to 40 μm at the rate of one sweep per second. The signal was amplified, and sampled at 520 time increments, equally spaced from the beginning of the sweep. The potentials at each of the 520 points were digitized, and stored in separate memory banks of a miniature computer that was part of the interferometer, in the form of a 16-bit binary word, of which the first bit was a sign bit. The signals for 300 sweeps were coherently added in the memory. The instrument provided for addition and subtraction of interferograms in the memory, hence background corrections were made directly, before the interferograms were reduced. After the complete interferogram had been stored, and background corrections made, it was read out of the memory one point at a time by the TWX coupler, which converted each 16-bit binary word into a 6-bit octal word with negative numbers in 7 's complement, of which the first bit was again a sign bit, and fed it, in ASCII code to a Teletype machine, where it was recorded on punched paper tape and in typed form.

6. Atmospheric Absorption

Absorption due to water vapor and carbon dioxide in the atmosphere is significant at certain wavelengths in the 0.25 to 30 μm range, and variations in such absorption could cause significant errors if ignored.

Major absorption bands of H_2O occur at 1.87, 2.70 and 6.22 μm , and minor bands at 0.94, 1.1, 1.38 and 3.2 μm . Major absorption bands of CO_2 occur at 2.7, 4.3 and 15.0 μm , and generally in the region between 11.4 and 20 μm . Weaker bands are present at 1.4, 1.6, 2.4, 4.8, 5.2, 9.4 and 10.4 μm .

The wavelengths shorter than 4.0 μm at which measurements were made were selected to avoid the atmospheric absorption bands, and hence errors due to such absorption.

The measurements at wavelengths longer than 4.0 μm were made in a water-free, CO_2 -free atmosphere. The entire optical path, including the source, spectrometer and detector, was enclosed with sheet plastic, and CO_2 -free dry air was fed into the enclosure at a rate of about 0.75 liter per second. The atmospheric purification train is described in reference [10]. A minor modification was made in the purification train, in order to increase the efficiency in removing CO_2 . The order of absorbing towers now is (numbers refer to figure 14 in reference [10]) 10. Ascarite CO_2 absorber, 11. Ascarite, 12. Silica gel. All other components are as shown in the figure. After the entire system had been exposed to the laboratory atmosphere for several days, continuous purging for 24 hours was required to reduce atmospheric absorption to a point where it could not be detected. This long purging time is believed to be due to the time required to remove adsorbed films from optical elements, particularly the window to the spectrometer, because the water vapor bands could be detected long after a hygrometer showed the atmosphere to have a relative humidity of less than one percent. After the system had been purged for 24 hours, only about 30 minutes of purging were required after opening the cover to remove one sample and insert another to restore the system to a condition where the atmospheric absorption could not be detected.

4. OPTICAL ALIGNMENT

Very careful optical alignment was required in order to assure proper operation of the equipment. Two special pieces of equipment were used to aid in this alignment, a machinists level with a sensitivity of about 0.0025° , and a small helium-neon laser.

The equipment was mounted on an optical table that was nominally level. The first step was to locate the sample mount assembly on the table, level the base plate by means of the leveling screws in the mounting plate, and clamp the mounting plate in position. A sample was mounted in the sample holder, and the holder positioned in the key slot so that the sample intersected the axis. The laser was then positioned so that its beam was centered on the axis, which was 8 cm above the top of the base plate and normal to the sample. The two reference mirrors were then mounted in their holders, the sample holder was moved out of the beam, and each reference mirror in turn was mounted in the base plate and adjusted to be normal to the laser beam.

The two spherical mirrors were in adjustable mounts which permitted them to be tilted about horizontal and vertical axes. The two mounts were placed side by side on an aluminum plate, in a position such that they were about 65 cm from the slot in the sample holder assembly, and equally spaced on either side of the axis. The aluminum plate was then raised until the centers of the mirrors were in the horizontal plane through the axis, leveled, and clamped in position. The laser was then moved to a position where its beam was in the horizontal plane through the axis, and passed through the position of the source. The laser was left in this position for checking alignment when changing from one size of sample to another, or when changing system components in going from one spectral range to another. A plane mirror with its surface vertical was placed in posi-

tion to reflect the laser beam to the center of the first spherical mirror. The sample holder was then removed from the beam, and a reference mirror mounted in the base plate facing the spherical mirrors. The base plate was then moved on the slides until the reference mirror was 71 cm from the spherical mirror. The tilt of the first spherical mirror about a horizontal axis was then adjusted until the reflected laser beam was level, and the position of the plane mirror was adjusted, if necessary, to bring the laser beam to the center of the reference mirror. The beam reflected by the reference mirror was then incident on the center of the second spherical mirror. Its tilt was adjusted to keep the laser beam level when reflected. The sample holder with a sample in place was then placed in the beam, and the second reference mirror was mounted with its surface in a position such that the two reference mirrors were equidistant from the front surface of the sample. The tilt of the second reference mirror was adjusted, if necessary, to make the positions of the laser beam on the second spherical mirror coincide when the sample was in or out of the beam. The spectrometer was then placed in position, its height adjusted until the horizontal plane through the axis passed through the center of its entrance slit, and clamped in position. The averaging sphere (or aperture plate) was then placed in position, and a second plane mirror with its surface vertical was used to reflect the laser beam to the center of the entrance aperture.

The source was then placed in position, with the aperture in front of the source centered on the horizontal plane through the axis, and the position of the source behind the aperture was adjusted until the first spherical mirror was well centered in the beam. The sample holder was removed from the optical path, and the base plate was moved forward or back until a sharp image of the aperture was formed on

the reference mirror. The second spherical mirror was checked to see that it was uniformly irradiated, the second plane mirror to see that none of the beam missed the mirror, and the entrance aperture of the averaging sphere to see that all of the beam was within the aperture when the sample was in and out of the beam. When satisfactory alignment had been achieved, the 10 cm diameter aperture was centered on the second spherical mirror, and the 7.5 cm aperture was centered on the first spherical mirror. This formed the limiting aperture for the system. When the 38 mm square samples were being measured, the 7.5 cm aperture was replaced by the 5.0 cm aperture. The felt-covered baffles were then put in position to prevent scattered light from the source from reaching the detector.

Two positions of the reference mirrors were required, 25.4 mm from the sample face for the 38 x 38 mm samples, and 95.25 mm from the sample face for all other samples. The location and size of the irradiated areas on the samples are shown in figure 8.

The axial ray from the spherical mirror was incident on the sample from an angle of 8.91° from the normal. When the 7.5 cm aperture was used in front of the spherical mirror, the angle of incidence of the cone of incident rays varied over the beam from 5.85° to 11.97° and when the 5.0 cm aperture was used, from 6.87° to 10.95° . The reflectance of metal surfaces varies only slightly with angle of incidence in this range.

A shutter was provided in front of the source in each case, which was closed when the background reading was taken.

Immediately before making a reflectance measurement, a sample was mounted in the sample holder, and the sample holder inserted into the key slot in the base plate, but out of the optical path. The reference mirrors were placed in the proper position for the sample to be measured. The base

plate was moved forward or back as required to obtain a sharp image of the source on the rear reference mirror. The position of the beam was checked on each mirror and the entrance aperture of the averaging device. Minor adjustments in alignment were made, if required, although this was rarely the case.

In the case of the disc samples, which were too small to be measured individually, two samples were mounted in the sample holder, side by side. In this case each sample was measured at least twice, in combination with a different sample each time so that the reflectance of each sample could be determined separately. (See equation 3).

5. TEST PROCEDURE

1. Recording Data

After the optical alignment had been checked, the raw data from which the reflectance was computed was re-recorded. Three different procedures were used, in the ranges of 0.25 to 1.2 μm , 1.2 to 4.0 μm , and 5 to 35 μm .

In the 0.25 to 1.2 μm range, the wavelength drum and slit drive of the monochromator were set to give the desired wavelength and slit width. The room lights were turned off, and the measurements were made in a darkened room. The shutter in front of the source was closed, and the background signal was recorded for 32 successive data points. The shutter in front of the source was then opened, and, with the sample out of the beam, the one reflection signal was recorded for 32 successive data points. The sample holder was then moved into position where it was in contact with the stop, and the three reflection signal was recorded for 64 successive data points. The sample holder was then removed from the optical path, and the one reflection signal was again recorded for 32 successive data points. The shutter in front of the source was then closed, and the background signal was again recorded for 32 successive data points. In each case the readings were recorded by the Teletype on punched paper tape and in typed form. The wavelength drum and slit drive were then moved to the settings for the next wavelength, and the process was repeated until measurements had been completed at all wavelengths in that range. The wavelengths at which measurements were made in the 0.25 to 0.58 μm wavelength range are shown in Table 1 together with the spectral bandpass at each wavelength. Similar values in the wavelength range of 0.58 to 1.2 μm are shown in Table 2. A similar procedure was followed in the wavelength range of 1.2 to 4.0 μm , except that the background was recorded

for 50 successive data points, the one reflection reading with the sample out for 50 successive data points, the three reflection reading with the sample in the beam for 100 successive data points, the one reflection reading again for 50 successive data points, and finally the background again for 50 successive data points. The wavelengths at which measurements were made are shown in Table 3, together with the spectral bandpass at each wavelength.

The entire optical system used for measurements in the 5 to 30 μm wavelength range was enclosed and maintained in a water-free, CO_2 -free atmosphere during test. A long-sleeved rubber glove attached to the wall of the enclosure permitted the sample to be moved into or out of the beam without disturbing the atmosphere. However, it was necessary to open the enclosure when samples were changed. In this case the interferogram for the entire wavelength range from 4 to 40 μm was scanned at once. The procedure was to scan the background, with the control switch on the console set to subtract the values from the computer memory, for 150 scans, then change the switch to the add position and record the one reflection data with the sample out of the beam for 300 scans, then again change the switch to the subtract position and record background for another 150 scans. The data were then read out of the computer memory and stored on punched paper tape and in typed form. The same procedure was then followed for the three reflection data with the sample in the beam, again recording negative background for 150 scans, positive reflectance for 300 scans, then negative background for 150 more scans. This procedure was repeated six times with each sample.

2. Reducing Data

The data were reduced on the NBS electronic digital computer. In the 0.25 to 4.0 μm range, the data were recorded in decimal form in ASCII code on punched paper tape. The

data were first transferred from paper tape to magnetic tape, and then converted from decimal form to binary. The mean and standard deviation were computed separately for the background, B, the one reflection data, ρ_1 , and the three reflection data, ρ_3 . The reflectance, ρ , was computed as:

$$\rho = \frac{\rho_3 - B}{\rho_1 - B} \quad (1)$$

Computation of the random error was more complicated. The standard deviation of the final reflectance, σ_ρ , was computed from the standard deviation of the three reflection values, σ_3 and the standard deviation of the one reflection values, σ_1 , as:

$$\sigma_\rho = \rho/2 \left[\frac{\sigma_3^2 + \sigma_B^2}{(\rho_3 - B)^2} + \frac{\sigma_1^2 + \sigma_B^2}{(\rho_1 - B)^2} \right]^{1/2} \quad (2)$$

(See reference [11]).

In the case of the disc samples, which were measured two at a time, the ρ in equation (1) represents not the reflectance of a single sample, but the square root of the product of the reflectances of the two samples. For samples A, B and C, three measurements were made, giving $\sqrt{\rho_a \rho_b}$, $\sqrt{\rho_b \rho_c}$ and $\sqrt{\rho_a \rho_c}$. The reflectance of each was then computed as

$$\rho_a = \frac{\sqrt{\rho_a \rho_b} \sqrt{\rho_a \rho_c}}{\sqrt{\rho_b \rho_c}} \quad (3)$$

and so forth. The inclusion of three measurements in each reflectance evaluation increased the random error in the result which was computed by the standard propagation of error method [11].

The precision of measurement of the large (7.6 x 10.2 and 3.8 x 3.8 cm) samples, expressed as the standard deviation of the mean of six replicate measurements on a single sample, varied from about 0.0002 to 0.0005 in the wavelength range of 0.257 to 0.577 μm , and from about 0.0010 to 0.0052 in the wavelength range of 0.60 to 4.0 μm . Corresponding values for the small disc samples were about 0.0004 to 0.0020 at wavelengths from 0.257 to 0.577 μm , and 0.0021 to 0.0095 from 0.60 to 4.0 μm .

The non-random error, or bias, is believed to be less than the precision of measurement reported above. The four principal sources of bias are: 1) non-linearity of the detector-amplifier-recorder system, 2) drift of the response with time, 3) variations in the atmospheric absorptance with time, and 4) differences in the spectral reflectances of the two reference mirrors. Linearity was checked, and no non-linearity was found. Non-linearity on the order of 0.002 should be detected. The fact that fluxes of about equal value were being compared indicates that errors from

this source would be small, except for gold at wavelengths shorter than about $0.7 \mu\text{m}$, even if measurable non-linearity was present. Drift on order of a few tenths of a percent per hour has been observed. Such drift is linear with time, at least to a first approximation. The scheme used in making the measurements compensates for any linear drift that may be present. Atmospheric absorption is always present in the normal laboratory atmosphere, and varies quite significantly with time when a living person is present. Human breath contains significantly more carbon dioxide and water vapor than the normal laboratory atmosphere. Errors due to variations with time in atmospheric absorptions were avoided by making measurements only at wavelengths where absorption due to carbon dioxide and water vapor is essentially absent. The reference mirrors differ in reflectances by as much as 0.001 at some wavelengths. The ratio of the reflectances was measured at each wavelength at which the mirrors were calibrated and a correction was made for the observed ratio. The residual error from this source should not exceed 0.0001.

The data from the interferometer spectrometer required entirely different treatment. As mentioned previously, the data were recorded for 520 points. The sweep generator in the controls for the interferometer generated a pulse at the beginning of each sweep, to serve as a reference line and start the timer which triggered the recording circuit. This pulse occasionally caused a large spurious signal to be recorded in the first or second memory bank, which, if used in reducing the data, would cause errors in the reduced spectrum. For this reason the first three data points were discarded in reducing the data.

The data on the punched paper tape were transferred to magnetic tape for entry into the NBS 1108 computer. A subroutine picked up the characters from the tape and stored them in the memory. Only data points from No. 4 to No. 515

were used. The computer converted negative numbers from 7's complement to true negative by subtracting them from 77778 in octal, then converted all numbers from octal to binary.

The Fourier coefficients A_K , B_K of the Fourier series

$$YE(X) = A_0/2 + \sum_{K=1}^{256} (A_K \cos KX + B_K \sin KX) \quad (4)$$

where $YE(X)$ is the amplitude of the interferogram at time or position X were computed^{3/}. The YE values are the recorded words, and the X values are the serial numbers from 1 to 512 of the recorded words. The program essentially solves 512 equations in 512 unknowns.

Next, monochromatic flux values $\eta_K \phi_K$ at wavenumbers (or wavelengths) corresponding to the points K ($K = 1$ to 256) were computed as

$$\eta_K \phi_K = \sqrt{A_K^2 + B_K^2} \quad (5)$$

The proportionality factor η_K in equation (5) is an instrument calibration factor that is wavenumber dependent, and ϕ_K is the monochromatic flux at the wavenumber corresponding to K . η_K is due to the spectral transmittance of the spectrometer window, the spectral reflectance and transmittance of the beamsplitter in the interferometer, the spectral response of the detector and polarization effects, and is constant at any one value of K .

^{3/}The computer program for this computation was written by R. J. Arms, and is designated subroutine FSER (N,L,Y,YE,A), and is available at NBS.

There was significant scatter in the $\eta_K \phi_K$ values, which appeared to be completely random. In order to reduce this scatter, the data were smoothed by taking as the value for each point the mean of five adjacent points centered on the desired K value. The reflectance, ρ_K , at each K value was computed as

$$\rho_K = [\eta_K(\phi_K)_3 / \eta_K(\phi_K)_1]^{1/2} = [(\phi_K)_3 / (\phi_K)_1]^{1/2} \quad (6)$$

where $\eta_K(\phi_K)_3$ is the smoothed monochromatic flux value at K for the three-reflection measurements and $\eta_K(\phi_K)_1$ for the one-reflection measurement.

The wavenumber, W, is related to K by the equation

$$W = -10.9741 + 10.1522K \quad (7)$$

for values of K from 25 to 256 from the wavelength calibrations. The wavenumbers corresponding to K less than 25 are less than 250, or the wavelengths are greater than 40 μm . The wavenumbers corresponding to K greater than 247 are greater than 2500, or the wavelengths are less than 4 μm . The interferometer spectrometer is restricted to wavelengths in the 4 to 40 μm range, hence only points between K = 25 to 247 were used.

The six ρ_K values obtained for each K value in the six replicate measurements on each sample were averaged, and the standard deviation of the mean was computed, with an average value of about 0.004. The 223 mean values of ρ_K corresponding to K values from 25 to 247 were then plotted as a function of wavelength, and a smooth curve was drawn through them. The values reported for each sample were then read off from this curve.

Our values for the reflectance of the best sample of aluminum averaged about 0.0035 higher than those of Bennett, Bennett and Ashley [5] for aged high-vacuum aluminum

deposited at a pressure of about 10^{-5} torr, and about 0.0024 lower than those of Bennett, Silver and Ashley [12], for fresh aluminum deposited at a pressure of about 10^{-9} torr. Our highest values for the infrared reflectance of gold averaged about 0.0018 lower than those of Bennett and Ashley [13] for gold deposited at a pressure of about 5×10^{-10} torr. This is considered reasonable agreement, particularly in view of the different conditions of application of the coatings and the reported precision of measurement.

All reflectance values were corrected for differences in the reflectances of the two reference mirrors. This correction was in most cases less than 0.001.

Figure 9 shows the average spectral specular reflectance of the aluminum and gold coated samples. The individual values for each sample will be reported in the Certificate accompanying that sample.

Tables I, II, III and IV show the wavelengths at which reflectances are measured, together with the slit width used with the prism monochromators, and the spectral bandpass at each wavelength.

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TABLE I
Ultraviolet Range

| Wavelength μm | Slit Width mm | Bandpass nm |
|-----------------------------|------------------|----------------|
| 0.2537 | 0.1 | 0.2 |
| 0.2650 | 0.1 | 0.25 |
| 0.2805 | 0.1 | 0.3 |
| 0.3023 | 0.1 | 0.4 |
| 0.3340 | 0.1 | 0.5 |
| 0.3651 | 0.1 | 0.6 |
| 0.4048 | 0.1 | 0.9 |
| 0.4358 | 0.1 | 1.2 |
| 0.5460 | 0.1 | 2.6 |
| 0.5770 | 0.1 | 3.1 |

TABLE II

Visible and Near Infrared

| Wavelength μm | Slit Width mm | Bandpass nm |
|-----------------------------|------------------|----------------|
| .600 | 0.1 | 4.0 |
| .650 | 0.1 | 4.5 |
| .700 | 0.1 | 5.0 |
| .800 | 0.1 | 6.0 |
| .825 ^{1/} | 0.1 | 6.0 |
| .850 ^{1/} | 0.1 | 6.5 |
| .900 | 0.1 | 7.0 |
| 1.000 | 0.1 | 7.5 |
| 1.200 | 0.17 | 15.0 |

^{1/}Measurements at these wavelengths for aluminum mirrors only.

TABLE III

Near Infrared

| Wavelength μm | Slit Width mm | Bandpass nm |
|-----------------------------|------------------|----------------|
| 1.200 | 0.17 | 18 |
| 2.000 | 0.15 | 13 |
| 3.300 | 0.34 | 20 |
| 4.000 | 0.80 | 38 |

TABLE IV

Infrared

| Wavelength μm | Mechanical Travel μm | Resolution μm |
|-----------------------------|------------------------------------|-----------------------------|
| 5 | 500 | 0.05 |
| 10 | 500 | 0.20 |
| 15 | 500 | 0.40 |
| 20 | 500 | 0.9 |
| 25 | 500 | 1.3 |
| 30 | 500 | 1.8 |

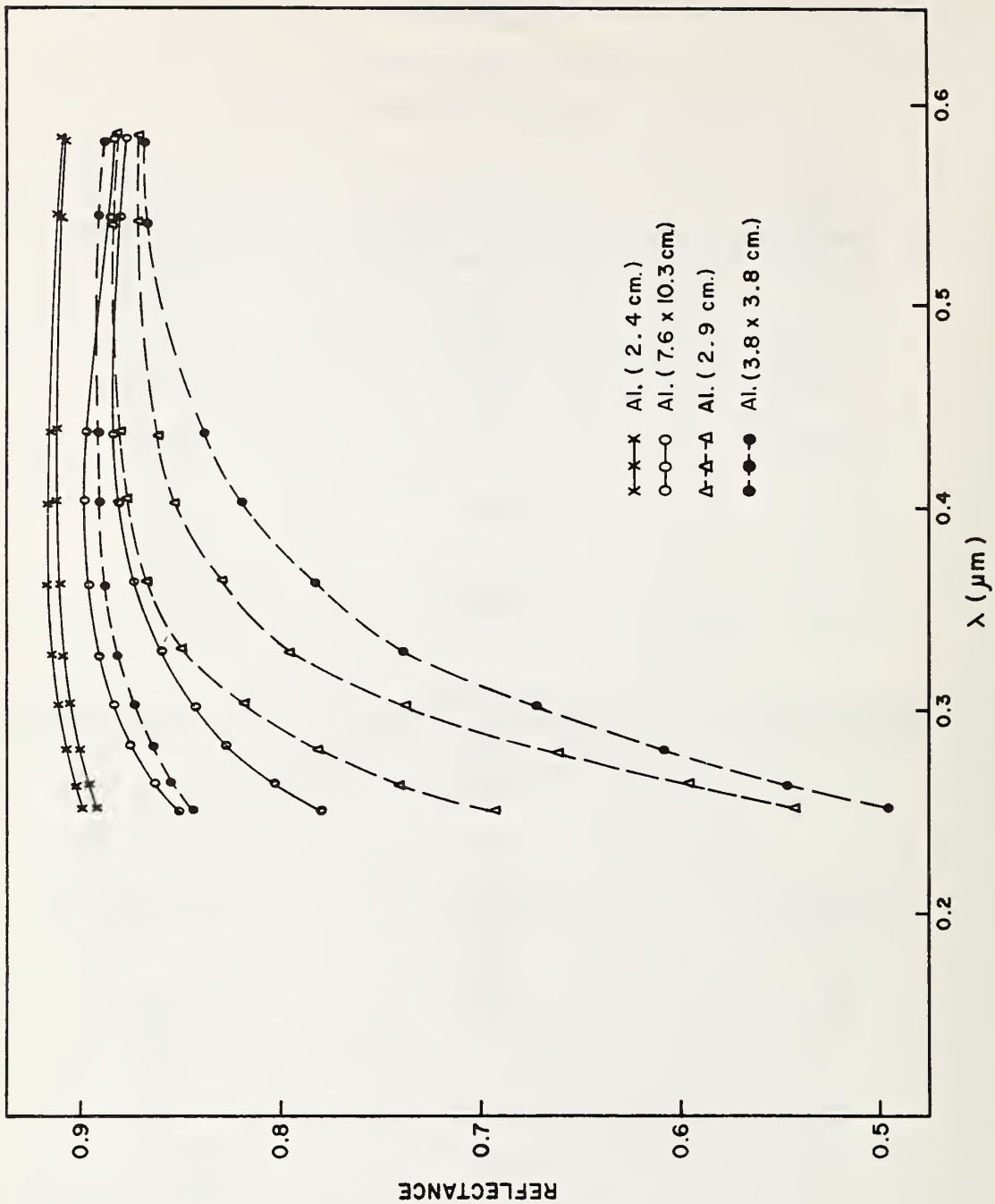


Figure 1. Range of reflectance values obtained in the ultraviolet on different sized samples of aluminum mirrors.

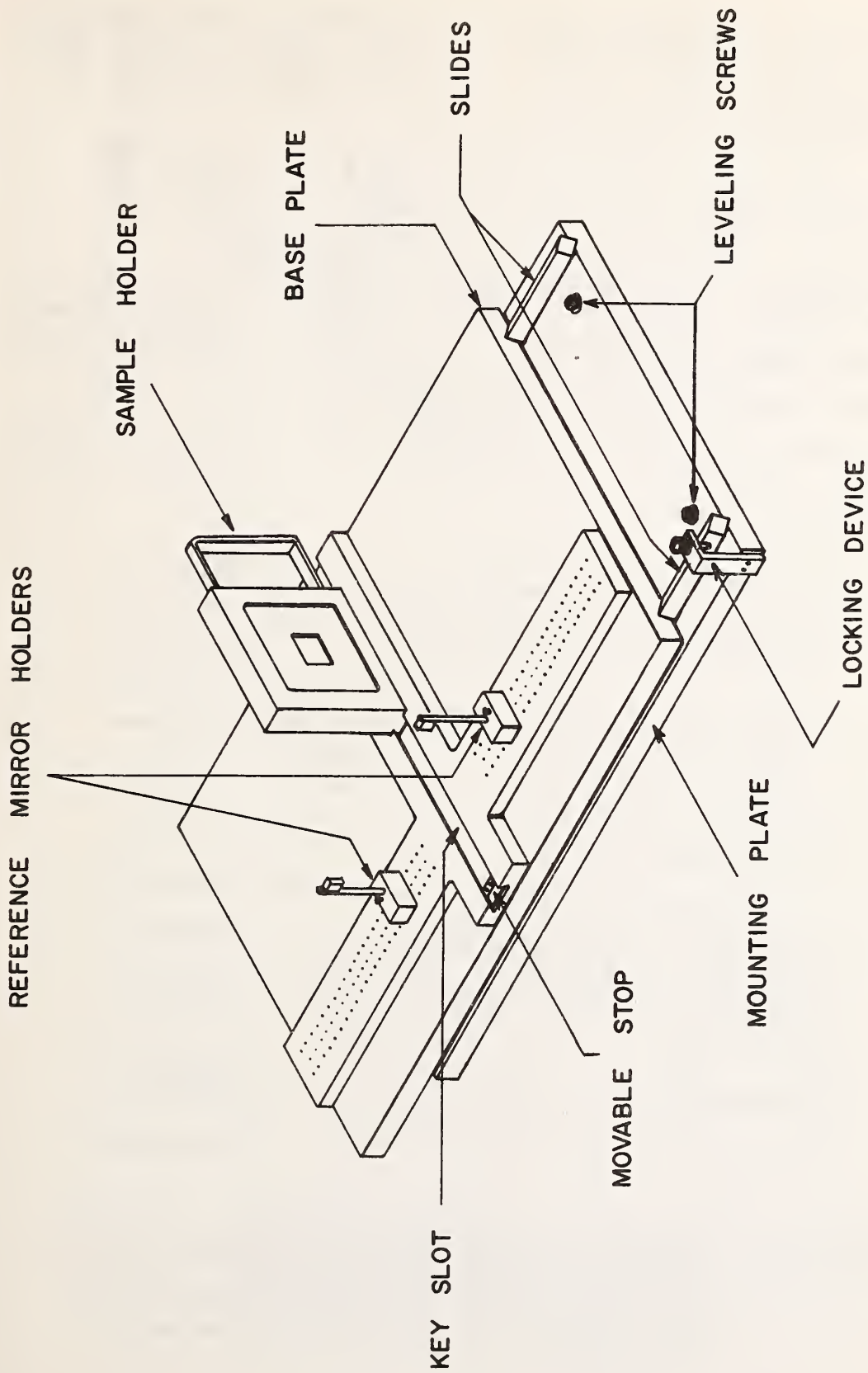


Figure 2. Sketch of the sample mount assembly. The base plate has been moved back from its normal position in order to show the leveling screws.

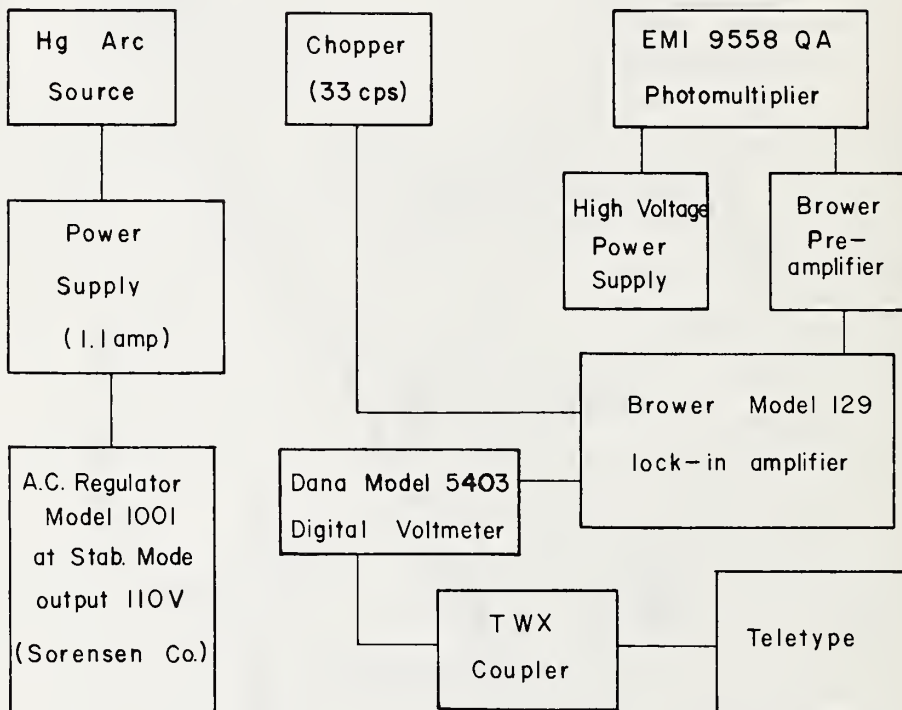
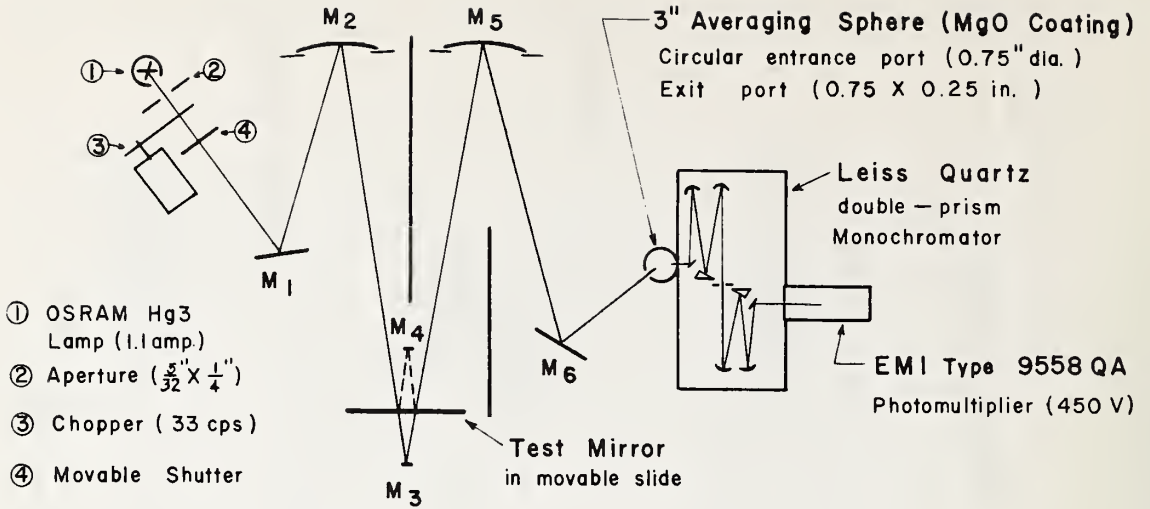


Figure 3. Schematic diagram of the equipment used for reflectance measurements in the wavelength range of 0.25 to 0.58 μm , together with a block diagram of the source power supply and recording circuit.

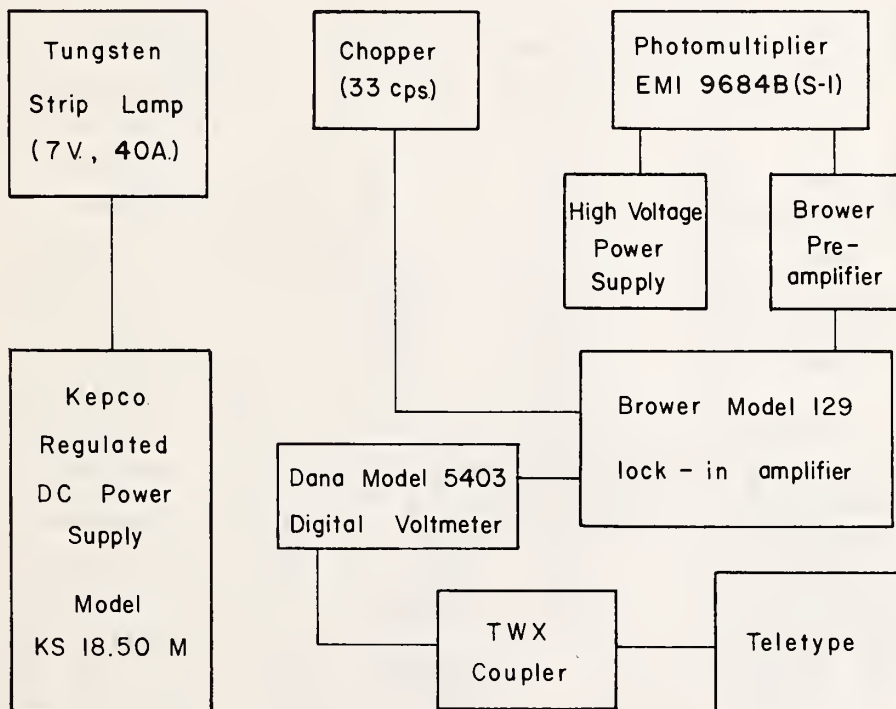
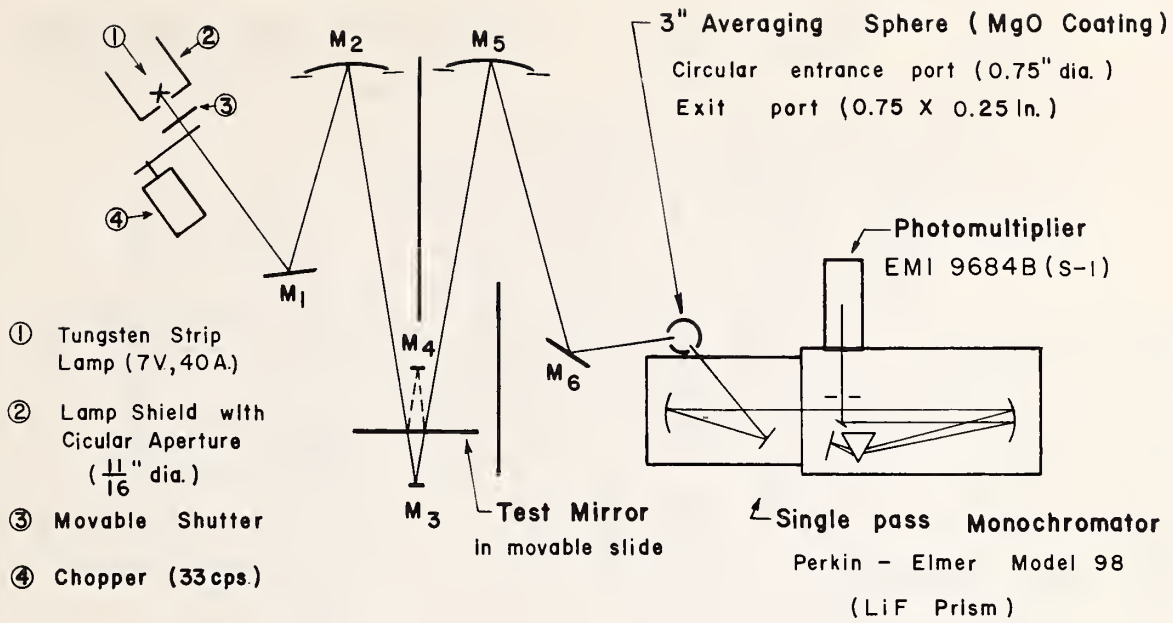


Figure 4. Schematic diagram of the equipment used for reflectance measurements in the range of 0.6 to 1.2 μm , together with a block diagram of the source power supply and recording circuit.

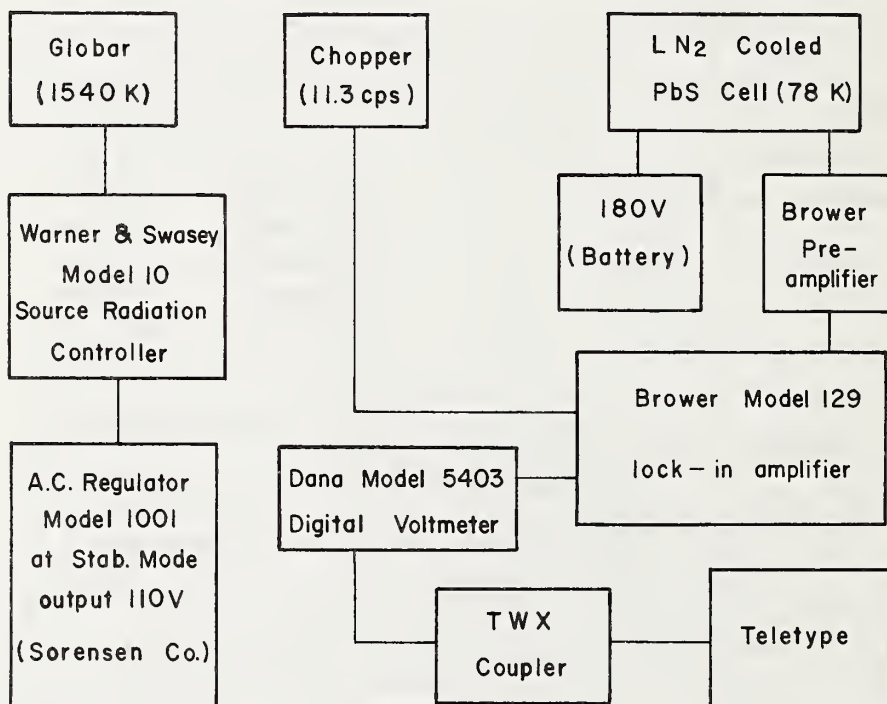
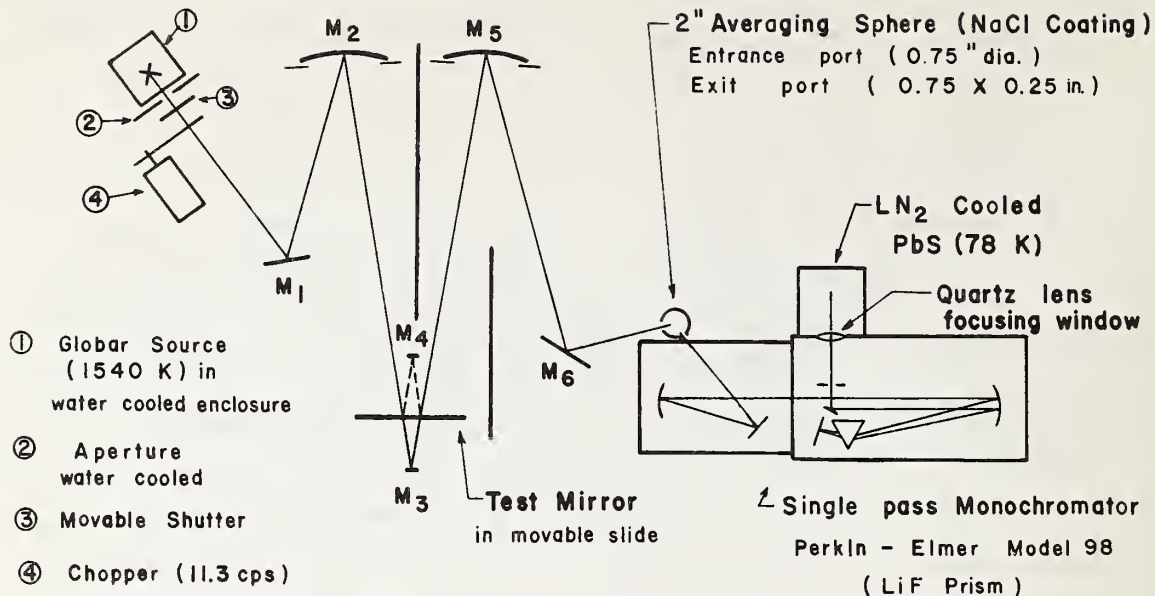


Figure 5. Schematic diagram of the equipment used for reflectance measurements in the 1.2 to 4.0 μm range, together with a block diagram of the source power supply and recording circuit.

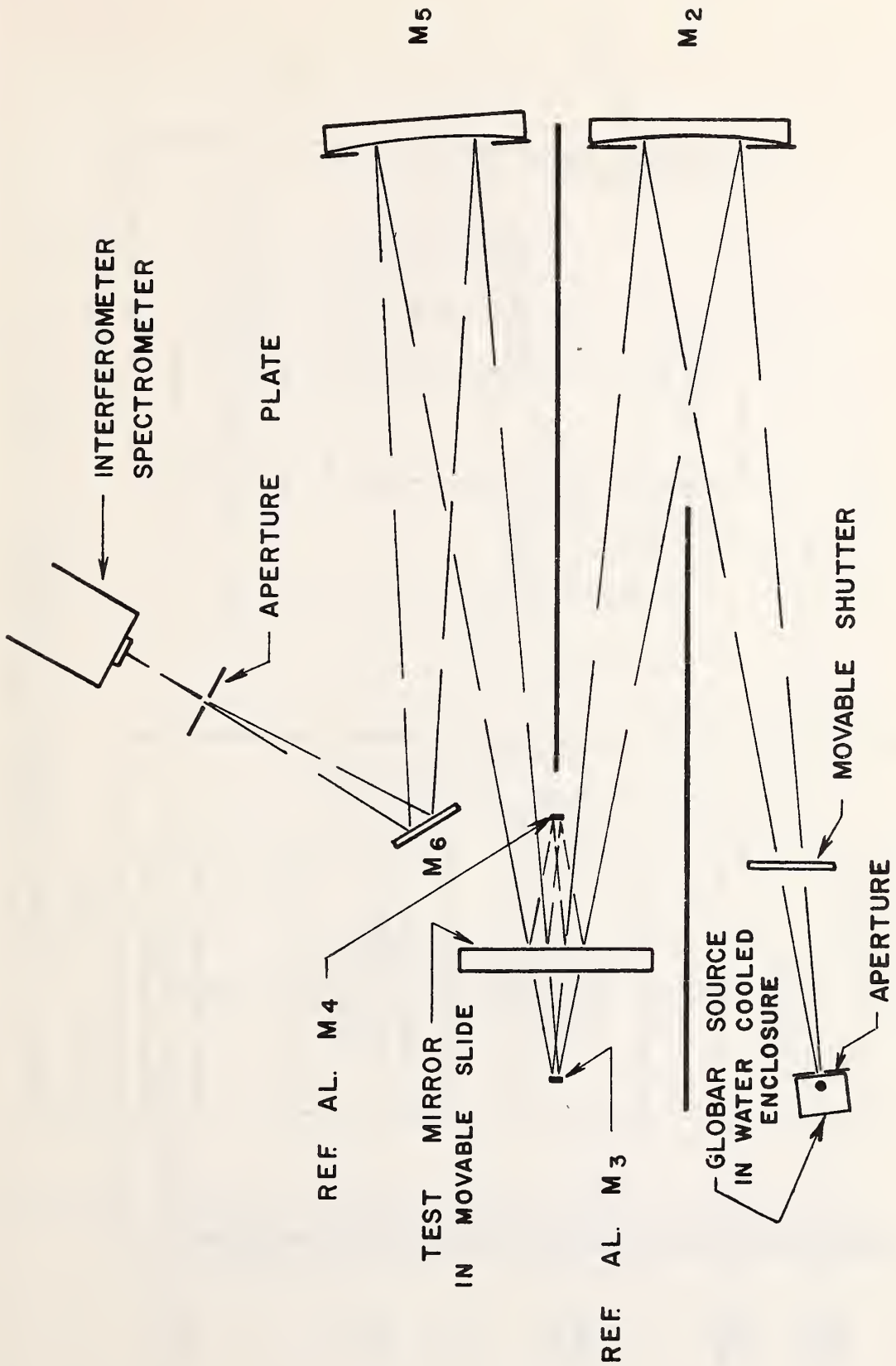


Figure 6. Schematic diagram of the equipment used for reflectance measurements in the 5 to 30 μm range.

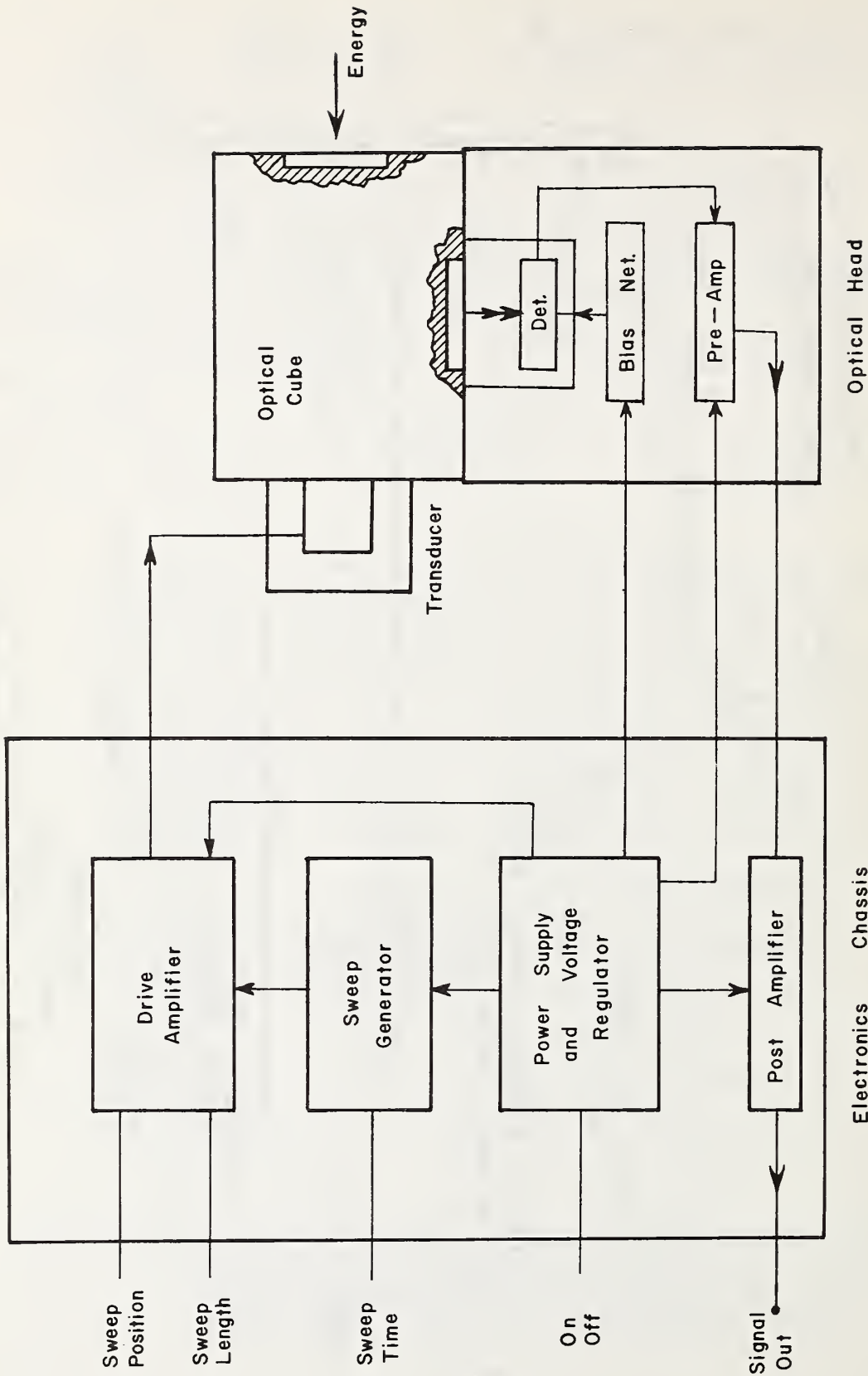


Figure 7. Block diagram of the control circuits for the interferometer spectrometer.

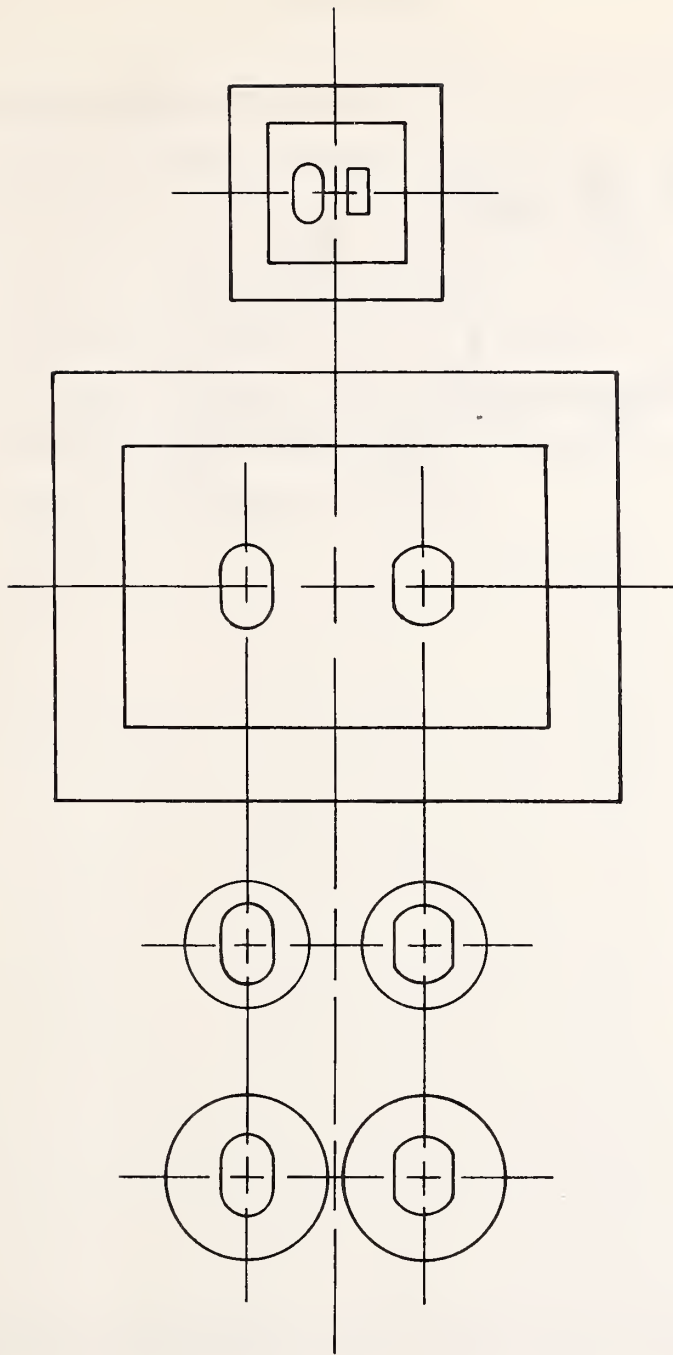


Figure 8. Size and location of the areas irradiated on samples of each size during reflectance measurements.

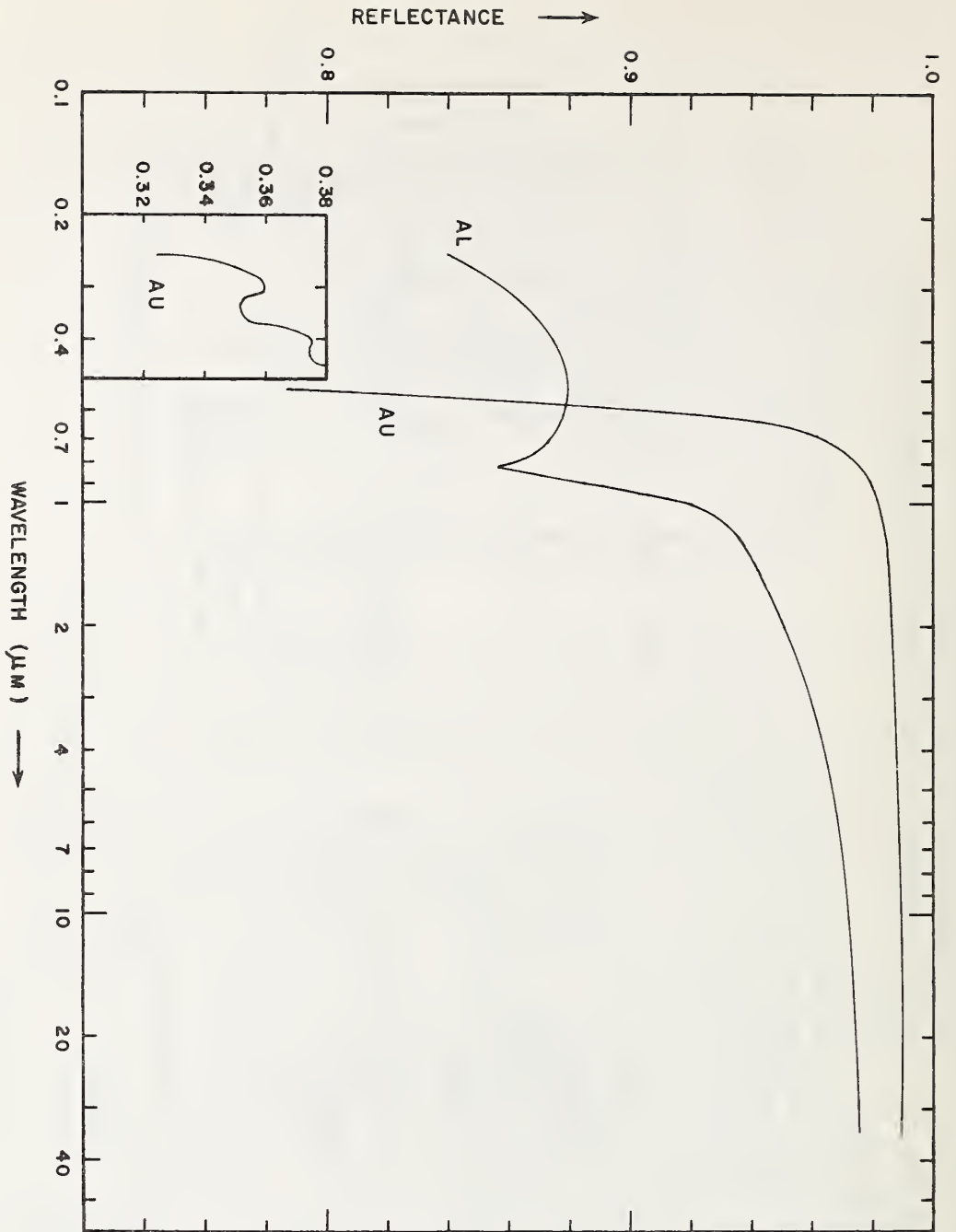


Figure 9. Average spectral specular reflectance of gold and aluminum reflectance standards.

7. APPENDIX

The following pages are copies of the Certificates issued with the Specular Spectral Reflectance Standards. The same first page is used with Numbers 2001, 2002, 2003, and 2004, and with 2005, 2006, 2007, and 2008, hence only one copy of each of these first pages is included.

A separate Certificate will be issued with each standard, and will include in the spaces provided the actual specular spectral reflectance measured on that standard at the indicated wavelengths.

National Bureau of Standards

Certificate

Standard Reference Materials 2001, 2002, 2003, 2004

Aluminum on Glass

(Standard for Specular Spectral Reflectance)

These Standard Reference Materials are for calibrating equipment used in evaluating the thermal radiation properties of materials. The mirrors were prepared by fast vacuum deposition of aluminum on fine-annealed borosilicate glass, the surface to be coated being flat to about 27 nm. The mirrors are prepared in four sizes.

| <u>SRM No.</u> | <u>Size of blank (cm)</u> | <u>Coated Area (cm)</u> |
|----------------|--------------------------------|-------------------------|
| 2001 | 7.6 × 10.2 × 1.9 | 5.1 × 7.6 |
| 2002 | 3.8 × 3.8 × 1.3 | 2.5 × 2.5 |
| 2003 | disk: 2.9 diameter × 1.0 thick | entire surface |
| 2004 | disk: 2.4 diameter × 0.6 thick | entire surface |

Each mirror is certified for near-normal (9°) specular reflectance at wavelengths ranging from 0.2537 to 30 micrometers and at corresponding resolved bandwidths from 1.0 to 1800 nanometers. The precision measure (σ_m) is the standard deviation of the mean of six replicate measurements. The certified values are given on the attached sheet. Details of experimental techniques used will be described in an NBS 260 publication.

Measurements of reflectance and other experimental work leading to the certification of these Standard Reference Material were performed by J. C. Richmond, and J. J. Hsia of the Heat Division, Institute for Basic Standards, National Bureau of Standards.

Washington, D. C. 20234
April 16, 1971

J. Paul Cali, Chief
Office of Standard Reference Materials

SRM No. 2001

Specimen material: Aluminum

Size: 7.6 X 10.2 X 1.9 cm

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0008 | 3 | 20 | | 0.0033 |
| 0.2650 | 1.0 | | .0007 | 4 | 32 | | .0052 |
| 0.2805 | 1.1 | | .0006 | 5 | 50 | | .0040 |
| 0.3023 | 1.2 | | .0005 | 6 | 72 | | .0040 |
| 0.3340 | 1.3 | | .0005 | 7 | 98 | | .0030 |
| 0.3651 | 1.4 | | .0002 | 8 | 128 | | .0030 |
| 0.4048 | 1.6 | | .0004 | 9 | 162 | | .0030 |
| 0.4358 | 1.7 | | .0002 | 10 | 200 | | .0030 |
| 0.5460 | 2.5 | | .0003 | 11 | 242 | | .0030 |
| 0.5770 | 2.5 | | .0003 | 12 | 288 | | .0030 |
| 0.600 | 4.0 | | .0030 | 13 | 338 | | .0030 |
| 0.650 | 4.5 | | .0020 | 14 | 392 | | .0030 |
| 0.700 | 5.0 | | .0018 | 16 | 512 | | .0040 |
| 0.800 | 6.0 | | .0015 | 18 | 648 | | .0040 |
| 0.825 | 6.2 | | .0014 | 20 | 800 | | .0040 |
| 0.850 | 6.5 | | .0013 | 22 | 968 | | .0040 |
| 0.900 | 7.0 | | .0010 | 24 | 1152 | | .0040 |
| 1.000 | 7.5 | | .0009 | 26 | 1352 | | .0040 |
| 1.200 | 15.0 | | .0038 | 28 | 1568 | | .0040 |
| 2.000 | 13.0 | | .0035 | 30 | 1800 | | .0040 |

SRM No. 2002

Specimen material: Aluminum

Size: 3.8 × 3.8 × 1.3 cm

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0008 | 3 | 20 | | 0.0033 |
| 0.2650 | 1.0 | | .0014 | 4 | 32 | | .0052 |
| 0.2805 | 1.1 | | .0008 | 5 | 50 | | .0040 |
| 0.3023 | 1.2 | | .0003 | 6 | 72 | | .0040 |
| 0.3340 | 1.3 | | .0005 | 7 | 98 | | .0030 |
| 0.3651 | 1.4 | | .0002 | 8 | 128 | | .0030 |
| 0.4048 | 1.6 | | .0003 | 9 | 162 | | .0030 |
| 0.4358 | 1.7 | | .0002 | 10 | 200 | | .0030 |
| 0.5460 | 2.5 | | .0002 | 11 | 242 | | .0030 |
| 0.5770 | 2.5 | | .0002 | 12 | 288 | | .0030 |
| 0.600 | 4.0 | | .0030 | 13 | 338 | | .0030 |
| 0.650 | 4.5 | | .0020 | 14 | 392 | | .0030 |
| 0.700 | 5.0 | | .0018 | 16 | 512 | | .0040 |
| 0.800 | 6.0 | | .0015 | 18 | 648 | | .0040 |
| 0.825 | 6.2 | | .0014 | 20 | 800 | | .0040 |
| 0.850 | 6.5 | | .0013 | 22 | 968 | | .0040 |
| 0.900 | 7.0 | | .0010 | 24 | 1152 | | .0040 |
| 1.000 | 7.5 | | .0009 | 26 | 1352 | | .0040 |
| 1.200 | 15.0 | | .0038 | 28 | 1568 | | .0040 |
| 2.000 | 13.0 | | .0035 | 30 | 1800 | | .0040 |

SRM No. 2003

Specimen material: Aluminum

Size: 2.9 cm diameter

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ |
|----------------------------|-------------------------------|-------------|----------|----------------------------|-------------------------------|-------------|----------|
| 0.2537 | 1.0 | | 0.0016 | 3 | 20 | | 0.0058 |
| 0.2650 | 1.0 | | .0028 | 4 | 32 | | .0090 |
| 0.2805 | 1.1 | | .0016 | 5 | 50 | | .0070 |
| 0.3023 | 1.2 | | .0006 | 6 | 72 | | .0070 |
| 0.3340 | 1.3 | | .0010 | 7 | 98 | | .0053 |
| 0.3651 | 1.4 | | .0004 | 8 | 128 | | .0053 |
| 0.4048 | 1.6 | | .0006 | 9 | 162 | | .0053 |
| 0.4358 | 1.7 | | .0004 | 10 | 200 | | .0053 |
| 0.5460 | 2.5 | | .0004 | 11 | 242 | | .0053 |
| 0.5770 | 2.5 | | .0004 | 12 | 288 | | .0053 |
| 0.600 | 4.0 | | .0087 | 13 | 338 | | .0053 |
| 0.650 | 4.5 | | .0052 | 14 | 392 | | .0053 |
| 0.700 | 5.0 | | .0040 | 16 | 512 | | .0070 |
| 0.800 | 6.0 | | .0035 | 18 | 648 | | .0070 |
| 0.825 | 6.2 | | .0038 | 20 | 800 | | .0070 |
| 0.850 | 6.5 | | .0035 | 22 | 968 | | .0070 |
| 0.900 | 7.0 | | .0026 | 24 | 1152 | | .0070 |
| 1.000 | 7.5 | | .0021 | 26 | 1352 | | .0070 |
| 1.200 | 15.0 | | .0095 | 28 | 1568 | | .0070 |
| 2.000 | 13.0 | | .0063 | 30 | 1800 | | .0070 |

SRM No. 2004

Specimen material: Aluminum

Size: 2.4 cm diameter

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0016 | 3 | 20 | | 0.0058 |
| 0.2650 | 1.0 | | .0028 | 4 | 32 | | .0090 |
| 0.2805 | 1.1 | | .0016 | 5 | 50 | | .0070 |
| 0.3023 | 1.2 | | .0006 | 6 | 72 | | .0070 |
| 0.3340 | 1.3 | | .0010 | 7 | 98 | | .0053 |
| 0.3651 | 1.4 | | .0004 | 8 | 128 | | .0053 |
| 0.4048 | 1.6 | | .0006 | 9 | 162 | | .0053 |
| 0.4358 | 1.7 | | .0004 | 10 | 200 | | .0053 |
| 0.5460 | 2.5 | | .0004 | 11 | 242 | | .0053 |
| 0.5770 | 2.5 | | .0004 | 12 | 288 | | .0053 |
| 0.600 | 4.0 | | .0087 | 13 | 338 | | .0053 |
| 0.650 | 4.5 | | .0052 | 14 | 392 | | .0053 |
| 0.700 | 5.0 | | .0040 | 16 | 512 | | .0070 |
| 0.800 | 6.0 | | .0035 | 18 | 648 | | .0070 |
| 0.825 | 6.2 | | .0038 | 20 | 800 | | .0070 |
| 0.850 | 6.5 | | .0035 | 22 | 968 | | .0070 |
| 0.900 | 7.0 | | .0026 | 24 | 1152 | | .0070 |
| 1.000 | 7.5 | | .0021 | 26 | 1352 | | .0070 |
| 1.200 | 15.0 | | .0095 | 28 | 1568 | | .0070 |
| 2.000 | 13.0 | | .0063 | 30 | 1800 | | .0070 |

Certificate

Standard Reference Materials

2005, 2006, 2007, 2008

Gold on Glass

(Standards for Specular Spectral Reflectance)

These Standard Reference Materials are for calibrating equipment used in evaluating the thermal radiation properties of materials. The mirrors were prepared by fast vacuum deposition of gold on fine-annealed borosilicate glass, the surface to be coated being flat to about 27 nm. The mirrors are prepared in four sizes.

| <u>SRM No.</u> | <u>Size of blank (cm)</u> | <u>Coated Area (cm)</u> |
|----------------|--------------------------------|-------------------------|
| 2005 | 7.6 × 10.2 × 1.9 | 5.1 × 7.6 |
| 2006 | 3.8 × 3.8 × 1.3 | 2.5 × 2.5 |
| 2007 | disk: 2.9 diameter × 1.0 thick | entire surface |
| 2008 | disk: 2.4 diameter × 0.6 thick | entire surface |

Each mirror is certified for near-normal (9°) specular reflectance at wavelengths ranging from 0.2537 to 30 micrometers and at corresponding resolved bandwidths from 1.0 to 1800 nanometers. The precision measure (σ_m) is the standard deviation of the mean of six replicate measurements. The certified values are given on the attached sheet. Details of experimental techniques used will be described in an NBS 260 publication.

Measurements of reflectance and other experimental work leading to the certification of these Standard Reference Materials were performed by J. C. Richmond and J. J. Hsia of the Heat Division, Institute for Basic Standards, National Bureau of Standards.

Washington, D. C. 20234
April 16, 1971

J. Paul Cali, Chief
Office of Standard Reference Materials

SRM No. 2005

Specimen material: Gold

Size: 7.6 × 10.2 × 1.9 cm

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0005 | 3 | 20 | | 0.0033 |
| 0.2650 | 1.0 | | .0004 | 4 | 32 | | .0052 |
| 0.2805 | 1.1 | | .0005 | 5 | 50 | | .0040 |
| 0.3023 | 1.2 | | .0005 | 6 | 72 | | .0040 |
| 0.3340 | 1.3 | | .0005 | 7 | 98 | | .0030 |
| 0.3651 | 1.4 | | .0002 | 8 | 128 | | .0030 |
| 0.4048 | 1.6 | | .0003 | 9 | 162 | | .0030 |
| 0.4358 | 1.7 | | .0002 | 10 | 200 | | .0030 |
| 0.5460 | 2.5 | | .0003 | 11 | 242 | | .0030 |
| 0.5770 | 2.5 | | .0003 | 12 | 288 | | .0030 |
| 0.600 | 4.0 | | .0030 | 13 | 338 | | .0030 |
| 0.650 | 4.5 | | .0020 | 14 | 392 | | .0030 |
| 0.700 | 5.0 | | .0018 | 16 | 512 | | .0040 |
| 0.800 | 6.0 | | .0015 | 18 | 648 | | .0040 |
| 0.825 | 6.2 | | .0014 | 20 | 800 | | .0040 |
| 0.850 | 6.5 | | .0013 | 22 | 968 | | .0040 |
| 0.900 | 7.0 | | .0010 | 24 | 1152 | | .0040 |
| 1.000 | 7.5 | | .0009 | 26 | 1352 | | .0040 |
| 1.200 | 15.0 | | .0038 | 28 | 1568 | | .0040 |
| 2.000 | 13.0 | | .0035 | 30 | 1800 | | .0040 |

SRM No. 2006

Specimen material: Gold

Size: 3.8 × 3.8 × 1.3 cm

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0006 | 3 | 20 | | 0.0033 |
| 0.2650 | 1.0 | | .0010 | 4 | 32 | | .0052 |
| 0.2805 | 1.1 | | .0007 | 5 | 50 | | .0040 |
| 0.3023 | 1.2 | | .0003 | 6 | 72 | | .0040 |
| 0.3340 | 1.3 | | .0005 | 7 | 98 | | .0030 |
| 0.3651 | 1.4 | | .0002 | 8 | 128 | | .0030 |
| 0.4048 | 1.6 | | .0003 | 9 | 162 | | .0030 |
| 0.4358 | 1.7 | | .0002 | 10 | 200 | | .0030 |
| 0.5460 | 2.5 | | .0002 | 11 | 242 | | .0030 |
| 0.5770 | 2.5 | | .0002 | 12 | 288 | | .0030 |
| 0.600 | 4.0 | | .0030 | 13 | 338 | | .0030 |
| 0.650 | 4.5 | | .0020 | 14 | 392 | | .0030 |
| 0.700 | 5.0 | | .0018 | 16 | 512 | | .0040 |
| 0.800 | 6.0 | | .0015 | 18 | 648 | | .0040 |
| 0.825 | 6.2 | | .0014 | 20 | 800 | | .0040 |
| 0.850 | 6.5 | | .0013 | 22 | 968 | | .0040 |
| 0.900 | 7.0 | | .0010 | 24 | 1152 | | .0040 |
| 1.000 | 7.5 | | .0009 | 26 | 1352 | | .0040 |
| 1.200 | 15.0 | | .0038 | 28 | 1568 | | .0040 |
| 2.000 | 13.0 | | .0035 | 30 | 1800 | | .0040 |

SRM No. 2007

Specimen material: Gold

Size: 2.9 cm diameter

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0012 | 3 | 20 | | 0.0058 |
| 0.2650 | 1.0 | | .0020 | 4 | 32 | | .0090 |
| 0.2805 | 1.1 | | .0014 | 5 | 50 | | .0070 |
| 0.3023 | 1.2 | | .0006 | 6 | 72 | | .0070 |
| 0.3340 | 1.3 | | .0010 | 7 | 98 | | .0053 |
| 0.3651 | 1.4 | | .0004 | 8 | 128 | | .0053 |
| 0.4048 | 1.6 | | .0006 | 9 | 162 | | .0053 |
| 0.4358 | 1.7 | | .0004 | 10 | 200 | | .0053 |
| 0.5460 | 2.5 | | .0004 | 11 | 242 | | .0053 |
| 0.5770 | 2.5 | | .0004 | 12 | 288 | | .0053 |
| 0.600 | 4.0 | | .0087 | 13 | 338 | | .0053 |
| 0.650 | 4.5 | | .0052 | 14 | 392 | | .0053 |
| 0.700 | 5.0 | | .0040 | 16 | 512 | | .0070 |
| 0.800 | 6.0 | | .0035 | 18 | 648 | | .0070 |
| 0.825 | 6.2 | | .0038 | 20 | 800 | | .0070 |
| 0.850 | 6.5 | | .0035 | 22 | 968 | | .0070 |
| 0.900 | 7.0 | | .0026 | 24 | 1152 | | .0070 |
| 1.000 | 7.5 | | .0021 | 26 | 1352 | | .0070 |
| 1.200 | 15.0 | | .0095 | 28 | 1568 | | .0070 |
| 2.000 | 13.0 | | .0063 | 30 | 1800 | | .0070 |

SRM No. 2008

Specimen material: Gold

Size: 2.4 cm diameter

Specimen no.:

| Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m | Wavelength (micrometer) | Resolved Bandwidth (nm) | Reflectance | σ_m |
|----------------------------|-------------------------------|-------------|------------|----------------------------|-------------------------------|-------------|------------|
| 0.2537 | 1.0 | | 0.0012 | 3 | 20 | | 0.0058 |
| 0.2650 | 1.0 | | .0020 | 4 | 32 | | .0090 |
| 0.2805 | 1.1 | | .0014 | 5 | 50 | | .0070 |
| 0.3023 | 1.2 | | .0006 | 6 | 72 | | .0070 |
| 0.3340 | 1.3 | | .0010 | 7 | 98 | | .0053 |
| 0.3651 | 1.4 | | .0004 | 8 | 128 | | .0053 |
| 0.4048 | 1.6 | | .0006 | 9 | 162 | | .0053 |
| 0.4358 | 1.7 | | .0004 | 10 | 200 | | .0053 |
| 0.5460 | 2.5 | | .0004 | 11 | 242 | | .0053 |
| 0.5770 | 2.5 | | .0004 | 12 | 288 | | .0053 |
| 0.600 | 4.0 | | .0087 | 13 | 338 | | .0053 |
| 0.650 | 4.5 | | .0052 | 14 | 392 | | .0053 |
| 0.700 | 5.0 | | .0040 | 16 | 512 | | .0070 |
| 0.800 | 6.0 | | .0035 | 18 | 648 | | .0070 |
| 0.825 | 6.2 | | .0038 | 20 | 800 | | .0070 |
| 0.850 | 6.5 | | .0035 | 22 | 968 | | .0070 |
| 0.900 | 7.0 | | .0026 | 24 | 1152 | | .0070 |
| 1.000 | 7.5 | | .0021 | 26 | 1352 | | .0070 |
| 1.200 | 15.0 | | .0095 | 28 | 1568 | | .0070 |
| 2.000 | 13.0 | | .0063 | 30 | 1800 | | .0070 |

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| 16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.) <p>This report describes in detail the preparation and calibration of Standard Reference Materials 2001 through 2008. These standards of spectral specular reflectance were prepared commercially by vacuum depositing opaque coatings of gold and aluminum, respectively, onto polished substrates of fine-annealed borosilicate glass. The near-normal (9° from normal) spectral specular reflectance was measured by the multiple-reflection technique. Four different combinations of source, spectrometer, and detector were used to cover the wavelength range from about 0.25 μm to 30 μm and beyond.</p> | | | |
| 17. KEY WORDS (Alphabetical order, separated by semicolons) Aluminum; gold; infrared reflectance; interferometer spectrometer; mirrors; reflectance; reflectance standards; spectral reflectance; spectral specular reflectance; specular reflectance; standards; ultraviolet reflectance. | | | |
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