WADC TECHNICAL REPORT 59-510
PART II

Standardization of Thermal Emittance Measurements

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FOREWORD

This report was prepared by the National Bureau of Standards under USAF Contract AF 33(616) 58-20, Amendment 1. It was initiated under Project No. 7360 "Materials Analysis and Evaluation Techniques", Task No. 73603, "Thermodynamics and Heat Transfer." The contract was administered under the direction of Material Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with Mr. Robert A. Winn acting as project engineer.

This report covers work performed during the second year on the project, from 1 July 1959 through 30 June, 1960.

The authors are pleased to acknowledge the contributions of the following persons:

F. J. Shorten, working with Harrison and Richmond in the Mineral Products Division of NBS on the development of procedures and equipment (other than data processing), and on the preparation and calibration of standard specimens;

R. F. Johnston and A. G. Maki, working with Plyler and Stair in the Atomic and Radiation Physics Division on the check-calibration of standard specimens.

ABSTRACT

Work during the year was directed toward establishment of equipment and procedures for the evaluation of normal spectral emittance of specimens held at temperatures in the range of approximately 800° to 1400°K (980° to 2060°F), over the wavelength range of 1 to 15 microns. Some of the developments of the previous contract were modified to increase precision and reduce errors in the measurements. One such modification was to provide means whereby a reference blackbody furnace and the hot specimen could be interchanged at will as sources of radiant flux to be measured relative to that from the comparison blackbody furnace. The apparent emittance of the specimen and of the reference blackbody furnace were both measured relative to the comparison blackbody furnace. The apparent emittance of the specimen at each of numerous selected wavelengths was then divided by the corresponding apparent emittance of the reference blackbody furnace to obtain the reported spectral emittance of the specimen.

Assembly of the equipment for determination of spectral reflectance, under conditions approximating normal illumination and hemispherical viewing, was completed.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER

Jules I. Wittebort

J. I. WITTEBORT
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I. OBJECTIVES

The general objective of this project is to develop standard techniques, and to select or design suitable equipment, for the measurement of spectral emittance, spectral reflectance and solar absorptance of materials, particularly those used in aircraft, missiles, or space vehicles. The project also includes (1) the selection or development of electronic data-processing equipment for on-line or off-line computation, from spectral emittance data, of total emittance, solar absorptance, and absorptance of radiant energy having any other known spectral distribution of flux, and (2) the selection of suitable materials and the preparation and calibration of specimens as working standards of spectral emittance, to be used for comparison of the results of spectral emittance determinations by several different laboratories.

II. SUMMARY OF PREVIOUS WORK

The first annual summary report on this project, WADC Technical Report 59-510 (August 1959), included definitions of terms, a discussion of the basic theory underlying thermal emittance measurements and the principles of data reduction; also a statement of the status of work on the project.

A laboratory blackbody furnace and a specimen furnace were designed and built. The laboratory blackbody furnace had a computed efficiency of better than 0.99, with an opening approximately 2 X 10 mm in size. The specimen furnace comprised a water-cooled shell surrounding the specimen, and was specifically designed to reduce temperature fluctuation of the specimen due to air currents and to eliminate reflection of radiant energy from the walls of the furnace back to the surface of the specimen, at angles such that it might contribute to the measured beam by being again reflected, this time from the specimen.

The source optics of a Perkin-Elmer Model 13 spectrometer were modified so that the radiant energy for the two beams was supplied by the laboratory blackbody furnace and by a hot specimen, respectively. Under these conditions the instrument recorded directly the ratio of the radiant flux in the two beams, which, when certain conditions were met, was also the normal spectral emittance of the specimen.

The power input to, and hence the temperature, of the blackbody furnace was varied by manual adjustment of an autotransformer. The specimen was heated by passing a current through it, and its temperature was controlled to within $\pm 0.8^{\circ}K$ of that of the blackbody furnace by means of a current-actuating-type control, responding to any e.m.f. generated in a differential thermocouple, one junction of which was immersed in the metallic core of the blackbody furnace and the other spot welded to the specimen.

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Equipment was designed for the measurement of spectral reflectance under conditions approximating normal illumination and hemispherical viewing, and all of the necessary components were procured or ordered.

Materials were selected and tested for use as working standards of normal spectral emittance, as follows: (1) oxidized Inconel was selected for use as standards of high emittance, and several specimens were calibrated in one laboratory. (2) A number of long-time heating tests were made of materials being considered for use as standards of intermediate emittance; none of these materials proved to have all of the desired characteristics. (3) Platinum was selected for use as standards of low emittance, and two specimens intended for this use were calibrated in one laboratory.

Engineering studies were conducted on the design of an automatic data-processing system for on-line computation of one pre-selected characteristic of the specimen, such as total emittance or solar absorptance, from the spectral emittance data; also for recording of the spectral emittance data in a form suitable for additional processing with an off-line digital computer.

III. COMPONENT PARTS OF THE INVESTIGATION AND ORDER OF PRIORITY

The objectives of this project cover such a broad range of conditions, as mentioned in WADC Technical Report 59-510 (August 1959), that it would be impossible to make simultaneous progress in all phases of the work in a single year with the available funds and personnel.

The full range of temperatures at which measurements are to be made can be conveniently divided into four smaller ranges, with arbitrary limits, on the basis of the different equipment and techniques that are required in each range, as follows: 1) below room temperature, 2) room temperature to 800°K (980°F), 3) the range between this temperature and 1400°K(2060°F), and 4) higher temperatures.* Similarly the wavelengths at which measurements are to be made may be divided into three ranges, again on the basis of the different equipment that is required in each range, as follows: 1) from about 0.3 micron to about 1.0 micron, 2) from 1.0 to 15 microns, and 3) from 15 to 35 microns or more. In addition, tests will be made on at least two types of specimens, those that can be heated by passing a current through them, and those for which this method is not practicable.

Concurrently with the development of instrumentation and techniques for the measurement of normal spectral emittance, physical working standards are to be selected and calibrated for normal spectral emittance, and data-processing components are to be incorporated into the equipment for test and further development.

* Temperatures are given in this report in terms of Kelvin and Fahrenheit scales. Kelvin is used because the theoretical relationships between temperature and blackbody radiation are based upon absolute temperatures. The Fahrenheit equivalent is also given to facilitate engineering and design applications. Temperatures that are frequently referred to, including 800°K (980°F) and 1400°K (2060°F), are sometimes given in °K only, to avoid monotonous repetition.

In conformity with the current greatest need of the Air Force, priority was given to development of instrumentation and techniques for the measurement of normal spectral emittance of specimens that can be heated by passing a current through them, at temperatures in the range of approximately 800° to 1400°K (980° to 2060°F) over the wavelength range of approximately 1 to 15 microns, together with the selection and calibration of physical working standards of emittance in these ranges, and the development of a suitable data processing system.

Secondary priority was given to the development of instrumentation and techniques for the measurement of reflectance, under conditions approximating normal illumination and hemispherical viewing, of specimens at room temperature, over the wavelength range of approximately 1 to 15 microns.

The next task in order of urgency was considered to be the extension of the wavelength range of approximately 1 to 15 microns down to about 0.3 micron and up to about 35 microns. Development work in the temperature range about 1400°K (2060°F), and for specimens that cannot be heated by passing a current through them, was included in the longer range planning.

IV. STATUS OF WORK ON THE PROJECT

A. Normal Spectral Emittance

During the past year, work on the development of instrumentation and techniques for the measurement of normal spectral emittance has been concentrated on improving the equipment and techniques described in WADC TR 59-510 (August 1959), in order to reduce error and improve the precision of results.

Modification of External Optics

It was found necessary to use a specimen of known emittance in the specimen beam in order to check the adjustment of the scale of the instrument at frequent intervals. Accordingly, the external optical system was modified as indicated in Figure 1, to permit the reference blackbody furnace or the specimen furnace to be placed alternately in position to supply radiant energy for the specimen beam, the comparison blackbody furnace always providing radiant energy for the comparison beam. This arrangement permitted the reference blackbody furnace to be used as a known source with an emittance of 1.00 in adjusting the scale of the instrument.

Modification of Test Procedures

It was found by experience that it was not possible to adjust the spectrometer so that the "100% curve", obtained as the ratio of the radiant energy emitted by the two blackbody furnaces operating at the same temperature, was flat over the wavelength range of 1 to 15 microns. The best that could be obtained was a curve that was flat within ±2½% between 2 and 14.5 microns on the spectral scale except for slight excursions at about 4.3 and between 8 and 11 microns respectively. The curve was too low below 2 microns, the extreme deviation being about 8% from the correct value at 1 micron, and too high above 14 microns, rising to a maximum

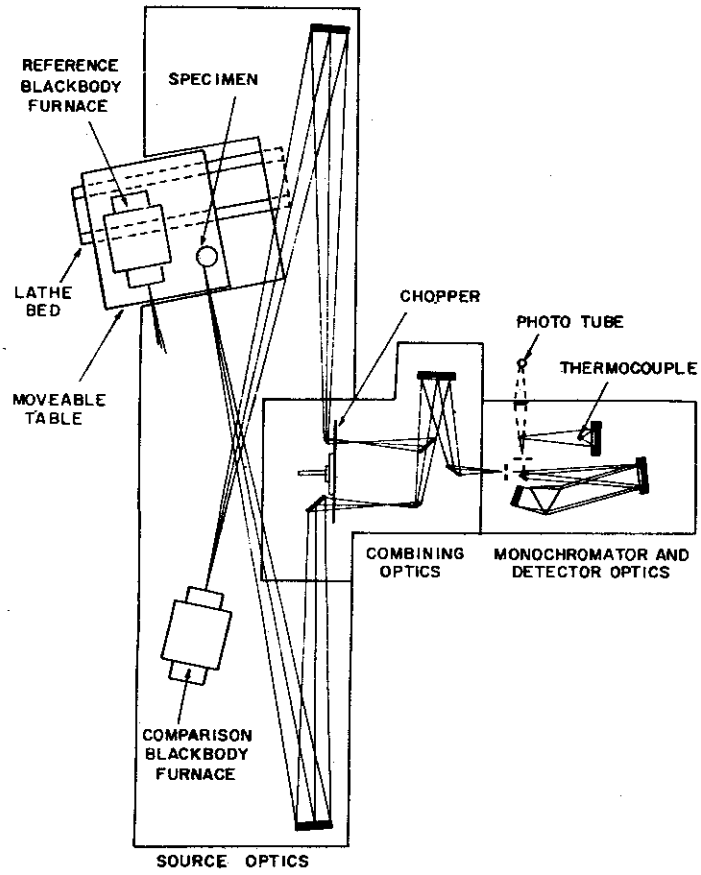


Figure 1.

Schematic diagram of the optical paths in the modified spectrometer. The specimen furnace is shown in operating position. The reference blackbody furnace can be shifted into operating position by sliding the movable table to a stop on the lathe bed.

Following geometric combination of the two beams they remain separated in time as they pass through the optical system in alternating pulses.

deviation of about 5% at 15 microns, as shown in Figure 2. Conferences with representatives of the manufacturer of the spectrometer led to the conclusion that this performance is as good as can be expected of the instrument.

The deviation of the "100% curve" from flatness is believed to be caused by one or more of the following factors: 1) The two beams are reflected by three different pairs of mirrors and by different areas on seven additional mirrors. Any variations in the spectral reflectance of equivalent mirrors, or of different areas on the same mirror at the locations where the respective beams are reflected, could cause deviations of the type observed. 2) The two beams pass through slightly different areas of the window on the thermocouple detector. Any difference in spectral transmittance of the areas traversed by the respective beams could cause deviations of the type observed. 3) The thermocouple target is not a perfect absorber; any variation in spectral absorptance of the two areas on which the respective beams impinge could cause deviations of the type observed.

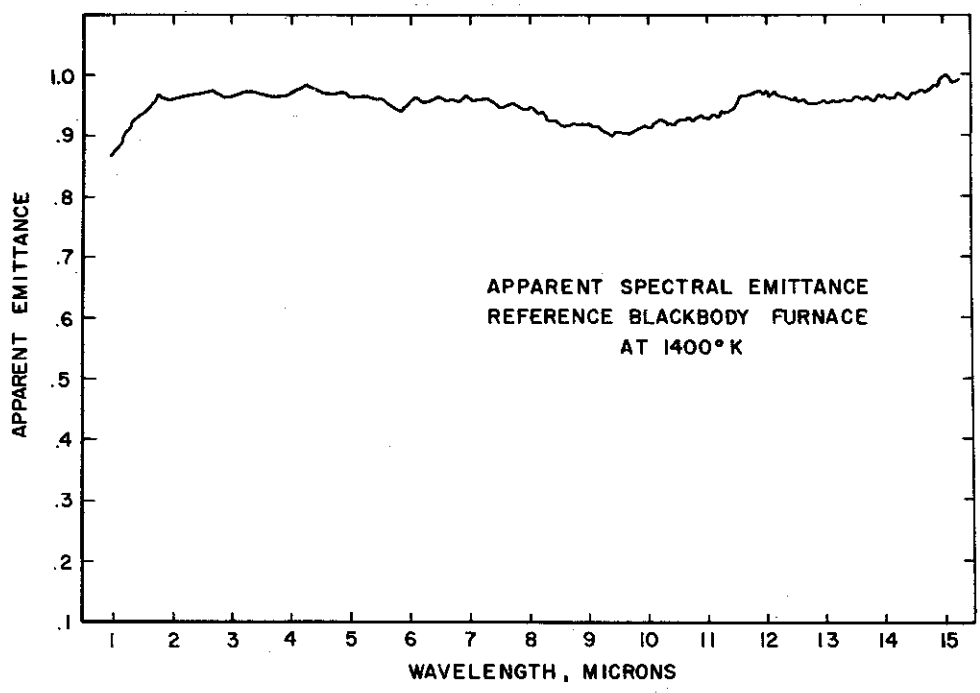


Figure 2. Apparent spectral emittance of the reference blackbody furnace at 1400°K. This is the calibration curve, or "100% curve", obtained when the reference blackbody furnace is used instead of a specimen as one of the two sources. Deviations of this curve from a straight horizontal line at 1.0 emittance (or 100%) are applied as corrections to curves obtained from test specimens. Among the possible sources of these deviations are: (1) variations in spectral reflectance of the mirrors, even over the surface of a single mirror on which the two beams cover different areas, and (2) variations in sensitivity of the receptor over its sensitive area which is not exposed exactly equally to the two beams.

The deviations of the "100% curve" from flatness are reproducible to about ±½%, over the wavelength range of 1 to 15 microns, provided the same instrument settings are used, and the two blackbody furnaces are at the same temperature. Hence the deviations of the "100% curve" from the ideal value of 100% can be compensated by the following procedure: 1) Obtain a spectral response curve for the two opposed blackbody furnaces at the desired temperature, T. 2) Replace the reference blackbody furnace with the specimen furnace, and heat the specimen to the desired temperature, T. 3) With the specimen and comparison blackbody furnace at temperature T, and with all instrument settings the same as at the time the initial curve was obtained, proceed with automatic plotting of the apparent spectral reflectance of the specimen. 4) Compute the ratio, at selected wavelength intervals, of the height of the specimen curve to that of the "100% curve", and plot these values as a function of wavelength. The curve obtained by connecting these points represents the normal spectral emittance of the specimen. This procedure was followed in obtaining the normal spectral emittance curves presented in this report.

Modification of Blackbody Furnaces

The opening in the blackbody furnaces as originally designed was 2 x 10 mm, which is exactly the size of the entrance slit of the monochromator at its maximum opening. With an opening of this size it was not possible to focus its image, by means of an optical system having unit magnification, onto the wide-open slit so that it overlapped the slit boundaries on all sides. In order to permit a larger opening, the cavity in each blackbody furnace was enlarged to a diameter of 19 mm (3/4 inch), and threaded. New end plates, with openings 9 x 18 mm, were installed. The net effect of both modifications reduced the computed efficiency of the blackbody furnace slightly. The efficiency for the new cavity, computed from the measured emittance of oxidized Inconel, within the following temperature limits, varied from a low of 0.9990 at 6 microns at a temperature of 780°K (945°F) to a high of 0.9999 at 14 microns at 1400°K (2060°F). It is believed that the error introduced by considering the laboratory blackbody furnace to be a true blackbody radiator is negligibly small.

The thermocouple hole at the back of each blackbody furnace core was drilled through into the cavity, and the thermocouple in each cavity was inserted to a depth such that the bead extended about 6 mm into the cavity. This change eliminated difficulties that had been encountered when the thermocouple was inserted into a small hole in the Inconel core.

The Inconel cores of the blackbody furnaces oxidize appreciably when operated at 1400°K for extended periods. Some of this oxide tends to flake off when the furnace is allowed to cool, and it has been necessary from time to time to remove the oxide that has collected within the cavity of the blackbody furnace. In order to reduce or eliminate such flaking of oxide in future work, new cores have been made of an 80-nickel 20-chromium alloy. This alloy is relatively difficult to machine, and the cores were cast; they will be installed in the blackbody furnaces the next time they require new resistance windings.

Grounding of Blackbody Furnace Cores

There was some intermittant instability in the electronic temperature control equipment, which permitted fluctuating temperature differences between the two blackbody furnaces of up to ±4°K, and between a specimen and the blackbody furnace of up to ±10°K. The trouble was traced to a pickup of 60-cycle potential in the thermocouple circuit. When the blackbody furnace was operated at 1400°K, the refractory insulators between the platinum resistance winding and the Inconel core, as well as those between the Inconel core and the thermocouple, became slightly conducting. The amount of current leakage was small, but the peak potential-to-ground of the resistance winding was approximately 50 volts, and the 60-cycle potential that was picked up by the grounded thermocouple was too large to be eliminated by the filters in the thermocouple circuit. This potential was eliminated by grounding the alloy cores of the two blackbody furnaces. With this modification the control equipment restricts the maximum temperature difference between the two blackbody furnaces to about ±0.25°K, and that between the blackbody furnace and a test specimen to about ±0.75°K. Possible further reduction of the latter fluctuation is under study.

A similar, but much smaller, 60-cycle potential was picked up from the specimens, which are heated by their resistance to alternating current. The voltage drop across a specimen during heating seldom exceeded 3 volts, so that when one side of the circuit was grounded, the potential-to-ground at the center of the specimen seldom exceeded 1 1/2 volts. Even this residual potential was eliminated by use of an isolation transformer with a floating ground, as shown schematically in Figure 3. However, this modification produced no improvement in the precision of the temperature; apparently the small 60-cycle potential from this source was eliminated by the electronic filters in the thermocouple circuit.

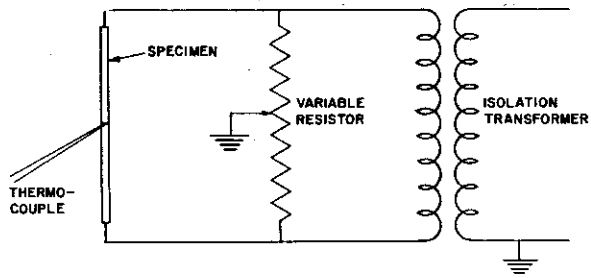


Figure 3. Schematic diagram of the system used to bring to ground potential the thermocouple. This system is known as a "floating ground" connection.

Use of High-Alumina Insulators

When ordinary porcelain insulators were used as electrical insulation between the thermocouple and Inconel core in the blackbody furnace at 1400°K some reaction occurred between the insulation and the oxidized Inconel, and between the insulation and the platinum, platinum-rhodium thermocouple. This difficulty was eliminated by substitution of high-alumina porcelain insulators for those previously used.

Sector-Disc Attenuator

Four special sector-disc attenuators were designed and constructed, and provision was made for installation in the specimen beam near the reference blackbody furnace. Each disc has eight blades; the openings between blades have widths of 33 3/4°, 22 1/2°, 11 1/4° and 5 5/8°. In operation, the discs pass 75, 50, 25 and 12 1/2 percent respectively, of the radiant flux in the intercepted beam.

The sector disc is rotated at a speed of more than 2500 r.p.m., and in a direction opposite to that of the chopper on the spectrometer. Thus the attenuator interrupts the beam at a frequency of more than 333 cycles per second, compared to the 13 cycles per second for the chopper. This rate appears to be fast enough to eliminate any coupling between the two effects in the detector and amplifier circuits.

The attenuators are used to expand the effective scale of the spectrometer, and thus to increase the precision of emittance determinations on specimens having low spectral emittance. For this purpose the appropriate attenuator disc is operated in the specimen beam for comparing radiant flux from the two blackbody furnaces. By proper adjustment of the amplifier gain, the automatically plotted curve on the strip chart under these conditions can be located between 0.9 and 1.0 on the strip chart scale. This curve then becomes a "75% curve", "50% curve", "25% curve" or "12 1/2% curve", depending on which attenuator disc was used. The

spectral emittance curve of the specimen is then taken in the usual way, with the attenuator removed from the specimen beam. Under these conditions the scale of the instrument has effectively been expanded by a factor of 4/3, 2, 4 or 8, and the precision of the measurement is increased by about the same factors. The normal spectral emittance curve is computed as described above, but each ratio so obtained is divided by the appropriate factor before plotting as the normal spectral emittance of the specimen.

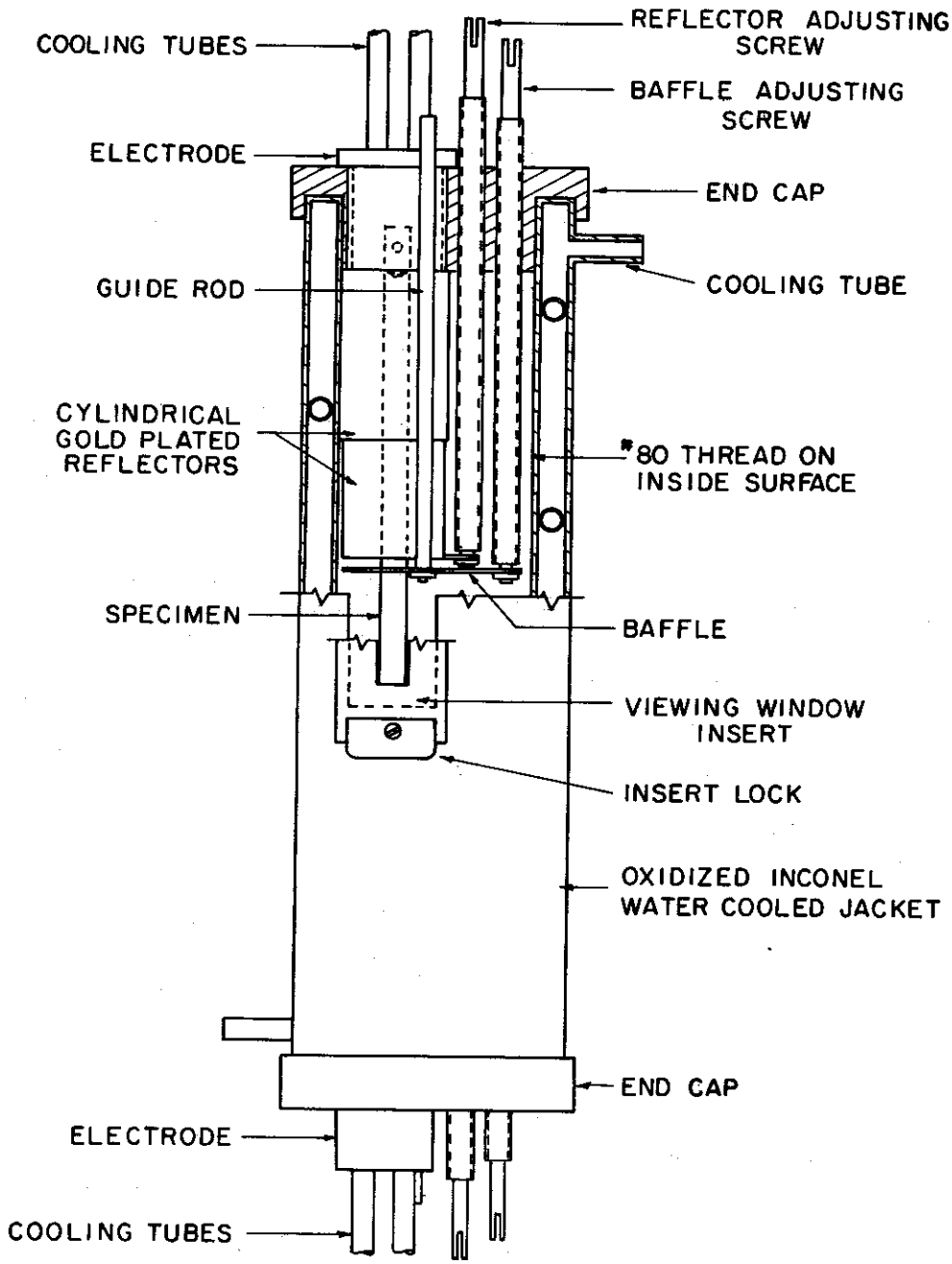
Modification of Specimen Furnace

When a specimen composed of metal such as platinum, that has a high thermal coefficient of resistivity, was heated in the specimen furnace by passing a current through it, there was a significant thermal gradient along the axis of the specimen, extending from the ends nearly to the middle. The temperature of the specimen decreased rapidly with distance from the central area in either direction, so that the portion of the specimen viewed by the spectrometer was not at uniform temperature.

Modifications were introduced into the specimen furnace as shown in Figure 4, in order to reduce the temperature gradient over the central 2-cm length of the specimen to a value so low that it could not be detected by means of an optical pyrometer. The modifications included (1) provision for adjusting the position of the baffles above and below the window in the specimen furnace, which are placed there to reduce convection currents in the furnace, and (2) the introduction of two adjustable reflectors, one surrounding each end of the specimen. Each reflector consists of two telescoping pieces of copper tubing, nominally 1 inch and 7/8 inch diameter, each 1 5/8 inch long. The inside of each tube is plated with gold, and is highly polished. In each case the large tube is permanently fastened to the end cap of the specimen furnace, and the small tube slides inside it when adjustment is made by means of a screw. In operation the reflectors reduce heat loss at the ends of the specimen, and thus reduce the thermal gradients over the observed portion of the specimen. This device was sufficiently effective so that at some temperatures, and with some alloys, it was possible to maintain a condition in which the temperature of the specimen increased along its axis, from the central portion. By proper adjustment of the convection baffles and reflectors, it has been possible to heat all specimens tested under conditions such that no thermal gradient over the central 2-cm length of the specimen could be measured with an optical pyrometer.

Control of Atmosphere

When the two blackbody furnaces were the sources of the respective beams of radiant energy, previously noted deviations of the recorded "100% curve" were pronounced at the wavelength intervals within which there is significant absorption of radiant energy by the water vapor and carbon dioxide in the atmosphere. Such absorption should be identical in the two beams if the spectral distribution of flux and optical path lengths are identical. However the two beams are separated in time, passing through the monochromator alternately at a frequency of 13 cycles per second. Since the spectrum is being traversed continuously, one energy pulse is displaced relative to the other on the wavelength scale, by a very small amount. The effect of this displacement is negligible over most of the



SPECIMEN FURNACE

Figure 4. Sketch of specimen furnace, partially cut away to show construction of adjustable gold-plated cylindrical reflectors and baffle at top. The outer shell and electrodes are water cooled. The specimen is located off-center in the cylindrical furnace in order to reduce reflection errors.

range, where the slope of the energy-vs-wavelength curve is not large. But at the wavelengths near those of maximum absorption, the energy-vs-wavelength curves become very steep, and the slight spectral displacement of the successive energy pulses can produce significant deviations in the ratios which are continuously recorded by the instrument.

In order to eliminate the effects of absorption by atmospheric water vapor and carbon dioxide, the entire external optical system of the normal spectral emittance equipment was enclosed in a lucite box. Small metal tubes were designed and constructed to connect (1) the lucite box enclosing the external optics, (2) the metal cover for the combining optics, and (3) the metal cover for the monochromator. Thus all of the optical paths in the instrument were enclosed within a single system of interconnecting housings which is nearly gas-tight. "Super-dry" nitrogen, free from carbon dioxide, was fed into the enclosure through several inlets, at a slow, constant rate, so that the enclosure was maintained at a slight positive pressure relative to the surrounding atmosphere. Gas-tight connections were installed to bring the cooling water, power leads and thermocouple leads through the metal base of the lucite box.

Operation at Wavelengths Down to 0.25 Micron

The sodium chloride prism was calibrated over the wavelength range of 0.45 to 15 microns. The prism is not recommended for use below about 1.5 microns, however, because the slope of calibration curve, in which the wavelength-micrometer reading is plotted as a function of wavelength, increases rapidly with decreasing wavelength below about 2 microns. The fused quartz prism was calibrated over the wavelength range 0.25 to 2.2 microns.

The thermocouple detector can be used at wavelengths over the range of approximately 0.6 to 15 microns (out to 40 microns with a cesium bromide window) but it loses sensitivity at wavelengths below about 1.5 microns. The photomultiplier detector with the 1P21 tube originally supplied with the instrument can be used over the wavelength range of 0.3 to about 0.6 micron. With a 1P28 tube, the lower limit is extended downward to about 0.25 micron. In order to obtain satisfactory operation of the equipment it was necessary to adjust the optical alignment each time the prisms or detectors were changed.

Several determinations were made of the normal spectral emittance of a platinum specimen over the wavelength range 0.25 to 15 microns.

Repairs and Additions to Normal Spectral Emittance Equipment.

A lead sulfide detector was ordered for use within the spectral region from 0.6 to 1.5 microns, where the available detectors do not have adequate sensitivity. However, in the range between 0.6 and 1.0 micron the lead sulfide detector also failed to meet the requirements fully, with the amount of radiant flux that was collected for measurement by the mirrors in the prevailing optical system.

The spherical mirrors of 36-inch focal length, which were furnished for use in the external optics of the normal spectral emittance equipment are 3 inches in diameter. The beam reflected by each mirror covers a solid angle of less than 5°, although the internal optics of the instrument can accommodate a beam covering a solid angle of about 15°. The use of mirrors of a size and focal length to produce a beam of the maximum solid angle that can be accommodated by the internal optics of the monochromator, would of itself increase the energy available for measurement by about an order of magnitude. This increase in energy is not necessary for determinations of normal spectral emittance of most specimens when (a) the temperature is above about $900^{\circ}K$ ($1160^{\circ}F$) and (b) the wavelength range of interest is between approximately 2 and 15 microns. For measurements at wavelengths below about 2 microns, or beyond about 15 microns, and for determinations at temperatures below about $900^{\circ}K$, an increase of this magnitude in the radiant energy reaching the detector may be critically important, at least for specimens that emit with certain spectral distributions of flux.

Accordingly, an order was placed for a transfer optical system which had recently been designed as an accessory for the Perkin-Elmer Model 13 infrared recording spectrometer, and which incorporates spherical mirrors that make full use of the relatively large numerical aperture of the monochromator optics of the instrument. Delivery is expected during August, 1960*.

Some difficulty occurred from time to time from binding of the slit micrometer so that the slit servo-motor could not drive it. Prompt manual adjustment was required to avoid detectable error from this source. Inspection of the equipment revealed that the threads on the micrometer screw were worn, and that the part should be replaced. A new slit micrometer screw assembly has been procured, and is to be installed in the near future.

Reduction of Stray Radiation

There was some indication, particularly when the photomultiplier detector was in use, that stray radiation, scattered on the interior of the monochromator, was reaching the detector and introducing an error. Baffles were installed in the monochromator to reduce such stray radiation, and subsequent tests indicated that errors from this source had become negligibly small.

B. Spectral Reflectance

Construction of special equipment for the evaluation of spectral reflectance under conditions approximating normal illumination and hemispherical viewing has been completed. A sketch showing the optical system of the equipment is shown in Figure 5.

*Funds for the purchase were made available by redistribution of available funds, involving drastic curtailment of the limited allotment for development of equipment and technique for the automatic computation of total emittance and solar absorptance from spectral emittance data.

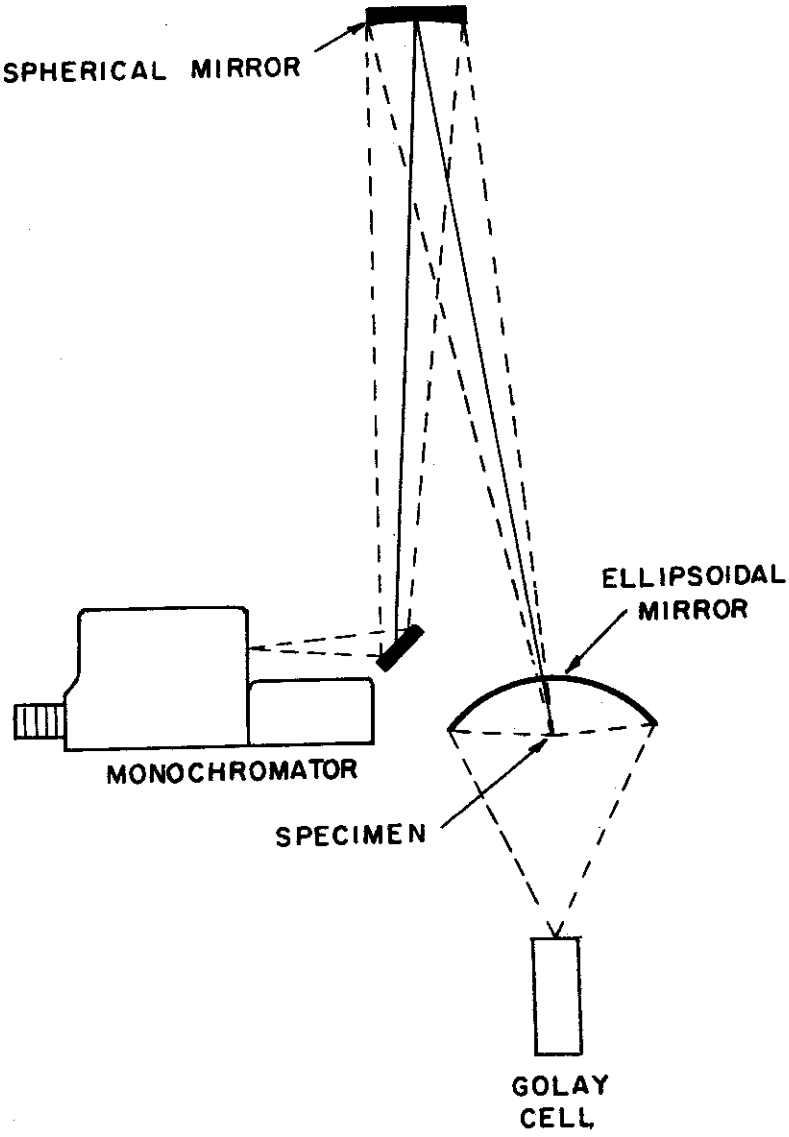


Figure 5. Sketch of optical path of beam from monochromator in apparatus for measuring the reflectance of specimens at temperatures that would be detrimental to the Golay cell.

This equipment comprises a source, a monochromator optical system, a reflectometer optical system, a detector, an amplifier, and a recorder. The source is a heated silicon carbide element that can be maintained at a uniform temperature by keeping the power input constant. Radiation from the source is chopped at 10 cycles per second, and is then focused onto the entrance slit of the monochromator. A Perkin-Elmer Model 83 monochromator, equipped with a sodium chloride prism and wavelength drive is used. The monochromatic radiant energy from the exit slit of the monochromator is focused in the reflectometer optics, by means of a 6-inch diameter spherical mirror, onto the specimen, which is located at the first focal point of an ellipsoidal mirror. The beam from the spherical mirror reaches the specimen through a small hole in the ellipsoidal mirror. The central ray of the incident beam strikes the specimen at an angle of about 7^{o} from the normal. Radiant energy reflected by the specimen is focused by the ellipsoidal mirror onto the entrance window of a Golay cell detector, located at the second focal point of the ellipsoidal mirror.

The signal from the Golay cell detector is amplified by a 10-cycle amplifier, then rectified to produce a d.c. potential, which is recorded on a potentiometer recorder.

Initially the equipment will be suitable for use only at room temperature. It is planned that the equipment will be modified to permit determinations on specimens held at temperatures above and below room temperature. The procedures to be used for calibrating the reflectance equipment have been selected, and equipment and materials required for the calibration have been procured.

Extensive measurements have been made at the Engineering Research and Development Laboratories, Ft. Belvoir, Va. on the reflectance of vapor-deposited rhodium mirrors and of "flowers of sulfur" over the wavelength range of 1 to 15 microns. Specimens of these materials have been procured, and will be evaluated. A comparison of our uncorrected data with the corrected data reported by Ft. Belvoir will yield one set of correction factors.

A modification has been introduced into the reflectometer to permit the Golay cell detector to be placed alternately at the first or second focal points of the ellipsoidal mirror. The intensity of the incident beam can be measured with the detector at the first focal point, and the apparent reflectance of a specimen can be determined with a specimen at the first focal point and the detector at the second focal point. Comparison of results obtained in this way with the reflectance of the same specimens determined in the Photometry and Colorimetry Section of the Bureau will yield a second set of correction factors.

Calibration of the reflectance equipment is proceeding as time permits.

C. Working Standards of Normal Spectral Emittance

Standards of Low Emittance

Polished platinum was selected for use as a standard of low emittance, to be used at temperatures not exceeding about 1400°K (2060°F) and at wavelengths in the range 2 to 15 microns. Platinum was selected because (1) it has relatively low spectral emittance within this wavelength interval; (2) it is relatively stable on heating in air, inert atmosphere or vacuum at temperatures up to about 1500°K (2330°F); (3) it does not change rapidly or extensively in emittance upon continued heating at temperatures up to 1500°K after it has been properly annealed at that temperature and (4) its temperature coefficient of electrical resistivity is known, and from its resistivity at a given temperature its spectral emissivity within this wavelength range can be computed with considerable reliability.

Two specimens of platinum were prepared and their spectral emittance measured over the range of 1 to 15 microns in both the Radiometry Section and the Ceramic Coatings Laboratory. Reasonably good checks with the computed spectral emittance curve were obtained, over the wavelength range of 3 to 15 microns by both laboratories. However it is important to bear in mind that these determinations are aimed primarily at the characterization of particular specimens that are to be used as working standards, rather than at the characterization of the metal, platinum.

A quantity of 40 ounzes Av. of platinum was supplied by W.A.D.D. for use in preparing specimens to be used as physical working standards of normal spectral emittance. Polished platinum specimens, 0.035 inch thick, have been fabricated, three each of each of the following sizes: discs 7/8 inch, 1, 1 1/8, and 1 1/4 inches in diameter, respectively; 2-inch squares, and strips 1/4 x 8 inches, 3/4 x 10 and 1 x 10 inches, respectively.

Standards of Intermediate Emittance

A number of composite specimens, representing a wide variety of high-temperature alloys, were prepared by applying several different ceramic coatings to heat-resisting alloys. These specimens were tested for stability during the heating in air at 1366°K (2000°F) until the coating flaked from the metal, or until the composite had withstood 247 hours of the heat treatment. During this treatment the specimens were removed from the furnace and allowed to cool in air for 30 minutes, twice during each 24-hour period.

Specimens to which coatings of flame-sprayed alumina or zirconia had been applied showed marked changes in appearance after relatively short periods of treatment, or the coating flaked from the metal during cooling, after even shorter treatment. Specimens to which NBS ceramic coating A-418 had been applied showed marked changes in appearance before completion of 247 hours of treatment, or the coating flaked from the metal on long-continued heat treatment. Specimens to which NBS ceramic coating N-143 had been applied showed only moderate visible effects from the heat treatment, the specimens having a substrate of Nichrome V showed no chipping, and only slight discoloration after 247 hours in air at 1366°K (2000°F).

Total hemispherical emittance measurements were made on specimens of N-143 coated Nichrome V, that had been subjected to heating in air for various times at 1366°K. The emittance of these specimens at or below about 866°K (1100°F) did not change significantly. However, the total hemispherical emittance at temperatures of 1088° to 1255°K (1500° to 1800°F) increased as much as 6% during 247 hours of pretreatment at 1366°K. Additional specimens of Nichrome V coated with N-143 were subjected to long-time heat treatment in air at 1172°K (1650°F). In this case the coating began to chip from the metal after heating periods of 200 to 300 hours.

In view of these results, other types of materials were investigated for use as working standards of intermediate emittance. It was noted that specimens of polished Kanthal A underwent only slight changes in appearance upon long-time heating in air at high temperatures. Specimens of this alloy were mechanically polished, weighed and subjected to heating in air at 1275°K (1835°F) for 640 hours. The specimens were removed and weighed at the end of 1/2, 1, 3, 5, 10, 20, 40, 80, 160 and 640 hours, respectively. The weight-gain data given in Table I show that during the final, 480-hour period of oxidation the total weight gain was only 153.4μg/cm^2, a rate of only 0.32μg/cm^2/hr.

TABLE I

WEIGHT GAIN DATA FOR MECHANICALLY POLISHED KANTHAL A IN AIR AT 1275°K (1835°F)

Time of Oxidation Hours	Total Weight Gain $\mu g/cm^2$ hour	Rate of Weight Gain Total Time $\mu g/cm^2$ hour	Rate of Weight Gain Last Period $\mu g/cm^2$ hour
1/2	77.7	155.4	155.4
1	86.9	96.9	18.4
3	103.9	34.6	8.5
5	113.5	22.7	4.8
10	137.2	13.7	4.7
20	159.1	7.95	2.2
40	186.6	4.66	1.37
80	230.2	2.88	1.09
160	300.2	1.88	0.88
640	453.6	0.71	0.32

The normal spectral emittance curves of Kanthal A specimens that had been oxidized for periods of 160 and 640 hours, respectively, at 1275°K (1835°F) are shown in Figure 6. Obviously the oxidized Kanthal had not become sufficiently stabilized after 160 hours of oxidation for use as a working standard of spectral emittance.

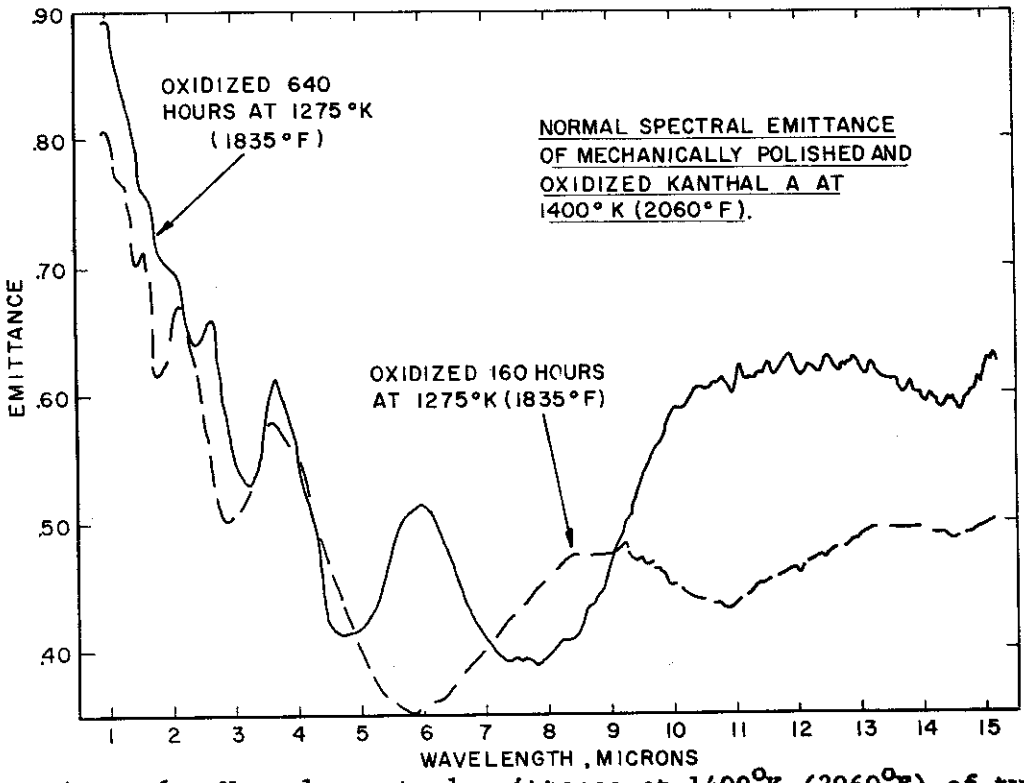


Figure 6. Normal spectral emittance at 1400°K (2060°F) of two specimens of Kanthal A that had been mechanically polished and then oxidized by heating in air for 160 and 640 hours, respectively, at 1273°K (1832°F).

Figure 6 does show, however, that if on further oxidation, or on oxidation at a higher temperature, sufficiently stable specimens can be produced from oxidized Kanthal, they will have several interesting properties that tend to make this composite particularly suitable for use as a working standard of intermediate emittance. The several sharp emittance peaks could be used as a check of the wavelength calibration of an instrument, and the wide range of spectral emittance at different wavelengths makes the curve suitable for a rough check of the scale linearity of an instrument.

The emittance peaks in the curves plotted in Figure 6 are typical of peaks that are formed by interference phenomena in a composite specimen consisting of a thin, transparent or partially transmitting coating over an opaque substrate. The shift to longer wavelengths of the peaks with variation in coating thickness (oxidation period) is consistent with this interpretation.

The data in Table I show that the average increase in weight during the 480-hour oxidation period from 160 to 640 hours was only 0.32 microgram per square centimeter per hour. If it is assumed that the oxidation obeys the parabolic rate law, as appears at least approximately true from the data, the rate of weight-gain at 640 hours is only 0.23 microgram per square centimeter per hour. These data suggest that it may be possible by oxidation under the proper conditions to produce oxidized specimens of Kanthal A that are sufficiently stable in emittance to warrant their use as working standards of normal spectral emittance.

Several additional specimens of Kanthal A were mechanically polished, and are being oxidized in air at 1366°K (2000°F). They will be removed and tested for normal spectral emittance after periods of 300, 400 and 500 hours.

Standards of High Emittance

Sandblasted Inconel that had been oxidized in air at 1366°K (2000°F) for five hours was selected for use in preparing working standards having high normal spectral emittance at all wavelengths. Specimens of this material were calibrated at temperatures of 780°K (947°F), 1050°K (1430°F) and 1400°K (2060°F) in the Radiometry Section of the National Bureau of Standards, as reported in WADC TR 59-510 (August 1959). Data on these specimens at 1400°K have been obtained in the Enameled Metals Section, and the agreement was reasonably good over the wavelength interval from about 2 to 15μ.

New specimens, three each in the following sizes and shapes, have been prepared from Inconel: - discs 7/8, 1, 1 1/8 and 1 1/4 inches in diameter, respectively, 2-inch squares, and strips 1/4 x 8, 3/4 x 10 and 1 x 10 inches, respectively. These are for use in equipment for emittance measurements that requires shapes different from that used in the NBS equipment reported here.

Measurements of Emissivity of Platinum; its Relationship to Resistivity

The Radiometry Section used two types of spectrometers in the comparison of radiation from test specimens with that from a blackbody. One of the instruments has a double-pass optical system which gives better resolution and less stray radiation. Besides the work on platinum there were some preliminary measurements in the Radiometry Section on low-emittance metal samples such as gold and aluminum. Techniques were developed for obtaining accurate measurements on low-emittance samples at temperatures as low as 523°K (482°F). For such low temperatures it is imperative that stray radiation be reduced to a minimum and that a correction be made for detector temperature, room temperature, and the slit temperature.

As a part of the Radiometry Section's approach to measuring the emittance of metals, studies were carried out on the possibility of utilizing calculations to reduce the experimental work and increase the accuracy of results. Table II gives values of the normal spectral reflectivity of aluminum at ambient temperature, as reported in the literature, for comparison with values calculated according to Schmidt and Eckert's modification[1/] of the Davisson and Weeks' equation

$$\epsilon = 0.365\ (\rho/\lambda)^{\frac{1}{2}} - 0.0464\rho/\lambda$$

where ϵ = emissivity, ρ = electrical resistivity, and λ = wavelength.

The calculated values of reflectivity were obtained by substituting (1-R) for ϵ in this equation, where R = reflectivity. There is reasonably good agreement between the calculated and observed values within the 4 to 12 micron range of wavelength covered in Table II.

Measurements on platinum similarly show a close correlation between experimentally observed emissivity and emissivity calculated from the Schmidt-Eckert equation. As illustrated in Table III, by means of this equation, one may calculate the emissivity of platinum for conditions under which measurements are not available, or are not easily obtainable. Thus spectral emissivity values for platinum have been calculated for very low temperatures, as given in the last two columns in Table III. In practice one should be cautious in the use of values thus calculated for conditions outside the range in which experimental evidence is available. The lower limit of the wavelength interval in which the Schmidt-Eckert equation is applicable is different for different metals and is a function of temperature. Furthermore, although the equation seems to work well for platinum and aluminum within the spectral limits shown in these tables, the computed spectral values for gold are about twice as large as the measured emissivity values. The cause of this discrepancy has not been resolved. Data reported in the literature, however, indicate that the experimental values are not grossly at fault.

Emissivities computed from the Schmidt-Eckert relationship are known to be in error at short wavelengths, the wavelength at which the errors become significant being a function of both the material involved and its temperature. The discrepancies at short wavelengths are attributed to the fact that the relation-

1/Schmidt, E., and Eckert, E., Forschung a.d.Geb.d. Ingenieurwes. 6, 175; 1935

TABLE II

NORMAL SPECTRAL REFLECTIVITY OF ALUMINUM

λ (Microns)	Bennett and Koehler[1]	Beattie[2]	Calculated
12	98.0	98.1	98.3
10	97.9	98.0	98.1
8	97.8	98.0	97.9
6	97.75	97.7	97.6
5	97.7	97.8	97.4
4	97.5	97.5	97.2

1/ J. Opt. Soc. Am. 50, 1 (1960)

2/ Phil. Mag. 46, 235 (1955)

TABLE III

EMISSIVITY OF PLATINUM

Wavelength (Microns)	Observed Emissivity 1400°K (2060°F)	Calculated Emissivity 1400°K (2060°F)	Calculated Emissivity 273°K (32°F)	Calculated Emissivity 243°K (-86°F)
1			0.110	0.103
2	0.179	0.165	0.079	0.074
3	0.139	0.137	0.065	0.061
4	0.117	0.119	0.056	0.053
5	0.100	0.107	0.050	0.047
6	0.090	0.098	0.046	0.043
7	0.090	0.091	0.043	0.040
8	0.093	0.085	0.0399	0.0375
9	0.091	0.081	0.0376	0.0354
10	0.087	0.077	0.0357	0.0336
11	0.080	0.073	0.0341	0.0320
12	0.075	0.070	0.0327	0.0307
13	0.068	0.067	0.0314	0.0295
14	0.064	0.065	0.0303	0.0284
15	0.065	0.063	0.0293	0.0275
16			0.0283	0.0266
17			0.0275	0.0258
18			0.0267	0.0251
19			0.0260	0.0244
20			0.0254	0.0238

ship depends upon the assumption that a metal acts as a homogeneous material, in its response to radiation of the longer wavelength; this assumption is valid at the longer wavelengths, but not at the shorter wavelengths.

D. Data Processing System

The automatic processing of normal spectral emittance data was not attained during the report period , because of the reallocation of funds that were originally provided for the purchase of data-processing equipment to other phases of the project, as explained in Section A (page 11). The system shown as a block diagram in Figure 7 was designed early in the year, but lack of funds for the purchase of components prevented its construction.

The system provides on-line reduction, by the selected ordinate method, (see WADC TR 56-510 for a discussion of the selected ordinate method) of the spectral emittance data into a single value for (a) total emittance, or (b) absorptance for radiant flux of any specified spectral distribution. In addition, the spectral emittance data will be recorded in digital form, suitable for entry into an off-line computer, if desired.

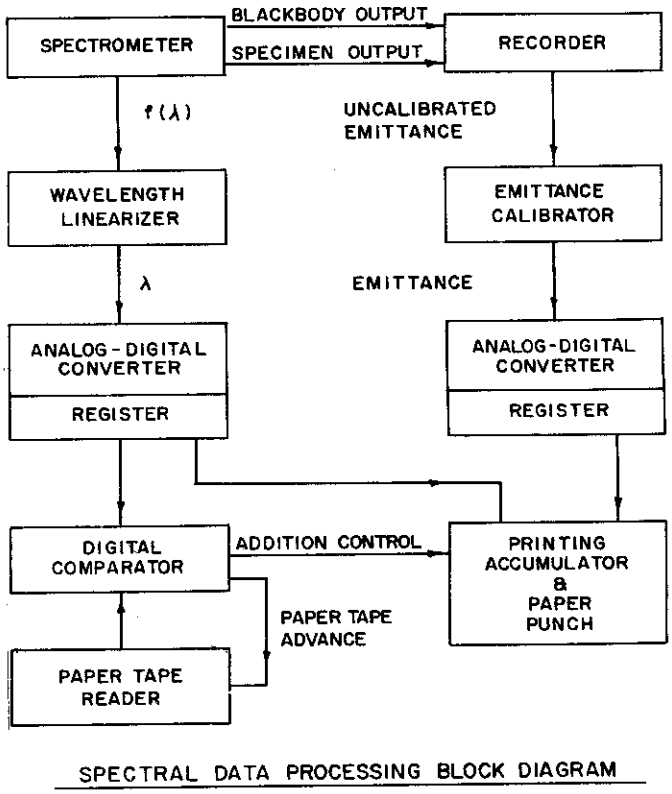


Figure 7. Block diagram of electronic processing equipment for spectral emittance data.

Operation of the system is as follows: The output of the spectrometer and its associated recorder is a plot of apparent emittance vs rotation increments of the wavelength screw-drive, which are non-linearly related to wavelength. The recorder deflection, after application of calibration factors, based on the apparent spectral emittance curve of the reference blackbody furnace (an example of which is shown in Figure 2) is proportional to emittance. The increments of wavelength for equal increments of rotation of the wavelength drive screw are dependent upon the characteristics of the prism used in the monochromator. Three prisms are used, fused quartz for the wavelengths of less than 1 micron, sodium chloride for the wavelength range 1 to 15 microns, and cesium bromide for the wavelength range 10 to 40 microns. It is desirable to record the emittance data, for processing, at uniform increments of wavelength. From a practical standpoint, it is also desirable to keep the modifications of the spectrometer to a minimum. The proposed solution is to attach a simple transducer to the wavelength

drive screw, and provide an external follow-up servo having a feed back element properly shaped to linearize the wavelength output. Appropriate feedback elements would be provided for each of the prisms mentioned above. The output of the servo would be used to drive an analog-to-digital converter. A similar A-D converter would be driven by the calibrated recorder deflection, either directly or through a servo follow-up drive. The outputs of these two A-D converters would feed an electrically operated printer and paper tape punch (through buffer registers if necessary) of which there are several commercially available. Such a device also has the ability to keep a running addition of entered quantities when ordered to do so. Up to this point in the process no calculations will have been made, but the data will have been preserved, both as a listing of emittance values vs wavelength, and also in the form of punched paper tape suitable for direct entry into off-line automatic computers.

For on-line results, a paper tape would be prepunched with the proper selected ordinates for any desired spectral distribution, such as that of a blackbody at the temperature of the specimen. This tape would be inserted into a tape reader, whose output would be fed into a digital comparator. The output of the A-D converter corresponding to wavelength would also be fed to the comparator. Whenever the wavelength at which emittance is being measured is the same as that for one of the preselected ordinates, the comparator will cause the printing accumulator to add and store the measured value of emittance. At the completion of the addition, the comparator will cause the tape reader to advance to a new setting, which will be retained until the wavelength drive reaches the next selected value, when the sequence is repeated. When a predetermined cut-off wavelength has been reached (beyond which the radiant energy may be neglected), the summation of selected-ordinate emittance values, will be printed out. In the case cited this sum will represent the total emittance of the specimen. In other cases it would represent the absorptance for radiant flux having the given spectral distribution.

The prepunched paper tapes could be stored and used repeatedly, and over a period of time a library of such tapes could be accumulated, for the computation of absorptance for a number of different sources of radiant energy.

V. PLANS FOR FUTURE WORK

As indicated in Section III of this Report, the general objectives of the project encompass an extensive field of investigation, which could not be completed in a one-year period.

It is planned to complete determinations of the normal spectral emittance of the working standards having low and high emittance, respectively, for use in the temperature range of approximately 780° to 1400°K (945° to 2060°F), after installation of the new transfer optics system is completed. Selection and preparation of the working standards of intermediate emittance for use in this temperature range will proceed concurrently.

During fiscal 1961 priority will be assigned to the following three phases of the program: (a) extension of the wavelength range of equipment previously developed on this project, downward to wavelengths below one micron, and upward to wavelengths above 15 microns, (b) extension of the temperature range downward by use of the reflectance equipment described in this report, the first of these experiments to be made at room temperature, and (c) completion of the design, procurement and installation of components for the data-processing system described in this report.

Coincident with each of these developments, physical working standards of normal spectral emittance throughout the added temperature and wavelength ranges will be prepared for submission to W.A.D.D.