NATIONAL BUREAU OF STANDARDS REPORT

7467

STANDARDIZATION OF THERMAL EMITTANCE MEASUREMENTS

PROGRESS REPORT No. 14
October 1 - December 31, 1961

Contract No. DO (33-616) 61-02
Task No. 73603

AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT 1009-11-10491

NBS REPORT 7467

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PROGRESS REPORT No. 14
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Task No. 73603

The two phases of this project are conducted under the supervision of the following persons, who have approved this project.

Horace M. Joseph
Electronic Engineer, Data Processing Systems Division

William N. Harrison - Coordinator
Chief, Enameled Metals Section, Mineral Products Division

to
AERONAUTICAL SYSTEMS DIVISION
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I. SUMMARY

Calibration of the platinum working standards of normal spectral emittance was completed. A statistical analysis of the data obtained in approximately 5400 measurements indicates that the error of measurement, expressed as a standard deviation, was approximately 0.4 in units of emittance x 100 (or 0.004 without the multiplier). This analysis also showed that the error of measurement was nearly constant over the temperature range of 800° to 1400°K, and over the wavelength range from 1 to 15 microns.

It was found that there were real differences in emittance between the different specimens measured, even though they had been prepared from the same batch of platinum and were subjected to the same treatment throughout. These differences, expressed as a standard deviation, were on the order of 1.2 in units of emittance x 100 (or .012 without the multiplier). The differences in emittance between specimens did not vary appreciably with temperature over the range 800° to 1400°K, but did vary significantly with wavelength.

Construction of the data-processing equipment was nearly completed, and installation during the next report period was promised.

II. INSTRUMENTATION

A. Normal Spectral Emittance Equipment

Some maintenance work on this equipment was required during the report period. A vibrator in the amplifier used in the differential temperature control developed a malfunction and was replaced. Also the 80 nickel-20-chromium alloy cores of the two blackbody furnaces were detached for removal of accumulated oxide, and replaced.

B. Reflectance Equipment

The design and construction of equipment for the measurement of reflectance under conditions approximating normal illumination and hemispherical viewing was described in WADC Technical Report 59-510 Part III. Construction of this equipment was financed in part by the Marshall Space Flight Center of NASA.

The vibration-dampening supports referred to in WADC Report 59-510 Part III were reasonably effective in isolating the Golay detector from building vibration. It was found on further investigation that part of the interfering vibration was caused by gears on the wavelength drive of the monochromator. This latter vibration was not eliminated by the supports since the monochromator is mounted on a table that is rigidly fastened to the framework supporting the detector. Such rigid connection is required in order to maintain optical alignment of the several mirrors in the system. A new air-spring mount for the detector was designed and built, and is functioning well. Oscilliscope tests of the equipment indicate, however, that the actual resonant frequency is slightly greater than the design frequency, probably due to an effect of the diaphragm material. Use of the new support system has indicated that vibration is no longer a serious problem, but further smoothing of the signal is to be undertaken by addition of an appropriate electronic filter.

It is desirable that the detector be placed alternately at the two foci of the ellipsoidal mirror, for checking the spectral reflectance curve of the standard. The sensitivity adjustment on the Golay detector is quite critical, and in order to control and stabilize this adjustment, a mechanical system for positioning the detector was designed, built and installed. Also, the specimen mount was modified to facilitate installation of the specimen-heating device. The ellipsoidal mirror was resurfaced, and handles were cemented to the outside, to facilitate lifting it. A new drum for the string drive was purchased and installed, which gives a considerably flatter "100% curve" over the wavelength range of 3 to 15 microns than the previous drive did.

A light-tight enclosure for the reflectometer to isolate the detector from the room light was constructed and installed. The rear panels of this enclosure were hinged to afford easy access for inserting specimens or adjusting the detector.

The work of calibrating the reflectometer is proceeding.

III. CALIBRATION OF WORKING STANDARDS

A. Platinum Specimens

Normal spectral emittance curves for the platinum working standards were established over the wavelength range of approximately 1 to 15 microns and at temperatures of 800°, 1100° and $1400^{\circ}K$.

The platinum specimens were received from the fabricator as 0.035-inch sheet, three each in the following shapes and sizes: discs 7/8, 1, 1 1/8 and 1 1/4 inches in diameter, 2 x 2 inch squares, and strips 1 x 10, and 3/4 x 10 inches in size, and six 1/4-x-8-inch strips. It had been specified that the specimens were to be supplied with highly polished surfaces. The finish actually supplied was not as smooth as had been desired, but it was decided to use the finish supplied, rather than send the specimens back for reworking. A 1/8-inch hole was drilled in one end of each strip specimen, to facilitate hanging during annealing.

Each specimen was washed in hot tap water to which a commercial detergent had been added, rinsed in running hot tap water, then in distilled water, and finally in ethyl alcohol. Rubber surgical gloves were worn at all times while handling the specimens, and the central portion, observed for emittance determinations, was not touched after cleaning. The specimens were dried in air and placed in a closed container, supported by the ends or edges only, for storage prior to annealing.

All specimens were annealed in an electrically-heated, silicon-carbide-element furnace. The strip specimens were hung by means of platinum hooks suspended from aluminum oxide rods in the furnace; the square and disc specimens were supported by the edges only on ceramic forms resting on a flat ceramic slab. All of the specimens were then enclosed in a ceramic muffle. Starting with a cold furnace, the temperature of the furnace was raised to 1523°K (1250°C) over a period of six hours, and held at that temperature for one hour. The power was then turned off, and the specimens were allowed to cool in the furnace, which required two days.

The specimens were removed from the furnace by means of cleaned platinum-tipped tongs and were placed in individual plastic holders, in which they were supported only by the ends or edges. Each plastic holder, containing a specimen, was then placed in an individual cardboard box, to protect it from contamination.

All of the specimens thus prepared, except the 1/4-x-8-inch strips, were delivered to the Aeronautical Systems Division by hand on October 31, 1961.

The six 1/4-x-8-inch strips were prepared for measurement by welding a platinum-platinum, 10% rhodium thermocouple to each specimen. A shallow groove was scratched in each specimen, normal to its axis and located at the mid-length. The 10-mil thermocouple wires were separately welded to the specimen by means of a condenser-discharge type of electronic spot welder. Each wire was laid in the shallow groove to position it for welding, and the welding operation was observed through a low-power microscope.

Precautions were taken at all times to avoid contamination of the specimens. They were handled as little as possible, and when handling was unavoidable the use of rubber gloves was continued, and even then the center portion of the specimen was not touched.

Three sets of curves were made for each strip specimen at each of three temperatures, 800°K, 1100°K, and 1400°K. Each set of curves consisted of (1) a "100% curve", obtained when the two blackbody furnaces at the test temperature were the sources for the respective beams, (2) a "zero curve", obtained when the specimen beam was blocked near the specimen furnace, and (3) a specimen curve, obtained with the comparison blackbody at the test temperature as one source and the specimen at the same temperature, as the other. Each curve was recorded over the range of wavelength drum settings of 16.7 to 1.2 turns (16.5 to 1.2 at 800°K), corresponding to a wavelength range of approximately 1.0 to 15.0 microns.

The normal spectral emittance was computed at each 0.1 turn of the wavelength drum from 16.7 to 12.0, and at each 0.2 turn from 12.0 to 1.2 turns, a total of 100 points. The wavelengths corresponding to these settings are given in column 1 of table 1. The normal spectral emittance was computed as the height of the specimen curve above the zero curve divided by the height of the 100% curve above the zero curve.

The data for the 18 determinations at each temperature and wavelength drum setting were tabulated, and the following values were computed: (1) E, the arithmetic average of the 18 measured values; (2) σ_t, the total standard deviation[1/] of the 18 values about the average of the 18 values; (3) e, the 95% confidence error of E; (4) σ_m, the average of six standard deviations, each computed from the three measured values on one of the six specimens, and (5) σ_s, the standard deviation of the six average values, one for each of the six specimens, about the grand average for all six. All of these computed values are given in table I.

The average normal spectral emittance of the six platinum specimens E, is plotted as a function of wavelength for readings at 800°K in figure 1, at 1100°K in figure 2, and at 1400°K in figure 3.

1/ All standard deviations computed in this study are precisely defined as "estimates of the standard deviation of the parent population from which the measurements were drawn." This quantity is assigned the symbol σ, to distinguish it from the root-mean-square standard deviation S.D. of individual values from the mean of a given sample.

It is apparent that the normal spectral emittance at each wavelength increases with an increase in temperature. This effect would be predicted from electrical conductivity, according to the Hagen-Rubens equation. The maximum that occurs at a wavelength of about 1.8 microns at 800°C shifted to shorter wavelengths with increase in temperature. The maximum at about 9.5 microns did not shift in wavelength with increase in temperature, but diminished in magnitude. A third maximum in the vicinity of 15 microns is not visible at 800°K, but appears at 1100°K and increases in magnitude with temperature. Spectral data at wavelengths longer than 15 microns will be required to establish with certainty whether or not this maximum shifts with temperature.

The lower curves in figures 1, 2 and 3 are plots of the 95% confidence errors[2/] of the average emittance values plotted in the upper curves.

The value σ_m is a measure of the reproducibility of the test procedure, or the precision measurement. The average value of σ_m, for all of the 100 wavelengths, is 0.35 at 800°K, 0.45 at 1100°K and 0.36 at 1400°K. Thus the overall precision of measurement for the equipment and test procedure is on the order of ± 0.4, in units of emittance x 100 (or 0.004 without the multiplier). In order to show any trend of σ_m with wavelength, the moving average for five values at adjacent wavelengths was computed, and plotted as a function of wavelength to obtain the lower curves in figures 4, 6 and 6, respectively. This curve is identified in each figure as the standard deviation due to error of measurement. The scatter due to errors of measurement, σ_m, does not vary appreciably with either wavelength or temperature.

The value σ_s indicates actual differences in emittance of the specimens that were measured. The average value of σ_s for all 100 wavelengths is 1.22 at 800°K, 1.29 at 1100°K and 1.18 at 1400°K; the grand average for scatter due to real differences between specimens is ± 1.23, in units of emittance x 100 (or 0.012 without the multiplier) and does not vary appreciably with temperature.

2/ The 95% confidence error has the following statistical significance. If the measurements were repeated a large number of times, say 1000 times, and the average and 95% confidence error was computed for each group of 18 measurements, then the limits of the group average ± the 95% confidence error would bracket the overall average of the 1000 groups of measurements about 19 times out of 20.

In order to show any trend of σ_s with wavelength, the moving average of five values at adjacent wavelengths was computed, and plotted as a function of wavelength to produce the upper curve in figures 4, 5 and 6, respectively. The curve is identified in each figure as the standard deviation due to differences in specimens. These curves show a definite relation of scatter to wavelength, and are similar in shape.

The values of σ_s are larger than the corresponding values of σ_m at each wavelength at each temperature, by an amount sufficient to demonstrate statistically that the observed differences between specimens are real, and could not occur due to chance fluctuations of the error of measurement.

B. Use of Emittance Data

The data given in table I and plotted in figures 1, 2 and 3 apply only to measurements made with the NBS equipment and procedures, on the six specimens that were measured. If other equipment and procedures are used on these specimens, the values for E and σ_s still apply. However, the values for σ_m will then depend upon the precision of the equipment and procedure used for the measurements, and the values of σ_t and e will depend in part on such measurements.

Platinum working standards of other sizes and shapes, which were prepared at the same time and from the same material as the 1/4-by-8-inch specimens from which the data in table I were obtained, were not measured at the National Bureau of Standards. They can, however, be used as working standards of normal spectral emittance, with the assigned values of E and σ_s given in table I. The true E value for any one of these specimens may vary from the corresponding E value given in table I by an amount that can be statistically predicted on the basis of the reported σ_s value, but the actual magnitude and sign of this difference for any individual specimen cannot be computed.

For equipment and procedure other than those here reported, new replicate measurements on different specimens, at different wavelengths, similar to those made to obtain the values reported in table I, are required to ascertain separately, by statistical methods, the scatter due to variation in specimens and that due to random error of measurement.

Systematic error of measurement, unlike random error, will produce average values consistently too low or too high. If newly determined emittance values are systematically higher or lower than the corresponding values in table I, an analysis will reveal whether or not the observed difference is statistically significant, or may be attributed to random errors of measurement.

IV. MATHEMATICAL EQUATIONS

The attempt to establish a suitable "program" for use with electronic computers in solving the equations relating the absorption-index and index of refraction of a metal to the contributions of bound and unbound electrons to the reflectance (emittance) characteristics of metals has continued. These equations are given as equations [2], [3] and [4] in WADC Technical Report 59-510 Part III.

Numerical experiments aimed at approximately locating acceptable values of the parameters were continued. It was established that the use of five parameters - corresponding to the assumption of the existence of essentially one "family" of free electrons and one "family" of bound electrons - is insufficient for a good fit of the experimental data throughout the 0.300 to 10.000 micron range of wavelengths. New programs were written, and experiments aimed at the utilization of a larger number of parameters, corresponding to additional "families" of electrons, are planned.

V. DATA PROCESSING EQUIPMENT

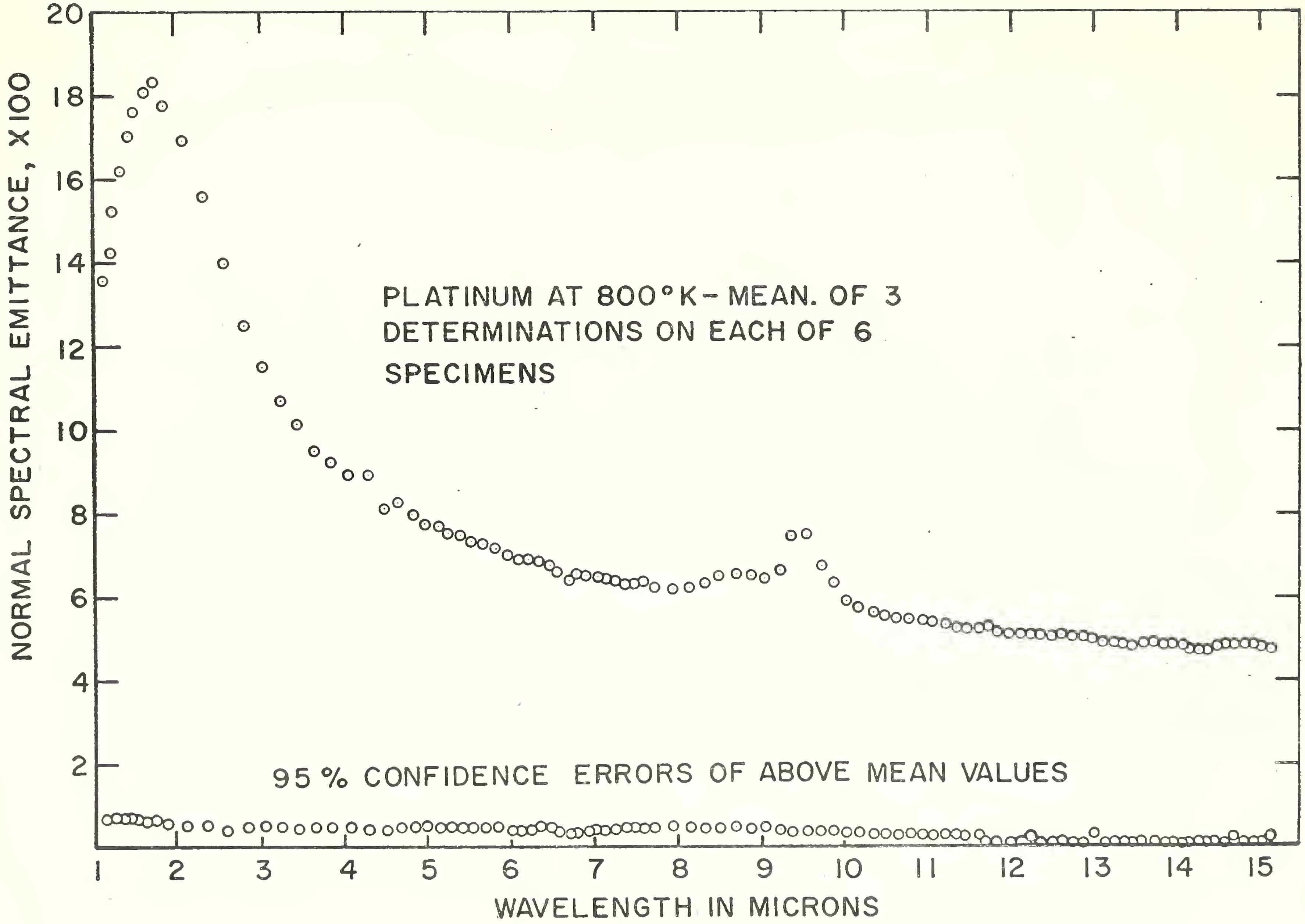
During the report period, the manufacturer has been completing the data-logging equipment for supplying recordings of spectral data runs on punched paper tape. Although delivery was originally expected in the middle of November 1961, a number of difficulties appeared which delayed completion of the equipment. At the request of the manufacturer, Mr. Horace M. Joseph of the National Bureau of Standards during December visited the laboratory where the equipment was being assembled. During his visit, the equipment was demonstrated, and essential portions operated successfully. It appeared that the equipment should be finished and delivered during the next report period.

The functions of these portions were the digitization of the drum dial rotation, the display of the resulting numbers, the adding of the numbers in an "accumulator", the punching of numbers on the paper tape, and the control of equipment-punching by use of an external reader of punched-paper tape. In addition, the punching and accumulation could be controlled by choosing to punch or accumulate at every 1st, 2nd, 5th, 10th or pre-selected (taped) drum dial position. The choice was independent and could be different for output punching and accumulation. (The output digitizer between the electronic counter display and the punch was not yet finished.)

There are two shaft encoders; one on the wavelength drum and one on the drum of the potentiometer controlling the pen position and the recorder. These shaft encoders utilize duplicate, coaxially mounted, transparent discs, each printed with curved black stripes from center to periphery. Two photocells at different positions are used to sense light passing between the stripes of both discs. One disc is stationary; the other rotates with its shaft. The assembly produces varying light intensity upon the photocells, depending upon the interference of the stripes. The photocells are carefully placed so that the signal appears first in one cell for clockwise rotation, and first on the other cell for counterclockwise rotation. These light signals are counted, and the counter output appears as pulses on appropriate channels according to the weight of the particular binary digit.

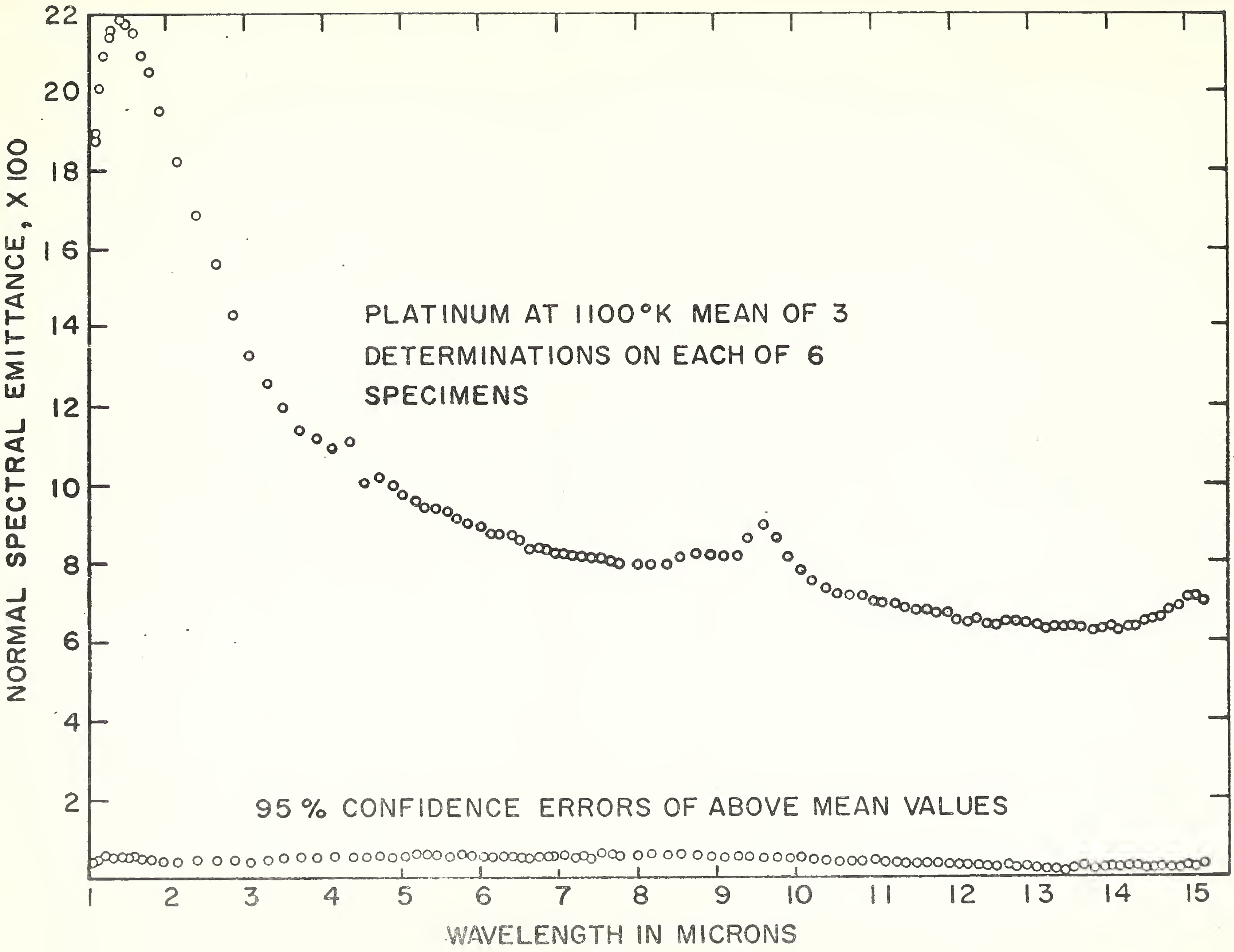
The digits are recorded on a paper tape in a code (as decided earlier) called the Friden Programmatic Single Case Code. This is a fairly common code. It would be fairly easy to adapt the equipment for use of any one of quite a number of other codes.

Figure 1. Average normal spectral emittance of six platinum working standards at 800°K (upper curve), and the 95% confidence errors associated with the plotted average values (lower curve).



NORMAL SPECTRAL EMITTANCE, X100
20
18
16
14
12
10
8
6
4
2
PLATINUM AT 800°K - MEAN. OF 3
DETERMINATIONS ON EACH OF 6
SPECIMENS
95 % CONFIDENCE ERRORS OF ABOVE MEAN VALUES
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
WAVELENGTH IN MICRONS

Figure 2. Average normal spectral emittance of six platinum working standards at 1100 $^{\circ}$K (upper curve), and the 95% confidence errors associated with the plotted average values (lower curve).



PLATINUM AT 1100°K MEAN OF 3
DETERMINATIONS ON EACH OF 6
SPECIMENS
95 % CONFIDENCE ERRORS OF ABOVE MEAN VALUES
NORMAL SPECTRAL EMITTANCE, X 100
WAVELENGTH IN MICRONS

Figure 3. Average normal spectral emittance of six platinum working standards at 1400°K (upper curve), and the 95% confidence errors associated with the plotted average values (lower curve).

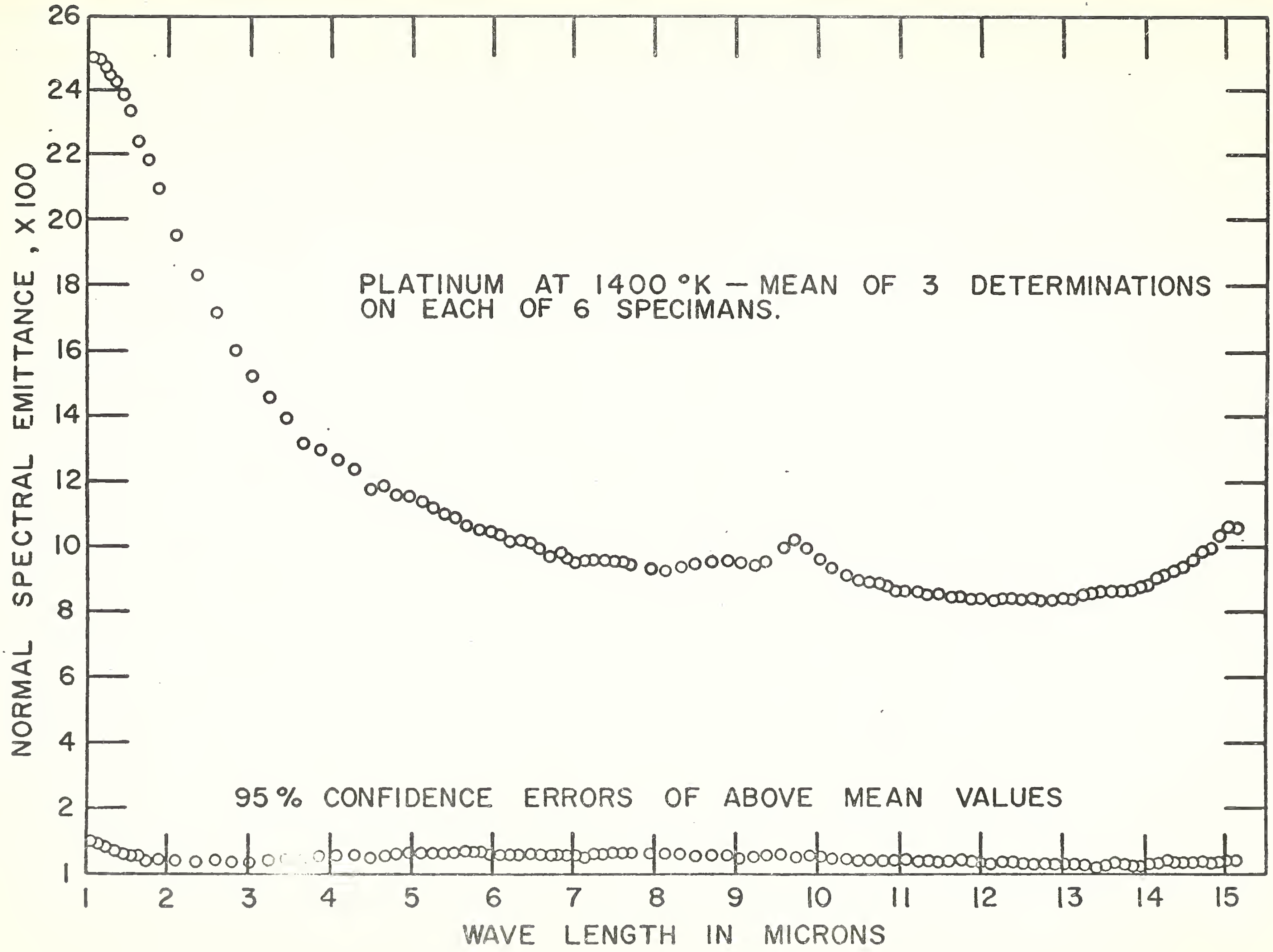
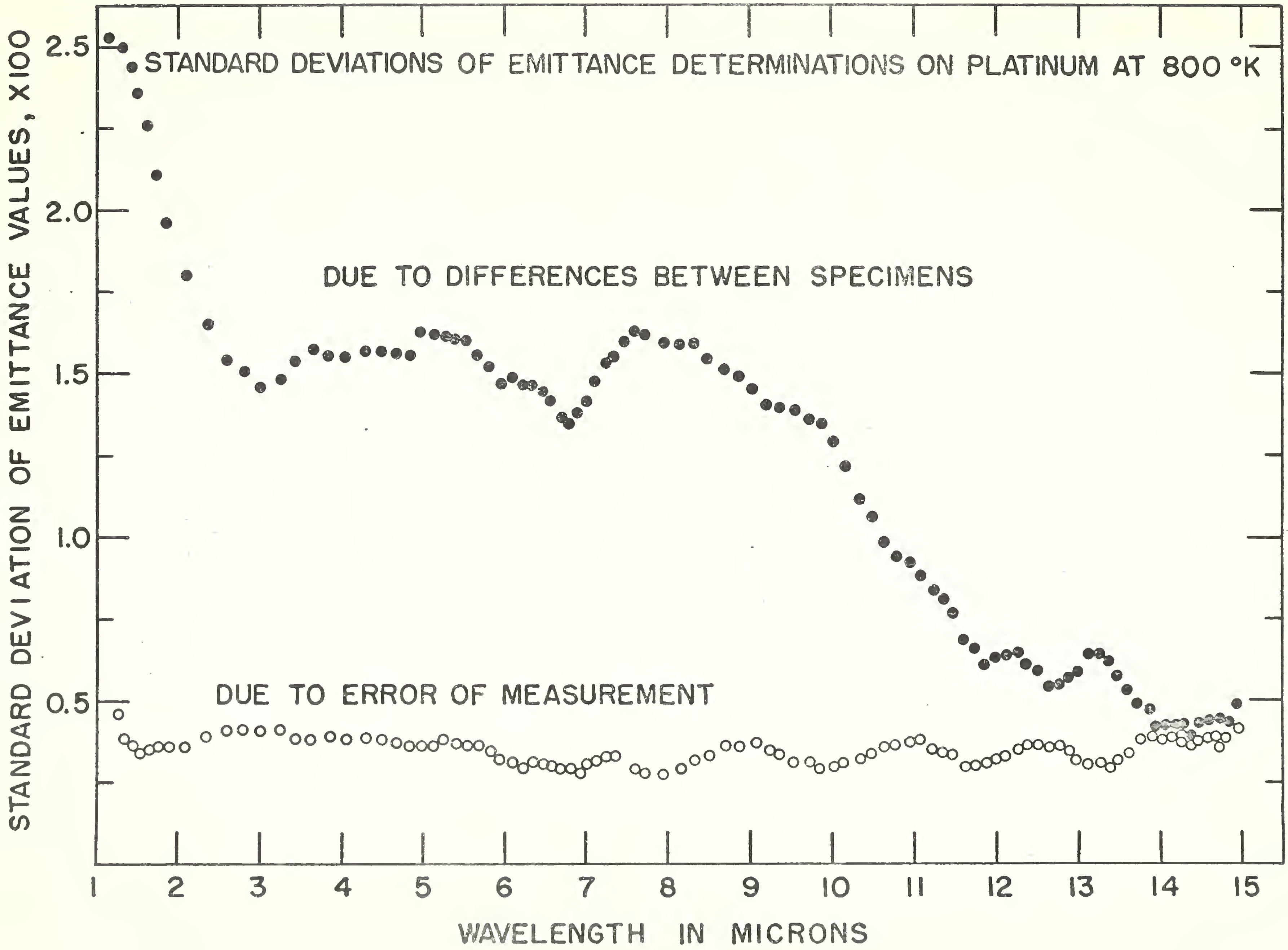

PLATINUM AT 1400 °K — MEAN OF 3 DETERMINATIONS
ON EACH OF 6 SPECIMANS.
95 % CONFIDENCE ERRORS OF ABOVE MEAN VALUES
NORMAL SPECTRAL EMITTANCE , X 100
WAVE LENGTH IN MICRONS
26
24
22
20
18
16
14
12
10
8
6
4
2
1
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15

Figure 4. Spectral distributions of two categories of standard deviations, each computed from 18 measured emittance values obtained at $800^{o}K$, three each on six platinum working standards. The upper curve represents standard deviations due to real differences in emittance between specimens, identified as σ_s in the text. The lower curve represents standard deviations due to random error of measurement, identified as σ_m in the text. In both curves each point represents the moving average of five adjacent values.



STANDARD DEVIATIONS OF EMITTANCE DETERMINATIONS ON PLATINUM AT 800 °K
DUE TO DIFFERENCES BETWEEN SPECIMENS
DUE TO ERROR OF MEASUREMENT
STANDARD DEVIATION OF EMITTANCE VALUES, X100
2.5
2.0
1.5
1.0
0.5
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
WAVELENGTH IN MICRONS

Figure 5. Spectral distributions of two categories of standard deviations, each computed from 18 measured emittance values obtained at 1100°K, three each on six platinum working standards. The upper curve represents standard deviations due to real differences in emittance between specimens, identified as σ_s in the text. The lower curve represents standard deviations due to random error of measurement, identified as σ_m in the text. In both curves each point represents the moving average of five adjacent values.

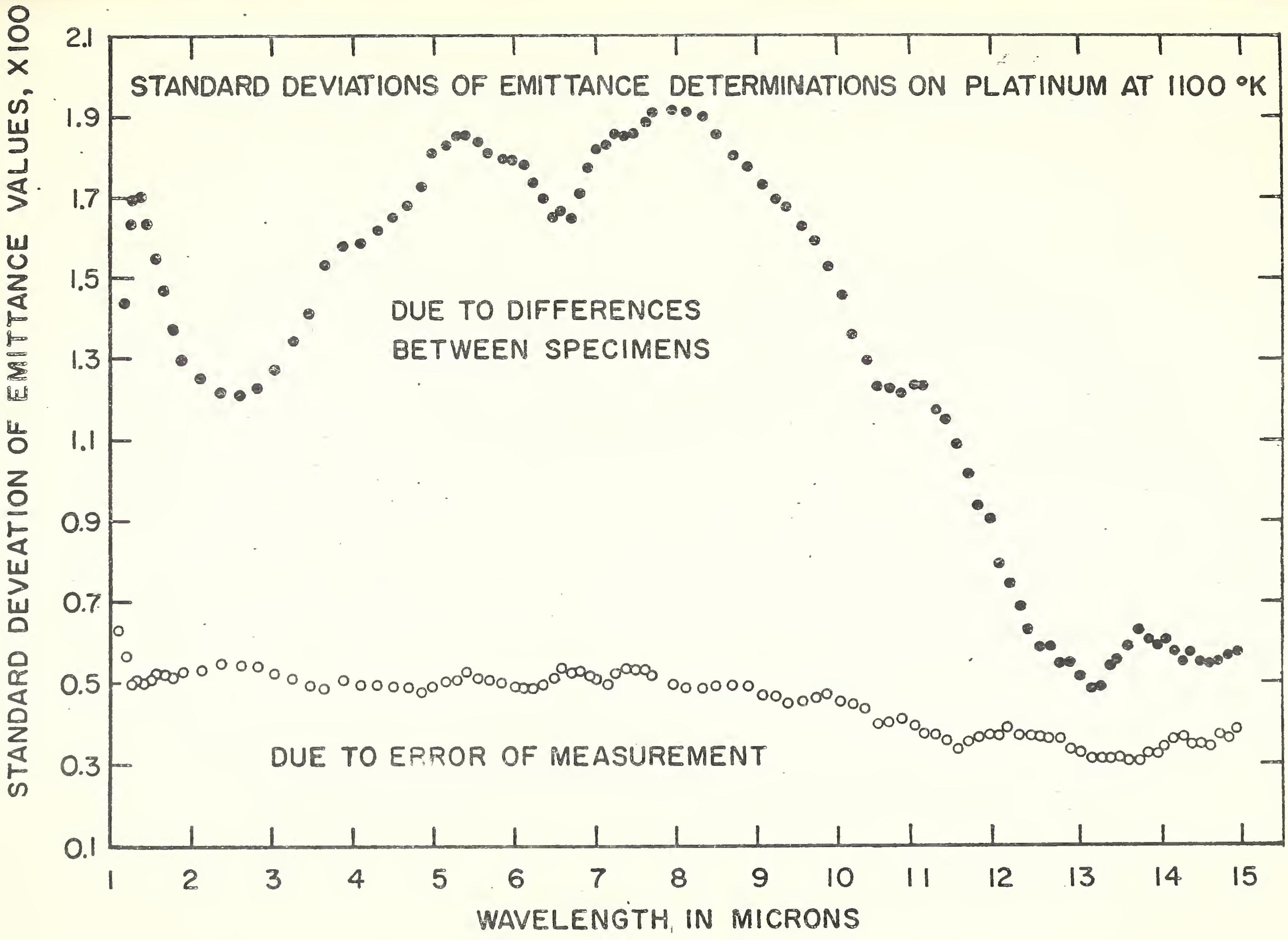
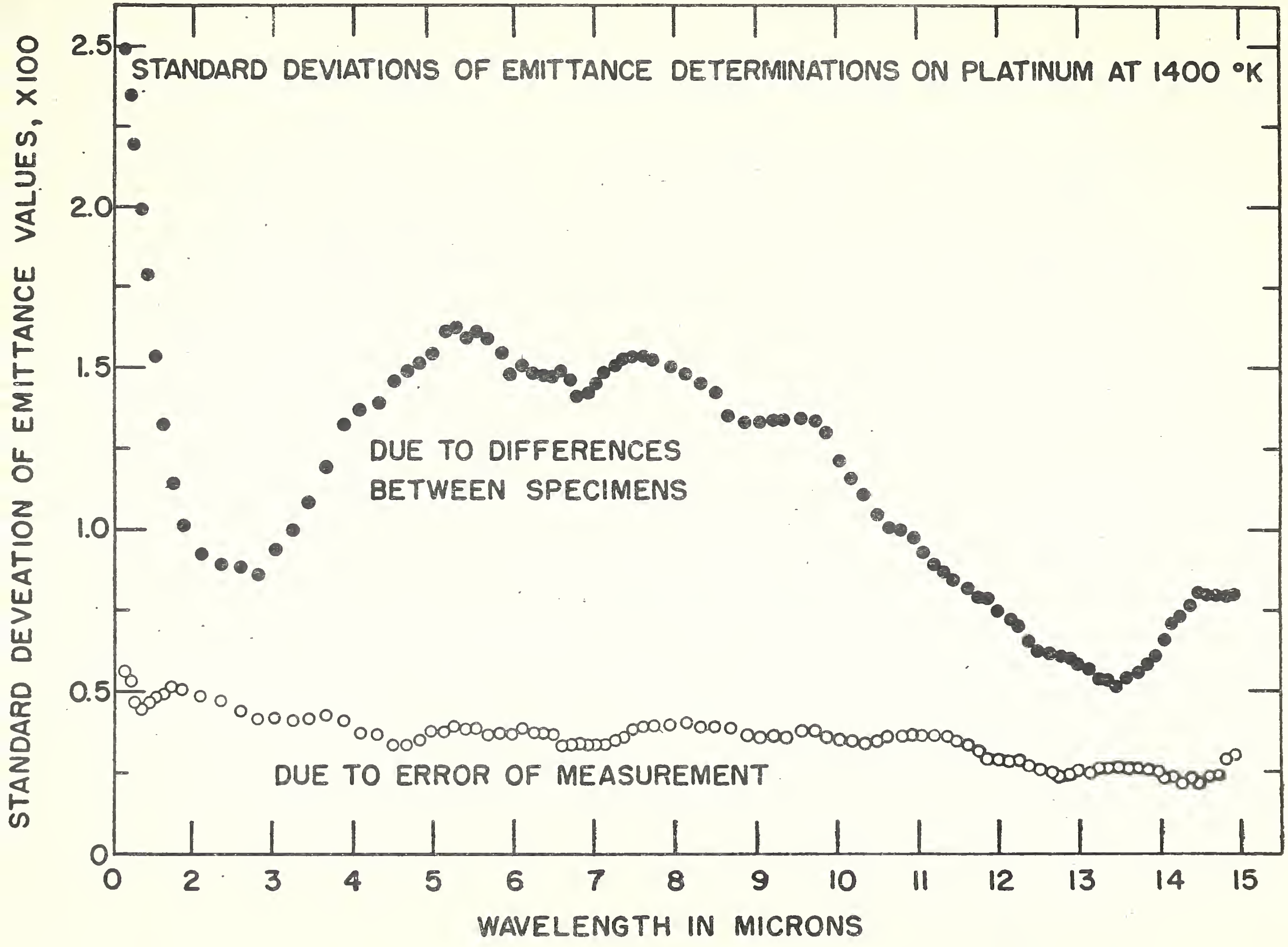

STANDARD DEVIATIONS OF EMITTANCE DETERMINATIONS ON PLATINUM AT 1100 °K
DUE TO DIFFERENCES BETWEEN SPECIMENS
DUE TO ERROR OF MEASUREMENT
STANDARD DEVEATION OF EMITTANCE VALUES, X100
2.1
1.9
1.7
1.5
1.3
1.1
0.9
0.7
0.5
0.3
0.1
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
WAVELENGTH, IN MICRONS

Figure 6. Spectral distribution of two categories of standard deviations, each computed from 18 measured emittance values obtained at 1400°K, three each on six platinum working standards. The upper curve represents standard deviations due to real differences in emittance between specimens, identified as σ_s in the text. The lower curve represents standard deviations due to random error of measurement, identified as σ_m in the text. In both curves each point represents the moving average of five adjacent values.


STANDARD DEVIATIONS OF EMITTANCE DETERMINATIONS ON PLATINUM AT 1400 °K
DUE TO DIFFERENCES BETWEEN SPECIMENS
DUE TO ERROR OF MEASUREMENT
STANDARD DEVEATION OF EMITTANCE VALUES, X100
2.5
2.0
1.5
1.0
0.5
0
0
2
3
4
5
6
7
8
9
10
11
12
13
14
15
WAVELENGTH IN MICRONS

U. S. DEPARTMENT OF COMMERCE
Luther H. Hodges, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

THE NATIONAL BUREAU OF STANDARDS

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Metrology. Photometry and Colorimetry. Refractometry. Photographic Research. Length. Engineering Metrology. Mass and Scale. Volumetry and Densimetry.

Heat. Temperature Physics. Heat Measurements. Cryogenic Physics. Equation of State. Statistical Physics.

Radiation Physics. X-ray. Radioactivity. Radiation Theory. High Energy Radiation. Radiological Equipment. Nucleonic Instrumentation. Neutron Physics.

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Organic and Fibrous Materials. Rubber. Textiles. Paper. Leather. Testing and Specifications. Polymer Structure. Plastics. Dental Research.

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Data Processing Systems. Components and Techniques. Computer Technology. Measurements Automation. Engineering Applications. Systems Analysis.

Atomic Physics. Spectroscopy. Infrared Spectroscopy. Solid State Physics. Electron Physics. Atomic Physics.

Instrumentation. Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

Physical Chemistry. Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Molecular Kinetics. Mass Spectrometry.

Office of Weights and Measures.

BOULDER, COLO.

Cryogenic Engineering. Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

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Radio Propagation Engineering. Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

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Radio Systems. Applied Electromagnetic Theory. High Frequency and Very High Frequency Research. Modulation Research. Antenna Research. Navigation Systems.

Upper Atmosphere and Space Physics. Upper Atmosphere and Plasma Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.

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