

T H E S I S

on

ABSORPTION AND REFLECTION FACTORS
OF BLACKENING MATERIALS USED IN
ENERGY MEASURING INSTRUMENTS

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INTRODUCTION

Blackening materials of some type are used on all radiation measuring instruments. The materials used are smokes, and lampblacks of different kinds applied either directly or with some binding agent. In the past few years a technique has been developed for depositing different metals in the form of dense black smokes. There has been no consistent study made during the last twenty or twenty-five years of the reflection, transmission, and absorption of these substances, although isolated references occur in the literature giving several of these factors at widely separated wavelengths. Recently there has been a very noticeable increase in the number of papers devoted to radiation measurements, due largely to the interest in rotation-vibration spectra of different substances. As a good radiation measuring instrument can be improved or made poorer by merely changing the thickness of the blackening material, it seemed highly desirable that a thorough systematic study of the entire subject should be made. Unfortunately, the construction and adjustment of the apparatus consumed so much time that the goal set has not been achieved. A good start has, however, been made.

The measurement of absorption, transmission, and reflection at different wavelengths requires some type

of spectrometer. The type chosen is the fixed arm Wadsworth prism spectrometer, using mirrors of medium focal length to conserve energy. A single prism is used for the same reason; high dispersion not being necessary as none of the substances show rapid variations of their properties with small changes of wavelength. The apparatus was set up on a massive concrete pier 8 x 10 feet in a double walled constant temperature room.

ARRANGEMENT AND CONSTRUCTION

Fig. 1 shows a diagram of the optical system, while Fig. 2 shows a photograph of the apparatus as assembled. The diagram and the photograph are labeled identically. The full lines in Fig. 1 indicate the path of the light beam when measuring emission or transmission; the dotted lines show the path when measuring reflection.

The spectrometer was made from an old U.S. Coast and Geodetic theodolite, having a 20 inch circle provided with measuring microscopes reading to one second of arc. A prism table was mounted with leveling screws on hole-slot-plane plates on the top of the theodolite. The prism (F) was mounted on a triangular piece of brass having three leveling screws and held in place by horizontal adjusting screws. The Wadsworth mirror (E) was held and adjusted by three hooks controlled by spring tension. This mirror like the others was front silvered

by Brashear's process.

The prism (F), approximately 60° and having a face $1\frac{3}{4} \times 2\frac{3}{4}$ inches, was cut from a clear rock salt crystal obtained from a mine at Detroit Michigan. The prism faces were polished in the usual manner with rouge on a pitch surface.

A tin house shown by the dashed lines in Fig. 1 covered the prism table and the path to the mirror (G). Removable shutters were inserted in this house just beyond the prism table when the instrument was not in use, in order to prevent the circulation of air and consequent absorption of moisture by the prism. The housing was painted black with Zapon and the side opposite the mirror covered with black velvet to intercept stray radiation. A lead strip covered with black velvet rested on the top and rear side of the prism. Another removable black shutter extended from above to just behind and below the velvet covered lead strip. This shutter could be removed to allow the direct passage of light through the housing above the prism for adjustment purposes.

At the principal focus of the mirror (G) which caught the dispersed energy from the prism (F) was placed the bolometer (H). The bolometer consisted of two platinum strips soldered to copper which in turn was mounted on a fibre frame. These two platinum strips,

blackened with camphor smoke were 0.5 x 12 mm and had a resistance of 1.16 and 1.17 ohms. The strips were made by the Wollaston process, that is, by plating silver on platinum and rolling as thin as possible. The sheet was cut on a dividing engine into strips 0.5 mm wide, and the silver was removed by nitric acid. The entire bolometer was mounted in a small wooden house to prevent changes caused by air currents. The house had a rocksalt window in front of the exposed strip and a glass window behind both strips for convenience in adjusting and checking the position of the bolometer in the focus of the last mirror (G).

Each of the bolometer strips served as one arm of a Wheatstone bridge, with resistance boxes (each 3.9 ohms) serving as the other two. For final balance, 14 inches of No. 26 bare copper wire was placed in series with one of the boxes, contact being made by a carefully built slider acting under constant spring tension. The resistance boxes and the slide were placed in a box and covered with cotton to maintain constant temperature conditions. A two volt 500 ampere hour battery supplied a constant current of 0.055 amperes or half of this value per strip. A Leeds and Northrup type H.S. galvanometer having a resistance of 18 ohms and a sensitivity of 8.8×10^{-8} amperes per millimeter deflection at a scale

distance of one meter was used. It was actually used at a scale distance of two meters. The image of a straight filament lamp on the ground glass scale was bright and exceptionally sharp. The maximum zero shift noted during a run lasting about an hour and a half was 15 mm. This of course, does not affect the results as the two values for each ratio were taken in immediate succession.

The bilateral slit (C) was kept at 10 mm height and 1 mm width throughout the investigation. In front of the slit was a shutter made of a butterfly valve from a carbureter. It was controlled by a lever. Between the slit and the shutter was placed the usual slide (N) used for transmission measurements. It consisted of a brass slide having two openings about $1 \times 2 \frac{1}{2}$ cm. Stops on the slide holder limited the motion so that either opening could be placed in the beam by operation of a handle. Over one of these openings was placed a piece of clear glass and over the other a similar piece, on the back of which material under test was deposited. The ratio of the deflection of the sample to deflection of the clear glass gives the transmission factor directly.

The source of energy for the first part of the work in the near infrared was a flat filament vacuum lamp (S), the tungsten filament being 2×40 mm. It was heated for the sake of steadiness by nine.2 volt 500 ampere hour

storage cells in series. The current used was 14.5 amperes which gave a temperature of 1457° C. as measured with a Leeds and Northrup optical pyrometer. The variation of current during a run was less than one per cent. The lamp was mounted on a ball and socket to facilitate adjustment.

In order to obtain the reflection factor of a substance, the incident and reflected energy must be measured in close sequence. To increase the convenience of making the necessary change a special holder for the lamp, the first mirror and the sample was built. Fig. 3 and 4 show that the total length of the light path and the angle of incidence of the beam on the mirror (A) are kept constant. The lamp and the sample are mounted at conjugate foci of mirror (A) so that they can be rotated about the center of the sample. The sample is mounted in gimbals and can be swung in or out of the beam. All necessary adjustments are provided for the lamp and mirror. To measure the incident energy, the apparatus is rotated into the position shown in Fig. 3, the photograph being taken from the position of mirror (B) Fig. 1; the light leaving the lamp is focused at the point above the axis of rotation (the sample being moved out of the beam) and passes through this focus to the mirror (B) which in turn brings the beam to focus on the slit (C)

Fig. 1. To measure the reflected energy, the apparatus is rotated to the position shown in Fig. 4, also taken from the position of the mirror (B) Fig. 1, and the sample is reintroduced into the beam. The light from the lamp is now focused on the sample and the portion reflected reaches mirror (B), which refocuses the beam on the slit (C) Fig. 1. The motion of rotation of the lamp and mirror is limited by stops, as is also that of the sample. The use of this auxiliary piece of equipment serves to speed up the measurements and also to decrease the fatigue of operation.

ADJUSTMENTS AND CALCULATIONS

The theodolite top was leveled by using a sensitive level. It is essential that the edge of the prism be parallel to the axis of rotation and that this axis lie in the bisector of the refracting angle of the spectrometer. A small hole was drilled through the prism table and through the triangular piece holding the prism and these holes set accurately in the axis of rotation; a small plumb bob was suspended directly over the axis of rotation with the plumb line passing through the center of the hole in the prism table, the hole-slot-plane plates carrying the prism table were then waxed into position while the plumb line was in position.

To bring the prism and the Wadsworth mirror into

their proper position, the most convenient piece of auxiliary apparatus is a rather low power large diameter (four or five inch) telescope. Such a telescope was not available and since the sensitive level available was mounted on long Wyes, the adjustment was somewhat intricate. A telescope with a Gaussian eye piece was leveled with the sensitive level. The mirror (E) of the Wadsworth mounting was adjusted until the cross hairs reflected from the mirror coincided with those viewed directly through the telescope. This made the mirror perpendicular to the axis of the telescope. The telescope was replaced by a smaller one mounted on the parallel ways of a microtome. It was also provided with a Gaussian eye piece. The telescope was made perpendicular to the mirror by adjusting leveling screws under the microtome. The small telescope could be moved along the ways keeping its axis parallel to itself. The spectrometer was rotated and the prism adjusted until the prism faces were perpendicular to the telescope.

The circle reading was taken when each face of the prism was perpendicular to the telescope. The prism angle (180° - difference between the two circle readings mentioned) was found to be $59^\circ 18' 55''$. The mirror (E) was rotated about a vertical axis until it bisected the angle between the prism faces.

The spectrometer settings were computed from the index of refraction of rock salt at the different wavelengths 0.1 micron apart. The index of refraction of rock salt at 20° C. was taken from curves by S.P. Langley and G.C. Abbot in the Annals of the Astrophysical Journal Volume I. The room temperature here was 24° to 25° C, but the temperature correction for the wavelengths used was found to be negligible. The following formula was used to calculate the angle of minimum deviation:

$$n = \frac{\sin \left(\frac{A + D}{2} \right)}{\sin \frac{A}{2}}$$

n = index of refraction

A = prism angle

D = angle of minimum deviation

$$\text{solving, } D = 2(\sin^{-1}(n \sin 1/2A) - 1/2A)$$

The value of D was computed with five place logs from wavelengths of 0.6 of a micron to 5 microns in steps of 0.1 micron. The D line of sodium was taken as the starting point. The difference between the angle of minimum deviation of sodium light and the angle of minimum deviation of some other wavelength gives twice the angle through which the spectrometer must be rotated to bring that wavelength into place occupied by the sodium line.

The bolometer should be placed so that the wavelength incident upon it has passed through the prism at minimum deviation. On the Wadsworth mounting, minimum deviation is indicated by the coincidence of a wavelength that has

passed through the prism with the undeviated beam. This adjustment was particularly easy in the present set up, as the undeviated beam could be obtained by merely removing the slide above the prism and sending sodium light through the spectrometer. The undeviated line was viewed by a long focus microscope and the spectrometer rotated until the sodium line was in the same position. The circle reading was of course taken and used as the starting reading. The bolometer, as mentioned before, had both front and rear windows, was brought into sharp focus in the microscope and was fixed in position by cementing its hole-slot-plane plates. This adjustment was examined almost daily, but was never found to vary. The previous computations of the spectrometer settings were checked by locating the CO_2 emission band in a Bunsen flame at 4.4 microns.

MATERIALS AND RESULTS

The samples taken were for convenience deposited on glass (the present investigation covers only the near infrared). A piece of glass about 1.3 x 3 cm was weighed and then smoked or painted with the blackening material and reweighed. After the run the thickness of the sample was increased and another weighing and run made. In many cases the reflection was too small to be measured, hence, the samples were usually examined by transmitted

radiation. The observations were usually made as follows. The spectrometer was set, the shutter (N) closed, and the zero reading of the galvanometer taken. The shutter (N) was then opened with the sample in the beam and the galvanometer reading taken again. The slide was changed so that the clear glass was in the beam and the galvanometer reading taken. The shutter (N) was then closed and if the galvanometer reading was the same as the zero reading at the start, the readings were accepted and a new spectrometer setting made. The deflection of the galvanometer with the sample in the beam, (glass surface toward the source) divided by the deflection with the clear glass in the beam gives the relative energy transmission of that particular thickness of blackening material. The reflected energy was measured when possible in much the same manner. The sample was placed on the gimbal (K) Fig. 3 at the focus of the beam from mirror (A). The direct energy deflection of the galvanometer was read with the sample (K) at one side. The equipment was then rotated and the gimbal (K) swung into the focus of the beam causing the reflected energy to be caught by the mirror (B) Fig. 4.

The following materials were studied:

Acetylene smoke	3 samples
Camphor smoke	7 samples
Zapon black	3 samples
Nitrokote	3 samples

The first curve sheet shows the relative energy distribution of the lamp as measured by the system at 14.5 amperes which is temperature of 1457° C. It will be noted that very little energy is available below a wavelength of 1 micron or above 3 microns.

The first blackening material used was that of acetylene smoke. Acetylene smoke gives a deposit of large velvety flakes. The sample glass was blackened by holding it some distance above a small acetylene flame. The next two samples were prepared by another layer on the preceding layer. The curves (curve sheet II) indicate that the efficiency as an absorber decreases as the wavelength increases and is caused mainly by increased transmission as the reflection was not sufficient to be measured.

The second material studied was Zapon black, a commercial black laquer that dries with a dull finish. The samples were prepared by brushing. Like acetylene and the remainder of the materials studied, the per cent transmission (Curve sheet III) increases with wavelength.

It requires 10 to 15 times the weight of Zapon per unit area to produce the same absorption as acetylene.

The third material studied was camphor smoke. The amounts deposited on the sample each time were so small that weighing was impossible on the balance available, a chemical balance on which 0.1 milligram could be detected.

The fourth material studied, Nitrokote, gave sufficient reflected energy so that transmission and reflection could both be studied. The per cent transmitted and the per cent reflected were added and the sum subtracted from 100, this yielding the per cent absorbed. The curves (curve sheets V, VI, and VII) indicate that the per cent of energy reflected is independent of the wavelength and thickness over the ranges studied. The coincidences of irregularities on the absorption curves indicate selective absorption.

CONCLUSIONS

An infrared spectrometer has been assembled, of sufficient sensitiveness to study absorbing materials of the limit of glass transmission.

Absorption curves for different thicknesses of acetylene smoke, camphor smoke, and Zapon black were obtained.

Absorption and reflection curves were obtained for nitrokote.

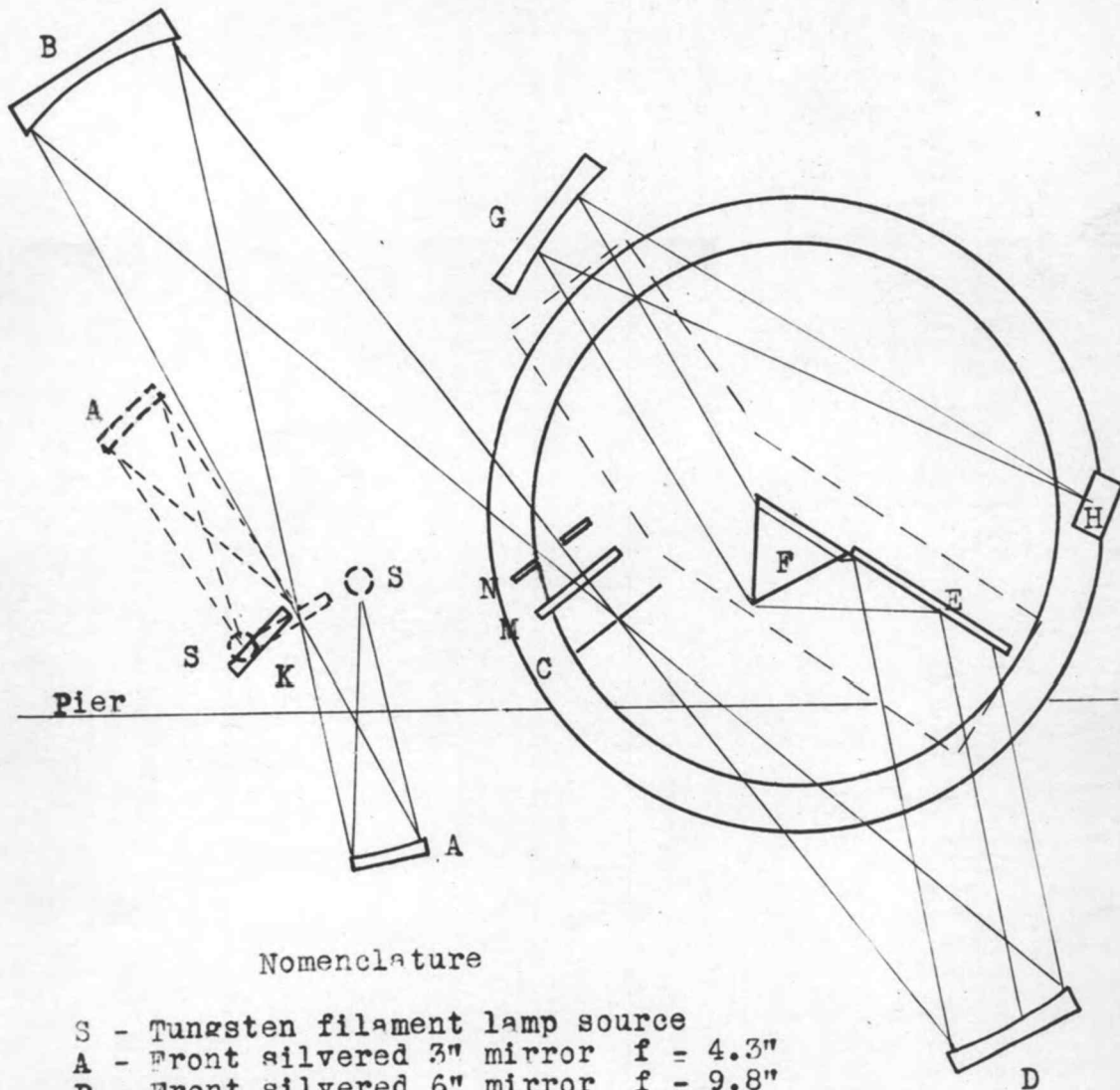
All materials studied show a decrease of absorption with an increase of wavelength.

The weight of material necessary for the same absorption ranks in the following order: camphor, acetylene, Zapon, and Nitrokote; the Nitrokote requiring more than 15 times the weight of camphor.

SUGGESTIONS

To complete the study of these and other materials, it is proposed to build a Thompson 4 coil galvanometer; which will increase the sensitivity of the present equipment by a factor of from 100 to 1000. The observations will be extended farther into the infrared and a Nernst glower substituted for the ribbon filament lamp. If more dispersion is needed the radiation will be sent through the prism the second time by a plane mirror and the bolometer moved to a place vertically beneath the slit.

Optical System



Nomenclature

- S - Tungsten filament lamp source
- A - Front silvered 3" mirror $f = 4.3"$
- B - Front silvered 6" mirror $f = 9.8"$
- C - Bilateral slit
- D - Front silvered 5" mirror $f = 19.7"$
- E - Plane silvered mirror $4\frac{1}{2}" \times \frac{1}{4}" \times 7\frac{1}{4}"$
- F - Rock salt prism, face $1\frac{3}{4}" \times 2\frac{1}{4}"$
- G - Front silvered 5" mirror $f = 19.7"$
- H - Bolometer
- K - Sample holder for reflection measurements
- M - Sample holder for transmission measurements
- N - Shutter

Fig. 1

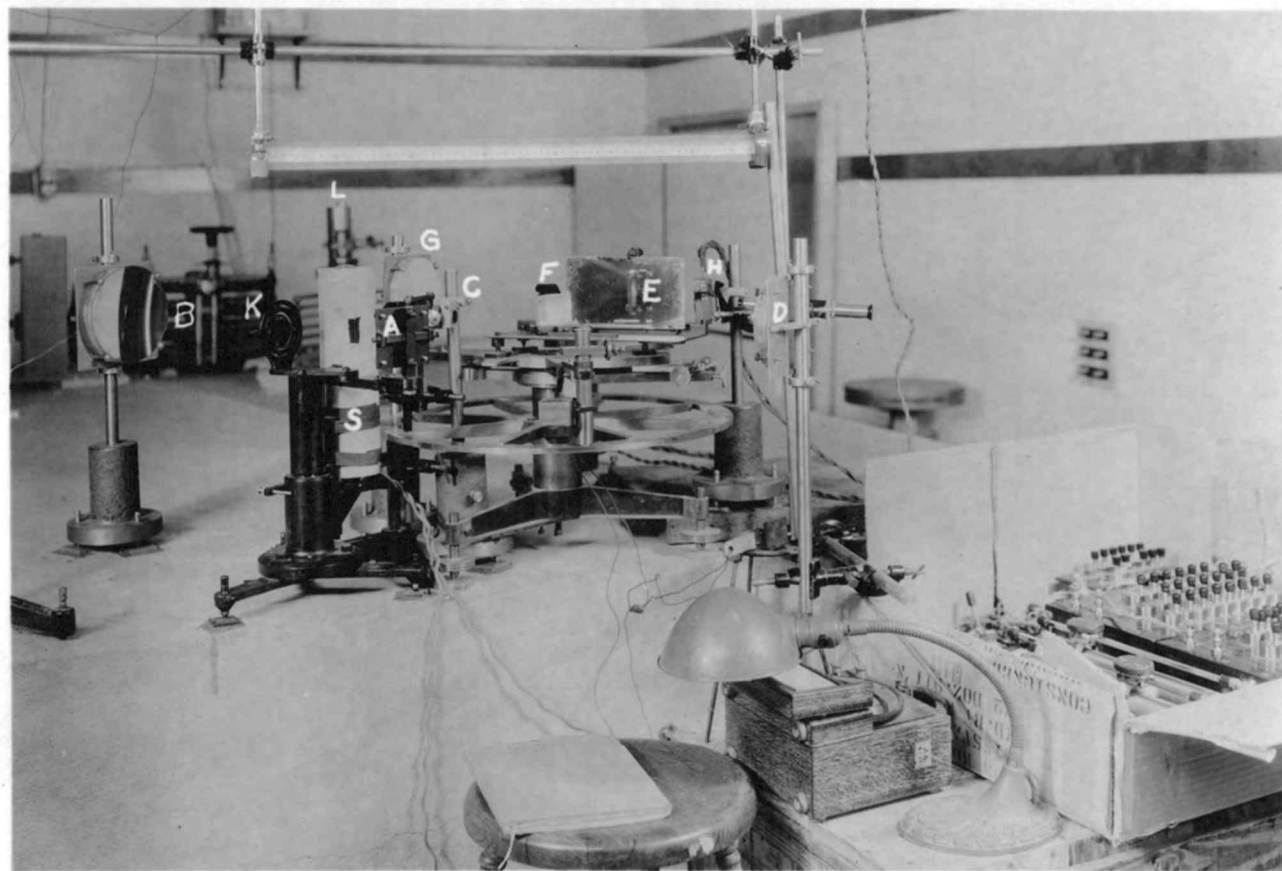


Fig. 2

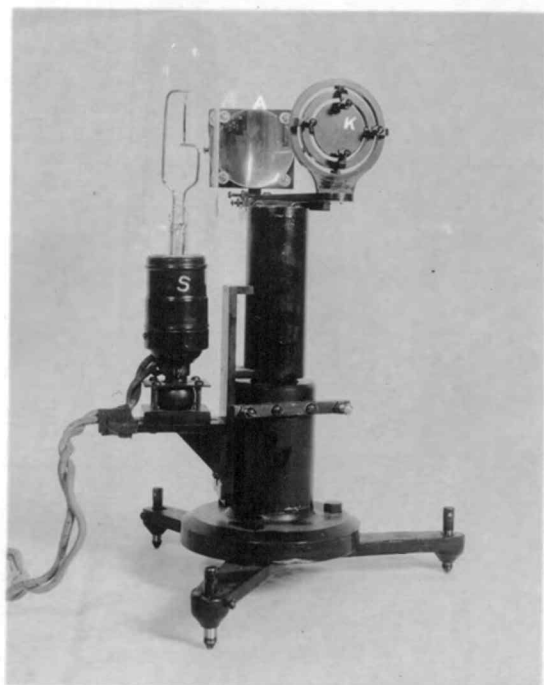


Fig. 3

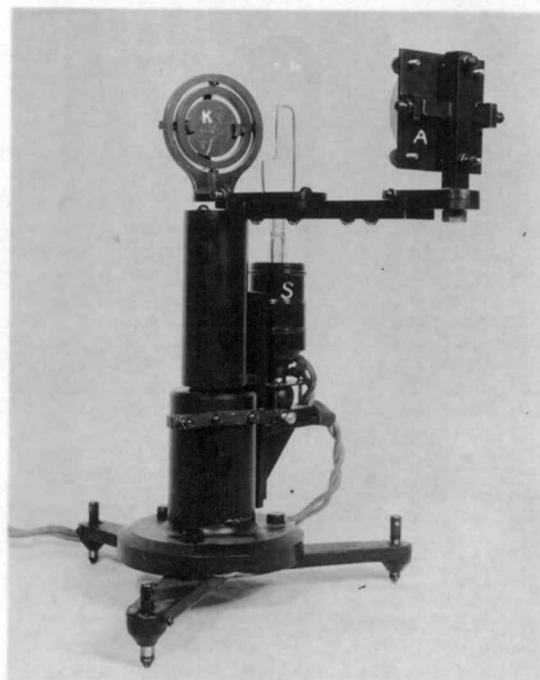
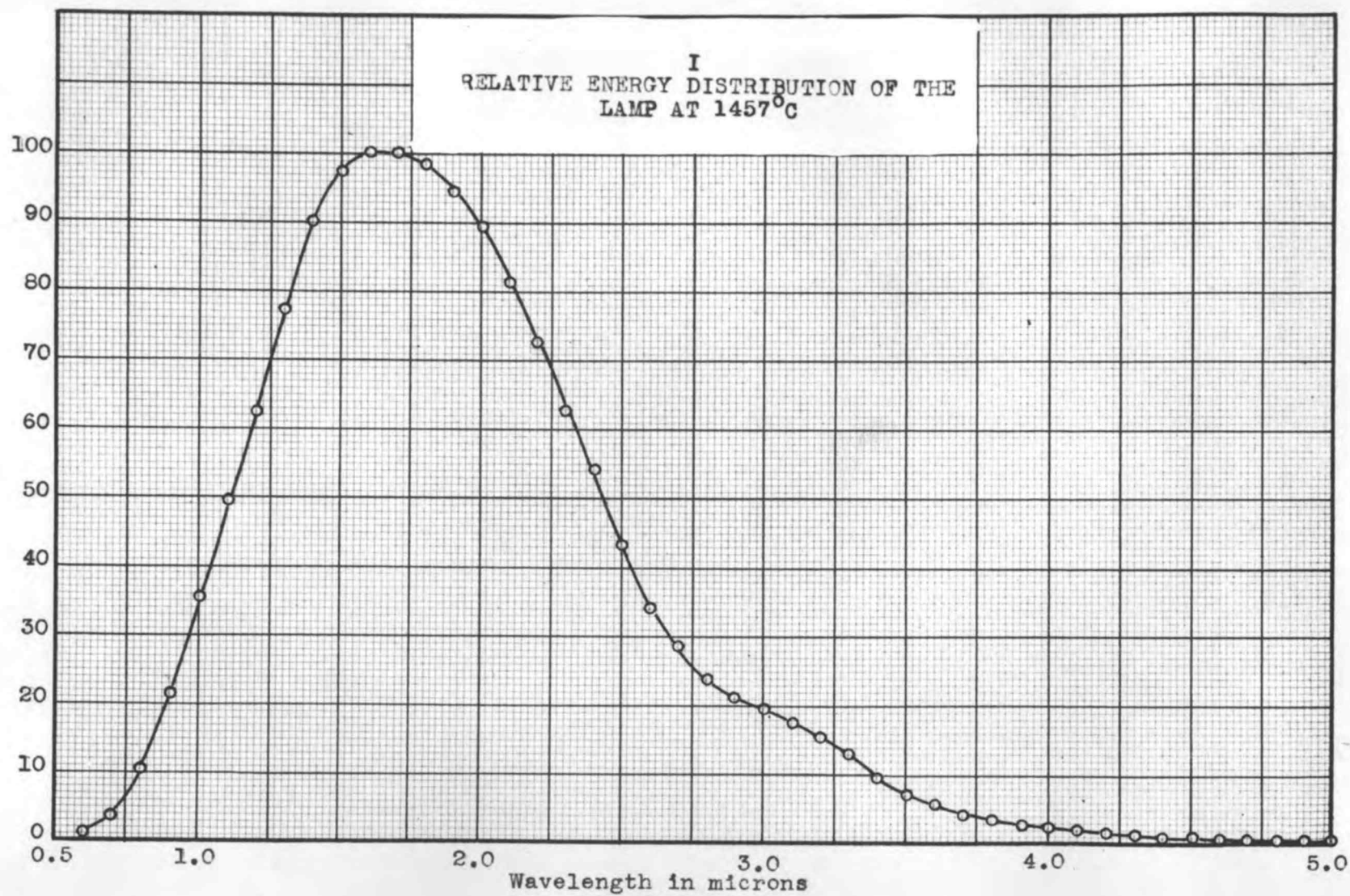
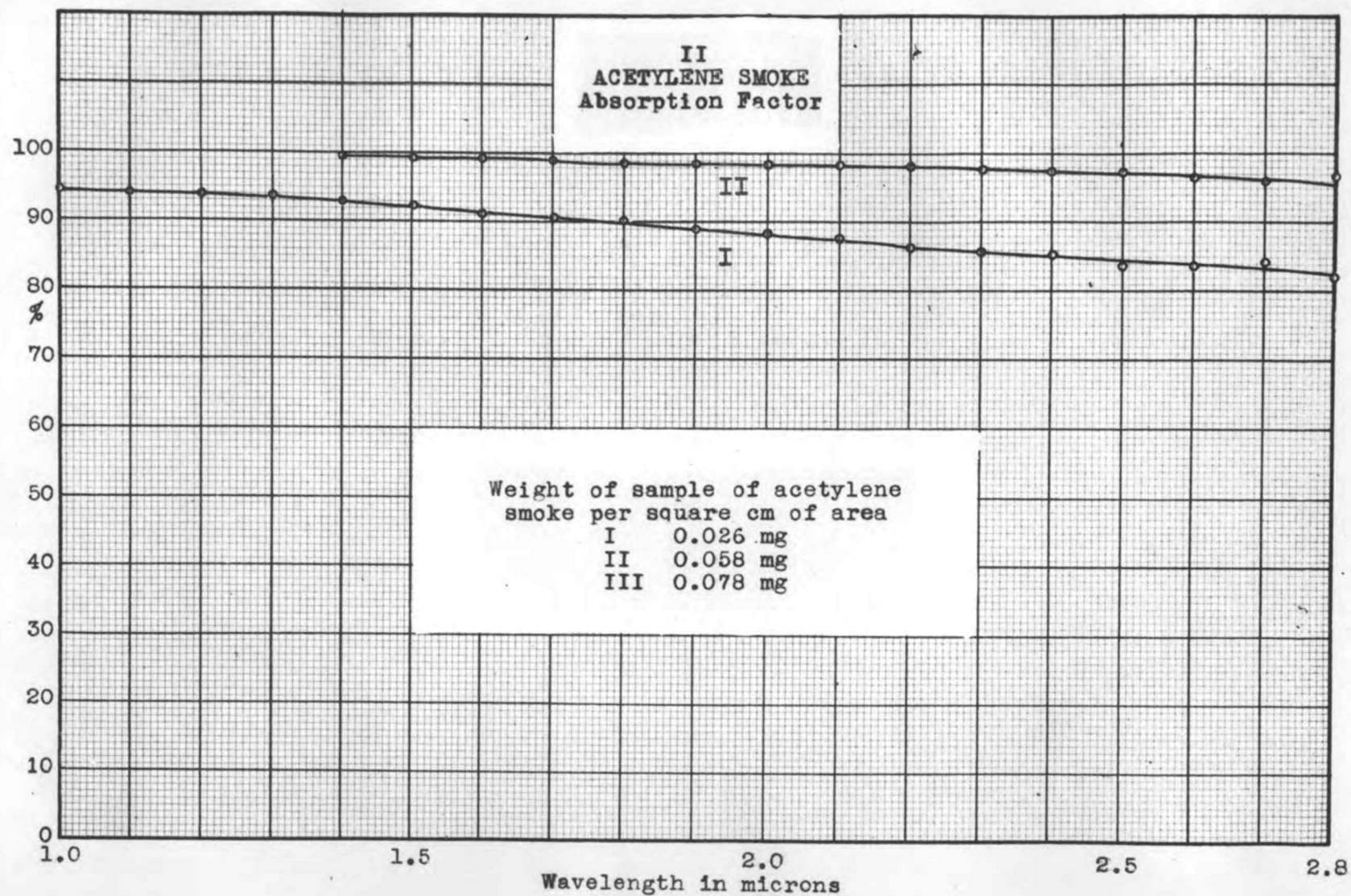
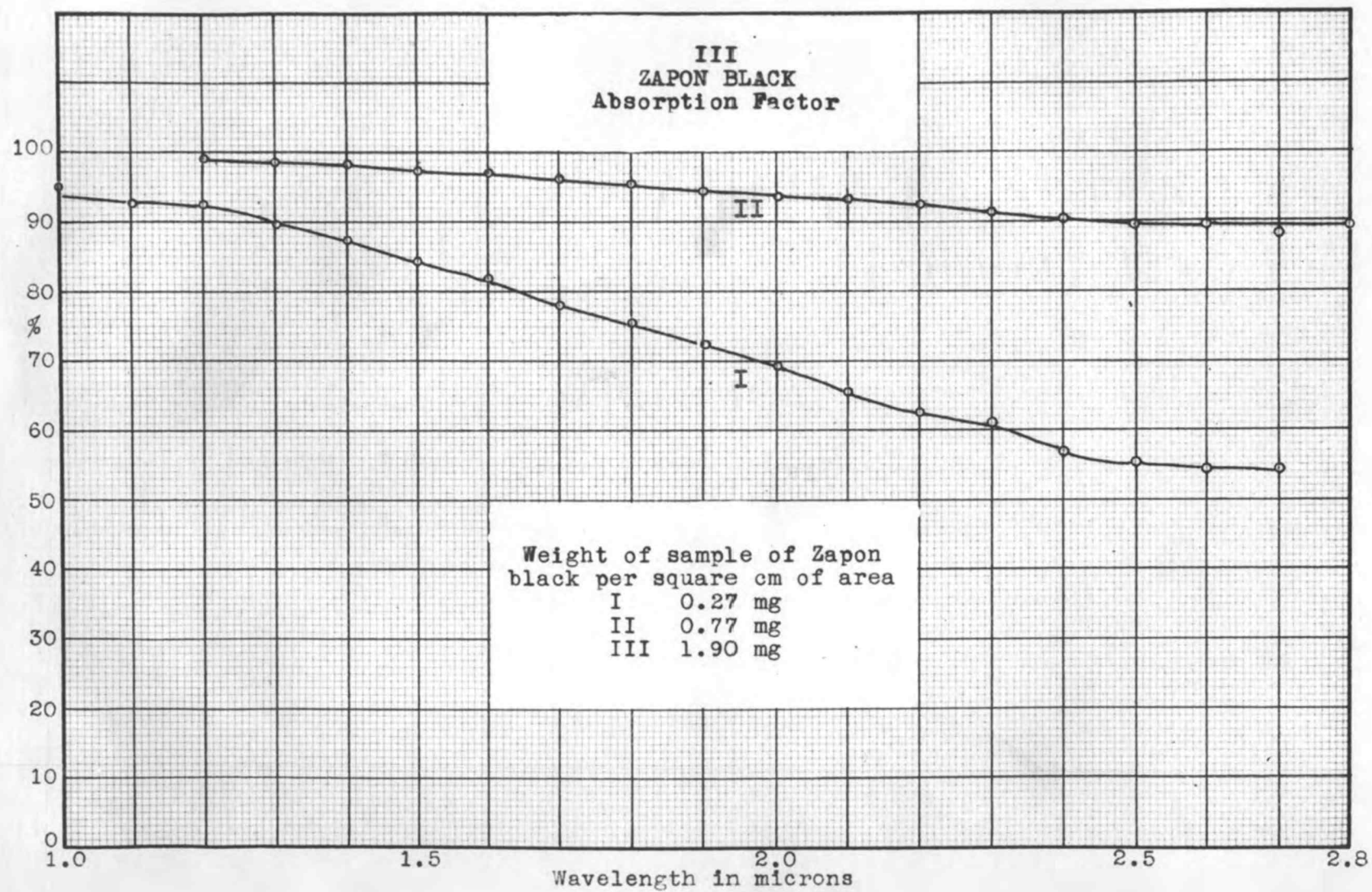


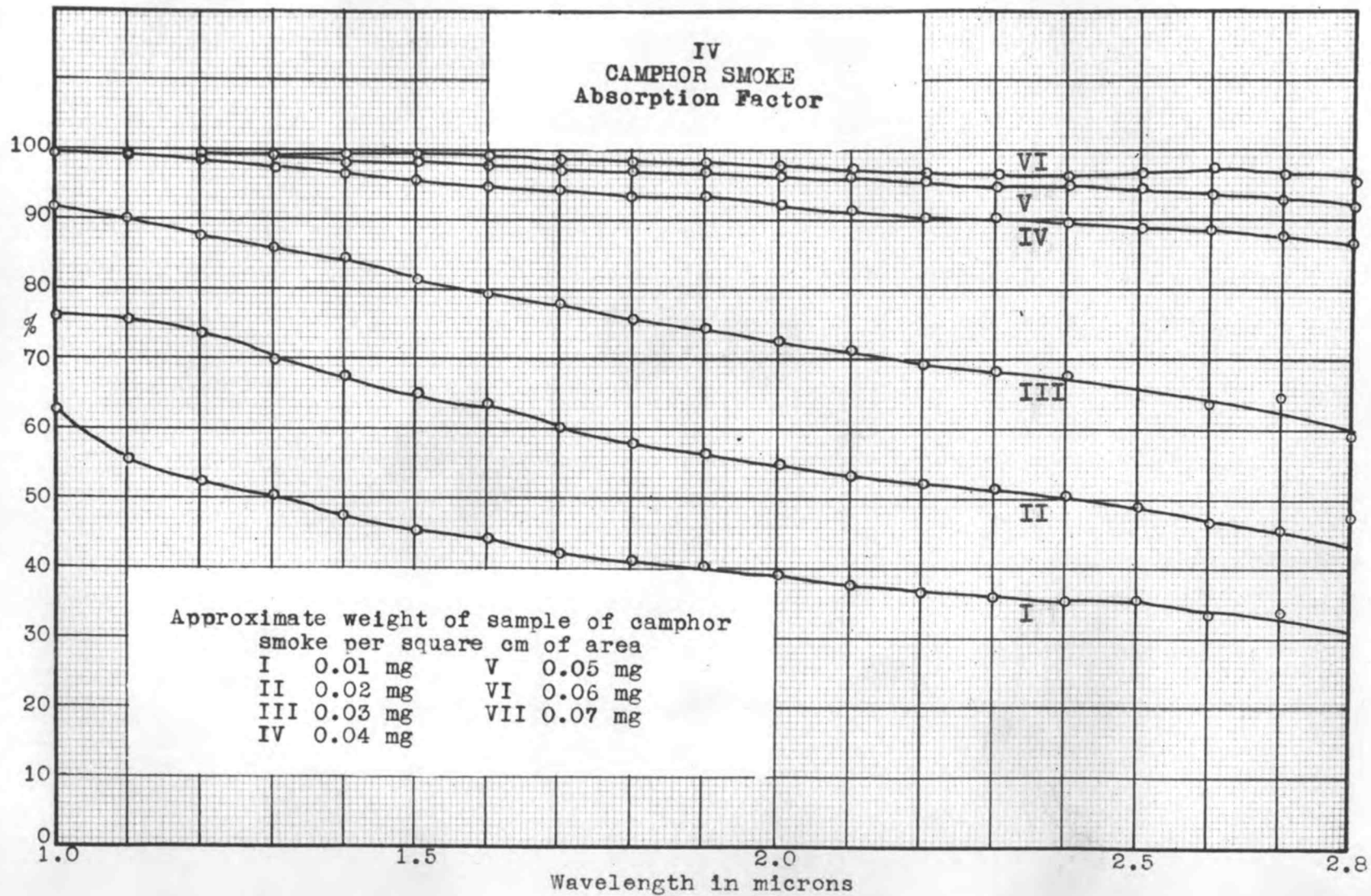
Fig. 4

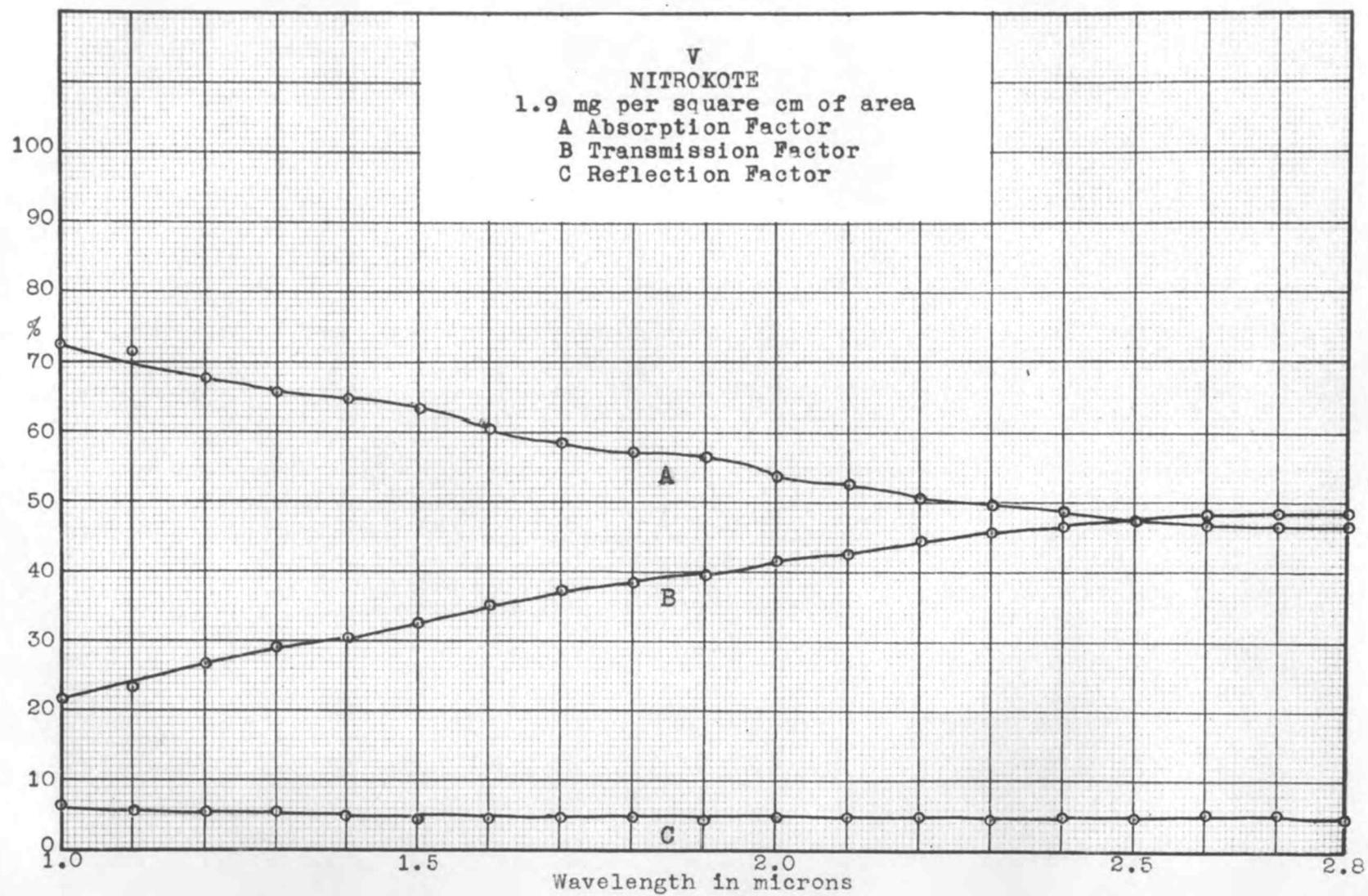


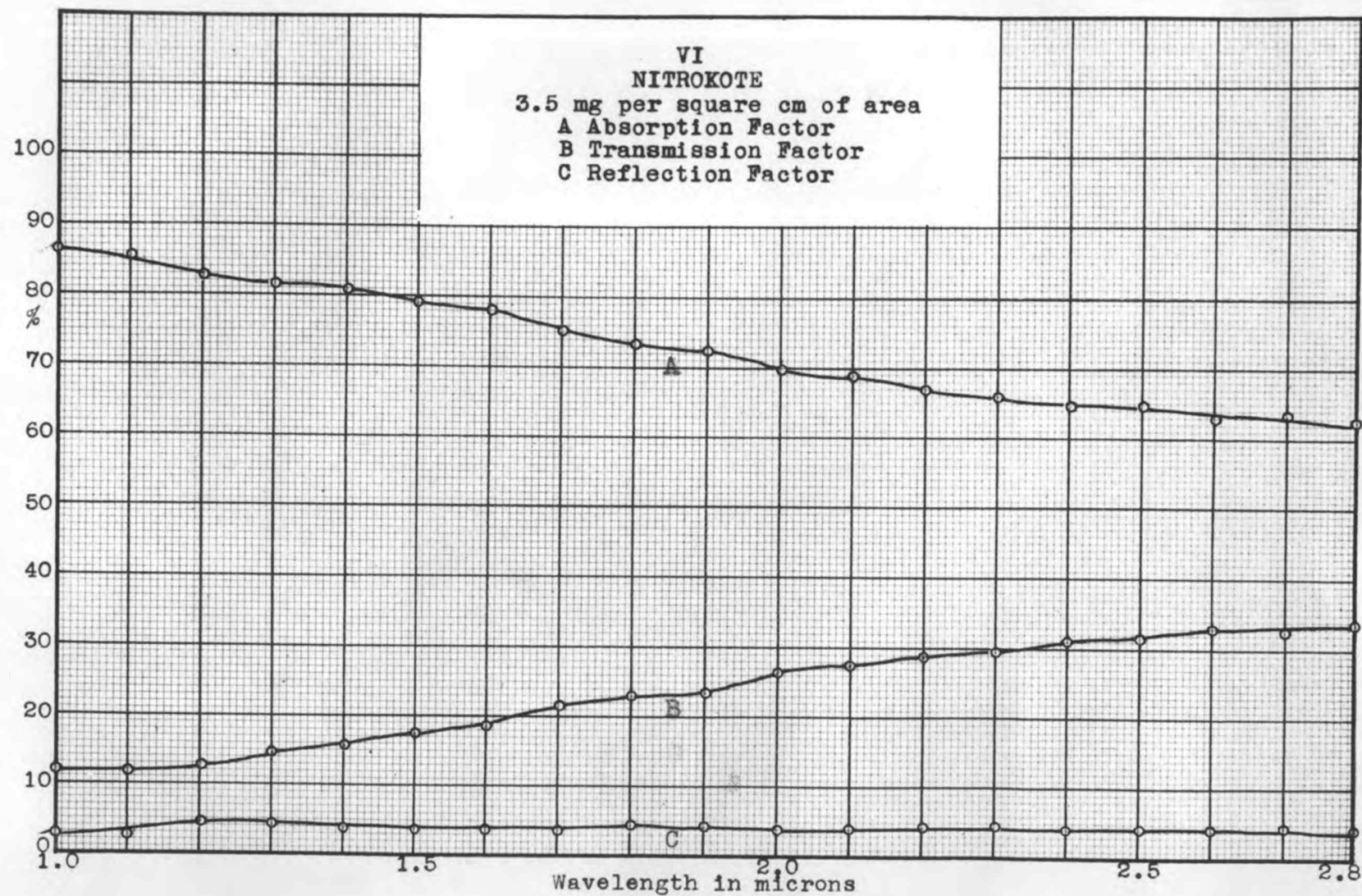


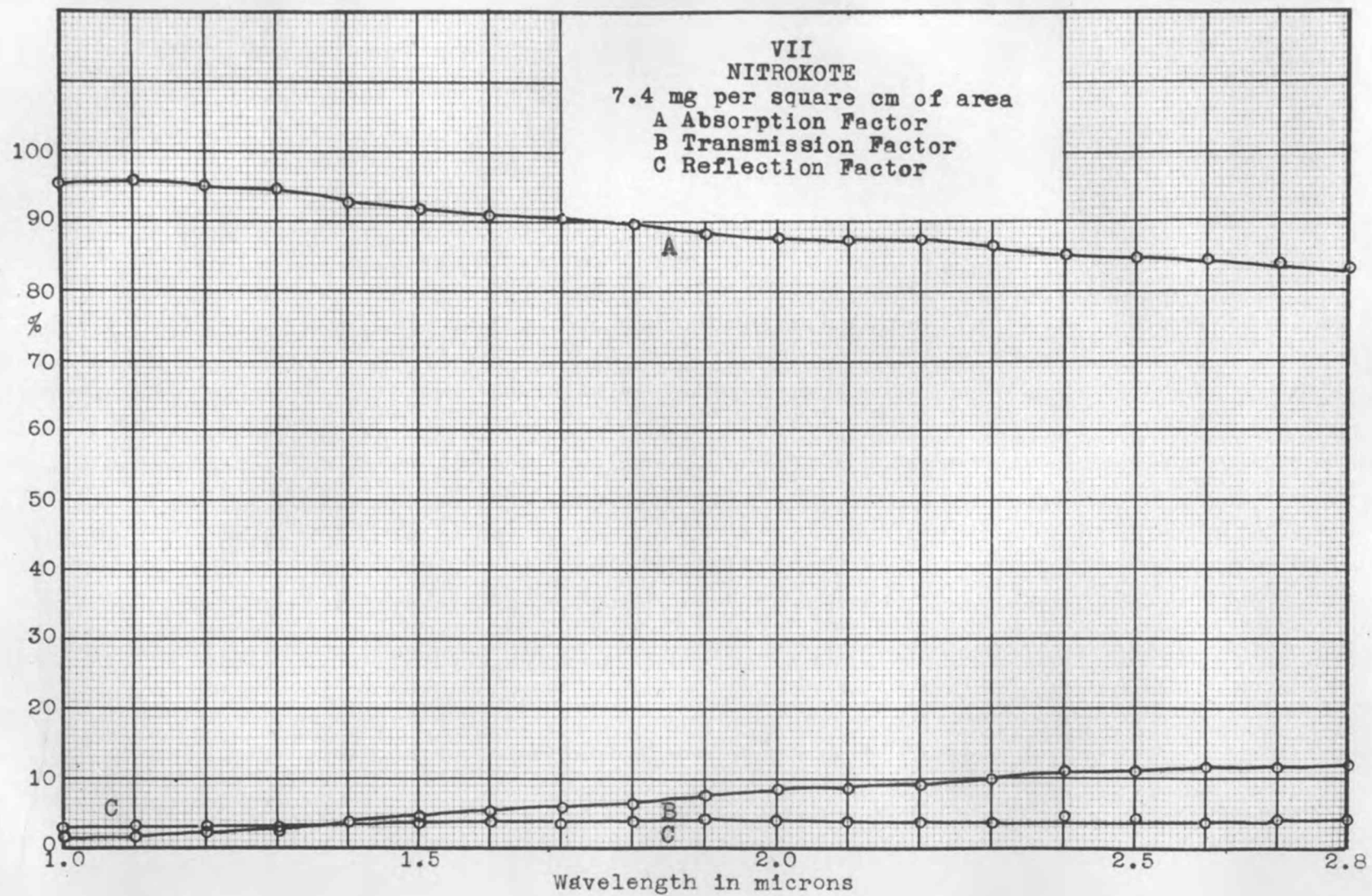


IV
CAMPHOR SMOKE
Absorption Factor









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