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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
Washington

Letter Circular LC-617

(Supersedes LC-393)

November 8, 1940

MEASUREMENT OF THE REFLECTANCE OF MANILA ROPE FIBER[1]

I. INTRODUCTION

National Bureau of Standards Letter Circular LC-393 describes a method for the measurement of the reflectance[2] of manila rope fiber for wave length 500 millimicrons. The measurements are used for grading rope and for control of quality in its manufacture. A reflectance requirement is part of Federal Specification T-R-601a.

A survey of the laboratories where measurements are made revealed that some difficulty was experienced in applying the test. Some of the difficulty could be ascribed to a lack of clarity in the description of the method.

This letter circular supersedes Letter Circular LC-393 and describes equipment and technique more recently developed for testing manila fiber for compliance with the reflectance requirements. It does not include a description of the latest photometric equipment since this equipment is still under construction.

1/ Prepared by Genevieve B. Reimann, Research Associate, Cordage Institute, and Ralph T. Mease, National Bureau of Standards.

2/ In this letter circular the term "reflectance" is used to designate the measured quantity, which is more accurately designated as the apparent reflectance relative to magnesium oxide, measured at approximately 500 millimicrons. This reflectance measured under 45° illumination and perpendicular viewing is referred to as "Becker" value in Federal Specification T-R-601a.

II. APPARATUS

The rope fibers are cut to short lengths to facilitate the preparation of a surface suitable for measurement. A meat slicing machine has been used for the purpose. However, the ends of the fibers may become frayed and darkened by heating caused by the power-driven rotary blade suggested in the previous letter circular. A more satisfactory and less expensive equipment for cutting the fibers has been developed by the Plymouth Cordage Company. It is a special vise for clamping a bundle of yarns which are held firmly while they are cut with a sharp knife. The length of the cut fibers is controlled by spacer plates bolted to the face of the vise and removed one at a time as the cutting proceeds. For convenience and speed of operation, the vise should be clamped to a firm table or work bench. This equipment is shown in figures 1, 2, and 3. A knife (shown in fig. 3) with a blade about ten inches long is satisfactory for cutting the fibers. It should be kept well sharpened and cleaned.

A Soxhlet extraction apparatus with an extraction chamber of 100-ml capacity is used for extracting the fibers.

The container for the sample of cut fibers whose reflectance is to be measured is shown diagrammatically in figures 4 and 5. It has a glass window through which the sample is illuminated and viewed during measurement. The cavity is 4 mm deep and the cover, opposite the glass, supports a membrane of stretched rubber to provide the right amount of pressure for holding the fibers in the container. It is not expected that the tension of the rubber dam will change importantly over a considerable period of time, but it should be renewed whenever it is suspected of having so changed. The tension need not be controlled very exactly since variations in the tension of the diaphragm changing its stretch by as much as 10 percent were found to be of no consequence with regard to differences in the reflectance measurements. With a rigid sample-retaining cover, the pressure on the specimen varies for very slight differences in the amount of fiber in the filled container, leading to difficulties in duplicating measurements because of differences in the compactness of the fibers.

A convenient way to form the rubber diaphragm and to hold it taut for fastening it in the frame of the sample-retaining cover is to cut a circular disc of sheet rubber dam 12 cm in diameter and fit it on a suitable stretching frame such as is shown at 4 in figure 6 and diagrammatically in figure 7. The eight upper movable sectors of the frame are lifted from the lower ones after removing the wood screws, and the circular platform placed in the center of the frame. The disc of rubber is laid with its center approximately in the center of the frame and on the platform. The eight sectors are put into place and the wood screws tightened so that the rubber disc is gripped securely near the circumference. Opposite pairs of sectors are now drawn out toward the edge of the frame a distance equal to 15 to 20 percent of the radius of the circle formed by the inner edges of the sectors when all are in positions corresponding to the two on the right in figure 7. After all sectors are drawn into place and the wing nut in each sector is tightened, the stretched rubber diaphragm is fastened between two layers of the sample-retaining cover. The cover frame may be made of wood, metal, or other suitable material. The one illustrated in figure 6 was made from layers of cardboard cut to proper shape and held together with cement and staples.

A "measuring section," illustrated in figure 5, is used to control the amount of fibers required. The measuring section is similar in form to the sample container proper and, when fitted on the sample container in the manner illustrated, increases the depth of the cavity to be filled with fiber to 8 mm.

The schematic drawing (A, fig. 8) of the photometric equipment required, originally a part of Letter Circular LC-393, shows the salient features of the equipment and the relative positions of the parts.

The photometer, 1, is a polarizing photometer of the Martens type. It projects through its support, 7, and has its axis perpendicular to the plane of the surfaces of sample and standard, 2. The sample and standard are illuminated by light from a lamp placed vertically above them with the light incident upon their surfaces at approximately 45°. They are viewed through the photometer which is mounted with its long axis normal to the plane of the surfaces of the sample and standard, and intersecting a line drawn through their centers. To eliminate possible polarization errors, the sample and standard are mounted in their plane in such a manner that a line drawn through the centers of their surfaces makes an angle of 45° with a horizontal line in the same plane.

The light source is a 1000-watt lamp which should be operated to give a color temperature of 2900 ± 50° K. One-thousand watt lamps which have a rated life of 1000 hours operated at normal voltage give color temperatures close to 3000° K. These lamps should be operated about 6 percent under rated voltage to give the color temperature 2900° ± 50° K.

The filter, 9, to be placed over the eyepiece of the photometer is a Wratten filter No. 75 cemented between layers of "B" glass and mounted in the cap, 8. When transmitting light from the 1000-watt lamp, the filter should have a wave length centroid of 500 ± 2 millimicrons based on standard luminosity data; its transmission should not exceed 0.5 percent at any wave length from 680 to 750 millimicrons.

Figure 8, B, is a drawing of a holder for sample and standard, showing a top view, 10, a side view, 11, and an end view, 12. The top view, 10, shows the surface of the sample, 14, and standard, 14', and a pivot, 15, about which the holder can be rotated to interchange sample and standard. The side view, 11, shows coiled springs, 16 and 17, for holding the sample and standard in place and a flat spring, 18 (see also 12), for locking the holder in either of the two positions.

A reflectance standard to serve as a comparison surface must also be provided. The standard should be of uniform reflectance, opaque, and of a permanent nature. Each laboratory should be equipped with a reference and a "working" standard. The apparent reflectance of these standards relative to magnesium oxide, for the particular illuminating and viewing conditions, must be known. When using the "working" standard in making measurements, it is covered by a sheet of clear thin glass which is cut from the same sheet of glass as that used in the sample container. Optical glass or microscope-slide glass 1 to 1 1/4 mm thick, transmitting not less than 90 percent throughout the visible spectrum, has been found satisfactory. The equipment should be placed in a room which can be darkened or so designed that glaring light cannot annoy the observer while measurements are being made.

III. PROCEDURE

At least three sections of the rope to be tested, taken not less than 2 feet apart, are required and they should be equally represented in the 8 or more grams of cut fiber which are required for extraction and measurement. The length of the sections will depend upon the size of the rope. Sections of rope 8 to 12 inches long are satisfactory. More than three cross sections of rope of small diameter are necessary to fill the vise and the vise has to be charged more than once when large ropes are tested.

The yarns are untwisted from the sections selected for cutting, colored markers if present are discarded, the fibers are straightened, laid approximately parallel, and the bundle is wrapped in paper (fig. 1). Strong paper of letterhead size is satisfactory for wrapping the bundle. Rubber bands may be used to hold it together. The paper is allowed to extend about an inch beyond the end of the bundle and is twisted to protect the ends of the fiber when inserting the bundle in the vise.

The steps in cutting the fibers are illustrated in figures 1, 2, and 3. The bundle of fibers is pushed through the vise from the rear. It is compressed by tightening the vise to an extent such that the height of the compressed section which will yield 8 grams of cut fiber is about 23 mm. A bundle which is much more or less compressed than this is difficult to cut properly. The nuts securing the spacer plates are released and the bolt heads are drawn into the countersunk holes. The projecting portion of the fiber bundle is cut off and discarded. The cutting is done with a firm, even stroke while holding the side of the knife against the face of the outermost spacer plate to guide it through the bundle. The first spacer plate is removed and the projecting fiber is cut. The second plate is removed, the fiber is cut, and this procedure is continued until all of the plates have been removed. The strips of paper are removed from the cut fiber which is then ready to be extracted.

The cut fibers are placed in an extraction thimble or in a cup formed from filter paper 14 cm in diameter, compacted with a glass stirring rod and laid in the chamber of a Soxhlet extraction equipment. The extraction flask is filled with petroleum ether to approximately two thirds its capacity. A few small pieces of porcelain (each about 1/5 the size of a penny) are placed in the flask to prevent "bumping." The extraction apparatus is assembled and the rope fibers extracted for at least two hours. The rate of extraction should be such that the extraction chamber fills and empties at least once every ten minutes. After a period of two hours, the filter paper or thimble containing the sample is removed from the extraction chamber, placed on a clean watch glass, opened, and the sample allowed to air-dry for at least two hours before measuring. The sample should be protected from dirt and dust while being dried.

Most rope fibers are likely to be freed of extractable materials in one hour or less if extracted at the rate specified. Others may require longer periods of soaking or softening of the oils, particularly if they are oxidized, to remove them from the fibers. The time of extraction is, therefore, to be considered as well as the number of fillings or siphonings of the extraction chamber.

The sample container in which the extracted fibers are to be measured is placed on a horizontal surface with the glass down. Small quantities of the fibers are sprinkled onto the glass in the manner illustrated in figure 4[3] to a depth of approximately 4 mm. The measuring section is fitted over the cavity of the sample container, and more fibers are added until a depth of 8 mm is reached, without disturbing the fibers in contact with the glass. The measuring section is then removed and the sample-retaining cover substituted for it.

The object of the sprinkling operation is to cause each fiber on the glass and forming the surface to be measured to lie with its long axis in a plane parallel with the plane of the glass surface. If some of the fibers stand on end, they prevent others from packing, thus producing shadows which register low reflectance for the sample. Orientation of many fibers in any one direction is also to be avoided. There may be a tendency for this to occur if the fibers become electrically charged when handled. Annoyance from static charges can be prevented by preparing the surface in an atmosphere of 60 to 75 percent relative humidity.

The filled sample container and the "working" standard with cover glass are inserted in the holders provided for them. The cap containing the filter is placed over the eye-piece of the photometer. The 1000-watt lamp is turned on. Looking into the photometer with the sample and standard in place in the holder, the observer sees a circular field, half of which is illuminated by light reflected from the standard and half by light from the sample.

Photometric measurements should not be made immediately after the eye has been exposed to strong light but the eye should first be permitted to become adapted to the conditions of observation.

The two areas in the field can be made to match in brightness by turning the photometer head. The position giving the match is read from the graduated scale and recorded. For convenience, all matches are made so that the readings will be between 0 and 90°, although it is possible to make matches in each of the four quadrants. Two such matches are made successively and recorded. The sample and standard are interchanged by rotating the holder for sample and standard (B, fig. 8) through an arc of 180°. The photometer head is again adjusted until the brightness of the two fields is the same and the position giving the match is recorded. Four such readings are taken. The sample and standard are again interchanged, and two more matches for brightness are made and recorded. The sample container is now removed from the instrument.

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[3] This method of forming the surface was developed by the Wall Rope Works, Inc., Beverly, New Jersey.

If during the course of measurement the first and last pairs of readings differ by more than ±0.5°, it is probable that the conditions of measurement have changed during the observation and that the results are not trustworthy. For example, if the eyes of the observer have not become accommodated to the conditions of illumination when taking the first pair of readings, the last pair may not be in agreement. When this occurs, the readings should be repeated.

IV. CALCULATION AND INTERPRETATION OF RESULTS

For convenience in calculation, the mean of the readings of the larger angle is called θ_1, and the mean of the readings of the smaller, θ_2. The reflectance of the sample for light of wave length 500 millimicrons, under the conditions of measurement, expressed as a percentage of that of a primary reference standard, magnesium oxide, is obtained by the following formula:

$$\text{Reflectance value (in percent)} = 100\ F \times \text{cotangent}\ \theta_1 \times \text{tangent}\ \theta_2,$$

in which F is the ratio of the brightness of the working standard to that of magnesium oxide under similar conditions. An example follows:

Observation No.	θ_1	Observation No.	θ_2
1	46.0°	3	32.3°
2	46.1	4	32.2
7	46.3	5	32.0
8	46.2	6	32.1
Mean	46.15°	Mean	32.15°

The factor, F, for the working standard (used while making the above readings) is 0.823. The calculation of reflectance can conveniently be made with logarithms; thus, log (0.823 x 100) is 1.9154; log cotangent 46.15° is 9.9826 - 10; log tangent 32.15° is 9.7983 - 10. Adding the three logarithms:

$$\begin{array}{l} 1.9154 \\ 9.9826 - 10 \\ \underline{9.7983 - 10} \\ 21.6963 - 20 = 1.6963 \end{array}$$

The number whose log is 1.6963 is 49.7, which is the calculated percentage of the light of wave length 500 millimicrons reflected by the sample towards the photometer relative to that which would be reflected under the same conditions by magnesium oxide. The numerical value 49.7 is considered one measurement on the rope sample.

The data in table 1 were obtained on an average grade of rope measured 25 times by an operator of average experience. The data may serve as an example for comparing the precision of similar measurements obtained from time to time by the same observer, and by different observers under the described conditions.

Table 1.

Measurement No.	Reflectance Value
1	44.7
2	45.4
3	45.3
4	44.7
5	45.5
6	45.0
7	44.1
8	44.7
9	44.9
10	45.1
11	45.1
12	44.8
13	44.8
14	45.1
15	44.5
16	44.1
17	45.1
18	44.5
19	44.2
20	45.0
21	44.6
22	44.3
23	44.7
24	44.1
25	44.7
Mean	44.76
Standard Deviation	0.40

A sample of data such as that given in table 1 serves also as a basis for finding the number of measurements in a group to be made in order to have confidence that the error of the arithmetical mean of the group does not exceed a given amount. Let σ represent the standard deviation obtained with a sample of data; t, a factor dependent on the confidence or probability (e.g., when confidence is 90, 95, and 99 percent, t is 1.645, 1.960, and 2.576 respectively); e, the error of the arithmetical mean of the group; and N, the number of measurements required in the group to give a degree of confidence that e does not exceed a certain amount. The formula for computing N is

$$N = \frac{\sigma^2 t^2}{e^2} \; .$$

The values of N in table 2 have been computed for several values of e, using the value 0.40 for σ obtained in table 1.

Table 2

Number of measurements, N, for confidence that the error, e, of the mean reflectance value does not exceed a given amount.

Confidence Percent	Values of N required to give stated confidence that the error, e, in reflectance units be not greater than:						
	0.1	0.2	0.3	0.5	0.8	1	1.03
99	106	27	12	5	2	2	1 (exactly)
98	87	22	10	4	2	1	
95	62	16	7	3	1		
90	44	11	5	2			
85	34	9	4	2			
80	27	7	3	2			
75	22	6	3	1			
70	18	5	2				
65	14	4	2				
60	12	3	2				
55	10	3	1				
50	8	2					

It should be emphasized that the error and reproducibility will probably differ for the samples of data obtained by different observers with varying degrees of skill and experience.

Correct values of N, based upon another observer's sample of data characterized by a different value of σ, may be computed as described, or, approximate values of N may be computed by multiplying the values of N given in table 2 by the ratio

$$\frac{(\sigma')^2}{(0.40)^2}$$

where σ' is the standard deviation obtained with the other observer's data.

The error, e, so computed results solely from lack of reproducibility in making the measurements; it can be taken as the final error only if other errors (such as those resulting from improperly adjusted equipment, incorrect illumination, and erroneous calibration factors for the reflection standards) have been eliminated.

V. REFERENCES

The following references should prove helpful to the technologist who is interested in the spectrophotometric and colorimetric problems arising in the measurement of manila rope fibers.

(1) Genevieve Becker. Procedure for the measurement of the reflectance of manila rope fiber for light of wave length 500 millimicrons. Letter Circular LC-393, Bureau of Standards, November 1933. The original procedure.

(2) R. E. Miskelly. A satisfactory method for cutting manila fiber for the Becker value test. Cord Age, Vol. XXXI, No. 6; June 1929. The Plymouth cutting device is illustrated and manner of its use described.

(3) Wm. D. Appel. A method for measuring the color of textiles. American Dyestuff Reporter, vol. 17, pp. 29-34, 49-54; 1928. A description of apparatus and method similar to the method for measuring the reflectance of manila rope fiber.

(4) Genevieve Becker and Wm. D. Appel. Evaluation of manila rope fiber for color. BS J. Research, vol. 11, No. 6, pp. 811-22; December 1933; RP627. A description of the original method for measuring the reflectance of manila rope fiber and other information relative to its use.

(5) Genevieve Becker. Spectral reflectance of the Philippine Island Government Standards for Abacá fiber. BS J. Research, vol. 11, No. 6, pp. 820-828; December 1933; RP628. A report of spectral reflectance measurements of a set of the official standards for the grades of abacá used in cordage.

(6) H. J. McNicholas. Equipment for routine spectral transmission and reflection measurements. BS J. Research, vol. 1, pp. 793-857; January 1928; RP30. Information pertinent to the use of a polarizing photometer.

(7) Report of the Optical Society of America Committee on Spectrophotometry. J. Opt. Soc. Am., vol. 10, pp. 169-241; February 1925. General information.

(8) Report of the Optical Society of America Committee on Colorimetry for 1920-21. J. Opt. Soc. Am. and Rev. of Scientific Instruments, vol. 6, pp. 527-596. General information.

(9) H. J. McNicholas. Absolute methods in reflectometry. BS J. Research, vol. 1, pp.29-73; July 1928; RP3. Theoretical discussion.

(10) Preparation and colorimetric properties of a magnesium-oxide reflectance standard. National Bureau of Standards Letter Circular LC-547. March 17, 1939.

(11) K. S. Gibson. Spectral luminosity factors. J. Optical Soc. Am., vol. 30, pp. 51-61; February 1940.

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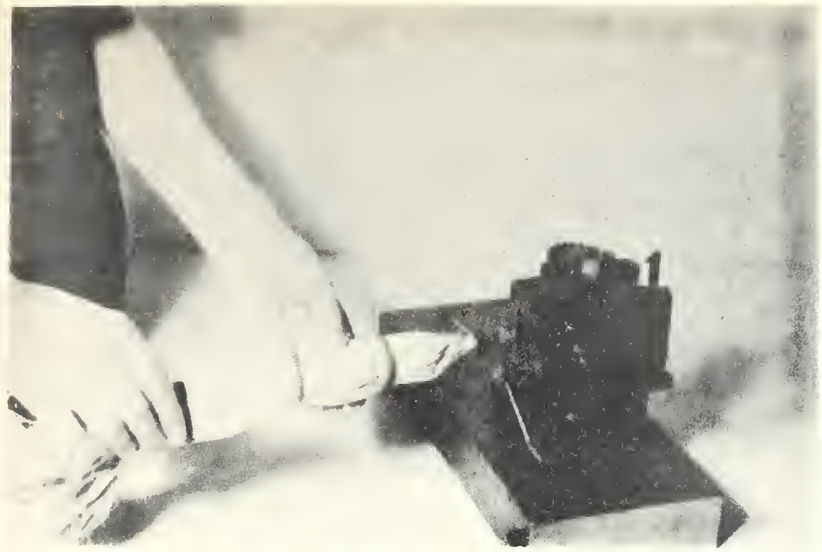

FIGURE 1.

Rear of vise with wrapped sample of rope yarns ready to be inserted.

Leather faced jaw has been lowered. Spacer plates, 1, bolted in place.

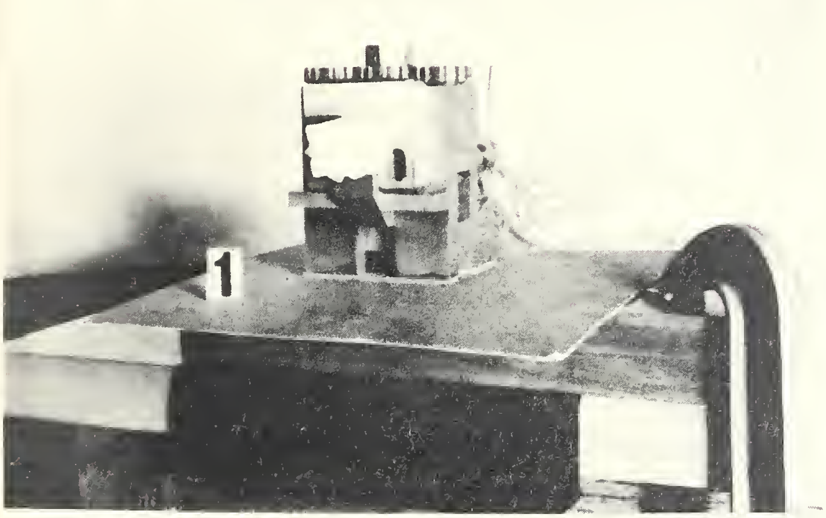

FIGURE 2.

Vise clamped to table, charged with fiber ready for cutting.

The leather faced jaw has been drawn against the specimen, holding it tightly. (1) is a piece of cardboard placed to catch the end first to be cut. Spacer plates are in place. Bolt heads withdrawn through holes in spacer plates. Rubber band near end has been removed.

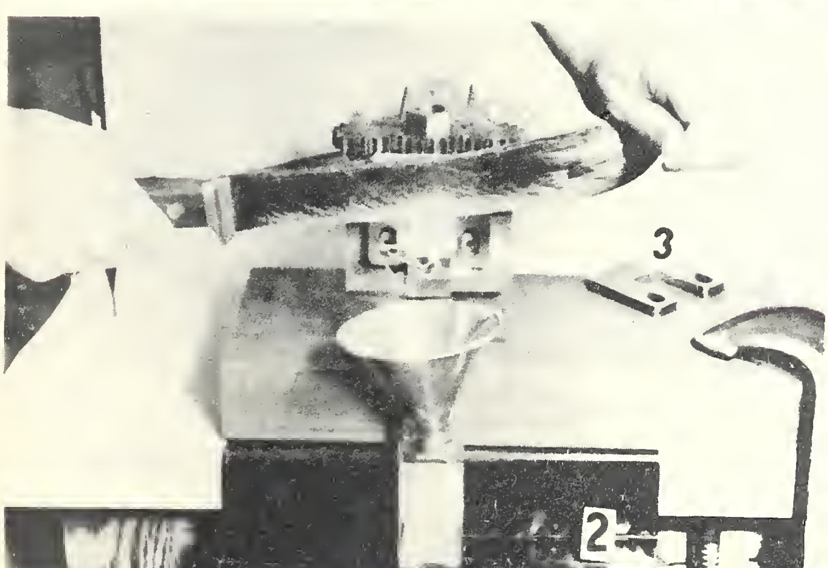

FIGURE 3.

Cutting 1-mm length of fiber.

The ragged end of paper and fiber protruding, fig. 2, has been cut off and the cardboard, 1, fig. 2, removed. Labeled specimen bottle with funnel to receive cut fibers is in place on shelf, 2. A group of spacer plates removed as the cutting proceeds is shown at 3. Note crinkled strip of paper formed by cutting.

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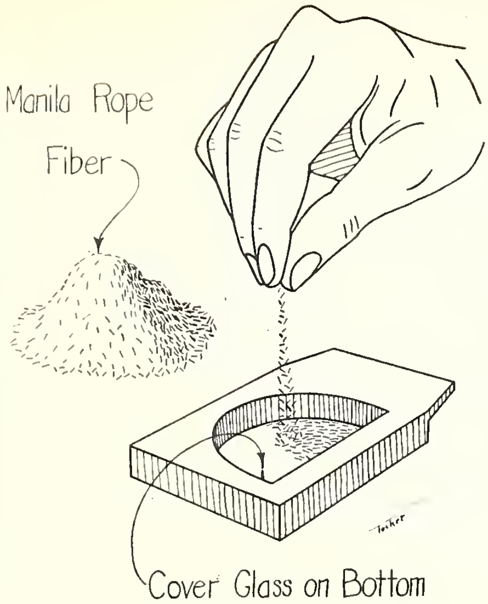

FIGURE 4.

Illustrating the manner in which the rope fibers are distributed (sprinkled) onto the cover glass of the sample container to form the surface to be measured.

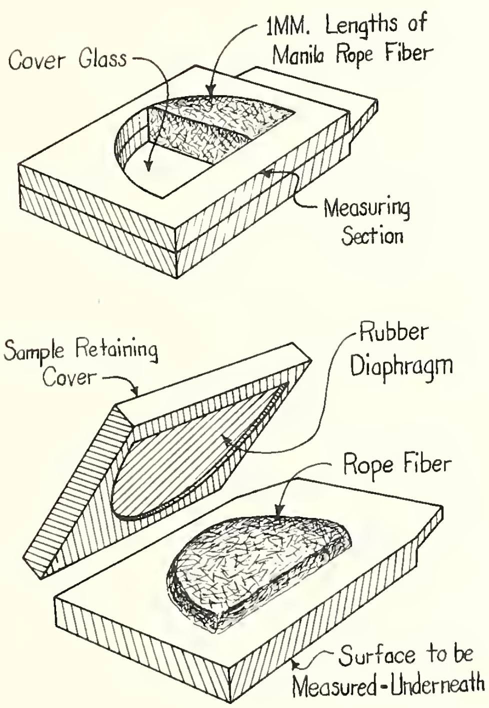

FIGURE 5.

Sample container.

Top drawing shows the sample container with the measuring section in place. One half of the fiber mat filling the container and section has been cut away, exposing the cover glass and revealing position of the section with regard to sample container. The cover glass is cut from microscope slide glass.

Bottom drawing shows the fibers in place in the cavity closed on the underside by the cover glass. The measuring section has been removed and sample-retaining cover is ready to be placed on the sample and container.

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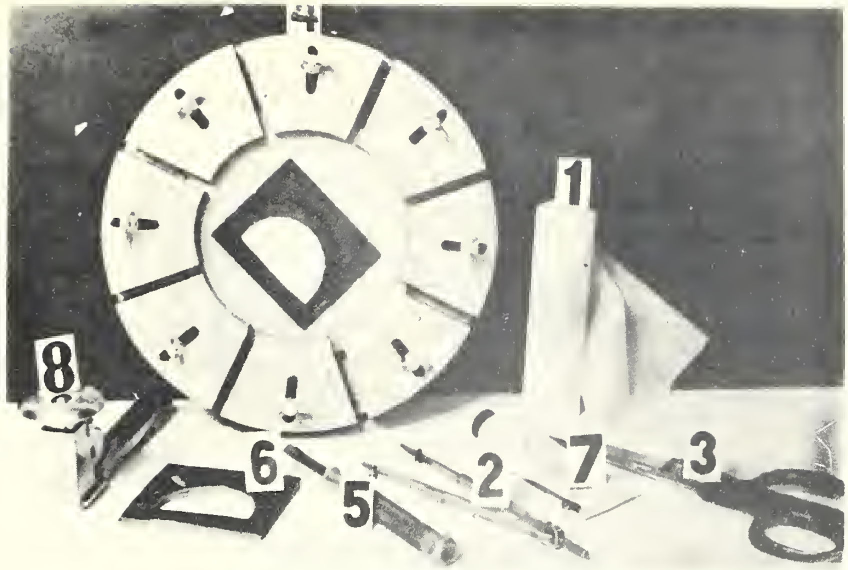

FIGURE 6.

Shaping the rubber diaphragm to form the sample-retaining cover.

A cardboard panel is shown stapled in place on the diaphragm, the staples passing through the diaphragm into panels on the opposite side of the diaphragm.

(1) Rubber dam. Medium, 5 in. wide. Rubber dam found satisfactory for the purpose was obtained from the S. S. White Dental Manufacturing Company, Philadelphia, Pennsylvania.

(2) Compass for marking off the circle of rubber dam to be inserted in the frame.

(3) Scissors for cutting the rubber dam.

(4) The frame with the rubber stretched and clamped in place by means of the sector clamps which are held in place by the wing nuts and bolts shown in the slot of each sector.

(5) Screw driver for tightening the wood screws which join the sectors.

(6) Cardboard panels for forming the sample-retaining cover.

(7) Rubber cement to hold the panels in position while being clamped to the diaphragm.

(8) Stapling machine for attaching the rubber dam to the sample-retaining cover.

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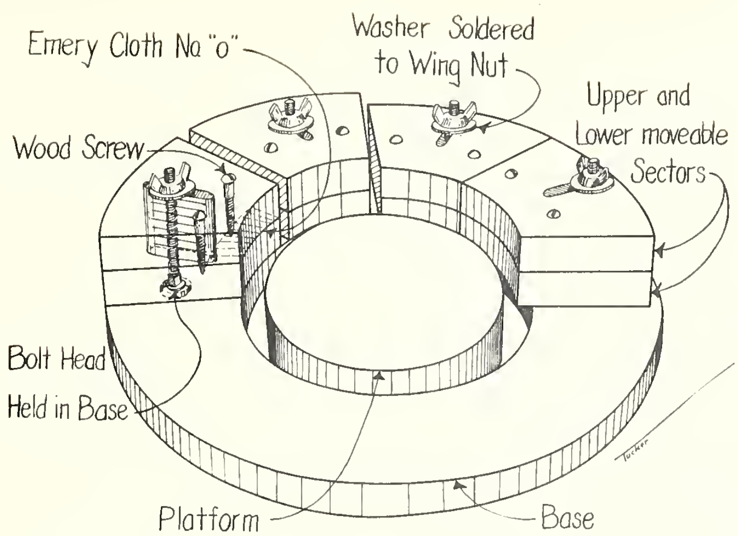

FIGURE 7.

Frame for stretching rubber diaphragm and holding it in place while fastening it to the sample-retaining cover.

Four of the radial clamps (see also 4, fig. 6) are shown. The two on the right are in a position with regard to the center of the frame, occupied by all the clamps when holding the rubber without tension. The two on the left are moved back in position occupied when the clamps hold the rubber under tension, ready to be fastened to the sample-retaining cover.

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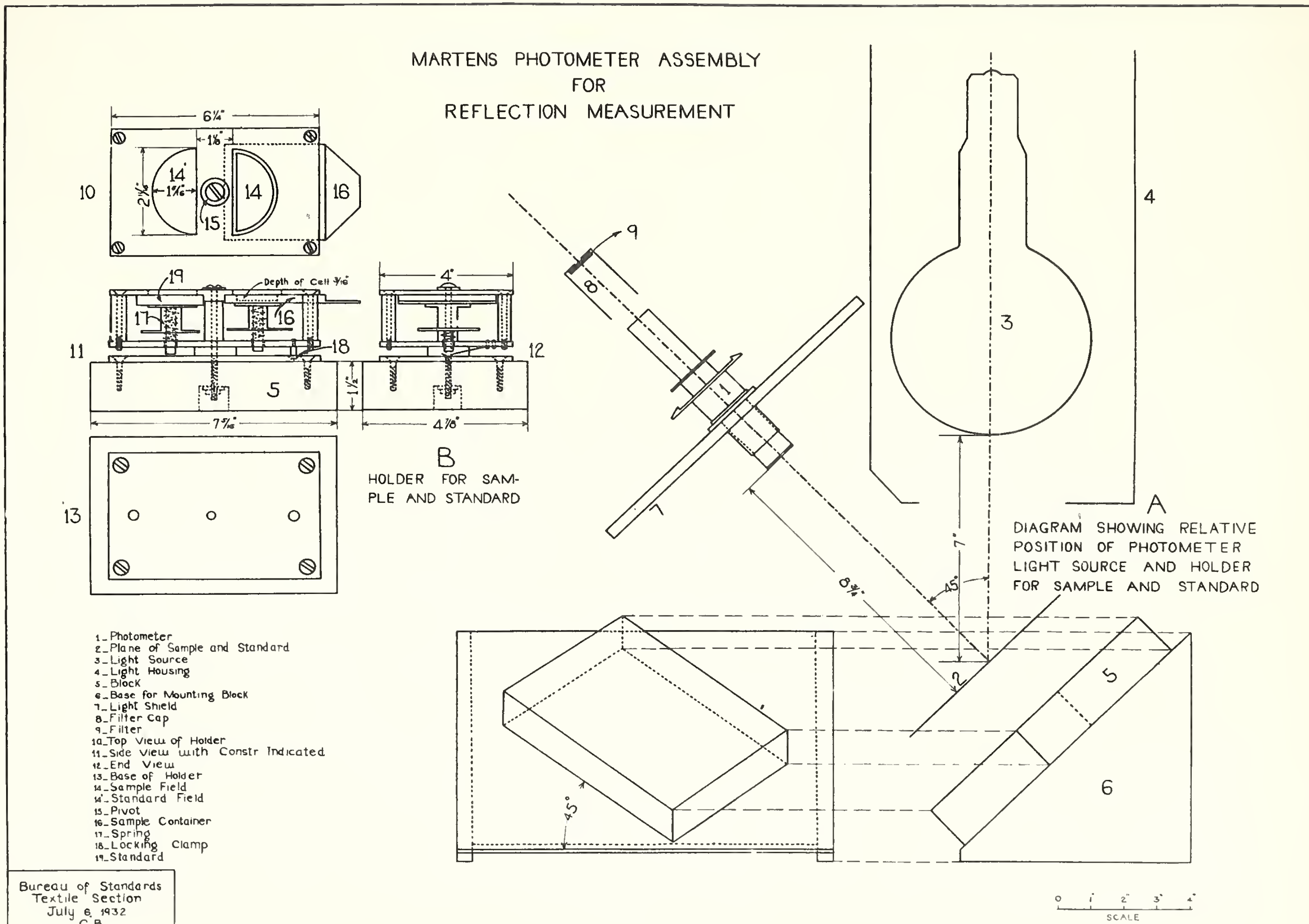
MARTENS PHOTOMETER ASSEMBLY
FOR
REFLECTION MEASUREMENT
6¼"
1⅛"
14'
14
15
16
10
19
Depth of Cell 5/16"
17
11
18
5
1½"
7 5/16"
4"
12
4⅞"
B
HOLDER FOR SAMPLE AND STANDARD
13
9
8
1
7
3
4
7"
45°
8¾"
2
A
DIAGRAM SHOWING RELATIVE POSITION OF PHOTOMETER LIGHT SOURCE AND HOLDER FOR SAMPLE AND STANDARD
5
6
45°
1_Photometer
2_Plane of Sample and Standard
3_Light Source
4_Light Housing
5_Block
6_Base for Mounting Block
7_Light Shield
8_Filter Cap
9_Filter
10_Top View of Holder
11_Side View with Constr Indicated
12_End View
13_Base of Holder
14_Sample Field
14'_Standard Field
15_Pivot
16_Sample Container
17_Spring
18_Locking Clamp
19_Standard
Bureau of Standards
Textile Section
July 6, 1932
G.B.
0 1" 2" 3" 4"
SCALE

FIGURE 8