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MICHIGAN
STATE HIGHWAY DEPARTMENT
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State Highway Commissioner

#173

MANUAL FOR REFLECTOMETER OPERATION AND MAINTENANCE

The reflectometer described herein for measuring reflectivity of white-pigmented membrane curing compounds was designed and built in the Research Laboratory as a development in continuing research on concrete curing methods. It is intended to provide a short, dependable method for acceptance testing of white membrane curing compounds in lieu of complete spectrophotometric analysis.

Highway Research Project 42 B-14 (2)

Research Laboratory
Testing and Research Division
Report No. 173
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MANUAL FOR REFLECTOMETER OPERATION AND MAINTENANCE

This reflectometer is designed to determine the luminous directional reflectivity of white membrane curing compounds. "Luminous directional reflectivity" is defined in ASTM method D307-44, and supplants the obsolete term, "apparent daylight reflectance", which occurs in the MSHD current specifications for white curing compounds. The method described herein is based on the ASTM method but reflectivity is determined from reflection values obtained at wavelength bands of light through three colored filters instead of at a continuous series of wavelengths throughout the visible spectrum. Many tests have shown a satisfactory agreement between results obtained by the abridged method and the longer ASTM analysis. Luminous directional reflectivity, R_s, is expressed as percent in relation to some arbitrary standard. Since freshly prepared magnesium oxide reflects more than 98 percent of incident radiation at all wavelengths, it is the standard usually employed for such tests and has been adopted here.

Included in this manual are a description of the reflectometer, instructions for its care and maintenance, and complete operating directions to determine luminous directional reflectivity.

APPARATUS

Description

An abridged spectrophotometric method is incorporated in this instrument, based on three primary colored filters. The prepared sample and standard white (a magnesium oxide coated plate) are mounted in the instrument in such a way that they can be alternately illuminated at an angle of 45 degrees. The instrument is shown in Figure 1. The reflected light is measured in a direction normal to the surface of the sample.

A blackened tube is used to shield the reflected light being measured as it passes through a hole in the wall separating the two sections of the instrument; thence through the particular filter in place to the photoelectric cell. The photocell is connected to the galvanometer and a shunt across the terminals which is activated by a microswitch as needed. The three filters are mounted side by side in a single holder which slides in guides so that each filter can in turn be placed in front of the photocell, as shown in the figure. With this arrangement, it is possible to obtain a complete set of readings showing the relative quantities of light reflected by the sample and standard for each of the three colors. A voltage regulator is used to maintain a constant illumination on the panel during the test.

Operation

The apparatus should be set up in a location where it will be free from vibration, smoke, or any bright light shining directly into the instrument vents.

The power line of the voltage regulator is connected to a 110-volt 60-cycle A.C. source. The reflectometer is plugged into the regulator. The galvanometer can be plugged directly into a 110-volt outlet.

Before the galvanometer is connected to the photocell terminals, the shorts across these terminals should be removed. The galvanometer hairline indicator should then be set to zero, using the knob marked "Adjust Zero", with reference to the lower scale (reading 0 to 80, left to right). The galvanometer should not be moved after this adjustment has been made since doing so will disturb the zero setting. The galvanometer is then connected to the photocell terminals with the wire leads provided, taking care that the polarity of these connections is correct. The terminal posts and lead wire connectors are color-coded to prevent error. The lamp is turned on by depressing both switches on the right side of the instrument. This also turns the fan on.

With the standard and sample inserted in the instrument, the lamp should be turned on for a minimum warm-up period of twenty minutes. This enables the voltage regulator, photocell, shunt, and galvanometer to stabilize to test conditions.

When the test is completed, the instrument should be cooled before moving it by turning off the lamp (bottom switch) and permitting the fan to run. When cooled, the fan switch should be turned off and all plugs disconnected, including the voltage regulator connection to the power source. The filter control rod is then pushed to the extreme left (red filter) position. If the instrument is to be moved, the standard panel should be removed. This will prevent particles of magnesium oxide from dropping on the black surfaces of the interior. Disconnect the galvanometer line, making certain to replace the shorts across the photocell and the galvanometer terminals.

When moving and storing the instrument, it must be kept in an upright position. Choose a storage area relatively free from dust, vibration, and a corrosive atmosphere. Prior to storing the aluminum panels, they should be cleaned in trichlorethylene or a similar solvent, to be ready for the next test.

TEST PROCEDURE

Preparation of Standard

As a standard white, a panel coated with magnesium oxide is obtained by burning magnesium metal ribbon in a well ventilated hood. The ribbon can be obtained in 1-oz. rolls, 1/8 in. in width from chemical supply houses.

The ribbon is taken from the roll and alternately folded back and forth until it forms a stick of six layers thickness, and about 5 or 6 in. long. Before breaking the end loose from the roll, it is spirally wound around the folded stick to hold it in shape, as shown in Figure 2C. Several of these should be made up before the actual coating of the plate commences. These sticks can now be easily handled with clean tongs while burning so that the magnesium oxide can be deposited on the panel. The panel should be held with tongs also, since it gets very hot. While burning the magnesium, it is not advisable to look directly at or near the

flame since the intensity of the visible light and the ultraviolet energy emitted are definitely injurious to the eyes. Welders' goggles or a piece of the glass from these goggles should be used as protection when burning the magnesium.

The magnesium is ignited in a Bunsen burner flame and then held to one side of the flame so that the magnesium oxide smoke rises freely and remains uncontaminated. The panel to be coated is held about 4 or 5 in. above the burning magnesium. As an aid to determine when the panel has been coated sufficiently, a black mark can be made on the panel prior to coating.

This fine powder coating of magnesium oxide must be handled quite carefully since it tends to flake from the panel. This is particularly true if the panel becomes too hot while applying the magnesium oxide, or if the previous coatings are too old. To avoid flaking when placing the standard in the instrument, about 1/16-in. of magnesium oxide must be very carefully removed from the top and bottom edges (Figure 2). To do this, use a razor blade, using a drawing motion. As a precaution, the standard should be placed in the instrument as soon as possible. The sample retainer should be steadied with one hand while sliding the panels in, aiding in protection of the coatings. A copy of the National Bureau of Standards circular entitled "Preparation and Colorometric Properties of a Magnesium-oxide Reflectance Standard" is attached.

Time can be saved by adding a new coat or two of magnesium oxide daily. The panel should be completely stripped and an entirely new standard coating built up weekly. The powder tends to lose its bond to the aluminum with time, making it difficult to handle without flaking off. Magnesium oxide is not absolutely stable as far as its color properties are concerned and any change in its color would affect the test.

> NOTE: Ceramic standards which have good color stability and which can be used as secondary standards in place of MgO are available from the National Bureau of Standards. We do not have these in the East Lansing Laboratory, however, because we felt that all the development work should be based on the primary MgO standard.

Preparation of Sample

In preparing the membrane curing compound samples to be tested, a standard procedure has been developed and adapted using two coats, a prime and a final coat. The prime coat is sprayed on until the panel appears covered. This coat is then dried in an oven at 140°F. for 15 minutes. The panel is cooled to room temperature before the final coat is applied. The final coat is dried in the 140°F. oven for 1 hour.

If a hiding power test is to be run on the sample, time and work can be saved by spraying the preliminary coat enough in advance of this test so that the panel is dry and ready for its final coat at the time the hiding power cards are sprayed. Each panel should be properly identified and about 1/16-in. of the bottom and top edges masked with tape during spraying so that the panels will slide easily into the retainer. This same tape can be used to hold the panel to a paper backing for convenience in handling. In spraying the test samples, the resulting surface should

be diffusive (coarse in texture) as shown in Figure 2A. This type of surface more nearly approaches the type of surface obtained in field applications. (A flat smooth-surfaced specimen will give results 2 percent or more lower in the value of R_g than the same sample sprayed to give a coarse-textured surface). To obtain this coarse texture, the panel being sprayed is held about 8 in. from the spray gun. The panel should be handled carefully, taking care not to touch the face. The test should be run as soon as possible after the sample has dried.

Procedure

Two controls are used to operate the instrument during the test -- the test panel selector (on the top front center of the instrument), and the filter selector rod (protruding through the right side near the bottom at the back).

To insure consistent and proper orientation of the test panels, a metal guide is secured on the top of the instrument. The guide has two "V" slots cut into it so that the test panel selector lever will slide into these slots. The lever must fall into these "V" slots to assure proper positioning of the panels. Since the instrument has been calibrated with respect to these "V" slots, any change in their position would necessitate a recheck on the instrument calibration. If recalibration becomes necessary, the instrument should be returned to the East Lansing Laboratory.

The filter control rod is directly connected to the filter frame which retains the three filters. To place each of the filters into position, the following procedure is observed: -

Green filter: Pull the control rod to the extreme right until the green filter-stop is touched.

Blue filter: Push the rod back into the box carefully until the blue filter-stop is touched.

Red filter: First depress the blue filter-stop release button (on the instrument top to the back left); then push the rod into the box until the red filter-stop is touched.

In each case, the control rod must be moved until the filter frame is brought up against the filter-stop.

After the instrument has been warmed up for the necessary 20 minutes, proceed with the test, using the following steps: -

1. Blue filter in position: MgO galvanometer reading, then sample galvanometer reading.

2. Green filter in position: Sample reading, then MgO reading.

3. Red filter: MgO, then sample reading.

4. Repeat 3 steps above, but taking sample reading first.

These steps are followed until three complete sets of actual readings are obtained for each filter.

After the data has been obtained, it should be checked over carefully, comparing each pair of readings to see that they compare reasonably with others for the same filter. If it is felt necessary, repeat any PAIR of readings (i.e., MgO and sample).

Computations

A sample of the data form is attached. In the extreme left column are listed the three filters in the instrument. The second and third columns are for galvanometer readings with the light incident on the magnesium oxide and the sample, respectively. The "MgO" and "sample" readings are made in pairs, three pairs being made for each filter. "R_λ" (spectral directional reflectivity) is the ratio of the sample to the MgO reading for each filter. The "av. R_λ" is obtained by averaging the three R_λ values for each filter.

H_λ and $\bar{y}_\lambda$ are the relative spectral irradiance of ICI Illuminant B and the luminosity function of the equal energy spectrum, respectively. The mean wavelength of light transmitted through the three filters found by the centroid method, is 495, 552.3, and 655 mμ for the blue, green and red respectively. Values for H_λ and $\bar{y}_\lambda$ for these three wavelengths taken from the tables in ASTM D 307 have been multiplied together and inserted in Column 6 of the sample data sheet for convenience. These three values for $H_\lambda\ \bar{y}_\lambda$ will be always the same for this particular set of filters when computing R_s for Illuminant B (natural sunlight) and may be incorporated permanently in the data sheet. The last column, headed "av. $R_\lambda\ H_\lambda\ \bar{y}_\lambda$" is the product of these three values. The sum of the values in the last column is divided by the sum of the values of the constants and multiplied by 100 to obtain r_s, an uncorrected value of the reflectivity. From a comparison of the results obtained with this instrument with those from a complete spectrophotometric analysis, a correction of 1.43 must be added to the uncorrected r_s determined in accordance with the foregoing procedure to find the actual value, R_s. The computational steps are listed below: -

1. Divide "sample" by "MgO" for each pair of readings to obtain R_λ.

2. Obtain the average of the three values of R_λ for each filter to obtain the "av. R_λ".

NOTE: (Compare "R_λ" values with "av. R_λ" values for each filter group. The usual maximum deviation is about 0.5 percent. If the deviation of the individual value from the average is greater than 1 percent, the test should be repeated.)

3. Multiply each "av. R_λ" by the constant "$H_\lambda\ \bar{y}_\lambda$" for each filter.

4. Divide the sum of the "av. $R_\lambda\ H_\lambda\ \bar{y}_\lambda$" products by the sum of the three constants "$H_\lambda\ \bar{y}_\lambda$" and multiply by 100 to obtain r_s in percent (the uncorrected luminous directional reflectivity.)

5. Add the correction factor, 1.43, to obtain the actual reflectivity value R_s and round off to the nearest unit. This is the figure reported.

Written in the form of an equation:

$$R_s = \frac{\Sigma R_\lambda H_\lambda \bar{y}_\lambda}{\Sigma H_\lambda \bar{y}_\lambda} .100 + 1.43$$

CARE OF THE INSTRUMENT

To keep the instrument in proper operating condition, the following should be observed: -

1. Oil the ventilating fan occasionally (1 drop of oil per bearing pad), wiping off the excess oil.

2. Keep the instrument interior clean and free from dust and magnesium oxide particles.

3. If necessary, paint the interior with blackboard paint. (The finish MUST be a dull, flat black).

4. Do not remove the back of the instrument unless it is absolutely necessary to clean the filters or photocell, or for necessary repairs.

5. Clean the lamp, mirror, filters and photocell with lens tissues annually (more often if necessary). Instructions for removing these parts for cleaning are given below.

Remove the left side of the instrument by removing all wood screws set into it through the top, front, back and bottom; also into the partition. Remove the filter-stop nearest the side removed; then loosen the lock nut on the filter control rod and unscrew the rod. Now, the filter rack can be slid out of the track for cleaning. The photocell unplugs. Figure 1D shows the instrument dis-assembled in this manner. The lamp, which is also easily accessible now, has a bayonet prefocused base. It must be depressed slightly and turned counter-clockwise for removal. The reflector can be cleaned in place, using care not to exert pressure enough to move it out of adjustment. All of these parts should be cleaned with a soft lintless cloth or lens tissue only. In replacing these parts, the reverse procedure is followed.

The instrument is supplied with aluminum panels numbered on the finger tabs. It is this numbered side which should be considered the face of the panel. These panels are made to slide into the sample retainer in the instrument.

REFLECTOMETER PARTS AND ACCESSORIES LIST

1 - G.E. Projection Lamp, 100 W; 100 T 8/108 sc

3 - Corning Filters; color specification Nos. 4-104, 4-102, and 2-78

1 - Weston Photronic Cell; model 594 RR

1 - Micro-switch; catalog No. BZ-2RL2

1 - Sola Constant Voltage Transformer; 120 V.A.; catalog No. 30806, serial No. D 04202

1 - G.M. Laboratories Galvanometer; int. res. 370 ohms, ext. crit. damp. res. 2640 ohms, sensitivity 0.033 microampere per mm., catalog No. 570-402

1 - Bausch and Lomb welding glass plate; 2" x 4-1/8", shade No. 12

20 - Aluminum test panels; 3-3/8" x 3-1/2", numbered

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RESEARCH LABORATORY
Testing and Research Division

519 R

Project No. _____

Date February 26, 1952

SUPPLEMENTAL LABORATORY NOTES White Membrane Curing Compound

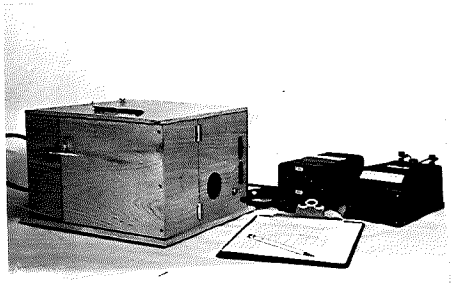
Sample No. 51 MR-12

Filter	MgO	Sample	R_λ	av. R_λ	$H_\lambda \bar{y}_\lambda$	Av. $R_\lambda H_\lambda \bar{y}_\lambda$
blue	39.9	29.5	0.739	0.740	24.747	18.312
	39.8	29.5	0.741	-	-	-
	39.8	29.5	0.741	-	-	-
green	37.4	28.3	0.757	0.756	101.499	76.733
	37.4	28.2	0.754	-	-	-
	37.3	28.2	0.756	-	-	-
red	62.7	47.4	0.756	0.758	8.530	6.466
	62.2	47.4	0.762	-	-	-
	62.2	47.1	0.757	-	-	-
					134.776	101.512

r_s = 101.512/134.776 = 75.32%

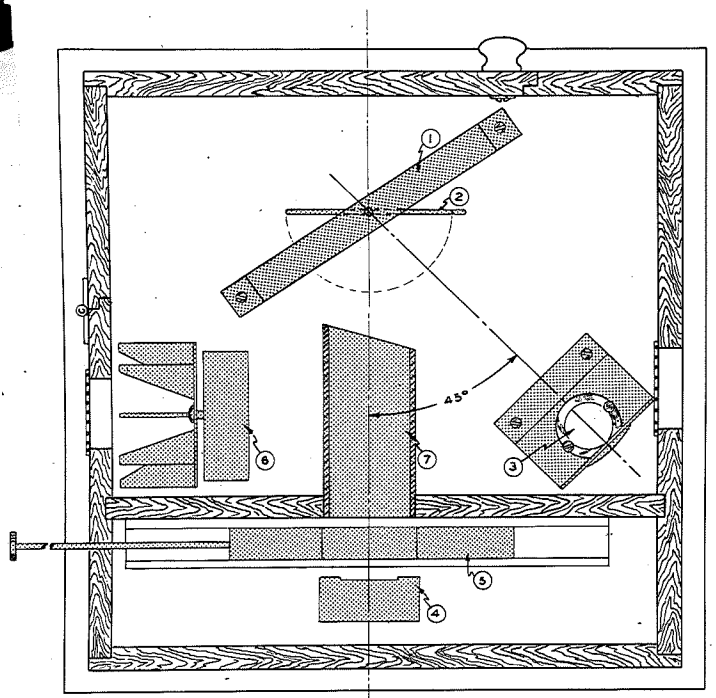
R_s = 75.32% + 1.43% = 76.75%

= 77%



REFLECTOMETER, VOLTAGE REGULATOR, AND GALVANOMETER ASSEMBLY

Ⓐ



1 SAMPLE MOUNT SUPPORT
2 SAMPLE
3 LIGHT SOURCE
4 WESTON PHOTOELECTRIC CELL
5 FILTERS
6 VENTILATING FAN
7 TUBULAR LIGHT SHADE

SCHEMATIC DIAGRAM OF REFLECTOMETER

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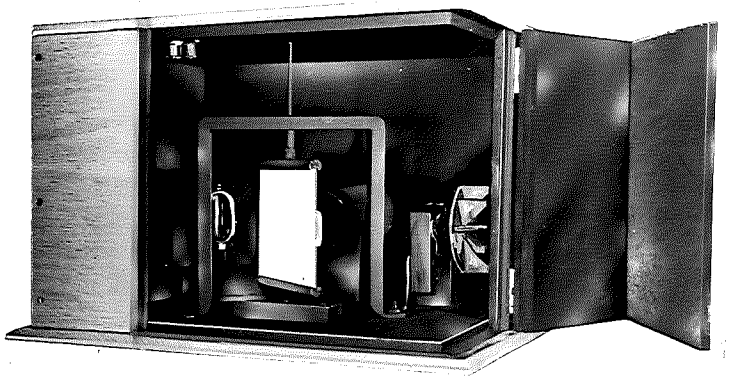
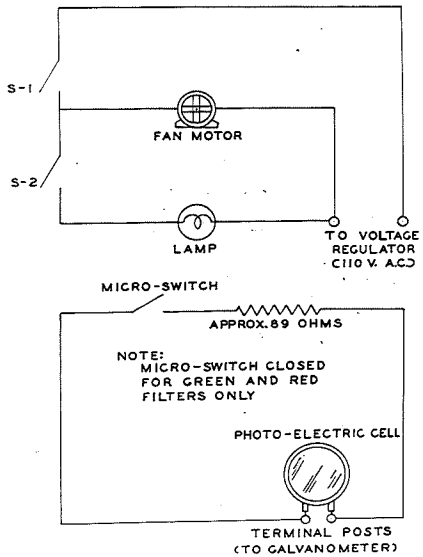
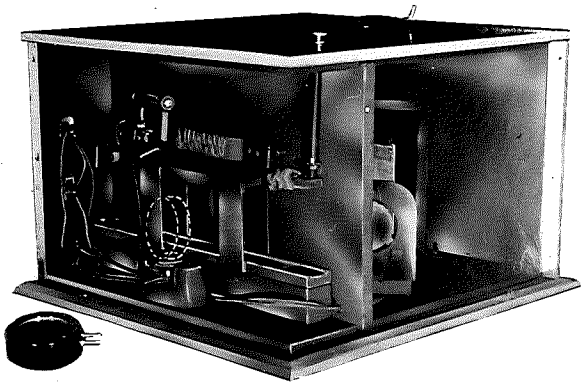


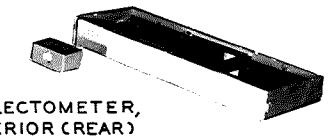
REFLECTOMETER, INTERIOR (FRONT)

Ⓒ



CIRCUIT DIAGRAMS





REFLECTOMETER, INTERIOR (REAR)

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MICHIGAN STATE HIGHWAY DEPARTMENT REFLECTOMETER

FIGURE 1

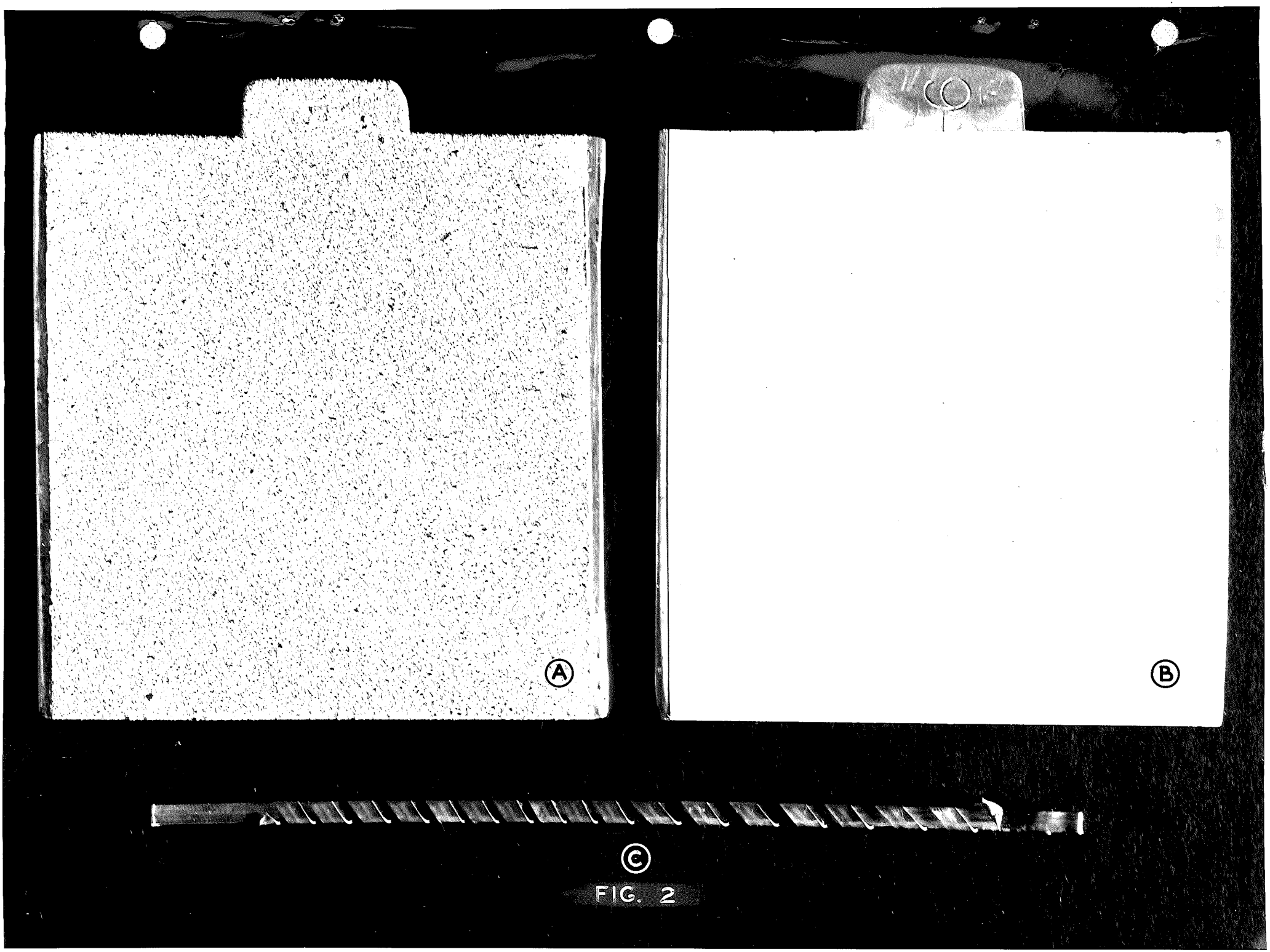

FIG. 2

HJK:EMR

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

Letter
Circular
LC-547
Superseding
LC-395

March 17, 1939

Preparation and Colorimetric Properties of a Magnesium-Oxide Reflectance Standard.

The smoke from magnesium freely burning in air deposited on a satisfactory base forms a uniform, fine-grained, diffusing surface of high reflectance. By observing a few simple precautions, this surface of magnesium oxide (MgO) may be made reproducible; hence, it serves as a convenient and reliable standard.

1. The magnesium should be obtained in the form of turnings of approximately 0.02 in. in thickness and 1/8 in. in width, preferably of spiral shape, and containing a minimum of magnesium dust. Suitable material may be obtained of the American Magnesium Corporation, 2210 Harvard Avenue, Cleveland, Ohio, the Aluminum Company of America, Gulf Building, Pittsburgh, Pennsylvania, Sterling Products Company, Easton, Pennsylvania, the Pfenstiehl Chemical Company, Waukegan, Illinois, and others.

2. The oxide must be deposited on a surface not affected in air by the heat from the burning magnesium. A satisfactory base may be made of (a) aluminum, (b) block porcelain, (c) sheet steel coated with white vitreous enamel, or (d) a baked surface of a sprayed mixture of magnesium oxide powder and distilled water (7, p. 21)*. Milk or opal glass is often unsatisfactory because it easily cracks from heating. Depolished surfaces are better than polished because the oxide adheres better; for the same reason, metallic surfaces are usually to be preferred to non-metallic. Surfaces of reflectance high and uniform throughout the spectrum are better than dark or chromatic surfaces because with the former a thinner layer of oxide is trustworthy. The thinner layer is desirable apart from speed of preparation because it does not chip off so readily.

3. Place a small quantity (about 5g) of the chips on a refractory dish (zirconium silicate and magnesite are suitable refractory materials) and ignite them with a hand blow torch or bunsen burner. Work the unignited chips beneath the flame until a slowly burning ball or clinker is formed; this gives a steady stream of smoke.

4. Place the surface to be coated about 8 to 10 cms above the flame and tilted about 30 degrees from the horizontal. Use of smaller distances results in a coarse-grained deposit and risks contamination by possible impurities in the magnesium (3).

(*) Numbers in parentheses, sometimes followed by a page number, indicate references in the bibliography.

5. Move either the combustion dish or the surface being coated from side to side in order to obtain a uniform deposit.

6. When the clinker has to be turned over or broken, in order to permit the magnesium to burn completely, the surface being coated should be temporarily removed, since the burst of flame is likely to carry up large dust particles.

7. Repeat the operation several times until a sufficient deposit is obtained. The layer should be so thick that further increase produces no sensible change in reflectance; the critical thickness is about half a millimeter (4, p. 17). Do not attempt to burn a large charge of magnesium at one time. Rather, build up the required thickness by a large number of small charges. In cases where it is inconvenient to measure the thickness of the coating place a small dot of india ink on the original surface near the edge, then deposit oxide until the spot cannot be seen in good illumination. If the original surface is dark, put on one coat of MgO first; a deposit of black smoke (from a candle or smoky gas flame), in a small spot near the edge, then supplies a similar test.

8. The operation should be carried out under a well ventilated hood in order to dispose of the excess oxide.

9. The operator's eyes should be protected from the high intensities of visible and ultraviolet radiant energy by suitable goggles (4, p. 30), or other means.

10. Magnesium ribbon may be used for small surfaces instead of turnings, but for large surfaces it requires careful manipulation to produce a uniform coating because of the irregular burning of the ribbon.

The properties of a surface so prepared are as follows:

1. It is a good diffusor (1, p. 59; 2).

2. Its light reflectance, 0.97 to 0.98, is very high (1, 4, 5).

3. The reflectance varies with wavelength in the visible spectrum by less than one percent (2,4) when the oxide is first prepared. (But see 5, below.)

4. The apparent reflectance for 45-degree incidence and normal viewing (standard conditions adopted by the International Commission on Illumination, Cambridge, 1931) is 1.00 (4, p. 29).

5. Its reflectance varies slightly with time. Although the reflectance is apparently constant with time between 550 and 750 mμ, it decreases at wavelengths less than 550 mμ; this may amount to as

much as 3 percent in the violet (6, p. 378), and causes the oxide to become slightly yellower with time. In such a case the changes in the trichromatic coefficients, x, y, z, and the luminous reflectance R, computed on the 1931 I.C.I. basis and for I.C.I. Illuminants A and C are as follows:

	Δx	Δy	Δz	ΔR
I.C.I. Illuminant A	$+0.001_1$	$+0.000_9$	-0.001_9	-0.001_3
I.C.I. Illuminant C	$+ .001_4$	$+ .002_0$	$- .003_5$	$- .002_3$

6. It is extremely fragile. However, if the MgO is deposited in a flat trough the edges are protected from chipping off.

The first four properties listed make this reproducible surface a convenient reference standard of reflectance; its usefulness is limited by the fifth and sixth properties (lack of constancy, fragility), which make it often desirable to use working standards of reflectance carefully calibrated in terms of the freshly prepared MgO.

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