Brightness of clay and other mineral pigments (45/0)  
*(REAFFIRMATION of T 646 om-11 as an Official Method)*  
*(Editorial changes from Draft 4 incorporated)*

1. **Scope**

1.1 This method describes a procedure for determining the brightness of clay or other mineral pigment that has been pulverized under controlled conditions and made into a uniformly compacted pigment plaque. This method is for use with minerals normally used in the manufacture of paper and is not intended for highly colored pigments.

1.2 The instrument employed has the same spectral, geometric, and photometric characteristics as that described in TAPPI T 452 “Brightness of Pulp, Paper, and Paperboard (Directional Reflectance at 457 nm).” The brightness scale applicable to this method is the same as the brightness scale described in TAPPI T 452.
1.3 In contrast to TAPPI T 534 “Brightness of Clay and Mineral Pigments (d/0° diffuse),” which uses an instrument with an integrating sphere to provide hemispherical illumination and perpendicular observation, this method utilizes an instrument which uses 45° illumination and perpendicular viewing.

NOTE 1: Brightness values obtained using this method will not agree with those obtained using TAPPI T 534.

1.4 The specimens must be prepared with close adherence to the instructions found in the Appendices.

1.5 This method utilizes a sample preparation apparatus which is identical to that required for TAPPI T 534. The measurement procedure is also similar.

2. Summary

This method provides: (a) a scale for the measurement of the brightness of clay and mineral pigments using 45/0 geometry, (b) a method for verifying the calibration of each instrument used for brightness testing, (c) procedures for the preparation of specimens from both dry powder or aqueous slurry including procedural variations for several specific clays and mineral pigments.

3. Significance

The brightness of clay or mineral pigment affects the brightness of the paper with which it is used. Although one might assume that the measured brightness is a property of the material, in fact the value obtained depends significantly on the manner of specimen preparation. For this reason, detailed instructions for drying and pulverizing the material and for pressing the test plaque are given in the appendices.

4. Definitions

4.1 **Brightness**, reflectance of an opaque thickness of material measured for blue light with a centroid wavelength of 457 nm under specified spectral and geometric conditions of measurement.

4.2 **Reflectance**, ratio of the reflected radiant or luminous flux to the incident flux in the given conditions.

5. Apparatus

5.1 **Reflectance meter**, adhering to the geometric, photometric, and spectral specifications stated in TAPPI T 452.

5.2 **Standards**, set of at least 5 pads of paper tabs and 2 polished white opal glass standards shall be supplied monthly by the standardizing laboratory with calibrations based on a master instrument. Additional opal

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1 Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the set
glass or ceramic standards (to be re-evaluated at least yearly), which have brightness values within 5 brightness units of each sample to be measured, shall be available upon request.

5.3  Sample (plaque) preparation apparatus as described in the Appendices.

6.  Calibration

6.1  Obtain a set of 5 paper and 2 opal glass standards at monthly intervals and as soon as they are received, calibrate the test instrument to the assigned value of the mid-range tab. If the instrument readings of the other standards differ from their assigned value by more than ± 0.3 point, adjust the instrument in accordance with the manufacturer's instructions so that the readings agree with the assigned values to within 0.3 point.

6.2  A frequent calibration check should be made with the opal glass or ceramic standard which is within 5 brightness units of the sample to be measured. The frequency of these checks will depend on the amount of use of the instrument and the accuracy required. Clean the opal glass or ceramic standards frequently (at least weekly) with the appropriate solution and dry with lint-free tissue.

NOTE 2:  To clean opal glass standards use a solution of 0.3% nitric acid, 95% ethyl alcohol (190 proof) and 4.7% distilled water. To clean ceramic standards, use window cleaner with ammonia.

NOTE 3:  Opal glass or ceramic standards should not be left over the instrument opening as it may harm the standard.

7.  Sampling and preparation of test specimens

7.1  From each test unit obtained in accordance with TAPPI T 657 “Sampling of Fillers and Pigments,” take a specimen of dry clay or pigment sufficient for the test.

7.2  In the event that the test unit is in a dispersed aqueous slurry form, determine the percent solids and remove a slurry equivalent to 100 g of dry pigment.

7.3  Prepare plaques (3 are recommended) by the procedure outlined in the Appendices. Leave the plaques in place as prepared until immediately prior to testing. They should be protected from circulating air, ultraviolet radiation, high humidity, dust, or other contamination which might affect the optical reflectance.
8. Measurement

8.1 When ready to test, “break” the plaque free of the plate. Lift the plaque cylinder vertically to prevent “burnishing” of the plaque surface, which will result if the cylinder is twisted or moved across the glass plate. Visually inspect the plaque surface to detect surface irregularities, contamination, or other flaws in the surface to be tested. Should any such be noted, discard the plaque and prepare another.

NOTE 4: Visual inspection and measurement should be made within 10 s of breaking the plaque free to minimize the effects of rapidly diminishing brightness when exposed to room air.

8.2 Carefully place each prepared test plaque into position on the reflectance instrument and measure its reflectance with reference to that of a calibrated working standard in accordance with the operating instructions supplied with the instrument.

9. Report

Report the average brightness of the plaques to one decimal place. Indicate that the measurements were obtained in accordance with TAPPI T 646. The report must also include the type of reflectance instrument used for the measurement and any deviations from TAPPI T 646.

10. Precision

10.1 The following data are taken from Reports 137 through 142 of the Paper and Paperboard Collaborative Reference Program conducted by Collaborative Testing Services, Inc. The data were modified to include only the 7 -10 laboratories that used the Anglo type pulverizer, as specified in this method. All samples were anhydrous, spray dried kaolin clays and ranged in brightness from 86 to 91 percent (measured in accordance with TAPPI T 452). Five test determinations were made per sample. Samples A1, A2 and A3 were from the same batch of clay; samples B1, B2 and B3 were from the same batch of clay etc.

10.2 The terms repeatability and reproducibility are used as defined in TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility.”
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**T 646 om-11**

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10.3 The user of these precision data is advised that it is based on actual mill testing, laboratory testing, or both. There is no knowledge of the exact degree to which personnel skills or equipment were optimized during its generation. The precision quoted provides an estimate of typical variation in test results which may be encountered when this method is routinely used by two or more parties.

11. Keywords

Brightness, Reflectance, Clay, Minerals, Pigment

12. Additional Information

12.1 Effective date of issue: to be assigned.

12.2 This method is intended to be used in conjunction with TAPPI T 452.

12.3 This method differs from TAPPI T 534 in that 45°/0° directional geometry is employed instead of d/0° diffuse geometry.
Appendix A. Preparation of kaolin clay powder plaques for reflectometry

A.1 Scope
A.1.1 This appendix describes a procedure for preparation of pressed white high reflectance plaques of specimens of kaolin clay to be used for reflectance measurements.
A.1.2 When samples of production runs are prepared for measurement, the samples should be obtained according to TAPPI T 657 “Sampling of Fillers and Pigments.”
A.1.3 There is no guarantee that plaques prepared by this method of preparation will be uniformly reflective even when new.

A.2 Apparatus
A.2.1 Apparatus required for preparation of dry specimens
A.2.1.1 Pulverizer
A.2.1.1.1 A removable specimen holding cup made of stainless steel, which has a 145 cc capacity. The pulverizing cavity (Fig. 2) shall be cylindrical in nature, having an 80 mm diameter and a 29 mm depth. The intersection of the bottom plane and the walls should be rounded so as to eliminate a crevice in which clay could collect. The top of the cavity should be sufficiently flat to eliminate clay build up while pulverizing. It should also be well sealed so as to eliminate the possibility of clay escaping or contaminants entering the cavity.
A.2.1.1.2 Three blades (Figs. 3-6) shall be configured as described below. Two flat blades are placed parallel to one another. They are to be soldered together at each end using a 0.68-in. long strip of blade material. The third blade, which is not flat but curved at each end, is oriented perpendicular to the first two attached blades. The blades and a spacer are located on a threaded driveshaft.
A.2.1.1.3 The pulverizer motor should rotate the blades at 25,000 rpm, thus generating a blade tip speed of 93 m/s (305 ft/s).
A.2.1.1.4 All parts of the pulverizer, with the exception of the blades, that come in contact with the clay are to be made of alloy 316 stainless steel. The blades are to be made of tempered stainless steel. Solder joints on the blade shall be silver soldered using cadmium free solder.
A.2.1.2 Plaque press
A.2.1.2.1 Cylinder, of brass, stainless steel, or other inflexible, non-reactive material, at least 25 mm (1 in.) long, with an inside diameter of a minimum of 19 mm (0.75 in.) with ends faced smooth and square to the long axis. The area of the cross section should be calculated from the inside diameter and recorded.
A.2.1.2.2 Plunger, metal, with flat, smooth end, with a diameter 1.5 mm (1/16 in.) smaller than the inside diameter of the cylinder and at least 25 mm (1 in.) long.
A.2.1.2.3 Press, such as an arbor press drill stand, or other type of press which applies pressure that is exactly vertical. It should have a chuck or similar device to securely hold the plunger and have clearance enough to accommodate the platform scale, glass plate, cylinder, and plunger.
A.2.1.3 Plate glass, about 150 mm (about 6 in.) square and 6-9 mm (1/4 - 3/8 in.) thick with a smooth, unscratched surface.
A.2.1.4 *Platform scale*, bathroom or other type, constructed so that the dial can be read from above and having a rigid platform that will not deflect under the pressure required.

Fig. 1. Pulverizer.
Fig. 2. Pulverizing cavity.

Fig. 3. Top view of flat blade.
Fig. 4. Side view of curved blade.

Fig. 5. Side view of flat blades assembled.
A.2.2 Additional apparatus required for preparation of wet specimen

A.2.2.1 Suction flask, 1500 mL or larger.

A.2.2.2 Buchner funnel, 150 mm diameter.

A.2.2.3 Filter paper, Whatman No. 5 or equivalent.

A.2.2.4 Drying dishes, shallow, easily cleaned container, 200-250 mm (8-10 in.) in diameter, made of corrosion-resistant material; for example, a 203 mm (8 in.) watch glass.

A.2.2.5 Drying oven, convection oven thermostatically controlled at 105° ± 3°C or microwave oven (600-700 watt) set to high.

A.3 Materials

A.3.1 If a wet specimen is being prepared, sulfuric acid (approximately 10% solution of H₂SO₄), or alum [approximately 25% solution of Al₂(SO₄)₃ • 14H₂O], iron-free, are required.

A.4 Sampling

A.4.1 From each test unit obtained in accordance with TAPPI T 657 “Sampling of Fillers and Pigments,” take a specimen of dry clay or pigment sufficient for the test.

A.4.2 In the event that the test specimen is in a dispersed aqueous slurry form, determine the percent solids and remove slurry equivalent to 100 g of dry pigment. Dilute the slurry to about 20% solids with distilled water and coagulate it with sulfuric acid or iron-free alum to a pH of about 3.0-3.5 if sulfuric acid is used or 4.0-4.5 if alum is used. The slurry sample should then be vacuum-filtered in a thin film on a 150-mm Büchner funnel to remove the excess water and dried as indicated in A.5.1.
A.5 **Procedure**

A.5.1 **Specimen preparation.** Spread out the specimen in a thin layer on the drying dish and dry in a convection oven or microwave to less than 1% moisture content. Caution; do not overdry. It is important to pulverize soon after drying to minimize reabsorption of moisture. If stored, the moisture content should be rechecked to be sure it is below 1% before pulverizing. Pulverize a 10-12 g sample for 30 s in the pulverizer described in A.2.1.1.

**NOTE A.1:** Caution: the pulverizer cup will get very hot. Handle with insulated gloves. There is, however, no evidence that heat causes brightness degradation.

A.5.2 **Plaque preparation**

A.5.2.1 Position the scale under the plunger press with the glass plate and cylinder(s) positioned to allow the plunger to vertically enter the cylinder. Before pressing, carefully position each cylinder in the center of the scale (off center pressing may result in measurement errors).

A.5.2.2 Pour the pulverized specimen into the cylinder to a depth of about 12.5 mm (about 0.5 in.) and then lower the plunger, while holding the cylinder firmly against the glass surface, being careful to let it settle slowly to avoid blowing finely pulverized material from the cylinder.

A.5.2.3 Continue depressing the plunger with gradually increasing pressure, until 30 psi or 210 kPa is obtained and hold at this pressure for 5 s. Release the pressure slowly over a period of about 5 s and withdraw the plunger.

A.5.2.4 Prepare any additional plaques by the same procedure.

**Appendix B. Preparation of calcined kaolin powder plaques for reflectometry**

B.1 *The method described in Appendix A should be used with the exceptions hereby specified.*

B.1.1 For dry pulverized calcined kaolin, further pulverization is not recommended. For calcined kaolin slurry, prepare as in A.4.2 and A.5.1.

**Appendix C. Preparation of CaCO₃ plaques for reflectometry**

C.1 *The method described in Appendix A should be used with the exceptions hereby specified.*

C.1.1 With reference to A.4.2, calcium carbonate slurry does not have to be coagulated with sulfuric acid or alum prior to filtering. Whatman 40 filter paper has been found to be acceptable in filtering precipitated, ground, or fine ground calcium carbonate.

C.1.2 With reference to A.5.1, precipitated, ground, or fine ground calcium carbonate will not yellow if dried to a “bone dry” condition of 105°C, therefore, overdrying is not a concern.
Appendix D. Preparation of TiO$_2$ plaques for reflectometry

D.1 The method described in Appendix A should be used with the exceptions hereby specified.

D.1.1 For A.5.1, use 22 g TiO$_2$ in place of recommended 10-12 g.

Appendix E. Preparation of alumina trihydrate plaques for reflectometry

E.1 The method described in Appendix A should be used with the exceptions hereby specified.

E.1.1 None

Appendix F. Preparation of structured pigment plaques for reflectometry

F.1 The method described in Appendix A should be used with the exceptions hereby specified.

F.1.1 None

Appendix G. Preparation of other pigments for reflectometry

G.1 For mineral pigments not specifically covered in this method, contact the pigment manufacturers for procedural information.

References

1. TAPPI Official Test Method TAPPI T 452 “Brightness of Pulp, Paper and Paperboard (Directional Reflectance at 457 nm).”


Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.