



Standard Test Method for Assessing the Color Strength and Dispersibility of Alkali Blue Pigment in Hot Melt Carbon Copy Paper Ink¹

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1. Scope

1.1 This test method provides a procedure to determine the color strength of dry or flushed alkali blue pigment in hot melt carbon copy paper ink compared to an alkali blue control mutually agreed upon by the purchaser and supplier.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 9.1 and 9.2.1.

2. Referenced Documents

2.1 ASTM Standards:

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems²

D 3460 Specification for White Watermarked and Unwatermarked Bond, Mimeograph, Duplicator, and Xerographic Cut-Sized Office Papers³

F 129 Test Method for Amount of Ink Deposit on Carbon Paper and Inked Ribbons, Other Than Fabric Type³

F 149 Terminology Relating to Optical Character Recognition³

F 221 Terminology Relating to Carbon Paper and Inked Ribbon Products and Images Made Therefrom³

F 497 Practice for the Use of the Electric and Electronic Typewriter as a Test Instrument³

F 597 Practice for the Evaluation of One-Time Carbon Paper in Carbon-Interleaved Business Forms by Use of an Electric Typewriter³

2.2 ANSI Standards:⁴

PH2.17 Density Measurements—Geometric Conditions for Reflection Density

PH2.18 Density Measurements—Spectral Conditions

3. Terminology

3.1 Definitions:

3.1.1 *steel scraper, n*—also referred to as a drawdown knife (see Fig. 1).

3.1.2 *drawdown, n*—a film of ink deposited on paper by a smooth edged blade to evaluate the characteristics of the ink (see Fig. 2).

4. Summary of Test Method

4.1 Samples of the control and the test material(s) are dispersed in a hot melt carbon paper ink, utilizing a laboratory batch-type heated shot mill apparatus or a heated ball mill. Resultant inks produced are compared by making drawdowns on grease proof translucent paper or by coating on carbonizing tissue in a proper and reasonable coating weight range. These are evaluated visually, with a densitometer or by use of the electric typewriter as a test instrument.

5. Significance and Use

5.1 This test method is intended to provide a means of evaluating the comparative color strength and dispersibility of alkali blue dry pigment or flushed color in hot melt carbon paper inks.

6. Interferences

6.1 Temperature limits must be maintained during dispersion and test sample preparation for reproducibility of test results.

6.2 Dispersion time must be carefully timed to a dispersion level agreed upon between supplier and consumer. For guidance, the following grind levels on the fineness of grind gage may be considered sufficient:

Grind Level	Gage Scale
5.5 to 6.0	Hegman (North Standard)
7.0 to 8.0	Production Club (PC)
1.25 to 0.75	Mils (0.001 in.)
32.0 to 25.0	Micrometres
12.5 to 7.5	NPIRI (2 mil gage)

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² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 15.09.

⁴ Available from American National Standards Institute, 25 W. 43rd St., 4th Flr., New York, NY 10036.



FIG. 1 Drawdown Knife



FIG. 2 Drawdown

6.3 In making the drawdowns for visual or densitometer assessments, or both, care must be exercised in holding the metal scraper in the same position each time and applying the same pressure. The control must always be included on each draw down.

6.4 Color hue differences between control and test material can introduce errors in visual strength estimates.

7. Apparatus

7.1 *Drill Press*, or similar drive capable of turning a $\frac{5}{16}$ -in. (8.0-mm) shaft at variable speeds with a maximum constant speed of 2000 r/min. Should have provisions for securing a 600-mL heating mantle beneath the drive mechanism.

7.2 *Three-Blade Stirring Propeller*, 2-in. (51.0-mm) diameter mounted on 12 by $\frac{5}{16}$ -in. (305 by 8.0-mm) shaft, stainless steel.

7.3 *Heating Mantle*, 600-mL.

7.4 *Autotransformer*, voltage range 0 to 120 V or other equivalent voltage controller.

7.5 *Metal Cans*, $\frac{1}{4}$ -pt (118-mL), 1-pt (473-mL), 1-qt (946-mL) and 1-gal (3.8-L), unlined with lids.

7.6 *Lids*, for pint cans with $\frac{5}{16}$ -in. (8.0-mm) hole in center (for use with shot mill).

7.7 *Stainless Steel Shot*, diameter size as mutually agreed upon by supplier and manufacturer.

7.8 *Strainer*, with mesh smaller than diameter of steel shot used.

7.9 *Ring Stand*, to support strainer.

7.10 *Metal Stem Thermometer*, dial-type 5° divisions, 0 to 300°F (2° divisions, - 22 to 149°C).

7.11 *Two Surface Thermometers*, dial, 5° divisions, 0 to 300°F (2° divisions, - 22 to 149°C).

7.12 *Laboratory Ball Mill*, having the following equipment:

7.12.1 *Jacket*, connected to a steam or hot water supply maintained at a specified temperature, $\pm 5^\circ\text{F}$ ($\pm 3^\circ\text{C}$). This specified temperature should be in the range of 190 to 235°F (88–113°C).

7.12.2 *Abrasion-Resistant Interior*.

7.12.3 *Vent Plug*.

7.12.4 *Grinding Cover and a Slotted Discharge Cover*, or a grinding cover with a discharge valve.

7.12.5 *Steel or Iron Balls*—Charge of $\frac{1}{4}$ to $\frac{5}{8}$ -in. (6 to 16-mm) steel or iron balls occupying 50 % of the mill's interior volume. Different ball sizes, a mixture of balls of different sizes, and different ball charge levels can only be used when agreed upon by the purchaser and supplier.

7.12.6 *Motor Drive*, with provision for mill speed adjustment so that mill can be set at its critical speed.

7.13 *Laboratory Oven*, capable of maintaining a constant temperature comparable to the ball mill operating temperature, $\pm 5^\circ\text{F}$ ($\pm 3^\circ\text{C}$).

7.14 *Stop Watch or Timer*.

7.15 *Gloves*, insulated heat resistant.

7.16 *Safety Glasses*.

7.17 *Safety Solvent Storage Cans*.

7.18 *Hot Plates*, two, capable of maintaining $205^\circ \pm 5^\circ\text{F}$ ($96^\circ \pm 3^\circ\text{C}$), with 10 by 10-in. (254 by 254-mm) top surfaces.

7.19 *Spatula*, 6-in. blade.

7.20 *Steel Scraper*, also referred to as a drawdown knife (see Fig. 1). This has a stiff blade, 4 in. (102 mm) at the bottom with a handle at the top. The edge should be honed to

smoothness with emery paper to produce a smooth, level contact surface. Similar in design to a putty knife.⁵

7.21 *Suitable Laboratory Coater.*

7.22 *Reflection Densitometer,*⁶ with spectral characteristics in accordance with ANSI PH2.18, capable of measuring reflection density in accordance with ANSI PH2.17.

7.23 Fineness of grind gage with any of the following scales: Hegman (North Standard), Production Club (PC), National Printing Ink Research Institute (NPIRI), Mills or Micrometers. A heated, thermostatically controlled gage may also be used.

7.24 *Weight,* 1000 g.

7.25 *Balance,* with a 0.001-g sensitivity at 50.000-g load, for weighing pigment or flushed color.

7.26 *Balance,* with 0.1-g sensitivity at 200-g load and capacity of 750.0 g for other weighings.

8. Materials

8.1 *Ingredients for Wax-Oil Carbon Paper Ink Composition,* without alkali blue, as agreed upon by the purchaser and supplier.

8.2 *Alkali Blue*—Control alkali blue dry pigment and alkali blue dry pigment to be tested, or control flushed alkali blue and flushed alkali blue to be tested.

8.3 *Grease Proof Translucent Paper,* 25-lb weight (17 by 22-in.; 500 sheets) (94.00 g/m²).

8.4 *Bond Paper,* Grade 4, Type I (Specification D 3460), 12 to 15 lb weight (17 by 22-in.; 500 sheets) (45.12 to 56.40 g/m²).

8.5 *Carbonizing Tissue,* having a basic weight of 7.0 to 9.0 lb (20 by 30-in.; 500 sheets) (16.38 to 21.06 g/m²)

8.6 *Mill Cleaning Materials,* such as ink oil, paraffin, slack wax, or other vehicles used in the ink formulation.

8.7 *Cleaning Solvents*—As selected using the nonmandatory information in the appendix of Test Method F 129.

8.8 *Cleaning Cloths.*

9. Procedures

9.1 *Laboratory Shot Mill Method:*

9.1.1 **Warning**—Protective gloves should be worn when handling hot apparatus. Safety glasses should be worn during this procedure.

9.1.2 Insert a 1-pt (473-mL) unlined metal can into the 600-mL heating mantle. Clamp the heating mantle with can, beneath the stirring drive mechanism. Place shaft and stirrer in the can and secure shaft to the drive mechanism. Position the stirrer ½-in. (13 mm) from the bottom of the can.

9.1.3 Determine the weights of the ingredients of the carbon paper ink needed to provide a total batch weight of 300 g. The composition of the carbon paper ink and the percent of alkali blue used is to be mutually agreed upon by the purchaser and supplier.

9.1.4 Weigh up the required amounts of all the ingredients except the alkali blue, which is weighed up separately on the more sensitive balance.

9.1.5 Add 750.0 g of steel shot and all the ingredients except the alkali blue to the can.

9.1.6 Connect the auto transformer to the heating mantle and adjust the temperature of the contents to 205 ± 5°F (96 ± 3°C).

9.1.7 Start slow agitation and allow the contents of the can to mix until all waxes are melted and mixture appears uniform.

9.1.8 Continue slow agitation and slowly add the alkali blue. Stir 5 min or until pigment is completely mixed and up to temperature.

9.1.9 Disengage the stirrer shaft from the drive mechanism and slip over it a can lid containing a ⅝-in. (8.0-mm) hole in the center. Lower the lid on the shaft and affix tightly to the can. Re-attach the stirrer shaft to the drive mechanism and position stirrer ½-in. (13.0 mm) from the bottom of the can.

9.1.10 Adjust the speed of agitation to 2000 r/min and grind, maintaining temperature of 205 ± 5°F (96 ± 3°C) by adjustment of the heating element, until an agreed upon fineness of grind gage reading is obtained for the ink. Optionally, a specific grinding time, mutually agreed on by the purchaser and supplier may be used. Also optionally, the control may be ground to an agreed upon fineness of grind gage reading and the test material(s) ground the same amount of time. The fineness of grind gage is preheated to 205 ± 5°F (96 ± 3°C) by placing on a hot plate. Temperature of hot plate surface should be checked with the dial surface thermometer (see Test Method D 1210 for fineness of grind test). Alternately, a fineness of grind gage with a built-in heating unit may be used.

9.1.11 When the grinding is complete, support the strainer on the ring stand above a 1-pt (473 mL) can and separate the carbon paper ink from the steel shot, allowing ink to flow into the can.

9.1.12 The steel shot should be cleaned immediately by agitating in a can with hot ink oil.

9.1.13 Repeat 9.1.2 through 9.1.12 for the control or for materials to be tested.

9.2 *Laboratory Ball Mill Grinding Method:*

9.2.1 **Warning**—Protective gloves should be worn when working with hot mill and hot carbon copy ink. Safety glasses should be worn when venting and opening the mill. Mill should be located in well-ventilated area to reduce solvent fumes. Waste solvent from cleaning should be put in an approved safety container.

9.2.2 Check that the ball mill is operating at its correct critical speed. The critical speed, S_c , in r/min is determined by the equation:

$$S_c = (54.19 / \sqrt{R})$$

where:

R = ball mill inside radius in feet.

9.2.3 Preheat the ball mill hatch covers to the mill operating temperature.

⁵ Available as Russell No. 254 or No. 530-4 from Fred L. Brooke Co., Park Forest, IL.

⁶ Among instruments that have been found satisfactory in this purpose are: Model 61, manufactured by the Cosar Corporation, Garland, TX; The GAM model 126P, manufactured by Graphics Arts Mfg. Co., Houston, TX, or the MacBeth Model RD-517 (or other comparable models), manufactured by MacBeth Corp., Newburgh, NY.

9.2.4 Heat the ball mill to the specified temperature by steam or hot water. Check the jacket inlet and outlet temperature. Check the temperature of the mill's contents.

9.2.5 Determine the weights of the ingredients of the carbon paper ink needed to provide a total volume equal to 25 % of the ball mill's capacity. The composition of the ink and the percent of alkali blue used is to be mutually agreed on by the purchaser and supplier.

9.2.6 Weigh the required amounts of all the ingredients except the alkali blue, which is weighed separately on the more sensitive balance.

9.2.7 Add all ingredients except the alkali blue to the ball mill. Secure the hatch cover on the mill. If the cover is equipped with a valve, make sure it is securely closed.

9.2.8 Start mill and run for 15 min to heat the contents to specified temperature. Check the mill's temperature.

9.2.9 Stop the mill with vent plug in the top position and carefully remove the plug. Check vent for blockage if no pressure is observed. Replace vent plug and manually rotate the mill so that the hatch is in the top position. Open hatch and add the alkali blue. Close and secure the hatch.

9.2.10 Start mill and grind until the agreed-upon fineness of grind gage reading is obtained for the ink. Optionally, a specific grinding time, mutually agreed on by the purchaser and supplier may be used. Also optionally, the control may be ground to an agreed upon fineness of grind gage reading and the sample(s) under test ground the same amount of time.

9.2.11 Stop mill (at end of grind time or for a fineness of grind test) and open as in 9.2.9. The fineness of grind gage is preheated to $205 \pm 5^\circ\text{F}$ ($96 \pm 3^\circ\text{C}$) by placing on the hot plate. The temperature of the hot plate surface should be checked with the dial surface thermometer. (See Test Method D 1210 for fineness of grind test.) If grind is complete, put preheated slotted hatch cover or replace hatch cover if equipped with a discharge valve and drain ink into a 1-pt (476-mL), 1-qt (946-mL) or 1-gal (3.8 L) can depending on the mill size. If grind is not complete, close the mill hatch and repeat 9.2.10 and 9.2.11 until agreed-upon grind level is obtained.

9.2.12 *Cleaning Ball Mill*—The mill should be cleaned by one of the following methods:

9.2.12.1 Two or more rinsings of oil, paraffin, or slack wax.

9.2.12.2 Two or more rinsings of the vehicle used in the test ink.

9.2.12.3 If complete cleanness and dryness is desired, use one or more rinsings with oil, paraffin, or slack wax followed by two rinsings with a cleaning solvent (see 8.7). Thoroughly dry mill by hosing with dry compressed air. Check for evidence of wetness by inserting blade of spatula under the ball charge, removing and examining blade for wetness and solvent odor. Blade should be dry and odor free. When rinsing mill, always vent mill before opening hatch.

9.2.13 Repeat 9.2.3 through 9.2.12 for the control or for materials to be tested.

9.3 *Laboratory Shot Mill or Ball Mill Procedure Using Flushed Alkali Blue Instead of Dry Alkali Blue Pigment:*

9.3.1 Procedures 9.1 or 9.2 can be followed, except that flushed alkali blue can be used instead of alkali blue dry pigment for both the control and the test material. The amount

used will be that necessary to provide a satisfactory level of color strength in the carbon paper ink, as determined by the purchaser. The total ink formulation can be adjusted by removing an amount of vehicle equivalent to that amount contained in the total amount of flushed alkali blue used. Alternately, an ink formulation designed specifically to utilize flushed alkali blue and mutually agreed upon by purchaser and supplier, can be used.

9.4 *Testing of the Hot Melt Carbon Copy Paper Ink:*

9.4.1 *Drawdown Method:*

9.4.1.1 Place the heat proof plate glass on one of the hot plates. Adjust the temperature of the glass surface and the surface temperature of the second hot plate to $205 \pm 5^\circ\text{F}$ ($96 \pm 3^\circ\text{C}$), monitoring with the surface thermometers.

9.4.1.2 Place samples of the inks from 9.1, 9.2, or 9.3 in $\frac{1}{4}$ -pt (118-mL) cans and place on the second hot plate. Heat samples to $205 \pm 5^\circ\text{F}$ ($96 \pm 3^\circ\text{C}$). Check the ink temperature with the metal stem thermometer.

9.4.1.3 Place a 6 by 8-in. (152 by 203-mm) piece of translucent grease proof paper on the plate glass of the first hot plate and place a 1000 g weight at the top to secure. Place a small amount of the ink containing the control near the top of the paper and place a similar amount of the ink containing the alkali blue test material in juxtaposition, approximately $\frac{1}{2}$ in. (13 mm) apart. Place the metal scraper above the inks. Holding the blade at a 15° angle from the vertical, pull down quickly from top to bottom, using uniform pressure. This procedure provides two touching adjacent films of inks of practical uniform thickness (known as a drawdown).

9.4.1.4 *Visual Evaluation of Drawdowns*—Hold the drawdown approximately 12 in. (305 mm) from a light source. Optionally, a light box may be used. By means of the transmitted light, compare the color strength of the ink containing the test material to that of the control. Note color strength differences. Repeat test, if desired, making pigment content adjustments.

9.4.1.5 *Optional Densitometer Evaluation of Drawdowns*—Back the drawdowns with $\frac{1}{4}$ -in. (6.35-mm) pad of bond paper. Adjust the densitometer on the bond paper to a zero reading before measuring the reflection density. Make all reflection density measurements with the bond paper behind the drawdown. Select a circular area, $\frac{1}{2}$ in. (13 mm) in diameter, in the center of each drawdown film. Using the visual response filter, take multiple measurements within the circular area and average. Individual measurements should be within 0.06 reflection density units. For color strength assessment, the average reflection density produced by the test material is compared to the control.

9.4.2 *Optional Image Quality Evaluation Using the Electric Typewriter as a Test Instrument*—If desired, the carbon copy paper inks containing the alkali blue control and test material(s) may be coated on carbonizing tissue utilizing a suitable laboratory coater. Coating weight on carbonizing tissue is to be controlled within ± 0.1 lb per 500, 20 by 30-in. sheets. (± 0.24 g/m²). The coated copy papers thus produced may now be evaluated according to Practice F 497, or Practice F 597.

10. Calculation

10.1 None required unless a quantitative value of the color strength of the test sample compared to the control is desired. In which case, the number of parts by weight of the test alkali blue sample to equal the strength of 100 parts by weight of the alkali blue control is determined from the pigment loading adjustment (9.4.1.4). Control of test sample:

$$= 100 \times \frac{\text{g test sample}}{\text{g control}}$$

11. Reporting

11.1 From choices of the evaluation procedures for the carbon copy paper ink, report the (I) estimated visual color

strength differences, or (2) the differences in reflection density (densitometer), or the quantitative pigment strength difference calculated in Section 10, and confirmed by (1) or (2).

12. Precision and Bias

12.1 Interlaboratory agreement has been found to be $\pm 10\%$ and intralaboratory $\pm 5\%$.

13. Keywords

13.1 alkali blue; carbon paper ink; color strength; dispersibility

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