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Practice for Evaluation of Vehicles for Pigment Wetting Using a Vacuum Modified Sigma Blade Mixer[1]

This standard is issued under the fixed designation D 6336; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers guidelines for the evaluations of vehicles for pigment dispersion using a vacuum modified sigma blade mixer, or vacuum flusher.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 280 Test Method for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments[2]

D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller[3]

D 1316 Test Method for Fineness of Grind of Printing Inks by the NPIRI Grindometer[4]

D 2066 Test Method for Relative Tinting Strength of Paste Type Printing Dispersion[4]

D 2067 Test Method for Coarse Particles in Printing Ink Dispersions[4]

D 4017 Test Method for Water in Paints and Paint Materials by Karl Fisher Method[3]

D 4040 Test Method for Viscosity of Printing Inks and Vehicles by the Falling Rod Viscometer[4]

D 4361 Test Method for Apparent Tack of Printing Inks and Vehicles by a Three-Roller Tackmeter[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *additives*—various materials that are used in relatively small quantities to condition the pigment or vehicle.

3.1.2 *break*—the action that takes place when water is separated from the pigment in a presscake.

3.1.3 *flushed color*—a color base in paste form prepared by flushing.

3.1.4 *flusher*—a mixing device that has two sigma shaped agitator blades parallel to each other, turning in opposing directions at different speeds.

3.1.4.1 *Discussion*—The mixing action of a flusher is that of kneading.

3.1.5 *flushing*—a method of transferring pigments from dispersions in water to dispersions in oil by the displacement of the water by oil.

3.1.5.1 *Discussion*—The resulting dispersions of flushing are known as flushed colors.

3.1.6 *pigment*—the fine solid particles of colorant used to give color to printing inks.

3.1.6.1 *Discussion*—The pigment particles are substantially insoluble in the vehicle and in water.

3.1.7 *presscake*—a mixture of pigment and water formed into a cake by passing through a filter press under pressure.

3.1.8 *vacuum cycle*—the time a flush is under vacuum to remove entrapped water.

3.1.9 *vehicle*—the liquid portion of an ink that holds and carries the pigment, provides workability and drying properties and binds the pigment to the substrate after the ink has dried.

4. Summary of Practice

4.1 Vehicle, pigment presscake, and additives are added into a sigma blade mixer and mixed until the water is displaced from the pigment presscake.

4.2 Step 4.1 is repeated two or three times until the capacity of the flusher has been reached.

4.3 The flusher is then sealed and a vacuum applied until the dispersion (flush) is free of moisture.

NOTE 1—Lithol rubine pigment undergoes a color conversion when essentially all water is removed.

4.4 Vehicle solvent and additives are added to adjust the strength, shade and body of the dispersion (flush) to that of a standard dispersion (flush).

5. Significance and Use

5.1 By following this practice it is possible to make reproducible flushes when using the same raw materials. Therefore, if someone wishes to evaluate the effect a different raw material has on a flush, it is possible to evaluate this effect by

[1] This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coating Materials, and Applications and is the direct responsibility of Subcommittee D01.37 on Ink Vehicles.

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

noting the change that occurs from a control flush to the experimental flush. This change can be, but is not limited to; such things as strength after vacuum, grind, grit, gloss etc. This practice can be used by ink companies, pigment companies or varnish companies. This practice is not meant to give absolute values but is meant to be used as a relative practice in which a control flush is made using a standard formula and the experimental flush is compared to the control flush. This practice is not meant to determine the absolute performance of a formula in production. Again it can be used to give a relative idea of how a formula will perform in production when a correlation has been established between laboratory flushing and production flushing.

6. Apparatus

6.1 *Laboratory Sigma Blade Flusher*, typically 1-L(1-qt) to 4-L(1-gal) capacity.

6.2 *Vacuum Pump*, capable of obtaining a vacuum in the flusher of 69 cm (27 in.) to 76 cm (30 in.).

6.3 *Wide Blade Spatula*, typically 5 by 10 cm (2 by 4 in.).

6.4 *Spatula*, typically 2.5 by 7.5 cm (1 by 3 in.).

6.5 *Scale*, capable of weighing up to 3 kg accurate to 1 g.

6.6 *Scale*, capable of weighing up to 1 kg, accurate to 1 g.

7. Materials

7.1 *Presscake*.

7.2 *Flushing Vehicle(s)*.

7.3 *Experimental Vehicle*.

7.4 *Flushing Additives*.

8. Procedure

8.1 Fig. 1 illustrates a typical formula for a 1-L laboratory flusher.

NOTE 2—It is common practice for formulas to be based on the amount of pigment calculated on a dry basis and not on the weight of presscake, since the amount of water in the presscake will vary from batch to batch. For example, a presscake can be referred to as 25 % dry or 25 % solids. This means that for every 100 kg of presscake there are 25 kg of pigment and 75 kg of water. Usually the entire quantity of presscake to be flushed will not fit in the flusher at one time. If this is the case, it is necessary to flush the required amount of pigment in a succession of breaks (see Fig. 1).

NOTE 3—Many formulas call for two or more kinds of oil or varnish or resin solution etc. Directions are usually very specific as to how much should be used, when the various items should be added, and the order in which they are added. It is normal practice to add these items in the same order as shown on the formula. The vehicle having the best pigment wetting property is usually added first. In some formulas, however, judgment is left to the operator, as predictions cannot be made.

NOTE 4—Flushing aids are very effective and should be used with care and good judgment.

8.2 *First Break*:

8.2.1 Add prescribed quantity of presscake to the flusher.

8.2.1.1 The presscake should be analyzed for dry weight or solids according to Test Method D 280.

8.2.1.2 All presscake should be weighed before it is charged to the flusher.

8.2.2 Agitate for 2 to 5 min. If using a multispeed flusher, agitate at low speed for 1 to 2 min then at high speed for 2 to 5 min.

8.2.3 Add flushing additive(s) if required.

8.2.4 Add vehicle in small quantities until break occurs. Remember you can always put vehicle in but never take it out. Therefore, always work the vehicle into the presscake well before adding more.

8.2.5 When the flush mass begins to form (water is flushed out) continue to mix until the water clears, then drain.

8.2.6 Run the flusher on high speed for 30 to 45 s and drain again. Do this until no more water is flushed from the system.

NOTE 5—First breaks generally use 45 to 55 % of the pigment and 55 to 60 % of the vehicle. First breaks are usually soft so it is very important to drain well. Also it is important that the mixer's sides should be scraped down thoroughly during the first break and all subsequent breaks, to make sure all pigment and presscake are mixed in properly. Proper draining will shorten the vacuum cycle.

8.3 *Other Breaks*:

8.3.1 Charge the required amount of presscake and mix thoroughly. No vehicle is to be added until the presscake charged has been mixed in well. The flush should have a dry appearance before any vehicle is added.

8.3.2 When the charged presscake has been thoroughly mixed, add the vehicle in small amounts until the break occurs. Use the minimum amount of vehicle to obtain the break.

8.3.3 After the break occurs run the batch with a stiff body (high viscosity) for 10 to 15 min. When the water clears drain once or twice.

8.3.4 Repeat 8.3.1-8.3.3 until all the required amount of presscake has been added.

NOTE 6—Typically subsequent breaks will take about 20 to 25 % pigment per break. The second break will take more pigment than the third break and, if needed, the fourth break will take less pigment than the third break etc.

NOTE 7—Typically the second break will take about 20 to 25 % of the vehicle and the third and all subsequent breaks will take little or no vehicle.

8.4 *Reduction*:

8.4.1 Add vehicle or solvent, or both, to the flush until the flush is just soft enough to mix in the flusher. If the flush tends to ride above the sigma blades of the flusher, the flush is too heavy. If the flush appears too soft when mixing, it is too low in viscosity.

8.4.2 Drain any water that appears during the reduction of the batch.

NOTE 8—Reduction is the reducing of the body of the batch so it mixes properly under vacuum. A poor reduction can affect the grind and strength when the batch is held under vacuum. It is not always necessary to reduce a batch if it mixes properly under vacuum.

8.5 *Vacuum*:

8.5.1 Seal the flusher so it is airtight, preferably in such a way the flush can be observed while mixing under vacuum.

8.5.2 Apply vacuum until 60 to 70 cm of mercury is obtained. The higher the vacuum the better, but make sure that 635 mm of mercury is the minimum used.

NOTE 9—The dryness of a flush can usually be determined by the lack of condensation on the walls and cover inside the flusher. If there is any doubt about the dryness of a batch, determine the moisture content in accordance with Test Method D 4017. For a flush to be considered dry there should be less than 2 % water remaining.

DEVELOPMENT FLUSHING FORM

Date:

Project No.

Product Code Number 9999
Presscake Used zyxw
Percent Solids 28.5%

I. Flushing Cycle	Amount used(a)	Break 1	Break 2	Break 3	Break 4
Presscake No. zyxw	160.0g	77.0g	37.0g	25.5g	20.5g
Vehicle No 1	35.0g	35.0g			
Vehicle No. 2	90.0g	60.5g	29.5g		
Vehicle No					
Additive No 1	5.0g	5.0g			
Additive No 2	.8g	.8g			
Solvent No 1	23.0g				
Total	308.0g(b)				

II. Reduction Cycle	
Vehicle No.	
Vehicle No.,	
Additive No 3	9.0g
Additive No	
Solvent No 1	1.0g
Solvent No	
Total	10.0g

III. Adjustments	
Vehicle No. 2	44.0g
Vehicle No	
Solvent No 1	20.0g
Solvent No	
Total	64.0g

TOTAL FORMULA 382.0g

By:

(a) These weights are calculated on the dry weight of the presscake.(pigment weight)
(b) For this formula, it is assumed that additives #1 and #2 are washed out of the system with the water.

FIG. 1 Development Flushing Form

8.6 *Standardization*:

8.6.1 After the flush is thoroughly dried, take a sample and determine the strength in accordance with Test Method D 387.

8.6.2 Based on the after vacuum strength, blend the flush so it is comparable to the control for strength, viscosity, and tack. Determine strength in accordance with Test Method D 2066, viscosity in accordance with Test Method D 4040, and tack in accordance with Test Method D 4361.

NOTE 10—It is suggested that a small sample of the batch be blended to

determine the proper combination of vehicles or solvent, or both, needed to achieve a comparable product to the control. Then use this formula to blend the batch in the flusher. The formula of the experimental flush should be as close to the formula of the control as possible.

9. Report

9.1 Report the following information:

9.1.1 *After Vacuum Strength*:

9.1.1.1 In accordance with Test Method D 2066, determine the strength of the flush after the vacuum cycle has been completed. Report the after vacuum strength of the control flush and the after vacuum strength of the experimental flush. Report the difference of these two flushes by subtracting the after vacuum strength of the control flush from the after vacuum strength of the experimental flush. This is the relative after vacuum strength.

9.1.1.2 Calculate the true strength of the experimental flush as follows:

$$\text{True Strength} = \text{percent strength experimental flush} \times \frac{\text{percent pigment in control}}{\text{percent pigment in experimental flush}} \quad (1)$$

NOTE 11—The use of 9.2-9.5 is left to the discretion of the operator. Other tests may be substituted for these tests if the operator feels they are more appropriate.

9.2 *Grind*:

9.2.1 Determine the grind of the standardized control flush and the grind of the experimental flush in accordance with Test Method D 1316. Report both grinds.

9.3 *Grit*:

9.3.1 Determine the grit of the standardized control flush and the standardized experimental flush in accordance with Test Method D 2067. Report both grit results.

9.4 *Tack*:

9.4.1 Determine the tack of the standardized control flush and the standardized experimental flush in accordance with Test Method D 4361. Report both tack readings.

9.5 *Viscosity*:

9.5.1 Determine the viscosity of the standardized control flush and the standardized experimental flush in accordance with Test Method D 4040. Report both viscosities.

NOTE 12—Comparing the true strength of the experimental flush gives an indication of the wetting efficiency of the experimental flush. If the experimental flush is stronger than the control, the formula change caused better pigment wetting. If the experimental flush is weaker than the control, the formula did not wet the pigment as well.

10. Keywords

10.1 dispersion; flush; flusher; pigment; presscake; sigma blade; vehicle