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ANALYSIS FOR PIGMENT VOLUME CONCENTRATION

BY

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REPRINTED FROM
OFFICIAL DIGEST, VOL. 34, NO. 455, DECEMBER 1962, P.1343 - 1354

RESEARCH PAPER NO. 177
OF THE
DIVISION OF BUILDING RESEARCH

OTTAWA

Analysis

For Pigment Volume
Concentration

By H. E. ASHTON*

The analytical procedure for measuring pigment volume con-
centration has been studied to improve the accuracy of the method.
Clear vehicles were tested first and included bodied linseed oil and
alkyd resins. The specific gravity procedure and the effects of driers
and anti-oxidant on the vehicle volume were examined. It was
found possible to determine the volume of vehicle with an ac-
curacy close to 100%. Pigmented samples were made in enclosed
mills to ensure known composition. Factors studied were measure-
ment of pigment volume, effect of specific gravity and particle size
of one and two pigments, range of PVC and composition of extrac-
tion solvent. To obtain the correct pigment volume it was necessary
to measure the specific gravity and weight of the pigment. The
final PVC's were about 0.4% high and the standard deviation be-
tween duplicates was 0.13%. The main source of error was in the
pigment volume determination and use of a more accurate density
method should eliminate the tendency to slightly high results.

INTRODUCTION

The introduction of the concept of critical pigment volume concen-
tration (CPVC) by Asbeck and Van Loo was an important step towards
more scientific formulation of pigmented coatings.[1] Since then several
papers have been published on different aspects of using CPVC,[2,3,4,5]
but they have been directed toward formulators because the use of PVC
can often reduce the time required to develop a new product.

There are times, however, when the analyst is interested in PVC.
This usually occurs when a specification authority wishes to use a PVC

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requirement and a method for its determination is therefore required. The work now reported resulted from such an occasion.

Several industrial members of the Committee on Paints of the Canadian Government Specifications Board had suggested that in some specifications pigment volume concentration would be more meaningful than the old requirements for pigment content by weight. The question of a suitable procedure was referred to the Test Methods Subcommittee; they decided to use the method given in the U. S. Federal Standard[6] as a starting point.

In this method the volume of total solids is determined on one sample by evaporating the solvent and weighing the dish and contents in air and in water. Another sample is then centrifuged in a graduated tube and the pigment volume obtained by adding a known volume of solvent and reading the total volume. The accuracy of measuring the solids volume is affected by two factors: the solvent evaporation, which is not generally complete in three hours, and the weighing of the solids and dish in water. In the latter case it is difficult to eliminate all air bubbles, especially those in contact with the rough surface of the dried coating. In addition, the specific gravity of the dish, being much higher than that of the solids, tends to influence the result. These considerations led to the suggestion of measuring the specific gravity of the isolated vehicle. Some members of the subcommittee questioned whether this would be possible and the Paint Laboratory of the Division of Building Research, National Research Council, was asked to investigate the modified procedure.

A program was accordingly undertaken to check the accuracy with which the PVC of known compositions could be determined. The measurement of the volume of clear vehicles was first examined. When a satisfactory procedure was developed, analysis was extended to pigmented materials which contained one, and later two, pigments. Other factors studied were the level of PVC and the specific gravity of the pigments.

TESTS AND RESULTS

General Procedure

Clear solutions of resinous materials were made up in volumetric flasks at a standard temperature of 20°C. Samples were removed by pipette only after the solutions had been kept at 20° for one hour. The specific gravity of the recovered vehicle was measured at the same temperature.

To determine the amount of vehicle solids most of the solvent was removed by distillation from a ground-glass jointed flask immersed in a hot water bath. The concentrated solution was transferred either to a 80-mm evaporating dish or to a 25-ml Hubbard-Carmick pycnometer. The remaining solvent and washings were removed first on the steam bath

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and then in a vacuum oven at 105°C and 26-to 30-in. vacuum. The solvent was considered to be removed when the total weight loss from two consecutive three-hour heating periods was less than 10 mg. The weight and specific gravity of the isolated vehicle were determined to obtain its volume.

Pigmented materials were prepared in a closed mill to eliminate loss of solvent. Samples were weighed into tared centrifuge tubes and centrifuged to separate the pigment and vehicle. The vehicle extract was concentrated and treated as above. The extracted pigment was warmed to remove the ether and dried to constant weight at 105°C. Its weight and volume were then measured. Where different treatments were being compared, samples were run in a random order.

Volume of Vehicle

Clear vehicles were investigated first because it was thought that measuring the volume of vehicle would be the most difficult part of the method. The most accurate way to obtain the vehicle volume is to weigh the vehicle and determine its density, whence volume $=$ weight $\div$ density. The accuracy is chiefly dependent upon the specific gravity procedure. For hard resins the usual manner of suspending a piece from a fine wire is adequate. For viscous vehicles which are prevalent in the paint field, suspension is impossible. Preliminary tests with bodied oils and alkyd resins using full and partially full pycnometers indicated that the procedures then available were not entirely satisfactory. The use of a petroleum thinner of low solvent power, low specific gravity and low volatility in place of water was found to improve the determination. Subsequent work led to a modified specific gravity method which can be used for viscous resins.[7] In the work reported here low odor solvent was used in all cases.

The best way to place the extracted vehicle in the widemouth pycnometer was then studied. Raw castor oil of predetermined specific gravity was weighed into a volumetric flask, dissolved in mineral spirits

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and benzene, and aliquots taken for different treatments. Some of the tests were also carried out on a bodied linseed oil and an alkyd resin. Evaporation of the solvent from an evaporation dish followed by transferring as much vehicle as possible to the pycnometer was found to be the fastest method. This method was also slightly more accurate in most cases than placing the concentrated vehicle in the pycnometer and distilling off the remaining solvent through a modified three-way head. The pycnometer method was slower and less accurate because it was difficult to transfer all the vehicle to the small pycnometer, while it was relatively simple to wash the sample into an evaporating dish.

Coatings contain driers and a practical analytical scheme should not be affected by their presence. The effect of driers on the volume measurement of the vehicle was therefore one of the first factors investigated. Concentrated solutions of a bodied linseed oil and of a solventless alkyd resin were made and aliquots taken for further dilution as shown in *Table* 1. The effects of the driers and of the procedure for evaporating the solvent are shown in *Table* 2.

The driers did not affect the bodied linseed oil result but did cause slightly high results with the more viscous alkyd resin. This probably occurred because the alkyd tended to skin over during the time required to remove the last traces of solvent.

Table 1—Vehicle Solutions With and Without Driers

Material	Sample 2 Bodied Linseed Oil	Sample 3 100% Alkyd Resin (Rezyl® 869)
Viscosity (reported)	Z-2	Z-6
Sp. gravity (determined)	0.9658	1.0706
Weight, g.	96.5017	107.0001
First dilution	to 250 ml with 100 ml mineral spirits and 50 ml benzene	to 250 ml with 100 ml mineral spirits and 50 ml benzene
	Sample 2A	**Sample 3A**
Second dilution	62.5 ml diluted to 250 ml. Therefore 50 ml contain: 4.8251 g = 4.9959 ml	62.5 ml diluted to 250 ml. Therefore 50 ml contain: 5.3500 g = 4.9972 ml
	Sample 2B	**Sample 3B**
Drier addition	125.0 ml oil solution 0.42 ml 24% lead 0.24 ml 6% cobalt diluted to 500 ml with benzene. 50 ml contain: 4.8251 4.9959 0.0337 0.0234 0.0129 0.0123 4.8717 g = 5.0316 ml	125.0 ml alkyd solution 0.42 ml 24% lead 0.26 ml 6% cobalt diluted to 500 ml with benzene. 50 ml contain: 5.3500 4.9972 0.0337 0.0234 0.0140 0.0133 5.3977 g = 5.0339 ml

Rezyl is a registered trademark of American Cyanamid Co.

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Table 2—Recovery of Clear Vehicles

Solution No.	Procedure	Weight, g	Volume, ml	% of Volume Recovered	Range
2A	Pycnometer	4.7439	4.8639	97.36	1.02
2A	Pycnometer	4.7300	4.8131	96.34	
2A	Evap. dish	4.7292	4.874	97.56	0.32
2A	Evap. dish	4.7260	4.8581	97.24	
2B	Pycnometer	4.9204	sample lost		
2B	Evap. dish	4.7974	4.8993	97.37	—
3A	Pycnometer	5.3018	4.9423	98.9	—
3A	Evap. dish	5.2871	4.9480	99.02	—
3B	Pycnometer	5.5763	5.0794	100.9	—
3B	Evap. dish	5.4813	5.1256	101.82	—

Addition of an anti-oxidant was suggested to overcome the skinning problem and thus reduce the evaporation time and minimize changes in the specific gravity of the vehicle. The anti-oxidant selected was 2, 6 di-tert butyl p-cresol (DBPC, BHT or Ionol-CP) ; solutions of 1 and 2% were made up in benzene. The effect of 10 ml of the 1% solution on the vehicle volume of the bodied oil (sample 2B) and the alkyd resin (3B) was determined as shown in *Table* 3. A higher viscosity alkyd (sample 5) was then tested with both 1 and 2% solutions. The results are given in *Table* 4.

From *Tables* 2 and 4 it can be seen that the evaporating dish is more accurate and slightly more reproducible than the pycnometer procedure. This was also true in additional tests not reported here. Driers did not influence the volume of linseed oil but with the more highly polymerized alkyds tended to give high results because of oxidation. This tendency was countered by the addition of anti-oxidant and for the Z-10 alkyd, 10 ml of 2% solution was required. This amount was used in all subsequent work.

Only one more test was run on clear vehicles. All tests had been carried out on vehicles which were somewhat fluid at 25°C. It was decided to try the procedure on a material that would be solid at room

Table 3—Effect of Anti-Oxidant on Volume Recovery
(10 ml 1% Solution DBPC Added: Evaporating Dish Procedure)

Solution No.	Weight, g	Volume, ml	% of Volume Recovered	Range
2B	4.7990	4.9527	98.43	0.19
2B	4.7912	4.9429	98.24	
3B	5.3206	4.9804	98.94	0.82
3B	5.3149	4.9390	98.12	

Table 4—Effect of Anti-Oxidant on Alkyd Solution

Material	100% alkyd resin (Duraplex® C-45LV)
Viscosity (reported)	Z10+
Spec. gravity (determined)	1.0886
Solution No. 5	55.8093 g alkyd
	0.5013 g 24% lead
	0.2594 g 6% cobalt
	50 ml mineral spirits
	Diluted to 500 ml with benzene

50 ml contain:

5.5809	5.1267
0.0341	.0237
0.0145	.0138
5.6295 g	= 5.1642 ml

VOLUME RECOVERY

Procedure	10 ml 1% DBPC Volume	% Volume	10 ml 2% DBPC Volume	% Volume	Mean %	Range
Pycnometer	5.2606	101.86	5.1938	100.57	101.22	1.29
Evap. dish	5.1645	100.01	5.1321	99.38	99.69	0.63
Mean %	—	100.94	—	99.98	—	—
Range	—	1.85	—	1.19	—	—

Duraplex is a registered trademark of Rohm & Haas Co.

temperature. In order to have a sample of known solids and specific gravity, a hard resin was added to the last alkyd. To determine the true specific gravity, the hard resin was melted at 65°C. in a vacuum oven until all air bubbles had disappeared. The density was measured by both the fine wire and solvent addition methods. The formula of the solution and the volume recovery are shown in *Table 5*.

The values were slightly high but were considered sufficiently accurate for the purpose. The method was then applied to pigmented materials.

Table 5—Recovery of Solid Resin
(Solution 6)

Resin	Spec. Grav.	Weight, g
Duraplex C-45LV	1.0886	13.9201
Poly pale ester gum 10	1.0855	13.8997

Diluted to 250 ml with 25 ml mineral spirits and balance benzene

50 ml contain 5.564 g = 5.1184 ml

Weight g	Volume, ml	Recovered	Percent of Volume Mean	Range
5.5807	5.1331	100.29		
			100.42	0.27
5.5825	5.1471	100.56		

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Determining Pigment Volume and PVC

Paints were made up in a "Quickie" ball mill[8] so that there would be no loss of ingredients. The materials were weighed to 0.1 g into the mill using a triple-beam balance. The steel balls were added at two or three stages during the loading of the mill to ensure adequate mixing, especially with paints containing a high percentage of pigment. When properly charged, a Hegman reading of $6\frac{1}{2}$ was obtained in 30 to 45 minutes from batches containing extenders. The formulas for the paints are listed in *Table* 6.

The pigments used were first dried at 105°C. to ensure absence of water. Their specific gravities were also measured to calculate the volume of pigment present in the samples. The apparatus described in ASTM method D-153[9] was not available so the same wide-mouth pycnometers and low odor solvent used to obtain the vehicle densities were used together with a vacuum oven with the heat shut off. Weld pycnometers might have been a better choice although it is slightly more difficult to place the pigment in them. The procedure was to add the pigment and only part of the solvent to the pycnometer and expose to a vacuum of 28 in. for two hours. The vacuum was then released and the pycnometer gently agitated and tapped to remove air bubbles. Vacuum was again

Table 6—Formulas for Paints

Paint No.	1	5	6	8	9
Pigments					
Titanox® RA	315.0	—	315.0	—	156.0
Foam A Barytes	—	111.5	—	—	162.0
ASP® 400	—	—	—	194.0	—
Vehicle					
Rezyl 869	80.5	26.75	—	—	—
Duraplex C-45LV	—	—	81.5	84.5	80.0
Mineral spirits	118.0	19.3	118.0	119.0	118.0
PVC	50.14	50.09	50.25	49.17	50.13

	10	11	12	Specific Gravity	
Titanox RA	156.0	125.0	94.0	4.1656	
Foam A	162.0	—	—	4.447	
ASP 400	—	200.0	20.0	2.584	
Duraplex C-45LV	56.0	41.0	110.0	1.0886,	1.0845*
Mineral spirits	118.0	119.0	82.0	—	
PVC	58.96	73.93	23.01	—	

* Different lots.

Titanox is a registered trademark of Titanium Pigment Corp., and ASP is a registered trademark of Minerals & Chemicals Philipp Corp.

Table 7—Analysis of Single Pigment Paints

Paint No.	Pigment Weight	Pigment Volume	Percent Recovered Vehicle Weight	Vehicle Volume	PVC Determined	% Error
1	100.71	121.8	98.36	98.73	55.37	+ 10.4
1	100.53	116.05	98.5	98.62	54.2	+ 8.1
1	100.7	116.83	99.15	98.74	54.34	+ 8.4
1	100.82	119.13	99.64	99.24	54.7	+ 9.1
5	100.27	110.52	101.03	100.34	52.5	+ 4.8
5	100.29	103.85	101.15	100.87	50.82	+ 1.5

applied for an hour after which the pycnometer was filled with solvent to obtain the total weight. Specific gravity values outside a range of 0.02 for four or more determinations were discarded.

The analytical procedure was applied to the first two paints that were successfully produced. One contained a hiding pigment with a particle size of about 0.4 microns. The other was made with an extender with a particle size at least 10 times larger. Both pigments had about the same specific gravity and were dispersed in the low viscosity alkyd. With these paints the volume of pigment was measured by adding 10 ml of kerosene containing 0.5% oleic acid and reading the total volume, as directed in the U. S. Federal Method.[6] The results in terms of the recovered percent by weight and percent by volume of the pigment and the vehicle are given in *Table 7*.

Obviously the main source of error was in the measurement of the pigment volume, particularly with the fine particle pigment. The vehicle weight and volume and the pigment weight were all close to the correct values. Paint 6 was then subjected to analysis in which the volume of pigment was measured both by addition of solvent and through its specific gravity, as shown in *Table 8*.

By using the weight and specific gravity of the pigment its volume can be determined much more accurately than by estimating the volume directly. The resulting PVC's were accordingly closer to the true values. Placing the kerosene and pigment mixture under vacuum, as suggested

Table 8—Effect of Pigment Volume Measurement on PVC
(Paint Sample No. 6)

Run No.	Procedure	Pigment Weight	Pigment Volume	Vehicle Weight	Vehicle Volume	PVC Determined	% Error
6-1	S. G.	100.49	102.06	101.67	101.37	50.42	+ 0.3
6-3	S. G.	100.5	103.01	101.64	101.22	50.69	+ 0.9
6-2	Addition	100.51	112.93	101.5	100.35	52.95	+ 5.4
6-2	S. G.	100.51	103.06	101.5	100.35	50.66	+ 0.8
6-4	Addition	100.5	114.87	101.88	101.63	53.31	+ 6.1
6-4	S. G.	100.5	103.69	101.88	101.63	50.5	+ 0.5

in the Federal Method, did not affect the results. The specific gravity procedure was used in all subsequent work.

With the first pigmented samples the extracts were placed directly in evaporating dishes and the solvents removed after each extract was added. This meant that most of the vehicle which was obtained in the first extract was heated four times which led to skinning in the vacuum oven. It was found preferable to keep the extracts in a florence flask and periodically distill off the solvent by immersing the flask in a hot water bath only to the level of the contents. After most of the solvent from the last extract had been removed, the concentrated vehicle was transferred to the evaporating dish using benzene as wash liquid. As well as avoiding exhaustion of the anti-oxidant, this procedure tended to flocculate the fine pigment which had been carried into the extract. It was then possible to decant off the vehicle.

The last five paints were prepared and analyzed to investigate the following factors: No. 8—low specific gravity pigment and effect of extraction solvent; Nos. 9 and 10—mixtures of coarse and fine pigments of the same specific gravity; Nos. 11 and 12—mixtures of coarse and fine pigments of differing specific gravities at extremes of the PVC range. The results are summarized in *Table* 9.

Four analyses were run on paint 8 because it had been noticed that it was not always possible to get a clear extract by centrifuging. Also when analyzing an aluminum silicate sample the vehicle recovery had been low by almost 10%. Up to this time the usual extraction mixture listed in ASTM D-215, Gardner, and Federal Standard 141 had been used. In an attempt to get better separation a mixture which the author had developed some years previously for gravity settling of pigments was used. The standard mixture was tried on runs 1 and 3, the special on runs 2 and 4. As shown in *Table* 9 the special solvent had

Table 9—Final PVC Determinations

Run No.	Factor	Pigment Volume	Vehicle Volume	Actual	Pigment Volume Content Det'd	Error	% Error	Range
8-1	Extractant	101.49	97.94	49.17	50.06	+0.89	+1.8	
8-3	Extractant	102.18	97.81	49.17	50.26	+1.09	+2.2	0.2
8-2	Extractant	101.56	99.48	49.17	49.68	+0.51	+1.0	0.1
8-4	Extractant	101.18	99.46	49.17	49.6	+0.43	+0.9	
9-1	Packing	101.6	99.67	50.13	50.61	+0.48	+1.0	0.1
9-2	Packing	101.46	100.01	50.13	50.49	+0.36	+0.7	
10-1	Packing	100.67	99.07	58.95	59.34	+0.39	+0.7	0.1
10-3	Packing	100.53	98.57	58.95	59.43	+0.48	+0.8	
11-2	High PVC	100.72	98.41	73.97	74.41	+0.44	+0.6	0.5
11-3	High PVC	100.82	95.74	73.97	74.95	+0.98	+1.3	
12-1	Low PVC	100.5	98.67	23.01	23.33	+0.32	+1.4	0.3
12-2	Low PVC	102.55	98.93	23.01	23.65	+0.64	+2.8	

somewhat better vehicle recovery. Since it had also been of value in other analyses, it was used for the remaining samples. The composition of the two mixtures was as follows.

Extraction Mixtures
(all parts by volume)

Standard		Special	
Ethyl ether	10	Benzene	6
Benzene	6	Ethanol (95%)	2.5
Methanol	4	Petroleum ether	1.5
Acetone	1		

Table 9 shows the lower specific gravity of the pigment in sample 8 did not affect the results. Neither did differences in particle size, which might lead to pigment packing, whether the pigments were of similar or different density. The procedure was not noticeably affected by high or low pigment volume concentration.

The results consistently tended to be high by about 0.4%. *Figure* 1 is a histogram of the differences between the determined and the actual PVC's for paints 6 to 12. Considering the small number, the approximation to the normal distribution curve is good. Run 11-3 can be rejected as anomalous on the basis of Dixon's Q test for extreme values.[10]

Errors in the determined PVC are chiefly attributable to the pigment volume. This is illustrated in run 12-2 where the recovered pigment weight was 100% but the pigment volume was 102.5%. Other runs showed the same trend although not to the same extent. The accuracy of the pigment volume depends upon the specific gravity measurement. In this work equipment normally available in a laboratory was used. Duplicates were run on each isolated pigment and the mean range was 0.02 although this value is the maximum allowable range in ASTM D-153.[9] It is considered that if a more reproducible method had been used, or if the method used had been refined, even better PVC results could have been obtained.

The standard deviation between duplicate PVC determinations was calculated to be 0.129%, excluding sample 11. Using the "studentized" range, the maximum permissible differences between duplicates should be 0.6% at the 99% confidence level. The usual 95% confidence range is not given because results from one operator are biased in favor of the accuracy of the method.

SUMMARY

A procedure which has overcome some of the original objections has been developed for the analytical determination of PVC. Its chief disadvantage is the long time required, even in a vacuum oven to remove all the solvent so that the true vehicle weight can be obtained. Contrary to the original assumption, pigment volume is the main source
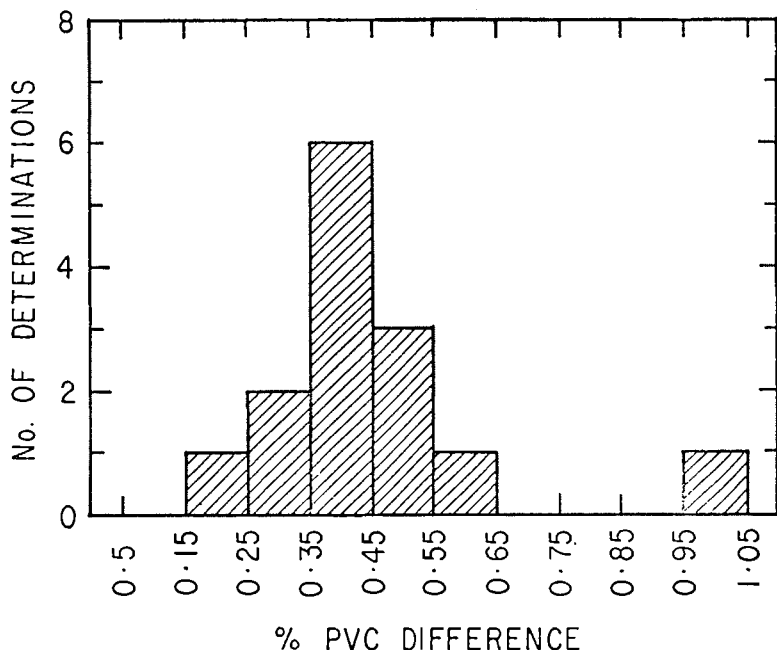
Figure 1—Histogram of difference from true PVC value

of difficulty. Finally, the footnote in method 4312 of Standard 141[6] should be revised since it has been demonstrated that the specific gravity procedure is more accurate than that given in the method for obtaining the pigment volume.

The complete method is as follows.

Weigh about 15 ml of the pigmented material into a tared 50-ml centrifuge tube. Add 10 ml of 2% DBPC in benzene and mix with a tared stirring rod. Add 15 ml extraction mixture, mix again and wash the rod with mixture to make a total volume of about 45 ml. Place the tube in a centrifuge tube shield containing water and balance with a duplicate tube. Whirl at a moderate rate until the pigment is well settled and decant the supernatant liquid into a tared 125 ml standard taper florence flask using benzene as wash liquid. Repeat the extraction twice with 40-ml mixture and once with 40-ml ethyl ether. During extractions distill most of the solvents from the flask by immersing it in a hot water bath to a depth equal to the level of contents. After the last extraction and distillation, transfer the concentrated resin solution to a tared evaporating dish, using benzene as wash liquid and leaving any flocculated pigment in the flask. Heat on a steam bath until only traces of solvent remain and then place in a vacuum oven. Heat at 105°C. and 26- to 30-in. vacuum until the total loss in weight from two consecutive 3-

hour periods is less than 10 mg. Cool in a desiccator and weigh. Warm in the oven and transfer as much resin a possible to a tared Hubbard-Carmick pycnometer. Determine the specific gravity of the isolated vehicle as described.[7]

Meanwhile dry the pigment in the centrifuge tube to constant weight after driving off the ether by immersing the tube in warm water. Cool in a desiccator and weigh. Add the weight of any pigment in the flask or adhering to the stirring rod. Grind the pigment to a fine powder and determine the specific gravity according to ASTM D-153.[9]

$$\text{Vehicle Volume (V.V.)} = \frac{\text{Weight of Vehicle}}{\text{Specific Gravity}}$$

$$\text{Pigment Volume (P.V.)} = \frac{\text{Weight of Pigment}}{\text{Specific Gravity}}$$

$$\text{Pigment Volume Concentration (P.V.C.)} = \frac{\text{P.V.}}{\text{P.V.} + \text{V.V.}} \times 100$$

$$\text{Percent Pigment by Weight} = \frac{\text{Weight of Pigment}}{\text{Weight of Sample}} \times 100$$

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