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Measurement of Particle Size and Distribution in a Suspension

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MEASUREMENT OF PARTICLE SIZE AND DISTRIBUTION IN A SUSPENSION

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INTRODUCTION

Within the last few years investigators, particularly in the field of paint and varnish chemistry, have been interested in the determination of particle size of paint pigments. From a knowledge of particle size and distribution the significance of the "hiding power" and other physical properties have been more thoroughly understood.

Swedberg particularly for purely theoretical reasons has done considerable work in this direction. Swedberg and Stamm[1] determined particle size of benzene in water emulsions. By observing changes of concentration with height and time in an illuminated cell containing the emulsion, they were able to calculate particle size and its distribution of mass. Concentration was found to be proportional to light intensity as observed photometrically, the light measured being the result of scattering at right angles to the source of illumination of the cell.

Stutz and Pfund[2] have determined the average size of particle by the light transmitted through a cell containing ZnO in suspension. They found that with decreasing of particle size the percentage of light transmitted decreased

and passed through a minimum at 0.24 microns regardless of the medium of suspension. This method involved the use of a ribbon filament lamp as source. The brightness of transmitted light through the cell was matched with the filament of an optical pyrometer.

The actual size of particle was determined by the method of Green[3] involving the counting and measuring of a sufficient number of particles to obtain a statistical average. Microphotographs were taken of about two hundred fifty particles and the photographs then projected on a screen with a magnification of 20,000-25,000 diameters. The measurement and distribution could then be readily made.

Using particles thus determined in the suspension, Stutz and Pfund were able to obtain a calibration curve for the pyrometer. The effect of different dispersion mediums was to shift the curve parallel to the ordinate axis, the amount of shift depending upon the refractive index of the liquid.

Gravimetric determinations of particle size have been made by other investigators involving weighing of particles by sedimentation. Odén[4] measured the amount of settling of a suspension automatically on a balance pan. Swedberg[5] gives a good description of this method.

PURPOSE OF INVESTIGATION

The purpose of the present investigation

was the determination of size of particle of "Glyptal" in suspension. Glyptal is the name given to a synthetic resin formed at an elevated temperature by condensation of glycerol (CH_2 OH. CHOH. CH_2 OH) and Phthalic Anhydride ($C_6 H_4 (CO)_2 O$) in the proportions of two to three respectively.

Glyptal or "A" stage glyptal as produced by this reaction is a transparent amber colored substance of density about 1.4. It is soluble in a number of organic solvents and possesses a flow point of 114-118°C. Upon further heat treatment the resin reaches the "B" stage possessing no flow point or solubility.

By incorporating a finely ground mixture of glyptal with certain pigments a fairly stable suspension with carbon tetrachloride is obtained. By dipping sheet metal in the suspension and baking at a fairly high temperature, an enamel finish, similar to vitreous enamels, may be produced. The uniformity and general appearance of the resulting finish is dependent upon the size and spacing of particles of the resin upon the surface of the metal. This in turn is dependent upon the concentration and size of particle in the suspension.

FIRST EXPERIMENTAL METHOD

A method similar to that of Stutz and Pfund[2] was tried out. The results obtained did not justify a very complete investigation by this means. The apparatus as used consisted of a gas filled tungsten lamp as light source,

a pair of biconvex lenses, the cell containing the suspension, and a pyrometer. The pyrometer was of the disappearing filament type and was loaned to the writer together with a milleammeter and calibration curves. These curves gave Black Body and True Tungsten filament temperatures plotted against milleammeter readings. The arrangement is shown in Figure I. The purpose of the lenses was to give a parallel beam of light in the cell.

Samples of glyptal were ground for one to two days in a porcelain ball mill and sieved over night in a mechanical sieve shaker. Particles were then classified according to the following Table I:

TABLE I - CLASSIFICATION OF SIZE OF PARTICLE

100% "A" Stage	85% "A" Stage 15% "B" Stage	66% "A" Stage 34% "B" Stage	100% "B" Stage
100-120 mesh (.149-.125 mm)⊙	100-120 mesh	100-120 mesh	100-120 mesh
120-150 mesh (.125- mm)	120-150 mesh	120-150 mesh	120-150 mesh
150-200 mesh (- .074 mm)	150-200 mesh	150-200 mesh	150-200 mesh
200- ? mesh * (.074-? mm)	200-? mesh	200- ? mesh	200- ? mesh
A	B	C	D

*All particles which passed a 200 mesh screen
⊙ Mesh openings - Tech.Papers Bureau of Standards No.321

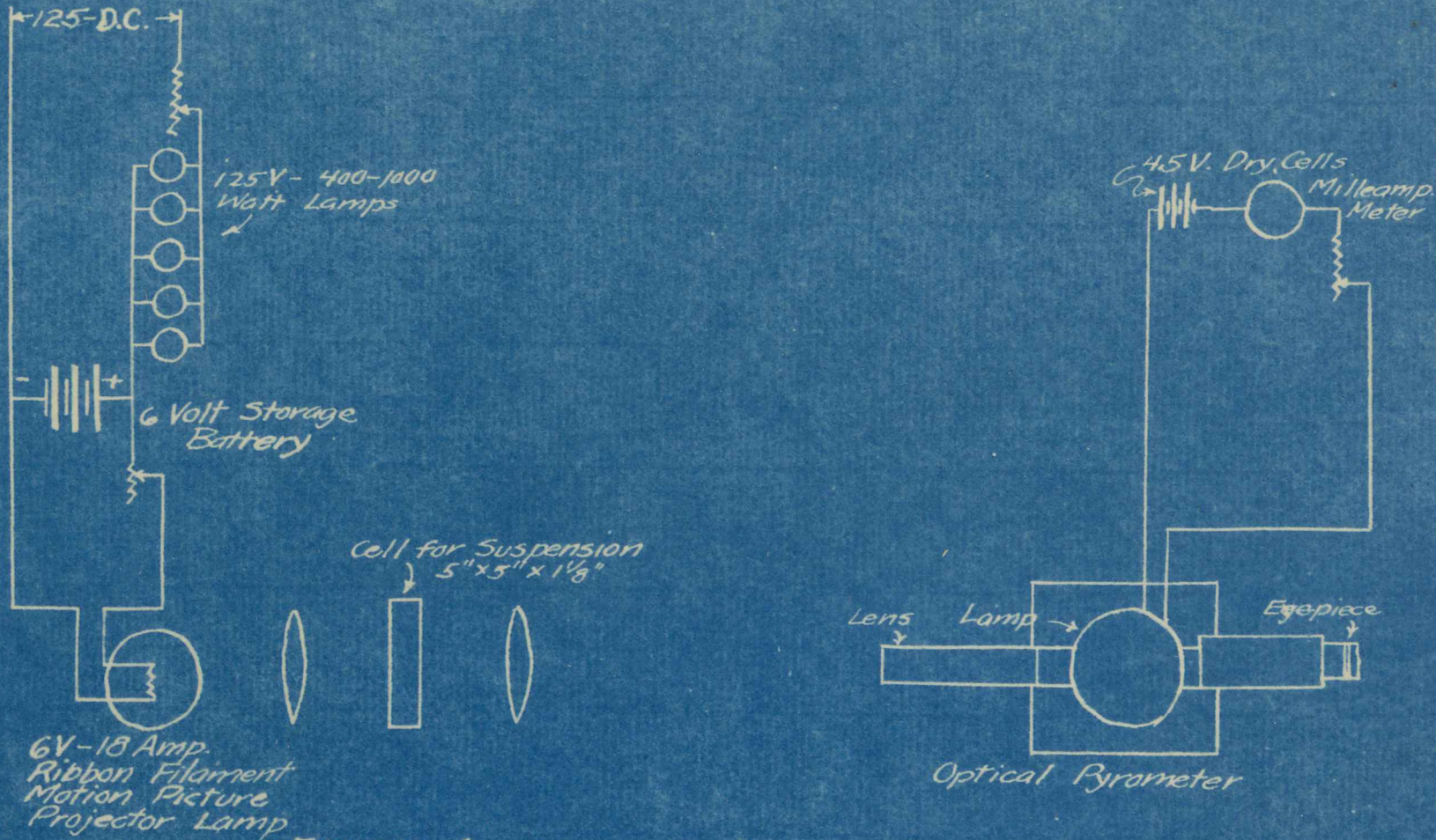

FIGURE 1 - Schematic Arrangement For Measuring Light Absorbtion or Transmission of a Suspension

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As screens below 200 mesh were not available no classification could be given.

The lamp source first used was a straight 8.5 mil tungsten filament, spring supported (to prevent sag) and gas filled. The lamp was designed to operate with a six volt storage battery at about four amperes. With this lamp it was found difficult to match the pyrometer filament, that is, repeated readings varied over two or three scale divisions of the milleammeter.

The area of the light source was then increased by using a motion picture projector lamp with a crimped ribbon filament. This gave very satisfactory check readings with no suspension in the cell. The lamp had a rating of 6 Volts- 18 amperes requiring a high capacity storage battery which was "floated" on the 125 Volt- D.C. shop line. The charging current for the battery was varied by a bank of high wattage lamps in series with the shop line.

In matching the ribbon filament of the lamp, a considerable area of filament near the center gave a constant temperature and so was utilized for all experiments. Likewise only one position of the cell was measured. The suspension was not stabilized by any agents and, of course, gradually became less dense at the area of illumination as the glyptal particles rose to the surface of the liquid.

Wien's Equation gives the following relation:-

$$\frac{1}{T_1} - \frac{1}{T_2} = \frac{\lambda_1}{C_2} \log_e (\text{Transmission})$$

where T_1 and T_2 are black body temperatures in degrees absolute and λ_1 and C_2 are constants. The percentage of light absorbed or transmitted can be readily calculated. It was suggested to the writer that the amount of light transmitted by the cell could be thus calculated and checked for various temperatures. For with a temperature of T_1 of the source at say 1600°K., and at 2000°K., the corresponding values of T_2, the temperature as measured with an absorbing medium between, should be proportionally lower.

The constants of this equation (Forsythe[6]) are λ_1 = .665 (the limiting wave length of the red eye piece glass) and C_2 = 14,350μ degrees, or simplified, the equation may be written:

$$9370 \left(\frac{1}{T_1} - \frac{1}{T_2} \right) = \log_{10} (\text{transmission})$$

Material of Class C was used for the experiment.

The following Table II gives some of the data obtained.

The results indicate no consistency in observation and in some cases show greater light transmission than the amount of light present at the source, that is, where the logarithm of transmission or $\left(\frac{1}{T_1} - \frac{1}{T_2} \right)$ is positive.

TABLE II - EXPERIMENTAL RESULTS OF LIGHT TRANSMISSION

GLYPTAL IN CARBON TETRACHLORIDE

Current (Amps.)	Conc. of Glyptal Grams/cc	Class and Mesh	Pyrometer Reading Milleamps.	Black Body Temp. °K	$\frac{1}{T}$	$\frac{1}{T_1} - \frac{1}{T_2}$	Remarks
16.6	---	---	459,459	2495	4.01×10^{-4}		Without cell present
16.6	---	---	439,438, 438	2410	4.15×10^{-4}		With empty cell present
16.6	---	---	420,420	2335	4.28		With cell and CCl_4
15.8	---	---	400,404, 405,405	2270	4.41		" " " "
15.0	---	---	388,389	2200	4.55		" " " "
14.0	---	---	367,367	2105	4.76		" " " "
13.0	---	---	346,345	2005	4.99		" " " "
17.9	---	---	441,441	2420	4.13		" " " "
16.6	---	---	421,421	2340	4.275		" " " "
16.6	.0005	C 200-?	410,404, 413	2250	4.39	-.1075	With cell & CCl_4 & glyptal
17.9	.0005	"	444,442, 438,440	2420	4.13	0.0	"
15.0	.0005	"	380,388, 387,387	2185	4.58	-.030	"
13.0	.0005	"	348,351, 353,350	2020	4.95	+.040	"
16.6	.0005	"	417,410 420,417	2325	4.31	-.035	"
16.1	.0005	"	310,315 308,311	1830	5.47		Shows absopption increased with decrease in particle size
16.1	.0005	150-200 C	407,407 407	2282	4.38		
16.6	.0005	"	417,421, 420,417	2332	4.29	-.015	
15.0	.0005	"	393,394, 392,392	2220	4.51	+.04	
13.0	.0005	"	353,350 355,349	2032	4.93	+.06	
15.0	.0005	"	391,391 394,392	2220	4.51	+.04	

Table II (continued)

rent Lamp ps.)	Conc.of Glyptal Grams/cc	Class and Mesh	Pyrometer Readings Milleamps.	Black Body Temp.°K.	$\frac{1}{T}$	$\frac{1}{T_1} - \frac{1}{T_2}$	Remarks
0	.0005	150-200 C	403,405, 405,406				Observations of cell after time interval of 2 days
6	.0005	"	436,431, 440,437				
0	.0005	"	363,363, 365,365				
0	.0005	120-150 C	361,354, 355,357, 357,360	2060	4.86	+.13	
0	.0005	"	400,397, 400,400	2250	4.45	+.10	
6	.0005	"	433,432, 434,432	2390	4.18	+.095	
9	.0005	"	452,457	2475	4.04	+.095	
9	.0005	"	357,360 357,356	2055	4.86	+.13	

This latter condition may be due to the fact that glyptal is transparent to ordinary light and with the size of particle measured, double reflection within the particle has reinforced the apparent brightness of the light source.

Actually, the particles being visible in the eye piece of the pyrometer were moving back and forth within the cell giving uneven illumination of the filament. This, of course, caused some difficulty in matching. For finer particle size this trouble would be eliminated.

SECOND EXPERIMENTAL METHOD - DISCUSSION

As mentioned previously, gravimetric measurement of particles settling in a suspension have been made in various ways. An adaption of Oden's method was utilized by Calbeck and Harned[7] very successfully for paint pigments.

As a chemical balance which could be modified to accommodate a settling apparatus was not available to the writer, determinations were made using long tubes. By a series of long tubes containing equal volumes of suspension the amount of settling with time could be readily obtained.

Each size of particle corresponds to a definite time of settling or in other words, the velocity of fall of a definite size of particle in the medium is a definite value. The velocity of fall is dependent upon the specific gravity of the particle, the specific gravity of

the medium, the viscosity of medium and the acceleration of gravity.

Stokes Law was derived on this basis. For the force of friction opposing fall is equal and opposite to the force of gravity of a particle falling at constant velocity

$$6\pi\eta rV = \frac{4}{3}\pi r^3(\rho_p - \rho_d)g$$

The left hand member is the force of friction and the right hand member is the force of gravity.

Stokes Law may then be stated:

$$r = \sqrt{\frac{9}{2}\frac{\eta V}{(\rho_p - \rho_d)g}}$$

r = radius of particle in cm.

η = absolute viscosity of medium in poise

V = velocity in cm/sec.

ρ_p = specific gravity of particle

ρ_d = specific gravity of medium

g = 980 cm/sec.^{2} is the acceleration of gravity

For a definite distance of settling of a particle the radii of the particle can be calculated for convenient time intervals. By choosing the liquid medium of suspension of the proper specific gravity and viscosity the time necessary for complete settling of the suspension should be readily adjusted.

The following Table III gives the physical

properties of several mediums of suspension for the glyptal.

TABLE III - VISCOSITY, DENSITY AND BOILING RANGE OF NON-SOLVENTS OF GLYPTAL

Liquid	Sp. Gr.	Boiling Range °C.	Viscosity (centipoise) 10°C.	20°C.	30°C.
Carbon Tetrachloride	1.600	75-100 (90%)	1.138	.975	.848
Carbon Bisulphide	1.270	25-70 (90%)	.405	.376	.352
Benzene	.880	82-100 (90%)	.763	.654	.567
68-70° Naphtha	.705	65-155 (90%)			
Water	.998	100	1.303	1.006	.8007
47% C_6H_6 * 53% CCl_4	1.254			.904	
43.1% C_6H_6 56.9% CCl_4	1.300			.904	
33.7% C_6H_6 66.3% CCl_4	1.3500			.920	
27.7% C_6H_6 72.3% CCl_4	1.400			.966	
14.3% C_6H_6 85.7% CCl_4	1.469			.980	

* C_6H_6 - CCl_4 mixtures determined at 26°C.

The viscosities of the pure substances were taken from the Smithsonian Tables. Viscosities of the CCl_4 - C_6H_6 mixtures were determined with a Stormer Viscosimeter, the accuracy of which is somewhat doubtful. Each value determined

is
however an average of ten measurements.

The following Table IV gives the Stoke's formula for several liquid mediums:

TABLE IV - RADII OF PARTICLE FROM STOKES LAW - 26°C

For CCl_4:-

$$r = \sqrt{\frac{\frac{9}{2} \times .009 \times h}{(1.600-1.377)60 \times 980\ t}} = .00173 \sqrt{\frac{h}{t}}$$

For Water:-

$$r = \sqrt{\frac{\frac{9}{2} \times .008737\ h}{(1.377 - .997)\ 60 \times 980\ t}} = .00162 \sqrt{\frac{h}{t}}$$

For Benzene:-

$$r = \sqrt{\frac{\frac{9}{2} \times .0061\ h}{(1.377 - .880)\ 60 \times 980\ t}} = .00097 \sqrt{\frac{h}{t}}$$

For 33.7% C_6H_6)
66.3% CCl_4):-

$$r = \sqrt{\frac{\frac{9}{2} \times .0092\ h}{(1.377 - 1.3500)\ 60 \times 980\ t}} = .00511 \sqrt{\frac{h}{t}}$$

For 47% C_6H_6)
53% CCl_4)

$$r = \sqrt{\frac{\frac{9}{2} \times .00904\ h}{(1.377 - 1.254)\ 60 \times 980\ t}} = .00237 \sqrt{\frac{h}{t}}$$

For CS_2

$$r = \sqrt{\frac{\frac{9}{2} \times .0036\ h}{(1.377 - 1.270)\ 60 \times 980\ t}} = .001625 \sqrt{\frac{h}{t}}$$

For 14.3% C_6H_6
85.7% CCl_4

$$r = \sqrt{\frac{\frac{9}{2} \times .00980\ h}{(1.469 - 1.377)\ 60 \times 980\ t}} = .002856 \sqrt{\frac{h}{t}}$$

It is evident from an examination of Table IV that benzene will give the most rapid settling period. The initial experiments by this method were made with average settling distances of 29.5 cm. using Class D glyptal (150-200 mesh) of Specific Gravity 1.377.

Under the conditions of the experiment and settling periods taken (which were much too short) the weight of material collected was not a function of the time.

For according to Table V the complete settling of all particles greater than .074 mm diameter should take place within the time intervals indicated.

TABLE V - RADII OF PARTICLE AND TIME SETTLING FOR SUSPENSION IN C_6H_6, CCl_4, H_2O, CS_2 AND MIXTURES OF C_6H_6 AND CCl_4

Time (Min.)	Radius of Particle (mm) (29.5 cm)	(5 cm.)	Liquid
60	.122	.049	CCl_4
120	.085	.034	
180	.070	.029	
240	.061		
480	.042		
960	.030		
60	.115	.046	H_2O or CS_2
120	.080	.033	
180	.066	.027	
240	.057		
480	.040		
960	.028		
60	.068		C_6H_6
120	.048		
180	.039		
240	.034		
480	.024		

TABLE V (Continued)

Time (Min.)	Radius of Particle (mm) 29.5 cm.	5 cm.	Liquid
60	.166	.058	47% C_6H_6)
120	.117	.048	53% CCl_4)
180	.096	.039	
240	.083		
480	.058		
960	.041		
60		.082	14.3% C_6H_6)
120		.058	85.7% CCl_4)
180		.048	
240		.041	
350		.037	

Experiments were then made for settling distances of 5 cm. for two mixtures of C_6H_6 and CCl_4 and CCl_4 alone.

SECOND EXPERIMENTAL APPARATUS AND RESULTS

The settling tubes were mounted as shown in Figure 2.

The apparatus was provided with inlet and outlet tubes for circulation of water to maintain constant temperature. The frame supporting the tubes was painted with a black varnish to aid in observation of liquid level and to protect the wood against swelling from the water in the bath. The glass plates were held in position by grooves in the sides of the frame. Attempts to make the frame watertight were unsuccessful although paraffin was used both on the inside and outside edges of the frame. A black

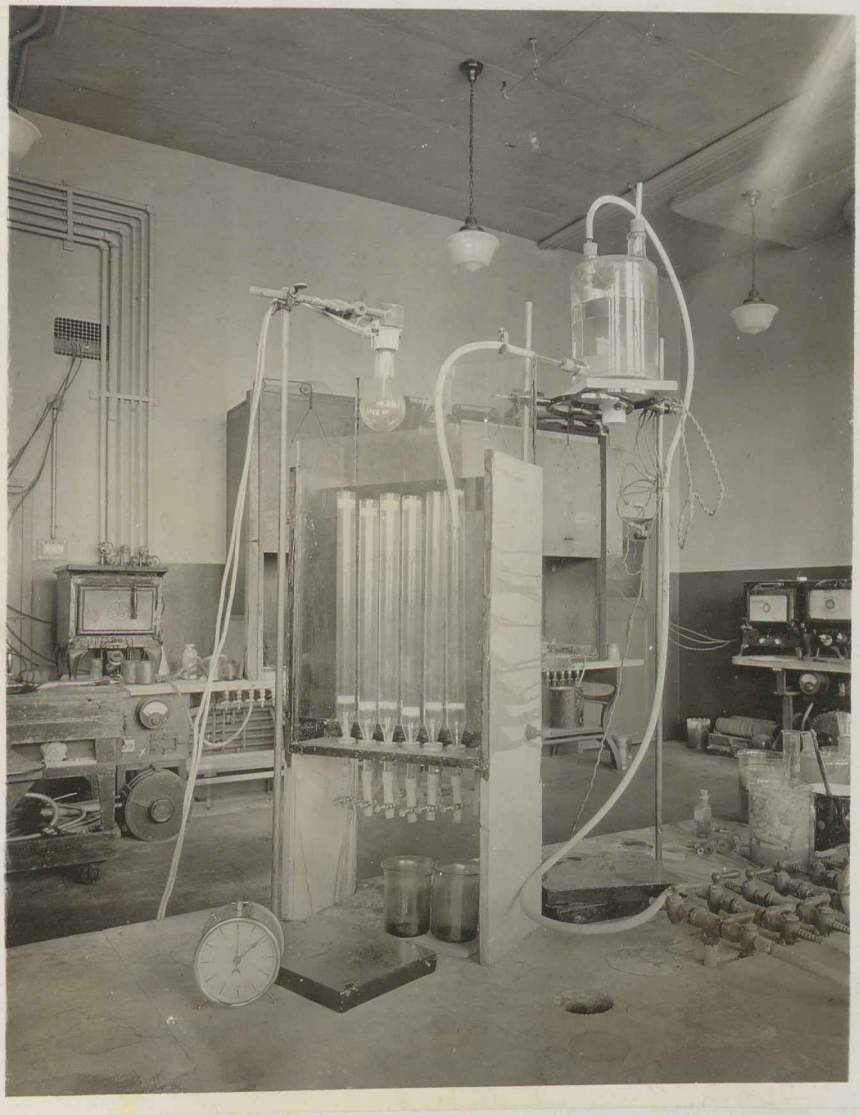

FIGURE 2. SEDIMENTATION APPARATUS

pitch dissolved in CS_2 was also tried without success.

Initial measurements were made with glass plates alone protecting the contents of the tubes from temperature change. The tubes were calibrated with markings on the glass at 25 cc. and 175 cc. levels as measured with pipettes of 25 and 150 cc. capacity. Variations in diameter of the tubes gave corresponding differences in level between markings.

A weighed amount of "B" stage glyptal was added to a definite volume of the medium of suspension and placed in a Wolff bottle. The opening at the bottom of the bottle was controlled by a screw clamp attached to a short length of rubber tubing. Air was circulated through the suspension at the two openings in the top of the bottle.

In the case of benzene, the suspension settled so rapidly that circulation of air was not sufficient to keep the suspension stirred.

For CCl_4, the particles tended to rise and the suspension was kept in circulation. In the case of H_2O, a foam was formed at the surface of the liquid which made level readings inaccurate.

The procedure followed consisted in filling the tubes, noting the time of filling, the time of settling and the time of empting liquid between level markings of the tubes.

The suspension was emptied into weighed beakers and evaporated to dryness and the beakers again weighed. Knowing the concentration of glyptal in the Wolff bottle and in the tubes, the percentage of glyptal by weight settling, at the time intervals of settling, could be calculated.

For both water and CCl_4 the evaporation to dryness took several hours in an oven at 100°C. Higher temperatures of evaporation than this are undesirable as the glyptal loses a small percentage of H_2O of condensation.

Table VI and Table VII give the results obtained with settling distances of 26-30 cm. and 5 cm. respectively. In Table VII the settling distance was constant and the volume corresponding to this distance was determined. The concentration of the glyptal was of course constant for any one series of experiments. Because of inconsistencies in the results of Table VI, the amount of glyptal in the remaining suspension, after removal of a volume corresponding to 5 cm. of tube length, was determined. This amount should be constant as this volume is 25 cc. in all cases. Also the total amount of glyptal in the suspension as measured by weight should correspond to the amount calculated.

It is evident that these relationships do not hold. V_1 corresponds to the volume of suspension between graduations on the tube - the total volume of suspension being equal to $(V_1 + 25)$ cubic centimeters.

TABLE VI - SETTLING RATES OF SUSPENSION - 150-200 MESH "B" GLYPTAL

A. SUSPENSION IN WATER

Tube No.	Time of Settling (Minutes)	Distance of Settling (Cm)	Wt. of Glyptal Settled (Grams)	Conc. of Glyptal in Suspension Grams/cc	Volume of Suspension (cc)
1	1	29.7	.1912	2.73×10^{-3}	175
2	2	27.8	.1205	"	175
3	4.5	27.3	.0410	"	175
4	8.5	29.3	.1775	"	175
5	16.25	27.5	.1614	"	175
6	1143.	26.5	.2373	"	175
B. SUSPENSION IN 68-70° NAPHTHA					
7	1.5	29.6	.1790	2.5×10^{-3}	175
8	2.5		.0951	"	175
9	5.0		.1989	"	175
10	8.5		.1280	"	175
11	16.5	20.4	.2358	"	175
C. SUSPENSION IN CARBON TETRACHLORIDE					
7	2.5	29.6	.4648	2.86×10^{-3}	175
8	4.5	30.0	.4705	"	175
9	10.25	29.3	.3906	"	175
10	23.5	29.3	.1500	"	175
11	45.5	29.2	.0838	"	175
12	58.0	29.9	.0911	"	175

TABLE VII - SETTLING RATES OF SUSPENSION - 150-200 MESH "B" GLYPTAL THROUGH 5 cm DISTANCE

SUSPENSION IN MIXTURE (47% C_6H_6
(53% CCl_4

Tube No.	Time of Settling (Min.)	V_1 Volume of Suspension (cc)	Wt. of Material Settled in V_1 (grams)	Conc. of Glyptal Grams/cc	Calc. Wt. Glyptal in V_1 (grams)	Wt. of Mat'l. from 25 CC. remain-ing	Calc. Wt. of Glyptal in 25cc. remain-ing (grams)	Total Wt. of Glyptal in (V_1+25) cubic cm. (grams)	Total Wt. Glyptal Calc. (grams)
	2	26.5	.1743	.00301	.07975	.0441	.07525	.2184	.1550
	4	27.5	.1697	"	.0828	.0312	"	.2009	.1580
	8	30.5	.2170	"	.0918	.0402	"	.2572	.1670
	16	27.0	.1915	"	.0813	.0188	"	.2103	.1565
	27	28.7	.2581	"	.0864	.1220	"	.3810	.1616
	64.5	29.0	.2276	"	.0873	.0839	"	.3115	.1625
	108	28.2	.2402	"	.0850	.0430	"	.2832	.1602
	168	28.5	.1885	.003005	.0857	.0633	.0751	.2518	.1609
	172	26.0	.1853	"	.0781	.0330	"	.2183	.1533
	247	26.5	.1460	"	.0796	.0479	"	.1939	.1548
	1410	27.0	.1090	"	.0811	.1024	"	.2114	.1563

The normal inside diameter of the tubes was one inch and the lower ends of the tubes were drawn down to one half inch diameter for a rubber tube and screw clamp controlling the orifice. Although the constriction in the tube from one inch diameter to one half inch diameter was very gradual a certain amount of suspension adhered to the walls of the tubes in this region. As the shape factor of tube was nearly constant for all tubes it was hoped that this error could be corrected by a constant difference in weight.

Another source of error involved the method of stirring the suspension in the Wolff bottle. As mentioned before, the glyptal particles settled very rapidly in benzene suspension so that the circulation of air through the bottle was insufficient.

For CCl_4 and the other suspensions, the glyptal was apparently well stirred. From an examination of Table VII it is evident that unequal quantities of glyptal were delivered to the different tubes.

A check was made upon the possibility of the CCl_4 and C_6H_6 mixture containing high boiling impurities which were not driven off upon evapporation at 100°C. A beaker containing the mixture of benzene and carbon tetrachloride alone - when evaporated at 100°C. for two hours gave no residue as determined by weight. On the other hand, when

evaporating the suspension to dryness, the glyptal turned to a dark brown color and it took several hours before the beaker gave no odor of benzene or carbon tetrachloride. That the suspension medium shows slight solubility for the glyptal or retains a certain amount of the latter by chemical reaction is evident from the great discrepancy in values as indicated in the last two columns of Table VII.

This latter source of error is the outstanding cause of discrepancy in the results obtained which error would seem unavoidable with the known non solvents of glyptal.

GENERAL SUMMARY AND CONCLUSION

A method of determining average particle size of glyptal in suspension by measurement of its light transmission in a cell is described.

A method of determining particle size and distribution of glyptal in a suspension by sedimentation is described.

Measurements of particle size of transparent particles, of the size determined, by the former method cannot be successfully applied. The method involves error due to internal reflections within the particle which reinforce the apparent brightness of the filament source. This is particularly true as particle size is

increased. This error may be increased by difficulty in matching with particles of a size which are visible without magnification as seen in the pyrometer.

This method might be checked with particle size in the neighborhood of 1-10 microns in the region in which Swedberg[1] worked with emulsions. That measurement by scattered light at right angles to the source may be successfully applied, is not without possibility.

The second method involves several errors, one of which alone would eliminate this means of measurement for glyptal particles. The sources of error may be enumerated as follows according to seriousness of error:

(a) Error due to solubility of chemical reaction between suspension medium and glyptal upon evaporation to dryness.

(b) Error due to settling of suspension in reservoir of settling tubes.

(c) Error in filling of settling tubes, that is, obtaining equal concentration of suspension in all tubes.

(d) Error due to shape of tube - suspension adhering to glass walls at the tube constriction.

(e) Error in filling to correct level and reading of liquid levels in the tubes.

In the case of clay suspensions in water, this latter method[8] (slightly modified) has been very successfully applied to particle size in the region of 14 microns and might be equally well adapted in certain

other cases.

For glyptal suspensions, the only method which would seem to be applicable is a sedimentation method[7] involving the weighing of suspension without evaporation on a balance pan.

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