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(Triannual Report)

DEVELOPMENT OF SPACE-STABLE THERMAL-CONTROL COATINGS (PAINTS WITH LOW SOLAR ABSORPTANCE/ EMITTANCE RATIOS)

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama

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DEVELOPMENT OF SPACE-STABLE THERMAL-CONTROL COATINGS (PAINTS WITH LOW SOLAR ABSORPTANCE/EMITTANCE RATIOS)

Contract No. NAS8-5379
IITRI Project C6014

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DEVELOPMENT OF SPACE-STABLE THERMAL-CONTROL COATINGS (PAINTS WITH LOW SOLAR ABSORPTANCE/EMITTANCE RATIOS)

I. INTRODUCTION

The general requirement under this contract is for thermal control surface coatings with very low, but stable, solar absorptance to infrared emittance ratios. The work is currently proceeding in two major directions; (1) inorganic pigment studies, and (2) methyl silicone photolysis studies. Secondary efforts involve (1) methyl silicone polymer synthesis studies in support of the photolysis investigations, (2) methyl silicone synthesis studies aimed at development of foamed structures, and (3) inorganic pigment binder investigations.

The principal aim of this report is to present various data generated in recent months -- particularly data that has not been reported to date. These data include (1) the results of test V61 which includes the very promising Owens Illinois Type 650 Glass Resin, (2) absorption spectra measurements (optical density in the ultraviolet and visible regions) of various paint constiuents, (3) particle size distribution analyses of various zinc oxide-pigment slurries measured on the Kaye Disc Centrifuge, and (4) the most recent polydimethylsiloxane synthesis and photolysis experiment.

II. METHYL SILICONE STUDIES

A. Irradiation of ZnO-Paints

Several zinc oxide-pigmented methyl silicone paints and a rutile titania-pigmented polyvinylidene fluoride (Kynar) were irradiated with ultraviolet for 2000 ESH (10X) in space simulation test V61. The results of this test are shown in Table 1.

Paint S-33, a specification paint prepared under contract to the Jet Propulsion Laboratory (Report IITRI-C207-25, Summary Report), was formulated from a freshly synthesized experimental "molecularly-distilled" resin R-9. This paint was observed to undergo the previously discussed reflectance increase in the infrared, α_2, which more than counterbalanced the degradation in the visible portion of the spectrum (resulting in a $-\Delta\alpha_s$). This coating has received greater emphasis in a current program for the Jet Propulsion Laboratory and will be discussed in some detail in the forthcoming Summary Report (IITRI-C6027-16 on that program). However, the stability of this system was again confirmed.

The specimen of S-13 designated #5082 was prepared from a paint manufactured on 11/2/64 and was cured using the lower catalyst concentration (Report IITRI-C6014-13, p. 48) and toluene solvent. The solar absorptance increase of 0.026 is consistent with previous results for a similar paint. In contrast, however, is the considerably greater stability exhibited by an identical paint cured with Shell Chemical Company's proprietary H2 catalyst. This catalyst has been observed to possess a shorter wavelength

Table 1

EFFECT OF ULTRAVIOLET IRRADIATION IN VACUUM ON ZINC OXIDE PIGMENTED METHYL SILICONE POLYMERS

Sample	Paint	Resin	Exposure ESH	Solar Absorptance α_1	α_2	α_s	$\Delta\alpha_s$
5077	S-33	R-9	0	0.102	0.104	0.206	
		(Butanol)	2000	0.106	0.099	0.205	-.001
5082	S-13	LTV-602	0	0.091	0.068	0.159	
			2000	0.121	0.064	0.185	.026
5084	S-13	LTV-602	0	0.089	0.073	0.162	
		(H2 Catalyst)	2000	0.094	0.073	0.167	.005
5080	S-13	LTV-602	0	0.106	0.089	0.195	
	(strained SP500)		2000	0.159	0.085	0.244	.049
5066	35% PVC	OI #650	0	0.098	0.092	0.190	
		(Lot 1025)	2000	0.100	0.082	0.182	-.008
5076-2	35% PVC	OI #650	0	0.086	0.058	0.144	
		(Lot 276)	2000	0.086	0.050	0.136	-.008
5078	r-TiO$_2$ pigmented		0	0.125	0.119	0.244	
	Kynar		2000	0.265	0.129	0.394	.150

absorption edge than the standard SRC-05 catalyst normally used with LTV-602 based paints.

The problem of instability introduced in the grinding of zinc oxide paints has received considerable attention and is noted in a previous communication on this program (report No. IITRI-C6014-8, Triannual Report). Both an increase in solar absorptance and decrease in ultraviolet stability has been noted as a function of the grind time and such factors as shear, load etc. The yellowing, or increase in solar absorptance, has been attributed to lattice distortion and interstitials brought on by scratching, grinding or compacting zinc oxide under pressure. The analagous problem of mill yellowing has been alleviated by reduction of grind time and ball-to-charge ratios. The problem of a concomitant decrease in stability to ultraviolet irradiation in vacuum, however, has been attributed to either mill contamination or to the accompanying defect structure itself. It appears from the results of test V61 that the loss in stability on grinding may be due to the defect structure. Specimen 5080 is an S-13 paint prepared from SP500 which was ground in an agate mortar and pestle and subsequently "hand-rubbed" into LTV-602 to make the paint. The $\Delta\alpha_s$ of 0.05 for #5080 is more severe than usually observed for S-13 prepared at low catalyst concentration (1 drop SRC-05 per 20 g paint).

B. Owens-Illinois Glass Resin Paints

Perhaps the most encouraging aspects of test V61 are the results obtained on zinc oxide-paints prepared from Owens-Illinois Type 650 Glass Resins (Table 1). Zinc oxide paints at 35% pigment volume concentration (PVC) not only exhibited the greatest stability in α_1 of any coating observed to date but undergo an increase in reflectance in the α_2-region to provide a substantial overall decrease in solar absorptance on irradiation for 2000 ESH.

Like the experimental resin R-9 synthesized in our laboratories, these resins provide reasonably hard, semi-gloss coatings at moderately high PVC's. Unfortunately, however, they require very careful curing schedules and handling procedures and they have been found to be dimensionally unstable at temperatures above 200 to 300°F -- especially when cooled to room temperature and/or liquid nitrogen temperature (immersion).

Type 650 Glass Resin is an ethanol solution of a 100% methyl polysiloxane condensation polymer which requires refrigeration in order to prevent gellation. Although the resins require precuring -- i.e., B-staging -- when used as casting materials, we have found precuring to be difficult in conjunction with their utilization in pigmented coatings. Furthermore, we have found that their physical properties vary from lot to lot in in the limited testing accomplished to date. The properties of paints prepared from various lots and at different pigment volume concentrations are presented in Table 2.

Work thus far has indicated a need for performing curing

Table 2

PROPERTIES OF OWENS-ILLINOIS TYPE 650 GLASS RESIN PAINTS PIGMENTED WITH SP500 ZINC OXIDE

Lot	PVC, %	Curing and Heating Schedule	Finish	Remarks
1004	30	1 hr @ RT + RT to 195°F in 1 hr + 24 hr @ 195°F + 195 to 300°F in ½ hr + ½ hr @ 300°F	Semi-gloss	Smooth, very hard surface after room temperature cure. After the 300°F cure, 60% of the specimens exhibited small cracks. After standing at room temperature for 16 hrs, the remaining 40% of the specimens developed small cracks.
1004	35	16 hrs @ RT + RT to 300°F in 2 hr with IR lamps	Matte	Coating was hard after 16 hours of room temperature cure. Heating with the IR lamps had no effect.
276	35	N/A	N/A	Paints (liquid) gelled in one week at room temperature.
1025	35	N/A	N/A	Paints (liquid) gelled in one week at room temperature.
1025	25	16 hrs @ RT	Gloss	Very hard coating which cracked after the 16 hours at room temperature.
1034	25	16 hrs @ RT	Gloss	Cracked after several hours at room temperature.
1034	30	1 hr @ RT + 1 hr at 140°F + 1 hr at 150°F	Matte	No change was observed at paints were cured (heated). No cracks observed. Cracked when immersed in liquid nitrogen.
1040	25	1 hr @ RT + 1 hr at 140°F + 1 hr @ 150°F.	Gloss	Cracked when cooled to room temperature after heating to 400°F
1040	30	1 hr @ RT + 1 hr @ 140°F + 1 hr @ 150°F	Semi-gloss	Cracked when cooled to room temperature after heating to 400°F.

studies on the Glass Resins. Steps are therefore planned which will utilize a B-staged polymer in the paint grinding process. The excellent optical properties -- both their initial properties and their ultraviolet stability in vacuum -- warrant more extensive examination of the Type 650 Glass Resin system.

C. Silicone Photolysis Studies

1. Oxidative Crosslinked Dimethylpolysiloxane

A flask equipped with a two-way addition tube fitted with a gas-inlet tube and a reflux condenser was employed to crosslink a cyclic polysiloxane after Hyde and Delong[1]. The flask was charged with the cyclic polysiloxanes (pentamer and higher) obtained from the hydrolysis of dimethyldichlorosilane. This material was heated by means of a Woods Metal bath to 220 - 230°C (pot temperature) for 16 hours while a slow current of air was continuously bubbled through the polymer. The resulting crosslinked material was of "jello-like" consistency, and was water-white with an excellent ultraviolet transmission -- 65% at 220 mμ and 40% at 200 mμ (see Figure 1).

Analysis: Calculated for $(CH_3)_2SiO$: C 32.38; H 8.16; Si 37.88
: C 32.34; H 8.06; Si 33.51

This solid polymer will be used in the next photolysis experiment in order that the photolysis can be carried out at or near room temperature.

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[1] J. F. Hyde and R. C. Delong, J. Am. Chem. Soc., <u>63</u>, 1194 (1941)

TRANSMITTANCE OF SILICONE POLYMERS

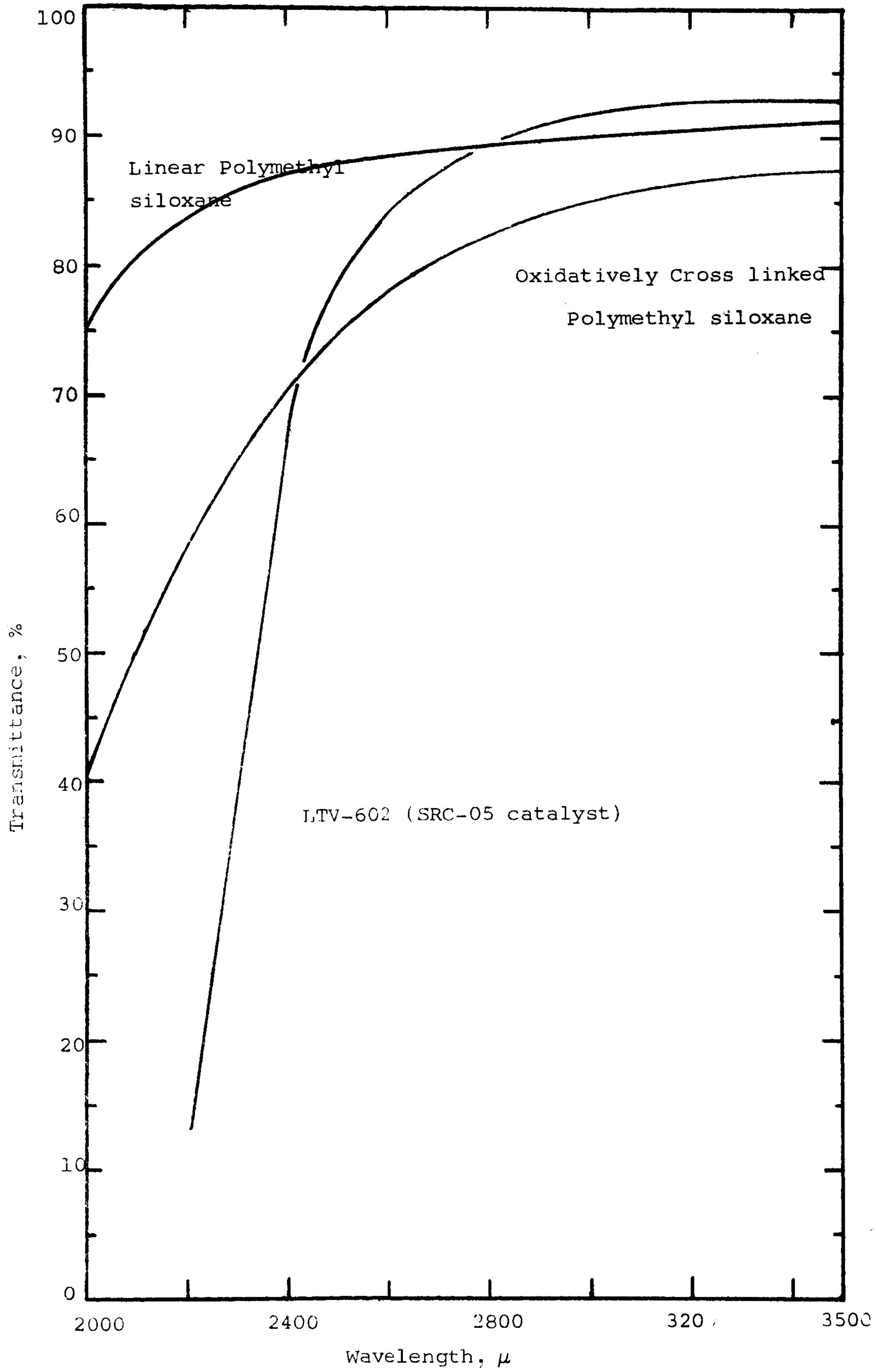

Figure 1

The transmission spectra of both the linear polydimethylsiloxane and the oxidatively crosslinked polymer are compared to LTV 602 cured with SRC-05 in Figure 1.

2. Ultraviolet Photolysis of Linear (Low Molecular Weight) Polydimethylsiloxane

Two duplicate samples in Suprasil quartz tubes were irradiated in liquid nitrogen for 2 hours in the apparatus described by Figure 9 in the last Triannual Report (IITRI-C6014-18). E.S.R. examination of one of the tubes showed no resonance lines. The sample gelled, indicating that crosslinking occurred. The fact that no E.S.R. spectra was obtained and that the specimen showed no visible color indicates that the crosslinking probably occurred by an ionic mechanism rather than free radical at this (LN_2) temperature (see discussion of polysiloxane photolysis in the last Triannual Report, IITRI-C6014-18). Considerable mass spectra data was collected during this irradiation experiment and this data is currently being analyzed.

III. PARTICLE SIZE ANALYSIS*

In recent publications [2,3] the use of a centrifugal photosedimentometer to measure the particle-size distribution of fine powders has been described in detail. The basic principle of the procedure is shown in Figure 2. A hollow disc is partially filled with clear fluid and rotated at the speed at which the sedimentation analysis is to be carried out. The disc is made wholly or partly of transparent materials, so that a beam of light can be shone through it (as shown in Figure 2). When the centrifuge is running steadily, the free surface of the clear fluid is, to the first order of magnitude, a cylinder whose axis coincides with the axis of rotation of the disc.

To carry out the size analysis of a powder, a small amount of a suspension of the powder is injected through the entry port in the top or side plate depending on axis orientation of the disc when steady running conditions have been obtained. Almost immediately, the injected suspension produces a uniform layer over the free surface of the fluid already in the tank. To obtain a stable injected layer,

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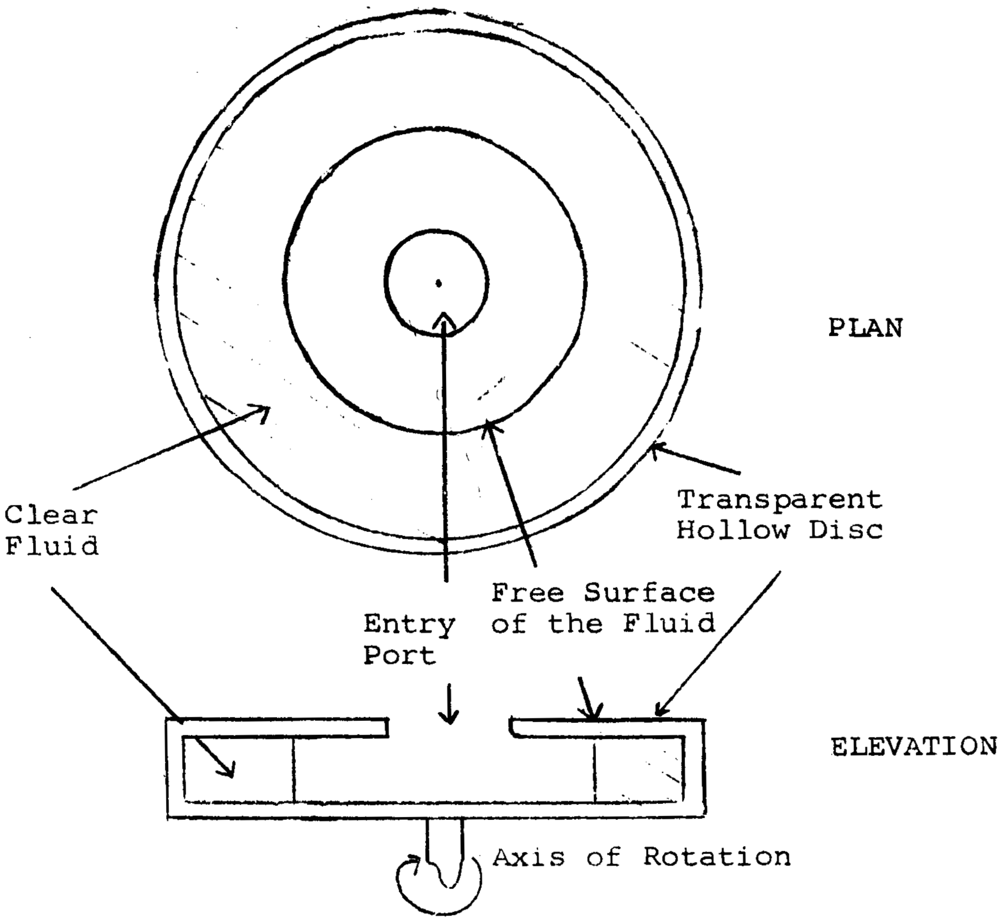
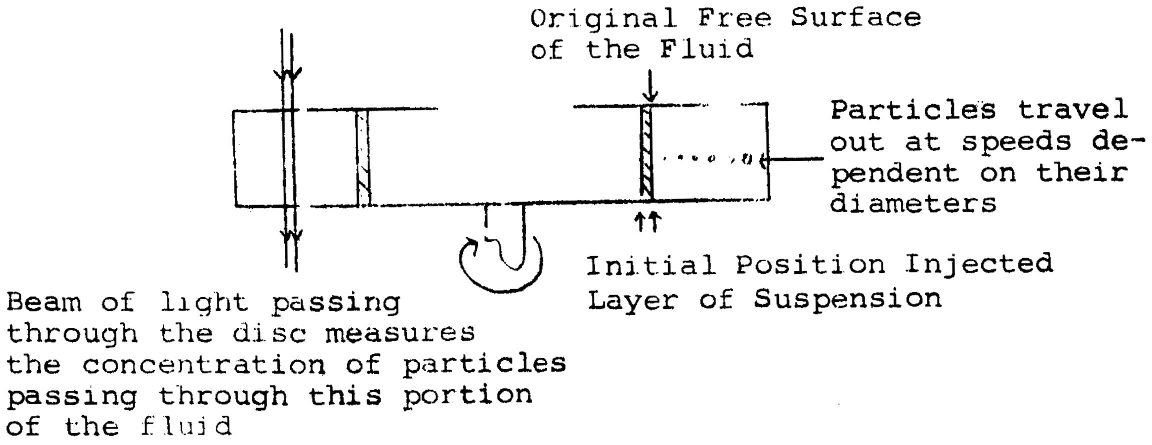
*Accepted for publication in "Paint in Varnish Production" -- entitled "Finger-printing Pigment Dispersions with a Disc Centrifuge" by B. H. Kaye.

[2] Burt, M. W. G. and Kaye, B. H., "Comparison of Particle Size Data Obtained Using a Centrifugal Photosedimentometer With Those From a Centrifugal Pipette Equipment", In Press.

[3] Groves, M. J., Kayer, B. H. and Scarlett, B., "The Size Analysis of Sub-Sieve Powders Using a Centrifugal Photosedimentometer", Brit. Chem. Eng., Nov. 1964.



(a) Before Injection of Suspension



(b) After Injection of Suspension

Figure 2
BASIC SYSTEM OF THE DISC CENTRIFUGAL PHOTOSEDIMENTOMETER

either the solids content of the injected suspension must be very low or other special conditions must be satisfied.[2] In the experimental work described in this paper these conditions were satisfied, and the discussion of the general technique is valid only for stable injected layers. The situation is shown in Figure 2b. The particles in the suspension begin to travel outward from the initial zone; their speeds depend on their diameters. The relationship between the particle diameter and the time required to reach the measurement zone where the beam of light is passed through the disc is expressed by a modified form of Stokes equation:

$$d^2 = \frac{18\eta \log_e S/B}{(\rho_p - \rho_L)\,\omega^2\, t}$$

where η = viscosity of fluid

d = diameter of the sphere of equal falling speed

ρ_p = density of particle

ρ_L = density of liquid

ω = angular speed of the centrifuge

t = time to travel from the injection zone, which is at distance B from the center of rotation, to the measuring zone, which is distance S from the center of rotation.

In the version of the disc centrifuge built at IIT Research Institute*the instrumentation used for measuring the attenuation of the beam is specially arranged so that linear movement on a pen recorder along the transmission axis is directly proportional to the mass of particles present in the beam. (A detailed discussion of the instrumentation is beyond the scope of this article.).

An example of the trace obtained from a pen recorder after an analysis has been carried out is shown in Figure 3. After the suspension is injected, the pen line continued to record maximum transmission until the largest, i.e., fastest sedimenting particles reach the light beam. The pen movement is a measure of the mass concentration of particles in the beam at any given time (shown in Figure 3).

Experimental investigations have shown that when white light is used, analytical results obtained by using a centrifugal photosedimentometer are comparable to those obtained by using the Slater-Cohen disc centrifuge [4] in which particle concentrations are assayed gravimetrically. This is shown by the data given in Table 3.

---

[*] Constructed on an in-house research program in association with Mr. L. Townsend and Mr. M. J. Salkowski

[4] Slater, C. and Cohen, L., "A Centrifugal Particle Size Analyzer", J. Sci. Inst., Vol. 39, pp. 614-617, Dec. 1962.

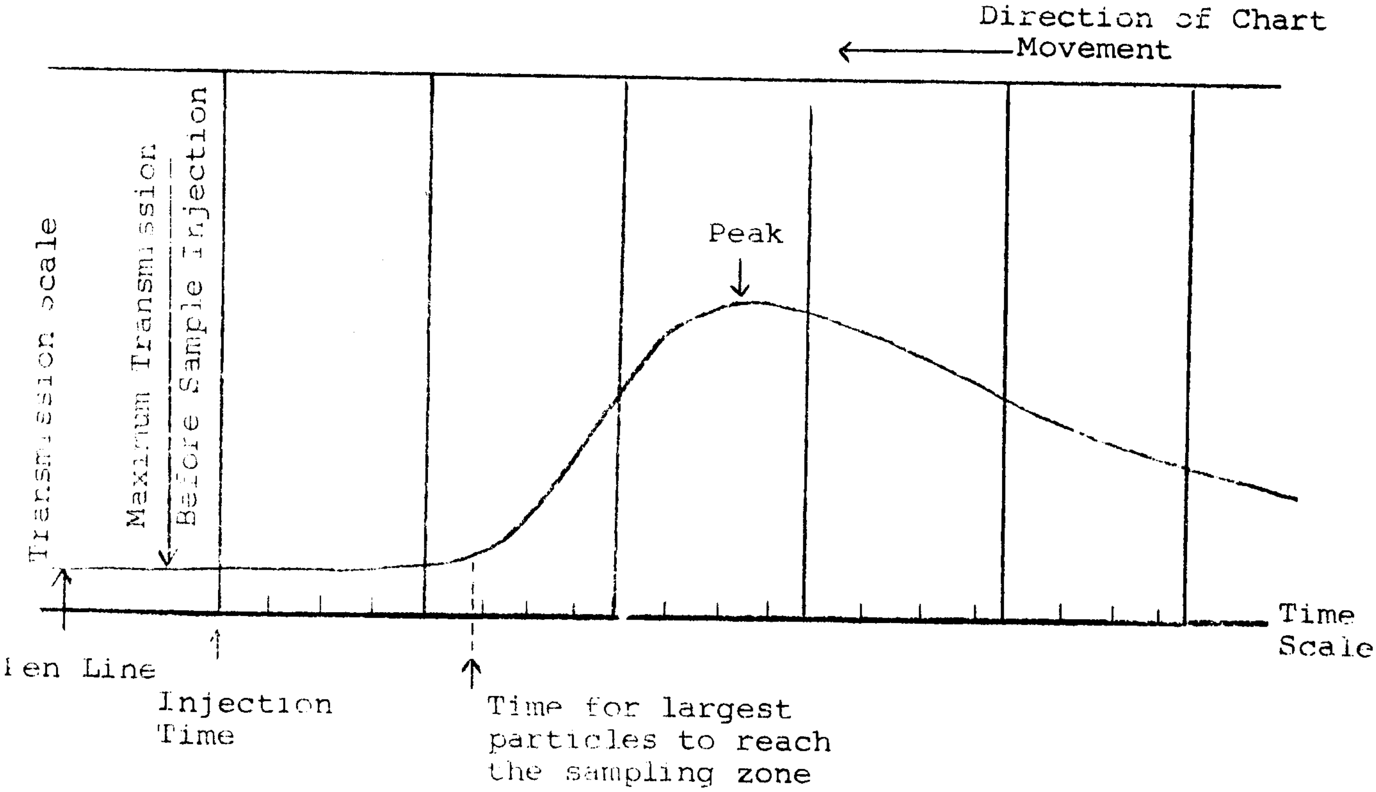


Figure 3

TYPICAL PEN RECORDER TRACE OBTAINED DURING A SIZE ANALYSIS ON THE CENTRIFUGAL DISC PHOTOSEDIMENTOMETER USING THE INJECTED LAYER TECHNIQUE

Table 3

COMPARATIVE DATA FOR TWO CENTRIFUGAL PARTICLE SIZE TECHNIQUES

Zinc Oxide				Barium Sulphate				Titanium Dioxide				Silica			
Stokes Diam.	Cumulative % Undersize			Stokes Diam.	Cumulative % Undersize			Stokes Diam.	Cumulative % Undersize			Stokes Diam.	Cumulative % Undersize		
	A	B	B'		A	B	B'		A	B	B'		A	B	B'
4.0	90	94	95	2.0	96	98	98	2.0	95	98	98	8.0	92	96	95
3.0	87	91	92	1.5	92	95	95	1.5	92	96	95	5.6	80	86	85
2.0	80	85	86	1.0	82	80	79	1.0	81	86	85	4.0	68	71	70
1.5	74	74	75	0.9	77	74	73	0.9	77	79	78	2.8	53	53	52
1.0	61	48	46	0.8	72	67	66	0.8	70	71	70	2.0	36	33	33
0.9	56	40	37	0.7	64	57	56	0.7	61	62	60	1.4	23	16	16
0.8	50	32	28	0.6	53	46	44	0.6	48	48	47	1.0	13	9	10
0.7	43	25	21	0.5	41	34	32	0.5	32	30	31	0.7	6	4	5
0.6	34	18	15	0.4	27	22	20	0.4	17	18	19	0.5	3	2	2
0.5	25	13	10	0.3	13	11	12	0.3	5	10	10				
0.4	16	8	6	0.2	3	5	4	0.2		4	5				
0.3	7	5	3												
0.2	2	2	1												

A = Analysis as determined on Slater Cohen Disc Centrifuge.

B = Analysis on the Kaye Disc Centrifuge.

B' = Analysis on the Kaye Disc Centrifuge.

NB Kaye Disc Centrifuge is an alternative for the centrifugal disc photosedimentometer.

This data abstracted from a paper by M. G. W. Burt and B. H. Kaye to be published soon.

The data for the Cohen Slater disc centrifuge is published by permission of the management of Simon Carves, Ltd.

The pen trace shown in Figure 3 can be used directly in several powder technology problems, and as a result it has been given a name of its own. It has become the practice among workers using the equipment to call this trace the fingerprint of the suspension. This may appear to be an exotic name, but it arises from the fact that if the purpose of the analysis is control of a manufacturing process, all that is required is a comparison of the trace of the manufactured powder with the traces previously obtained for acceptable and unacceptable powders obtained by using identical analytical conditions. If the product trace coincides with that of the acceptable powder trace, the process is functioning satisfactorily. In principle, the characteristics of the fingerprint could be applied to automatic control of a manufacturing process. The fingerprint also yields the following direct information on the characteristics of the injected suspension.

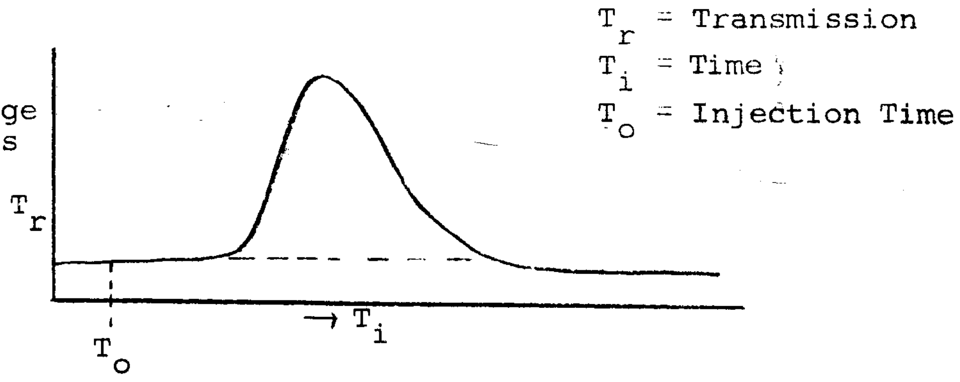
(1) The time which elapses before the pen starts to move is a direct indication of the size of the largest particle present in suspension.

(2) The peak of the trace shows the location of the particle-size group with the largest weight percentage of the particles in it.

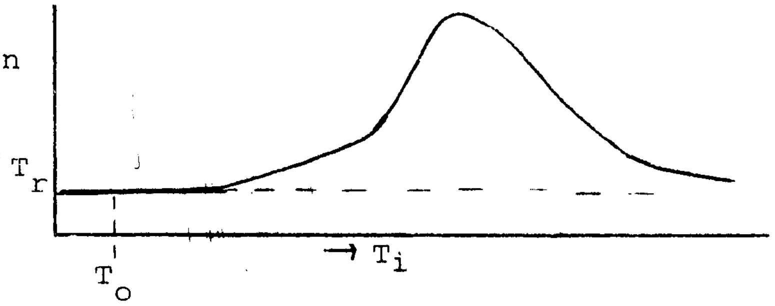
(3) The shape of the trace indicates the spread of particle sizes present in the suspension.

This latter point can be more fully appreciated by considering Figure 4 in which different types of traces are illustrated.

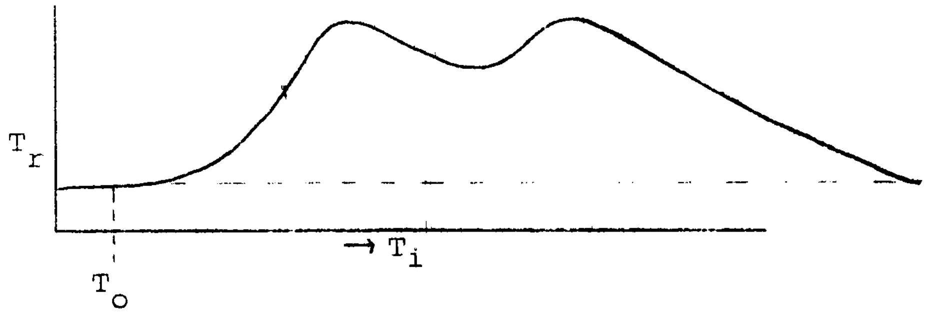
a. Only a relatively short range of particle sizes present in suspension



b. A few larger sized particles but major portion of particles in the smaller particle size range



c. Bimodal Distribution



d. Suspension contains large quantity of fines

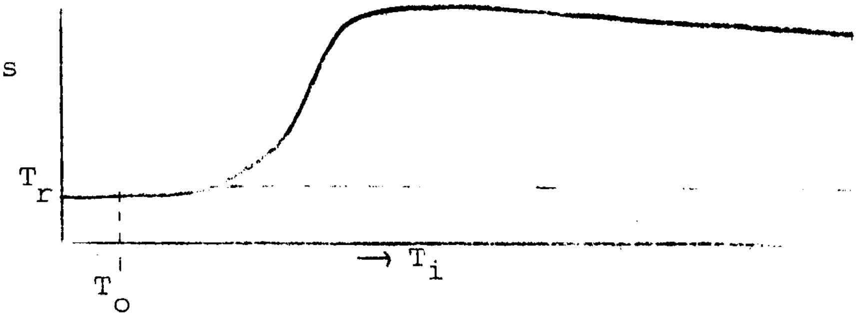


Figure 4
DIFFERENT TYPES OF FINGERPRINT TRACES

The rapidity with which information can be obtained from the pen trace is illustrated by the following experiment. A suspension of zinc oxide powder in water was prepared in four different ways. Four equal-weight portions of the same zinc oxide pigment were placed in four identical glass jars, which were 2 in. high and 2 in. in diameter. Two of the jars were half filled with 1/4-in. ceramic balls. Water was added to all jars until the surface was 1/4-in. below the top of the jar. A quantity (0.1 gm) of Alconox dispersing agent was added to one of the jars containing balls and to one containing pigment and water only. The four jars were sealed and placed on slowly rotating rollers for 16 hr. A sample from each jar was analyzed on the centrifuge. The four traces obtained are shown in Figure 5. From these four traces the following information could be obtained.

(1) The presence of the ceramic balls had no significant effect on the dispersion obtained.

(2) The dispersing agent considerably changed the largest particle present in suspension and the percentage of very fine particles.

(3) The use of the Alconox dispersant resulted in greater deagglomeration than did the mechanical action of the balls.

In the case of this particular experiment, this information was sufficient so that we could proceed to the next stage of the investigation. However, the particle-size distributions

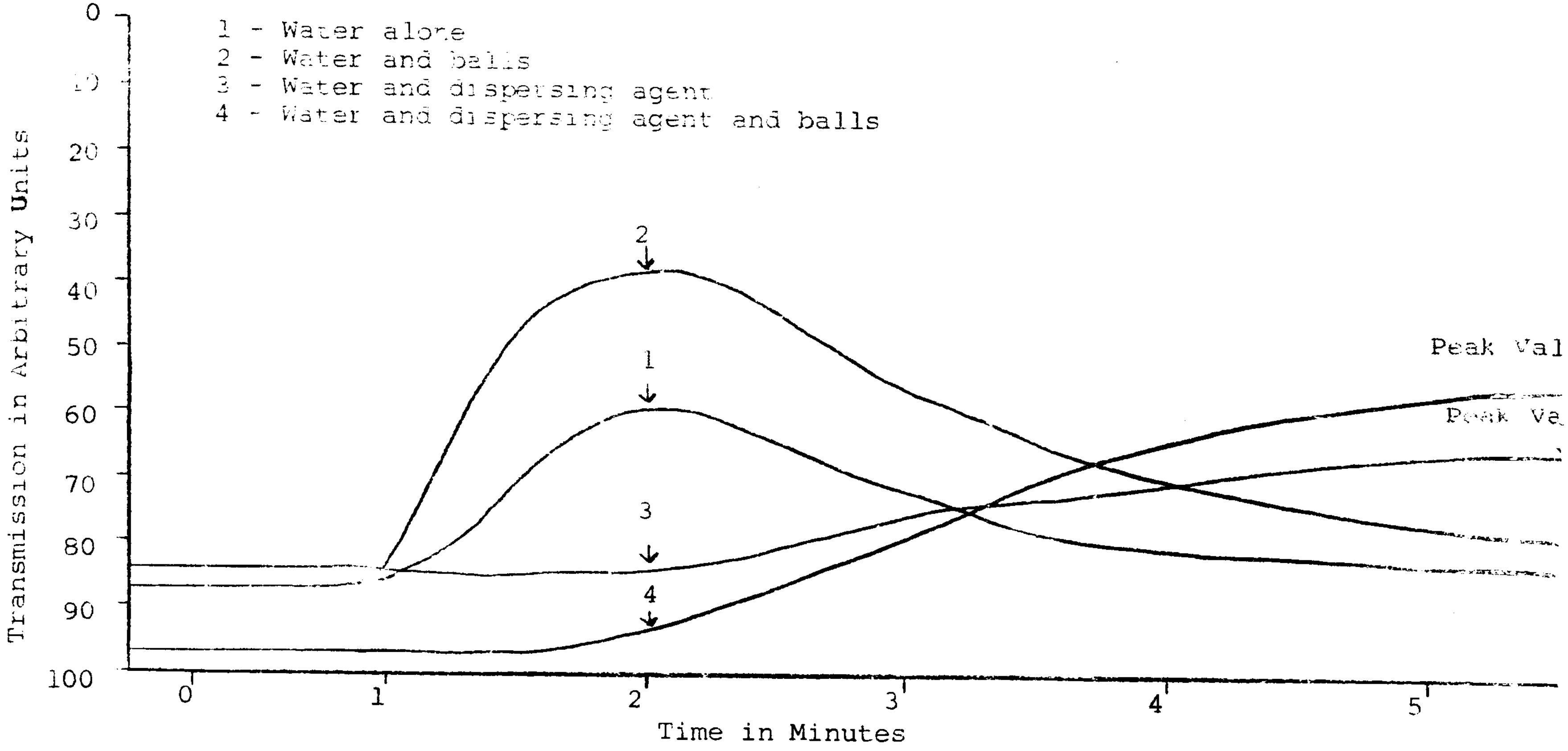


Figure 5

RECORDER PEN TRACES FOR THE FOUR SUSPENSIONS of the particles in suspension were calculated from these traces and are given in Figures 6 to 9. Comparison of these distributions show vividly that the effective factor in improving the dispersion was the addition of the dispersing agent, but this information was already displayed qualitatively in the pen recorder traces.

The size distribution of several other zinc oxide pigment dispersions was subsequently measured. These consisted of Alconox dispersions of (a) Calcined SP 500, (b) Eagle Picher Nr. 730, (c) American Zinc 55L0, and (d) 2.1 Micron SP 500. The size distributions are plotted accordingly in Figure 10.

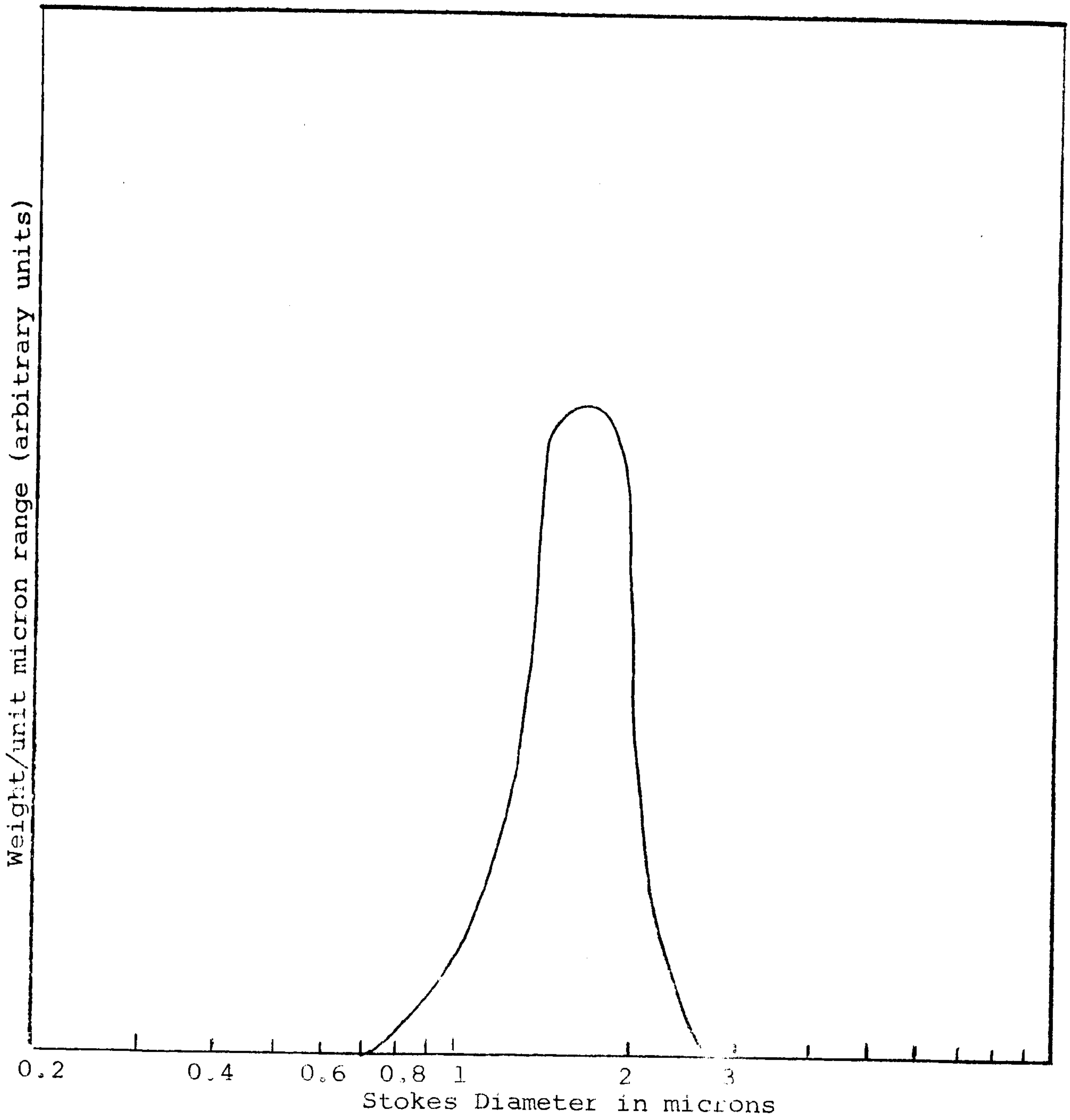


Figure 6

SIZE DISTRIBUTION OF ZINC OXIDE PARTICLES DISPERSED IN WATER ONLY

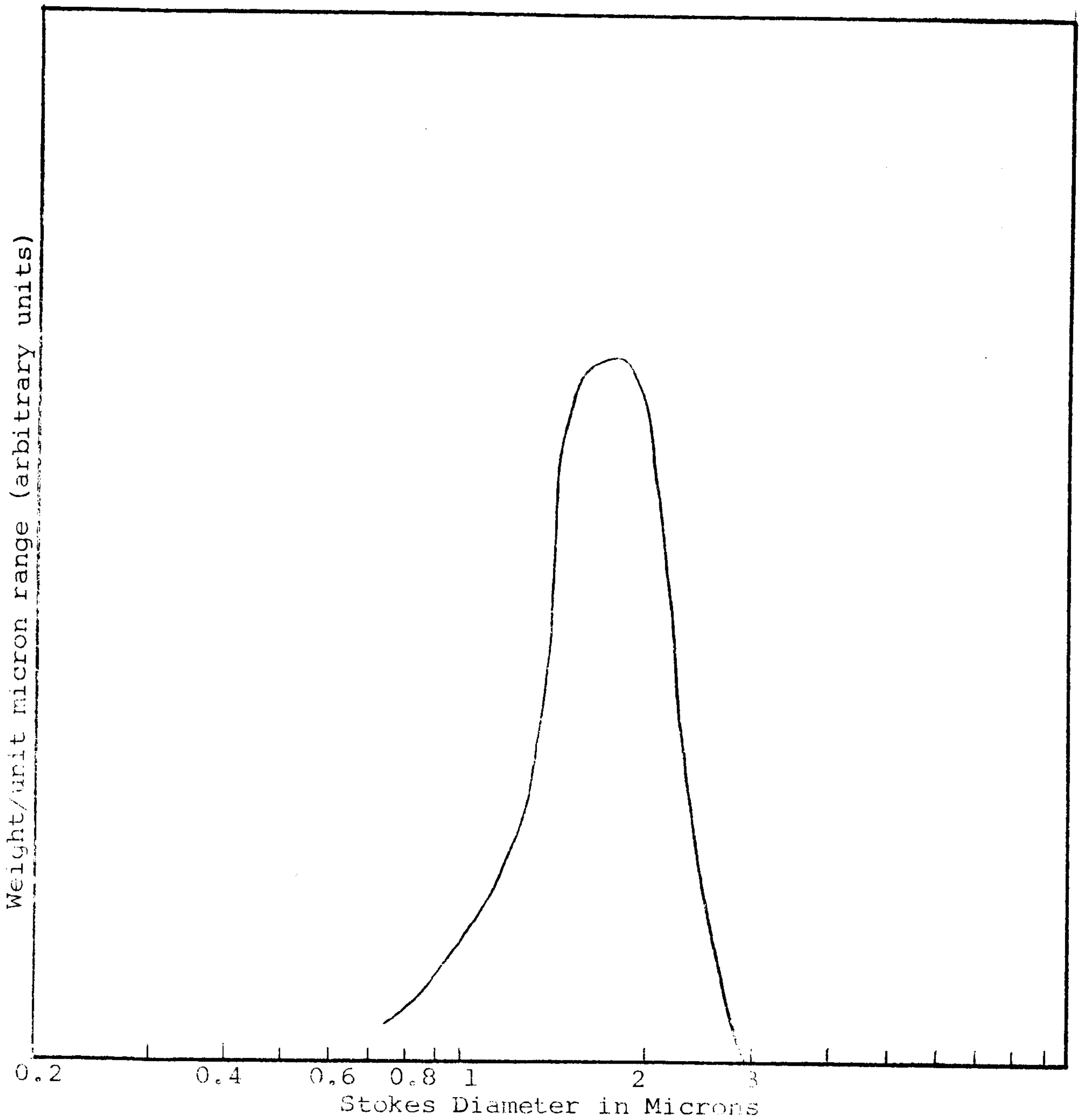


Figure 7

SIZE DISTRIBUTION OF ZINC OXIDE PARTICLES
DISPERSED WITH AID OF BALLS ONLY

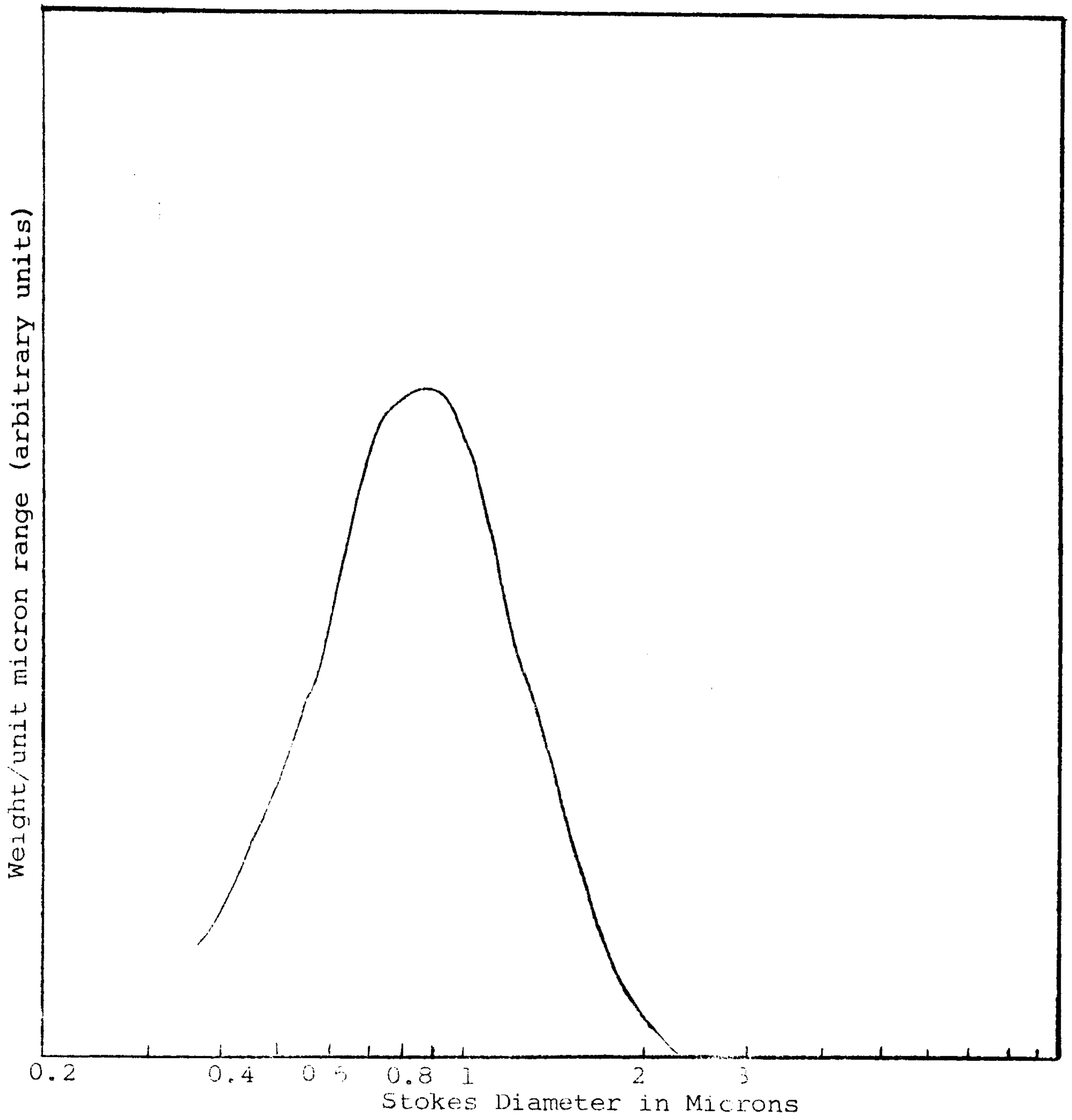


Figure 8

SIZE DISTRIBUTION OF ZINC OXIDE PARTICLES
DISPERSED WITH THE AID OF A WETTING AGENT ONLY

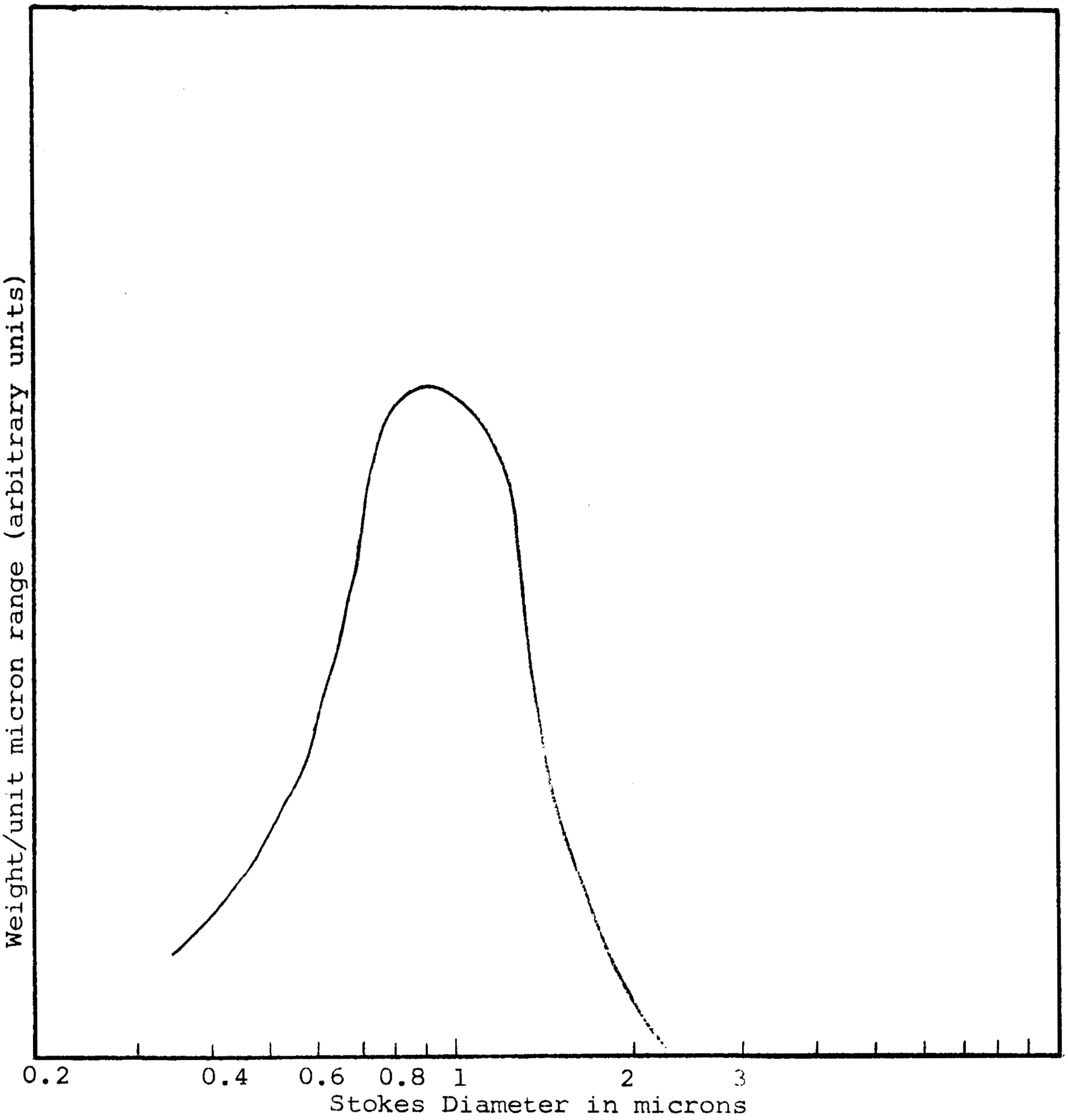


Figure 9

SIZE DISTRIBUTION OF ZINC OXIDE PARTICLES
DISPERSED WITH THE AID OF BALLS AND A WETTING AGENT

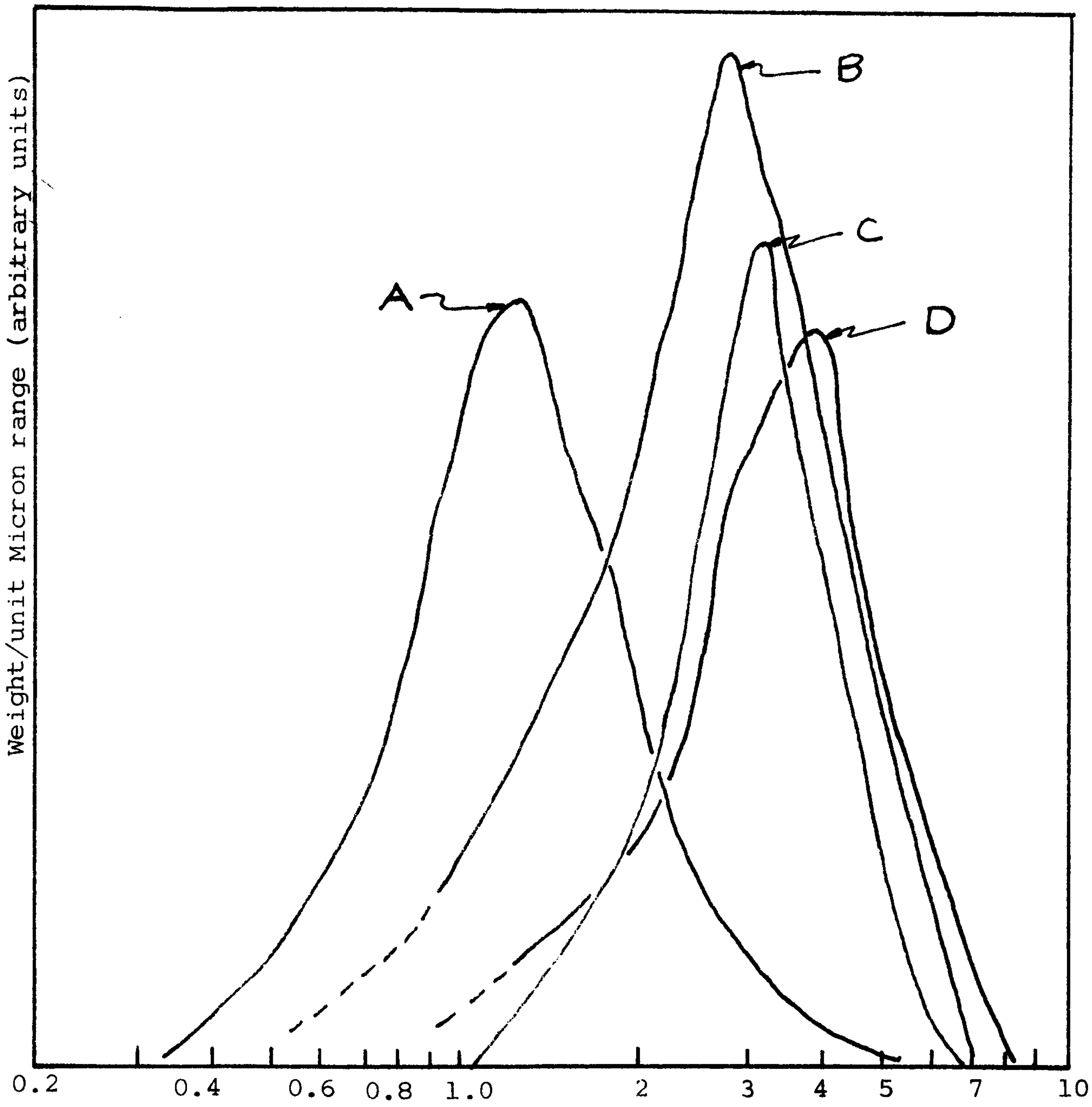


Figure 10

SIZE DISTRIBUTION OF SEVERAL ZINC
OXIDE PIGMENT DISPERSIONS

APPENDIX

The optical densities of various paint constituents are presented in Figures 11 through 15. The pertinent parameters associated with these measurements are presented in Table 4.

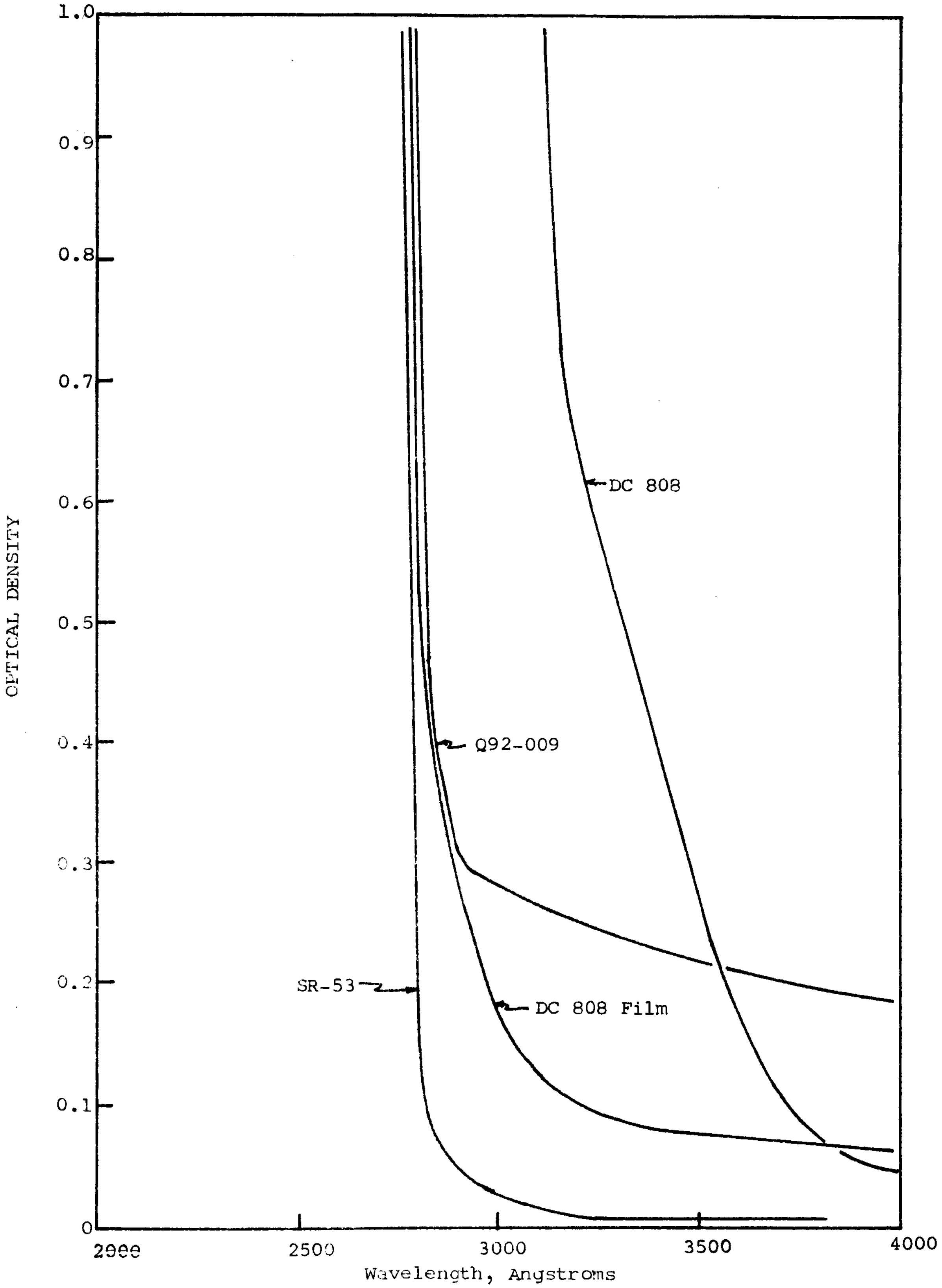


Figure 11

OPTICAL DENSITY OF SEVERAL SILICONE MATERIALS

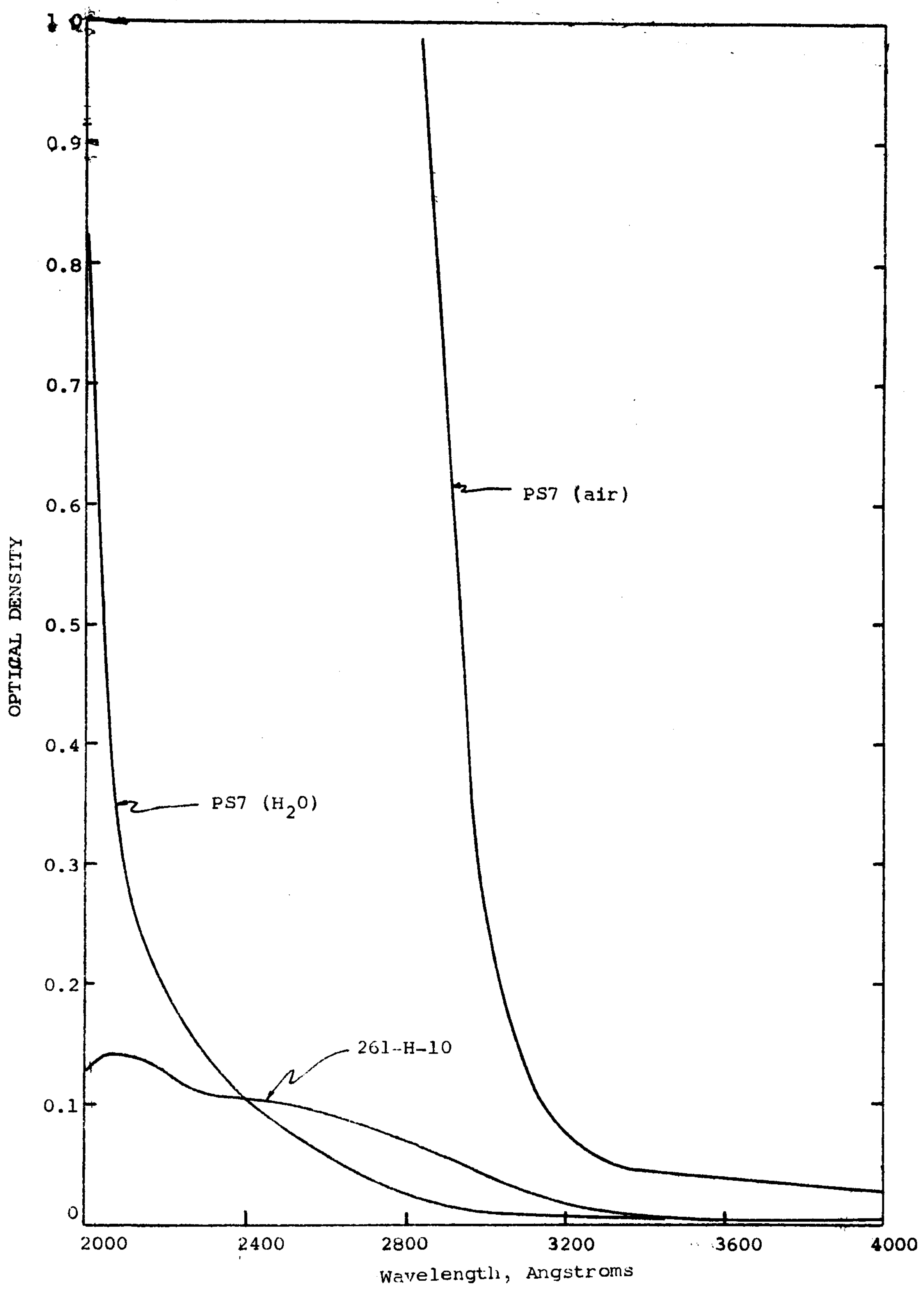


Figure 12

OPTICAL DENSITY OF INORGANIC BINDERS

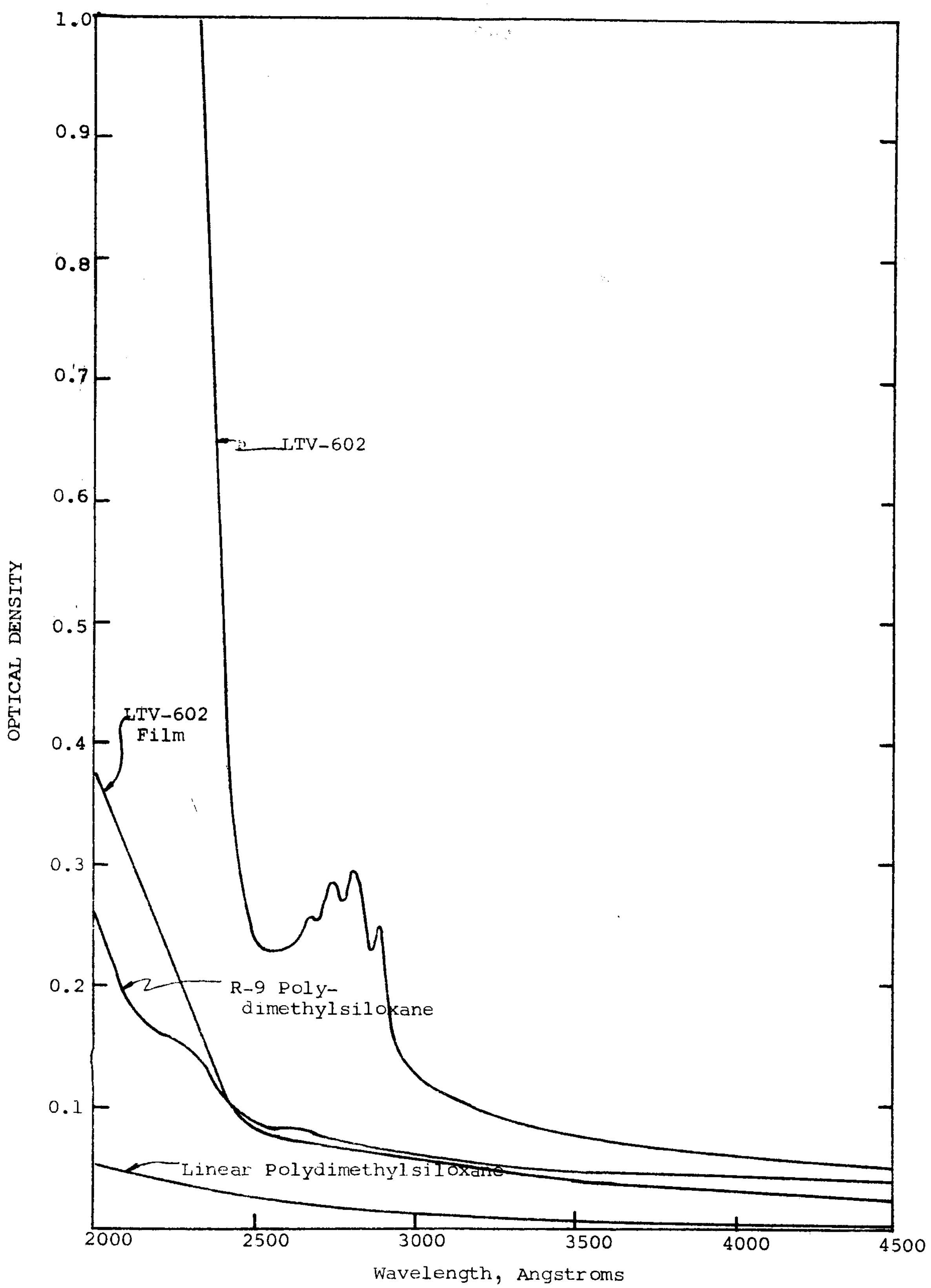
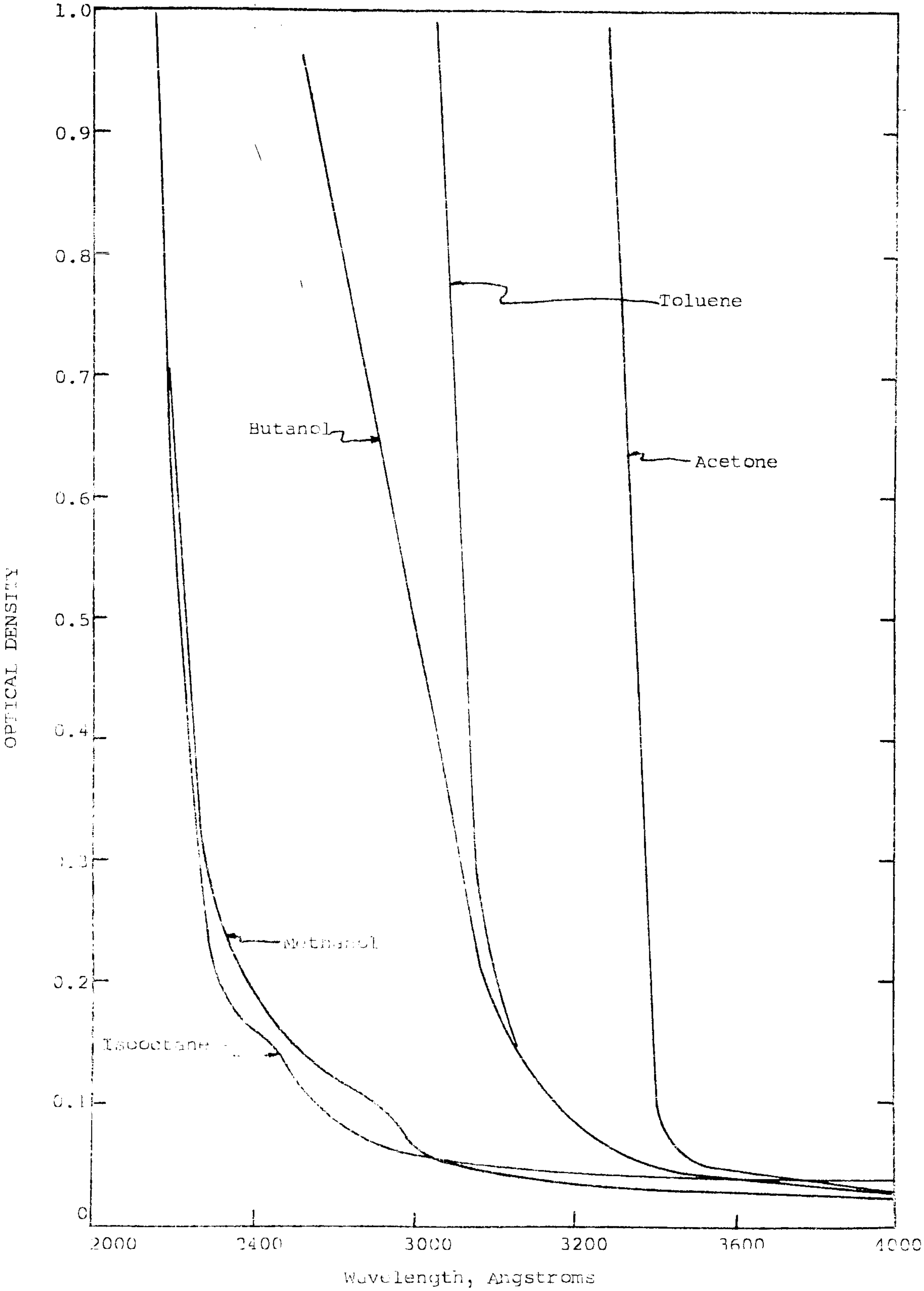


Figure 13

OPTICAL DENSITY OF METHYL SILICONE POLYMERS



OPTICAL DENSITY OF SEVERAL SOLVENTS (COMMERCIAL GRADE)

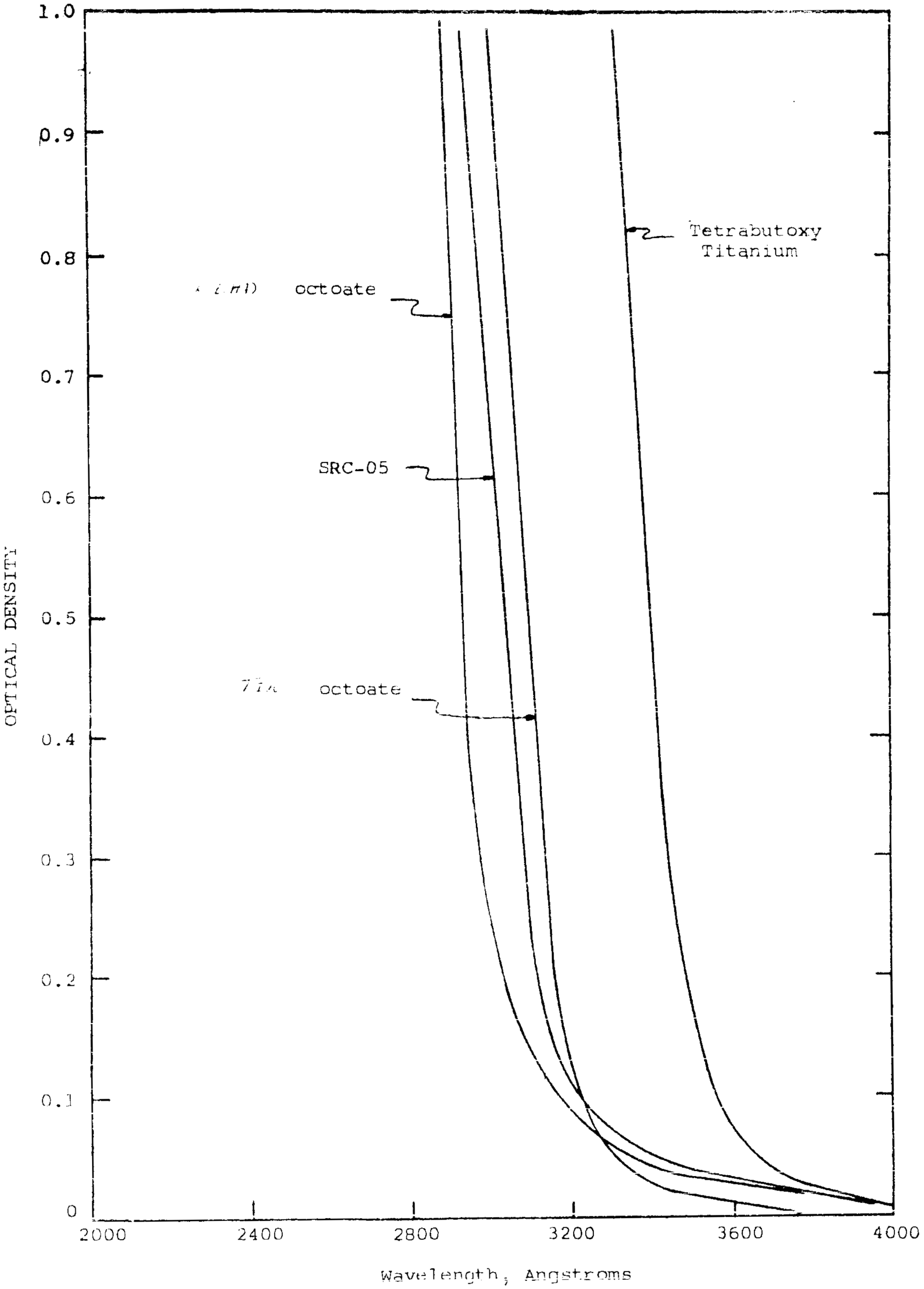


Figure 15

OPTICAL DENSITY OF SEVERAL SILICONE CURING AGENTS

Table 4

PERTINENT PARAMETERS FOR OPTICAL DENSITY MEASUREMENTS

Figure No	Material	Solvent	Support	Reference	Remarks
11	SR-53 silicone resin	isooctane	1-cm Quartz cell	isooctane	General Electric
11	Dow Corning 808 resin	dioxane	1-cm Quartz cell	dioxane	2 drops/ 5cc
11	Dow Corning 808 resin	-	suprasil slide	air	1-mil film (300°F)
11	Dow Corning Q92-009	isooctane	1-cm Quartz cell	isooctane	dilute
12	PS7 potassium silicate	water	1-cm Quartz cell	air	concentrated
12	PS7 potassium silicate	water	1-cm Quartz cell	water	dilute
12	261-H-10 Alkaline phosphate	water	1-cm Quartz cell	water	dilute
13	LTV-602 silicone	isooctane	1-cm Quartz cell	isooctane	dilute
13	LTV-602 silicone		suprasil slide	air	wet film/ no catalys
13	Linear polydimethylsiloxane	isooctane	1-cm Quartz cell	isooctane	See Figure
13	Polymethylsiloxane (R-9 type)		suprasil slide	air	wet film
14	Isooctane	-	1-cm Quartz cell	air	-
14	Butanol	-	1-cm Quartz cell	air	-
14	Methanol	-	1-cm Quartz cell	air	-
14	Toluene	-	1-cm Quartz cell	air	-
14	Acetone	-	1-cm Quartz cell	air	-
15	SRC-05 catalyst (LTV-602	isooctane	1-cm Quartz cell	isooctane	General Electric
15	Lead octoate	conc.	1-cm Quartz cell	air	Nuodex
15	Tin octoate	conc.	1-cm Quartz cell	air	Nuodex
15	Tetrabutoxy Titanium (TBT)	isooctane	1-cm Quartz cell	isooctane	duPont

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