Serial No. KN 52-2

Copy No.

E. I. DU PONT DE NEMOURS & COMPANY
256 VANDERPOOL STREET
NEWARK, NEW JERSEY

52-2 [illegible] file

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

A NEW APPLICATION OF LIGHT TRANSMISSION IN FOLLOWING PARTICLE SIZE REDUCTION

II. APPLICATION TO PIGMENTS OTHER THAN CPC

Period Covered MARCH 1951 TO DECEMBER 1951

FILE: 144

DATE: 2-25-52

NJ 14071

Serial No.

Copy No. 5. [illegible]

E. I. DU PONT DE NEMOURS & COMPANY
KREBS PIGMENTS DEPARTMENT
236 VANDERPOOL STREET
NEWARK NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

[illegible]

Period Covered [illegible] 20 [illegible] 51

FILE:

DATE: [illegible]-52

Copy No. 3. Library File
(144)

[illegible]

[illegible]

MARCH 1951 TO DECEMBER 1951

Charge: 1101-11-1 - 25%
1101-11-6 - 10%
1101-11-36 - 50%
[illegible]-11-[illegible] - 15%

SUBMITTED BY: A. R. HANKE　　DATE SUBMITTED: 2/2[illegible]

APPROVED BY : B. H. PERKINS　　DATE ISSUED: 2/25/52

DUP050068890

[illegible]

[illegible] in detail in KM-51-43. [illegible] m [illegible] pigments other than [illegible]

[illegible]

D [illegible]

[illegible]

[illegible] pigments to which this [illegible]

) [illegible] the [illegible] and [illegible] 5 [illegible]

) [illegible] Cl. Anthranilic Acid [illegible] (Ba) [illegible]

) Dianisidine Blue [illegible]

[illegible]) Auric Brown (F-4-P).

[illegible]) Molybdate Orange [illegible]

2. In all cases, the spectrophotometric curve [illegible] in such fashion as to give a smaller [illegible] index [illegible] treatment given the pigment or the [illegible] give a smaller pigment particle or a better dispersion. [illegible] of electron micrographs and specific surface measurements by nitrogen adsorption are shown to correlate with this index.

E[illegible]AL

[illegible] spectrophotometric curves.

The general technique of obtaining spectrophotometric curves [illegible] to KM-51-43 [illegible] will not be repeated here. In all [illegible] that all measurements were made on the [illegible] recording [illegible] photometer" using [illegible] light path cells placed farther away than [illegible] from the integrating sphere to minimize the error introduced by measuring some scattered light as transmitted light.

DUP050068891

[illegible]

[illegible] the [illegible] of the [illegible] visible portion of the [illegible], the particle size index is the [illegible] the [illegible] spectrophotometer. [illegible] size index [illegible]

Index = [illegible] / Optical [illegible]

[illegible] 100 is used simply to bring the [illegible] in the index.

[illegible] the extended [illegible] the effect of [illegible] ting [illegible] transmission measurements, [illegible] of the slurry to about 30 ml [illegible] ience, visual inspection of the [illegible] concentration is about right. [illegible] th in [illegible] light transmission is [illegible] solution is used in the comp[illegible] or of the "[illegible]" solution.

In this case the milled sample has an index of [illegible] for the original pigment. Note also that with [illegible] the wave-length at maximum light [illegible] wave-lengths. This is another general [illegible] served to a greater or lesser [illegible]

Fig. 2 illustrates dispersions of RT-525-D in [illegible] RT-525-D is the manganese salt of Red 2B + 20%. When [illegible] non-aqueous dispersion such as this, the technique is to [illegible] with a medium that will keep the pigment dispersed but which will not dissolve it. In this case, amyl acetate was ideally suited. The curve indicating a small particle size is that of the standard, whereas the large particle size curve is that of a laboratory preparation giving a pigment of larger average particle size in the enamel. The particle size index of the standard pigment is 15.1 as against 60.6 for the laboratory preparation. Average particle diameter calculated by specific surface measurement on these two pigments is 0.12 microns for the standard and 0.27 microns for the laboratory sample. This calculation assumes a specific gravity of 1.6 and is made using the equation in the footnote. Electron micrographs of these two samples are also shown in the same figure. No detailed study was made of the relationship [illegible]

[illegible] Specific surface measurements were obtained from the [illegible] [illegible] Station using the nitrogen adsorption technique. Particle [illegible] was calculated, assuming spheres, from the equation,

$$\text{Particle Diameter (microns)} = \frac{6}{\text{Sp. Surf. (sq. meters/gram)} \times \text{Sp. Gr.}}$$

[illegible] surface measurements, electron micrograph [illegible] particle size index but it can be [illegible] the proper [illegible] [illegible].

[illegible] 4.63 [illegible]

Fig. 3 shows the application of this [illegible] V.M. & P. [illegible] the importance of the [illegible] particle size index [illegible] by this light [illegible].

The vehicle solids in this [illegible] V.M. & P. [illegible] sufficient [illegible] solids. [illegible] dispersing action on the pigment. [illegible] Acetone and this is reflected in the [illegible] the [illegible] dispersion as against 15.2 for the [illegible].

[illegible] Dispersion [illegible]

This illustrates a dispersing technique [illegible] of [illegible] micrographs. [illegible] a [illegible] glass [illegible] solution [illegible] in T-3601 [illegible]. The resulting paste is worked with a spatula [illegible]. As the paste gets too thick due to solvent evaporation, T-3601 [illegible] drop at a time and the [illegible] of the spatula [illegible] continued. After 5 minutes, a [illegible] proper dilution with T-3601 for [illegible] further dilution is made with T-3601 [illegible] concentration for light transmission measurements.

Fig. 4 shows light transmission curves and electron micrographs of [illegible]-283-D, SD-83044 as compared to a laboratory sample of [illegible] particle size. As can be seen, there are two minima of about [illegible] magnitude. This allows a choice of minima in calculating the [illegible]. In this case, if the shorter wave-length minimum had been used, [illegible] little difference would be shown between the two pigments, [illegible] longer wave-length minimum is chosen and the particle size index [illegible] calculated from the equation:

$$\text{Particle Size Index} = \frac{\text{Minimum optical density (455 m}\mu\text{)}}{\text{Maximum optical density (varies around [illegible])}}$$

This makes the particle size index 12.8 for the standard and 16.[illegible] for the experimental pigment.

[illegible] T-3601 is made up of the following:

Butyl Acetate . . .	37.0%
Toluol.	35.0%
Ethyl Acetate . . .	12.5%
Butyl Alcohol . . .	12.0%
Ethyl Alcohol(23A).	3.5%

[illegible] sample. The calculation [illegible] on p. 2 using a specific gravity of 1.3[illegible].

[illegible] it can be seen that [illegible] index is not [illegible] size estimate, it is well to [illegible] difficult to obtain but [illegible] particle size index is [illegible] sur[illegible] it [illegible] of [illegible].

[illegible]

[illegible] brown pigment [illegible] visible [illegible] spectrum. The transmission curve [illegible] blue end to the red end. In this [illegible] will be had by taking [illegible] spectrum. Precision will [illegible] in regions where the [illegible] with [illegible] length, but even so, sufficient precision is poss[illegible] index.

Fig. 5 illustrates [illegible] lacquers dispersed in [illegible]. Both light transmission curves and electron micrographs are [illegible]. The curve labeled "large" has an index of 5.5 and the curve [illegible] "small" has an index of 3.1. The index in this case is def[illegible] equation,

$$\text{Particle Size Index} = \frac{\text{Optical Density at [illegible]}}{\text{Optical Density at [illegible]}}$$

There is no assur[illegible] that the degree of disp[illegible] both the electron micrograph dispersion[illegible] dispersions [illegible] the appearance of the [illegible] the particle size index.

F. [illegible] to Molybdate Orange [illegible]

Molybdate orange pigment has a larger particle size and larger specific gravity than those pigments previously discussed. This means [illegible] and [illegible] can [illegible] the [illegible] transmission. [illegible] adequate [illegible] has not quite been satisfactorily [illegible]. [illegible] it is [illegible] of the light transmission curves [illegible] diagram [illegible] the same principles apply.

[illegible] light transmission [illegible] to [illegible] The standard [illegible] 2500 has a [illegible] the specially treated sample [illegible] in this case is defined by the equation,

Particle Size Index = [illegible]

[illegible] surface measurements [illegible] diameter of 0.13 microns [illegible] The diameter was calculated using the formula in the [illegible] and a specific gravity of [illegible].6.

Again there is perfect correlation but the difference [illegible] of the electron micrographs [illegible] differences in the specific surface [illegible] size index. This [illegible] measuring [illegible] of affairs [illegible] is [illegible] in the case of [illegible] particles [illegible] are [illegible] single [illegible] measured by a nitrogen adsorption [illegible] micrograph of the experimental sample shows a [illegible] which may well account for the difference in particle size [illegible] the specific surface measurements.

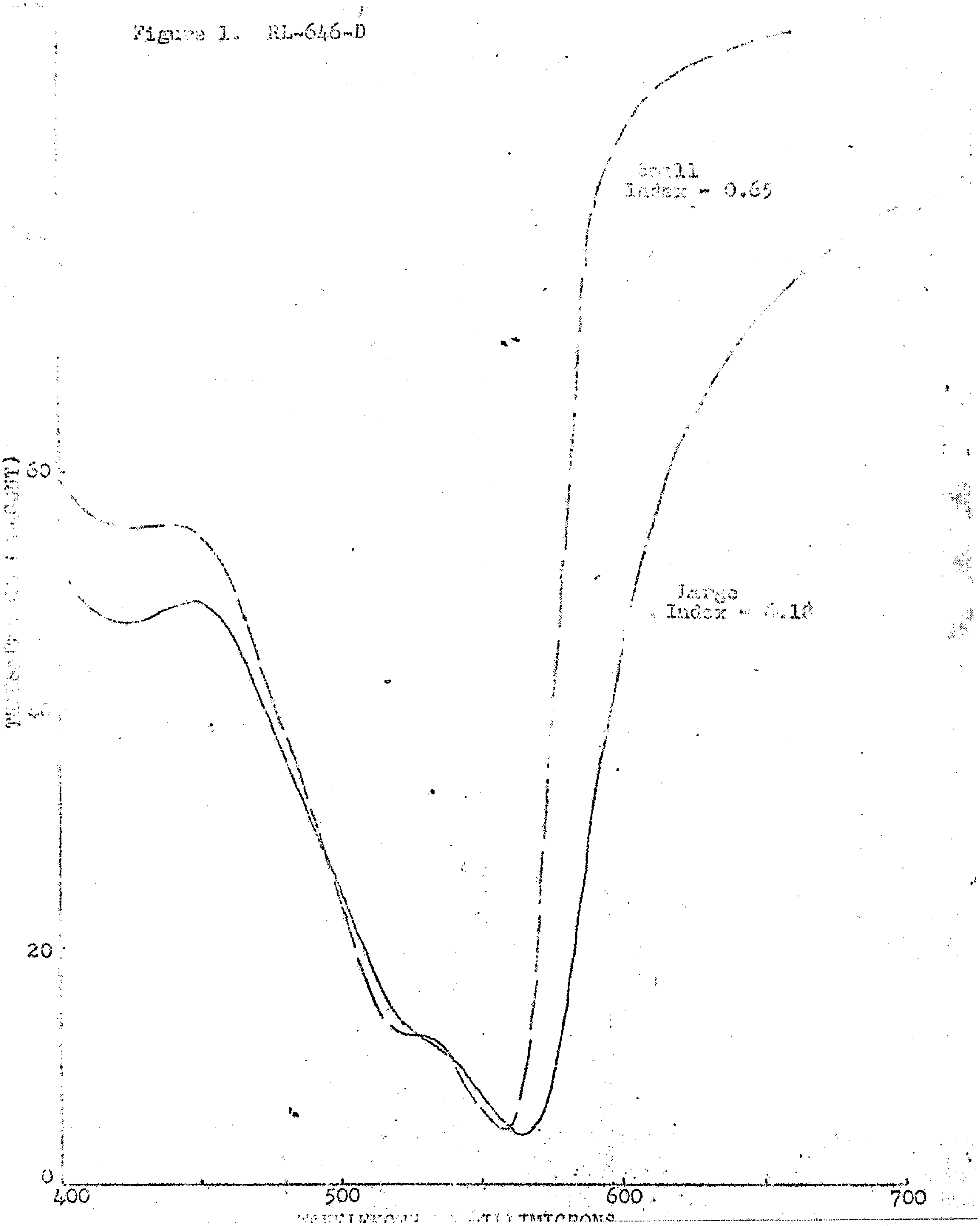
IV. [illegible]URE PROGRAM

As time permits some work is planned to [illegible] the light transmission measurements over very small light paths allowing a more direct comparison of the results obtained by the electron microscope and light transmission.

V. PATENT SITUATION

No action is being taken.

Figure 1. RL-646-D



DUP050068896

RT-525-D

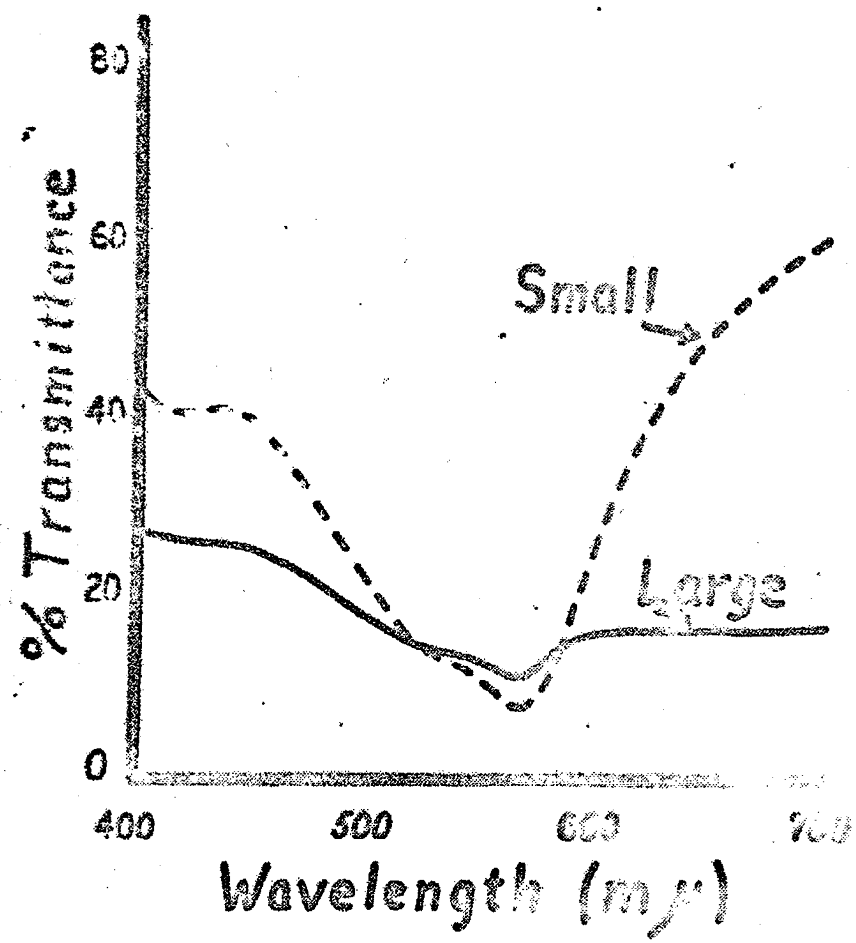


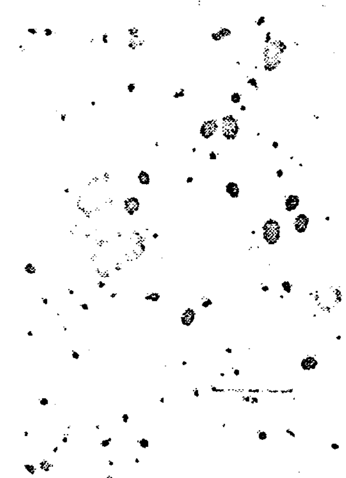
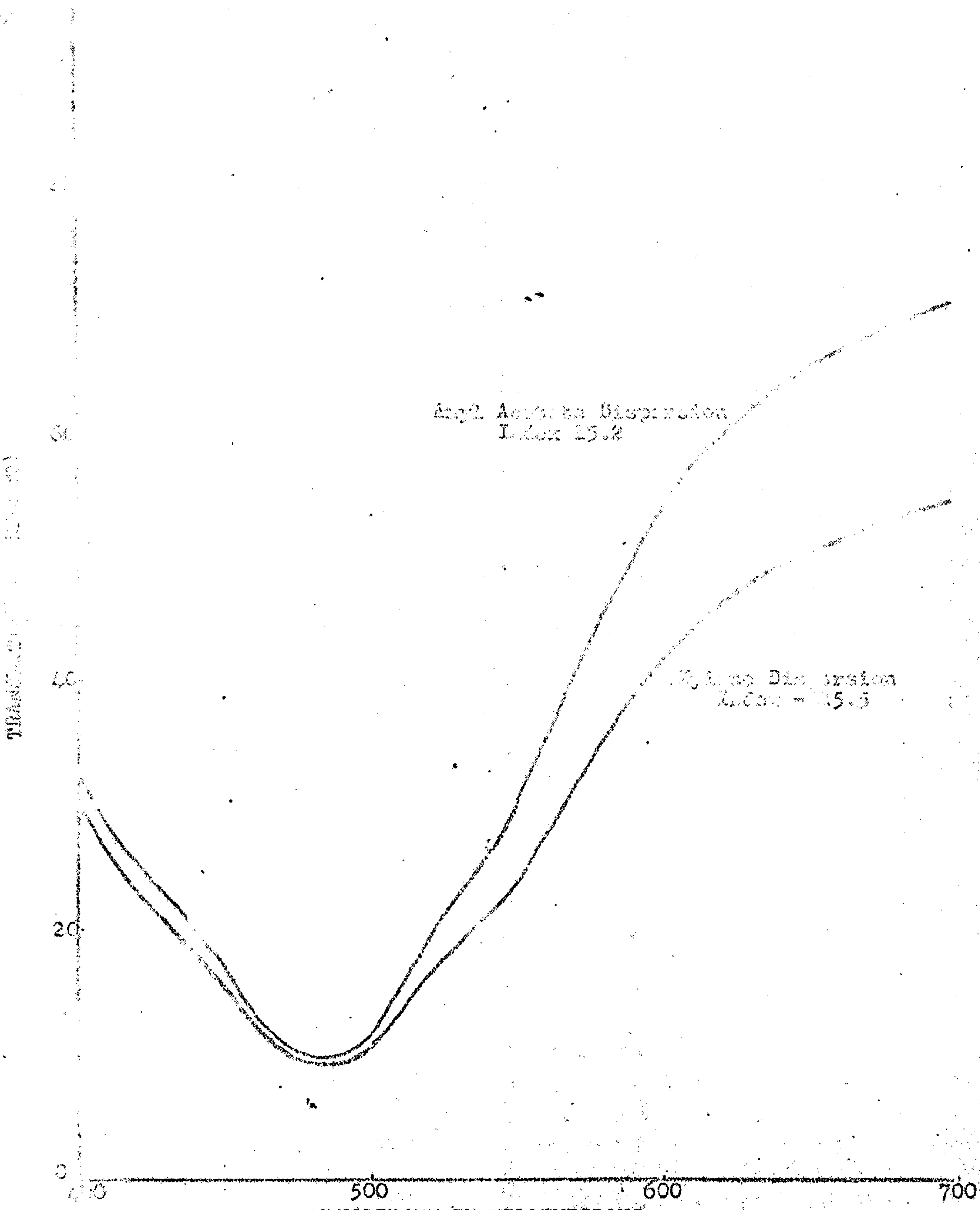
Figure	Dia. (μ)	Index
 Small	0.12	19.1
 Large	0.27	80.8

Figure 3. RT-508-D



BT-283-D

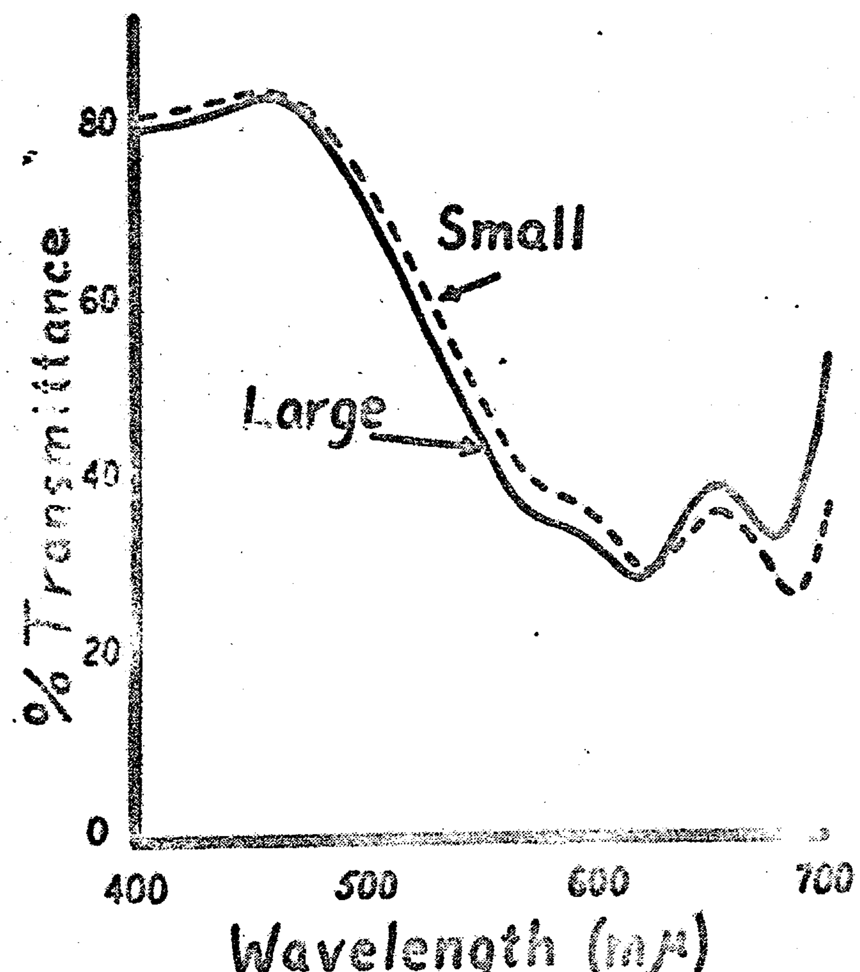


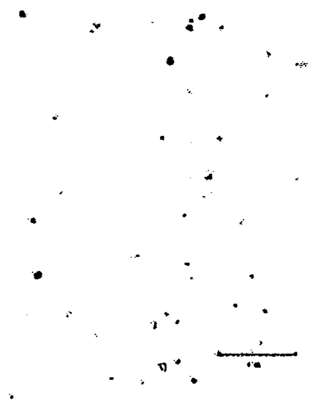
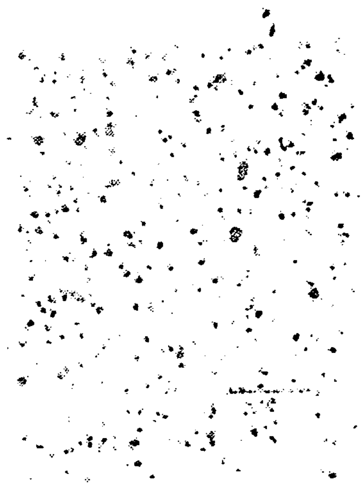
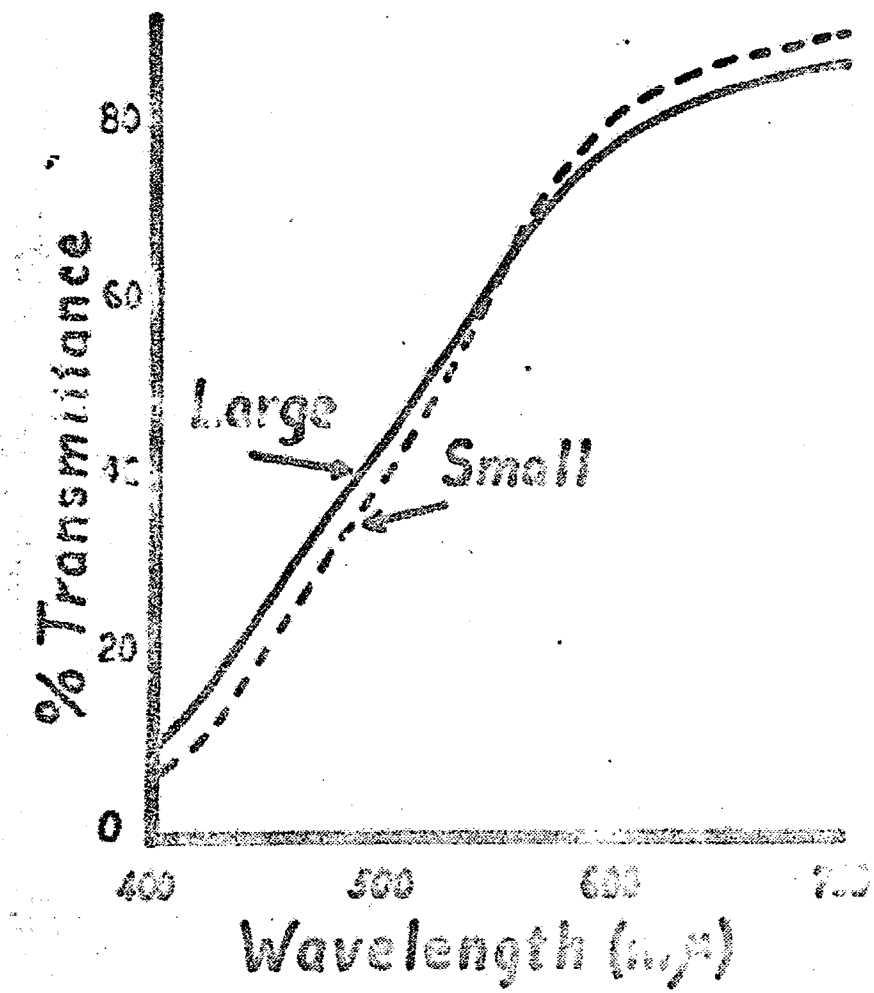
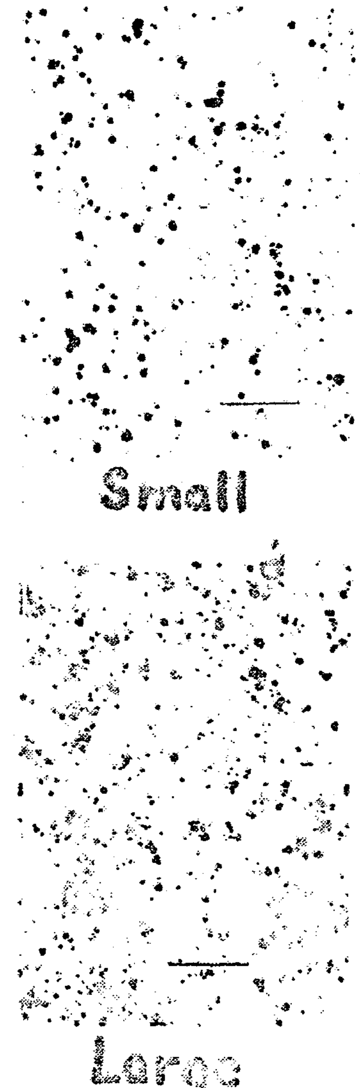
Figure	(μ)	Index
 Small	0.08	12.8
 Large	0.65	16.5

Figure [illegible]

Index

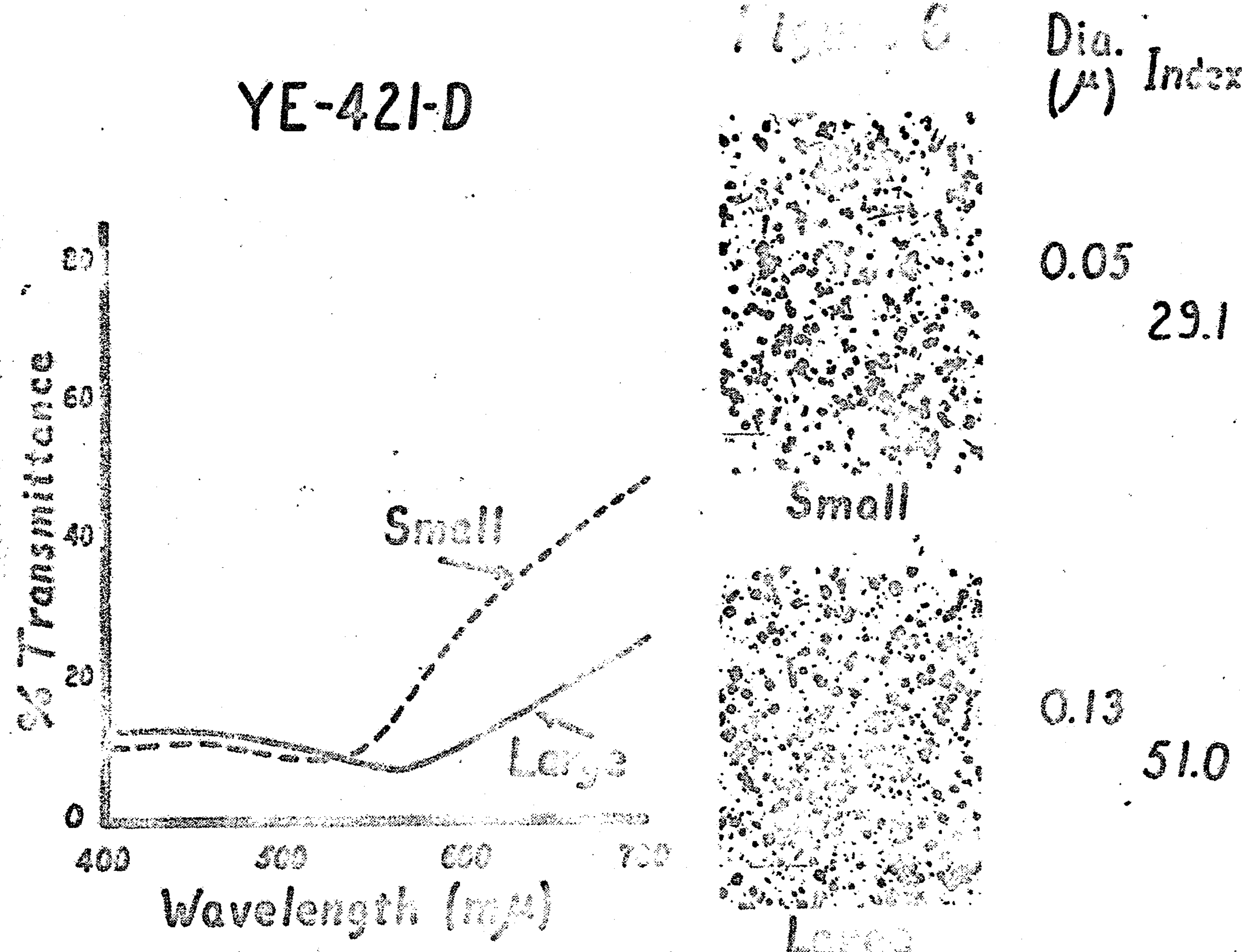
F-4-P





3.1

5.5


YE-421-D
% Transmittance
0
20
40
60
80
400
500
600
700
Wavelength (mμ)
Small
Large
Dia. (μ)
Index
0.05
29.1
Small
0.13
51.0
Large