Problem HS/DD/HT 666
Final Report
Section II
March 8, 1938

# Examination of MacGregor New H. T. S. White Lead

## Purpose of Examination:

To compare MacGregor's white lead with 18 dry and to determine its superiority and characteristics in typical paint formulations.

## Opacity Section:

The primary purpose of this section is to determine if Mac Gregor's white lead is as good as its tinting strength indicates, and if so, how much better it really is.

## Discussion - General Notes:

Previous work done by Mr. Floyd Nelson and members of the White Lead Laboratory staff. The paints used in this section were made by and obtained from Mr. Nelson.

The opacity of MacGregor white lead was investigated from three angles.

1. The wet opacity was determined by the reflectometer and normal hiding power curves plotted.

   The decision to run the opacity on the wet films was reached because preliminary work indicates that films sufficiently thick to reach 98% contrast would wrinkle on drying. It was also believed that the volatile would escape within an hour's time and that from that time on the opacity would change very slightly.

   The contrast ratios were also determined on the dried films because the drop in the contrast ratios determined from reflectometer measurements made immediately and at the end of one hour were very appreciable and it was desirable to know how much more opacity the films lost on drying.

2. The necessary reduction of a paint made with MacGregor's white lead to make it equal in hiding at 96% contrast to one made with 18 dry was determined. In other words, it was desired to know the quantity of MacGregor white lead required, per hundred gallons, to make a paint equivalent in hiding power at 96% contrast to one made using the normally required amount of 18 dry.

3. In order to clinch the argument, paint-outs were made on gray contrast sheets at a spread of approximately 470 square feet per gallon. The paints used for these paint-outs were reduced according to the data obtained under part 2 above so that they would be equivalent in hiding power.

Opacity Section - Part 1.

Procedure:

Only the variations from the regular reflectometer procedure will be noted here. The spinning of the films is changed in that instead of building up successive coats on one slide until 98% contrast is reached coats of varying thickness are spun on several different slides. A slightly varied spinning technique is required to spin one heavy coat instead of the thin ones usually spun. Enough slides are spun to give a series of paints ranging from 82% to 98% contrast.

Brightness readings were made immediately after spinning the film, after it had dried one hour, and after it had dried several days. It is impracticable to bring the wet films in contact with the sphere opening as is done in the case of dry films. This made it necessary to standardize the instrument and make the readings allowing a small clearance. Using 0.5 mm. as the practical working distance for measuring the reflectance of wet films we obtain a drop of 3 micro amperes, which is equivalent to 0.8% brightness at 98% but 0.6% at all normal brightness readings (normal to paint films). By standardizing the reflectometer against $MgCO_3$ = 98% with a 0.5 mm. space between the reflectance surface and the sphere opening we should obtain results reproducible within $\pm$ 1 micro ampere or a range of 0.4% brightness ($\pm$ 0.2%).

The excess paint was wiped from the slides as completely as possible and the weight taken at the end of one hour. Wet to dry factors were run for the same length of time and the wet weight of paint calculated from these results. The paints were gaining weight by oxidation at this time. See graphs of drying weights vs. time included in this report.

Experimental Work - Tabulations and Results:

Data received from Mr. Nelson concerning the manufacture of paint No. 666-4 (18 dry) and 666-5 (MacGregor).

Formulation

| Base | Vehicle |
|---|---|
| 45 av. oz. White Lead | 18 fl. oz. (90%) (44) |
| 13-1/2 fl. oz. Vehicle | 1 " " ( 5%) (1539) |
| | 1 " " ( 5%) (440) |

He used 32-3/4 av. oz. of the above paste for reduction with 7 fl. oz. of the mixed vehicle as above.

Gallon weights found: Paint No. 666-4 - 17.30 pounds
" " 666-5 - 17.26 "

It was necessary to calculate a 100 gallon formula and the theoretical gallon weight from the above data.

Base Paint as Formulated — 32.75 av. oz. of paste contain

| Item | fl. oz. | av. oz. | % by weight | av. oz. |
|---|---|---|---|---|
| 18 dry | 6.300 | 45.00 | 77.52 | 25.39 |
| (44) | 12.150 | 11.74 | 20.22 | 6.62 |
| (1539) | 0.675 | 0.68 | 1.17 | 0.38 |
| (440) | 0.675 | 0.63 | 1.09 | 0.36 |
| | 19.800 | 58.05 | 100.00 | 32.75 |

Gallon weight of base 23.45 lbs.

7 fl. oz. of added vehicle:

| Item | fl. oz. | av. oz. | % by volume |
|---|---|---|---|
| (44) | 6.30 | 6.088 | 90 |
| (1539) | 0.35 | 0.352 | 5 |
| (440) | 0.35 | 0.328 | 5 |
| | 7.00 | 6.768 | 100 |

Formula (paint as used)

| Item | fl. oz. | av. oz. | % by wt. | % by vol. | *100 gallon basis lbs. | gals. |
|---|---|---|---|---|---|---|
| 18 dry | 3.55 | 25.39 | 64.25 | 19.55 | 1110.89 | 19.44 |
| (44) | 13.15 | 12.71 | 32.16 | 72.41 | 556.56 | 72 |
| (1539) | 0.73 | 0.73 | 1.85 | 4.02 | 32.24 | 4 |
| (440) | 0.73 | 0.69 | 1.75 | 4.02 | 29.92 | 4 |
| | 18.16 | 39.52 | 100.01 | 100.00 | 1729.61 | 99.44 |

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Gallon weight of paint - 17.41 pounds

*The pigment was calculated on the basis of 80 gallons of mixed vehicle in order to avoid using small fractions of a gallon in the vehicle formulation.

Comparison of Contrast Ratio vs. Drying Time vs. Weight of Wet Paint and Square Feet Per Gallon.

| 18 Dry Slide No. | Contrast Ratios % Orig. | Imm. | 1 Hr. | Dry | Weight of Wet Paint | Sq. ft./gal. |
|---|---|---|---|---|---|---|
| 1 | 5.22 | 84.38 | 83.21 | 82.48 | 1.6838 gms. | 567.49 |
| 3 | 5.17 | 88.67 | 87.44 | 85.78 | 1.9250 " | 496.39 |
| 2 | 5.50 | 89.73 | 89.02 | 86.70 | 1.9882 " | 480.61 |
| 4 | 5.48 | 90.58 | 89.83 | 87.25 | 2.2229 " | 429.86 |
| 13 | 5.09 | 92.86 | 92.58 | 90.56 | 2.8242 " | 338.34 |
| 10 | 5.14 | 94.79 | 94.50 | 93.00 | 3.3303 " | 286.92 |
| 12 | 5.57 | 95.72 | 95.44 | 93.95 | 3.4209 " | 279.33 |
| 11 | 5.26 | 97.39 | 97.38 | 96.64 | 4.2781 " | 223.36 |
| 9 | 5.12 | 98.82 | 98.34 | 98.10 | 5.6197 " | 170.03 |
| MacGregor | | | | | | |
| 6 | 5.54 | 82.11 | 81.86 | 80.45 | 1.0114 " | 942.59 |
| 5 | 5.68 | 82.61 | 82.61 | 82.11 | 1.1245 " | 847.78 |
| 7 | 5.67 | 90.93 | 90.43 | 90;12 | 1.6521 " | 577.04 |
| 8 | 5.47 | 91.65 | 91.63 | 90.87 | 1.8022 " | 528.98 |
| 18 | 5.80 | 92.20 | 92.20 | 91.13 | 1.5995 " | 596.02 |
| 17 | 5.35 | 96.22 | 96.22 | 95.49 | 2.5646 " | 371.73 |
| 16 | 5.26 | 96.72 | 96.72 | 96.24 | 2.9746 " | 320.49 |
| 15 | 5.32 | 98.14 | 98.14 | 98.13 | 3.5992 " | 264.87 |
| 14 | 5.24 | 98.84 | 98.62 | 98.38 | 4.9067 " | 194.29 |

Conclusions: (Part 1)

Square feet per gallon coverage at - % contrast.

| | 98% | 96% | 90% | 86% |
|---|---|---|---|---|
| 18 dry | 190 | 255 | 430 | 520 |
| MacGregor | 260 | 370 | 620 | 760 |
| * | 36.84% | 45.10% | 44.19% | 46.15% |

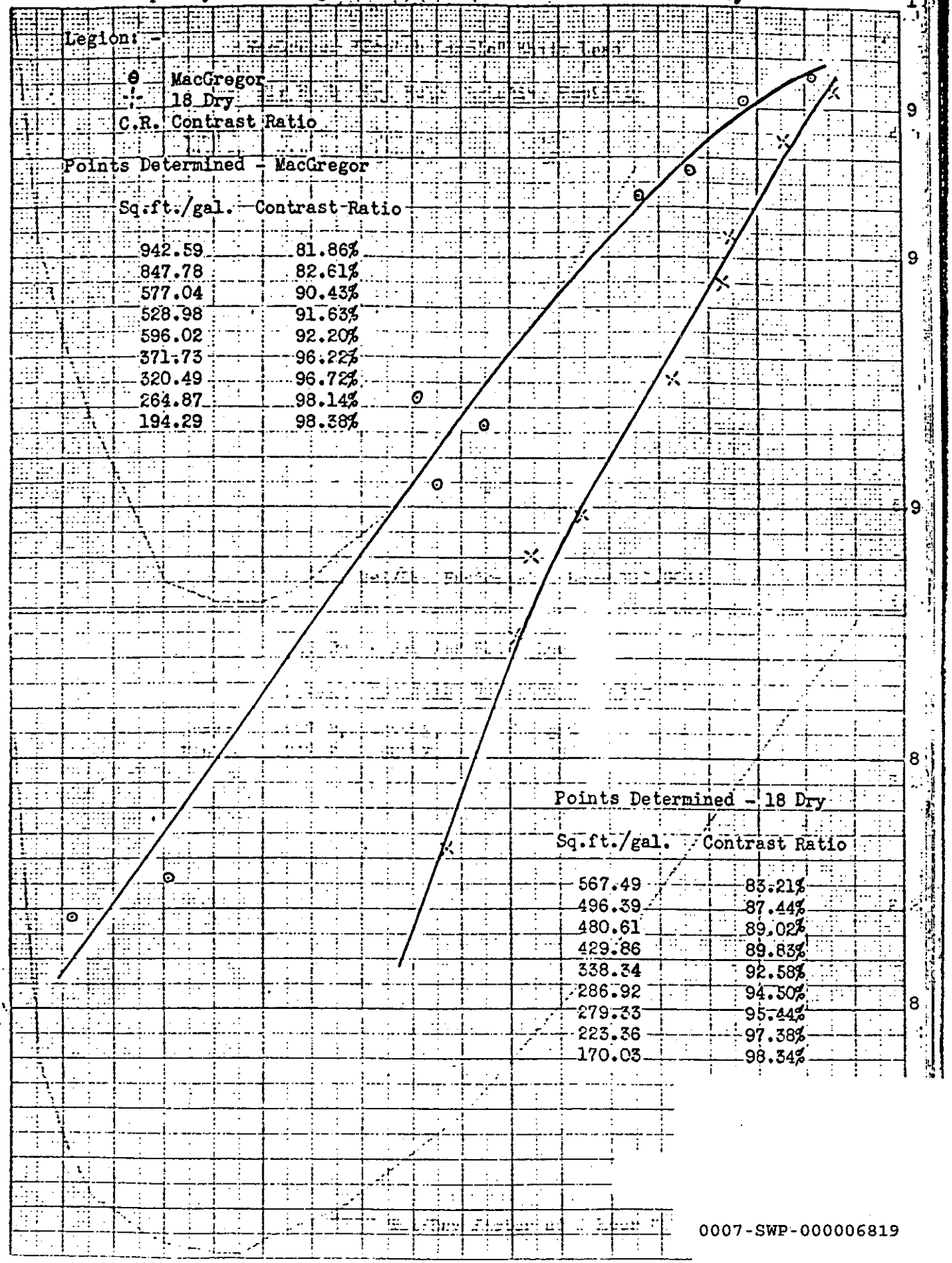
*Per cent increased coverage of MacGregor over 18 dry. Up to the point at which the curves become asymptotic they indicate MacGregor to have 45% more covering power than 18 dry.

MacGregor shows much less tendency to lose opacity on drying. See table of contrast ratios above.

One possible explanation is the difference in settling rate. The 18 dry settles from the film very rapidly, leaving a heavy layer of non-volatile covering the pigment. This cuts down the brightness which in a measure decreases the contrast. Paints made with Mac Gregor white lead show this tendency only slightly. It is problematical that MacGregor does not settle or settles only slightly during drying. This is probably due to its higher consistency.

A structural difference in the white lead particles might cause the difference.

## Opacity of MacGregor "Scotch Laddie" White Lead vs. 18 Dry

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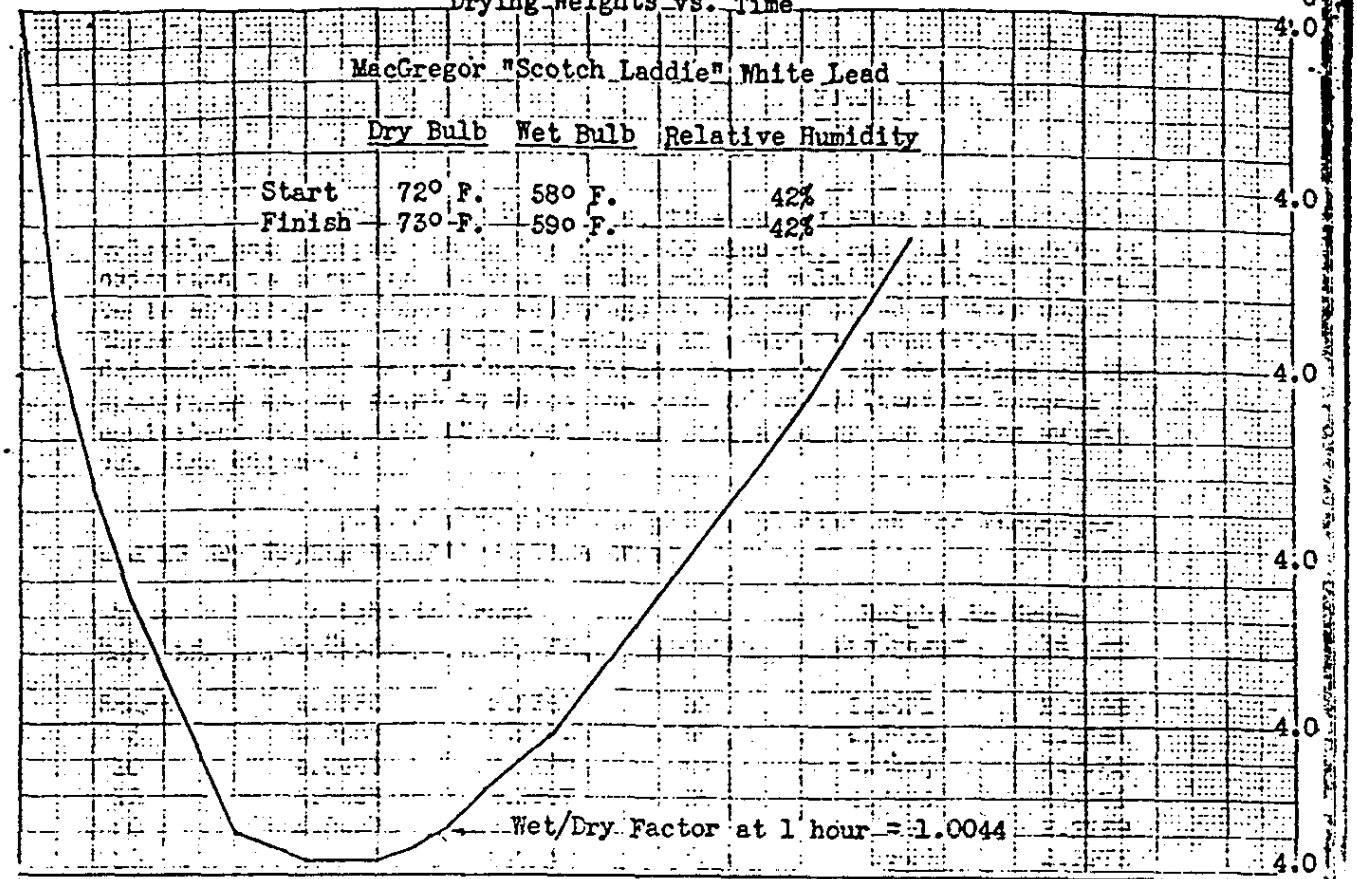
Drying Weights vs. Time

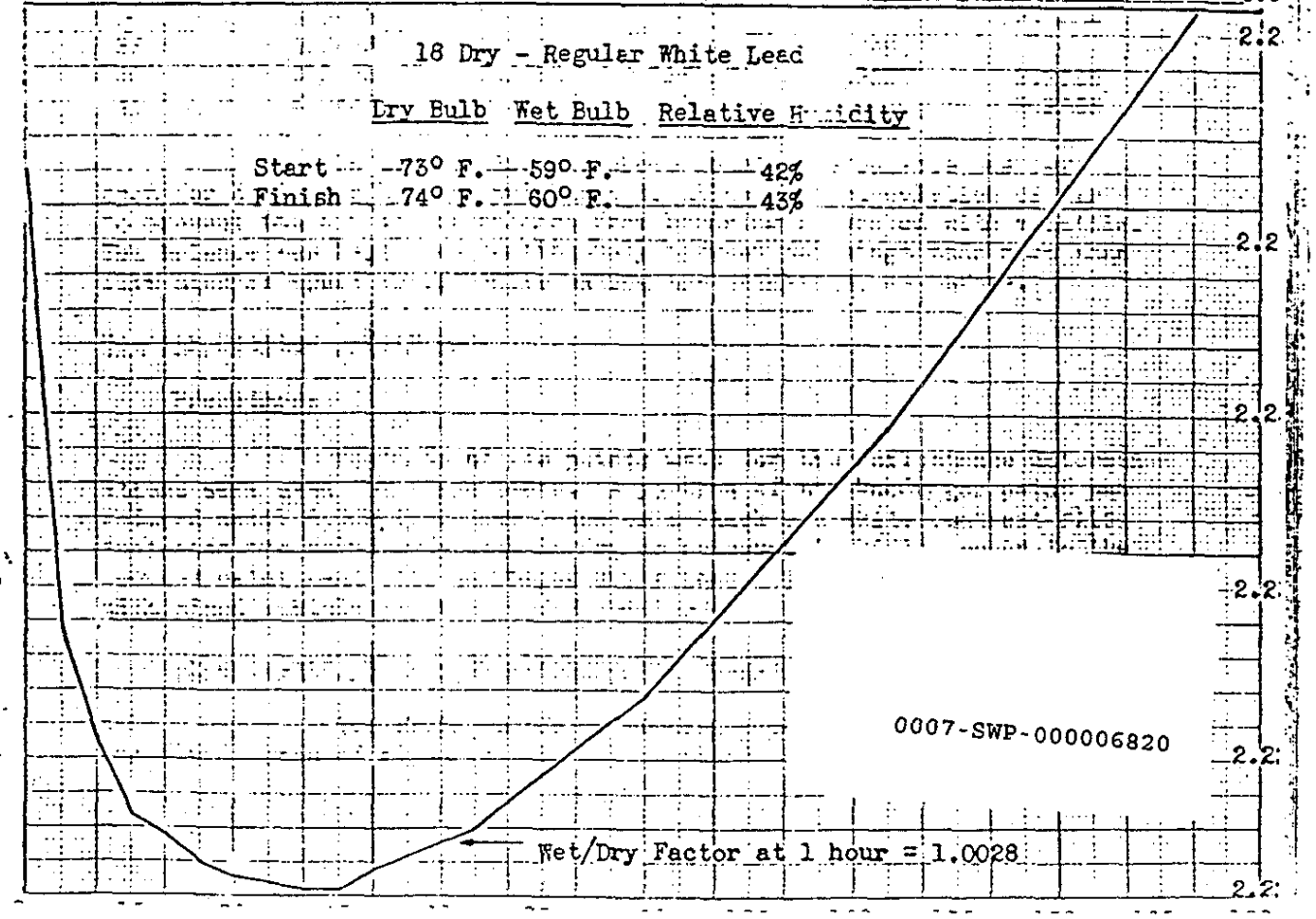
MacGregor "Scotch Laddie" White Lead

| | Dry Bulb | Wet Bulb | Relative Humidity |
|---|---|---|---|
| Start | 72° F. | 58° F. | 42% |
| Finish | 73° F. | 59° F. | 42% |




18 Dry - Regular White Lead

| | Dry Bulb | Wet Bulb | Relative Humidity |
|---|---|---|---|
| Start | 73° F. | 59° F. | 42% |
| Finish | 74° F. | 60° F. | 43% |

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The Drying Weight vs. Time curves show that most of the volatile has escaped within the first 40 minutes, and that the rate of oxidation is about 1/3 as fast as the rate of volatilization. It would be to advantage to make all weighings at the end of 40 minutes under these conditions. Generally Speaking, it would be advantageous to determine the wet to dry factors first, then make all weighings of the spun films at the point at which the rate of volatilization becomes equivalent to the rate of oxidation as indicated by the drying weight vs. time curves.

Data determining the points plotted on the MacGregor and 18 dry Drying Weight vs. Time curves.

| Time Minutes | Weight in Grams MacGregor | 18 Dry | Time Minutes | Weight in Grams MacGregor | 18 Dry |
|---|---|---|---|---|---|
| Start | 4.0550 | 2.2360 | 50 | 4.0431 | 2.2259 |
| 5 | 4.0503 | 2.2294 | 55 | 4.0433 | 2.2261 |
| 10 | 4.0483 | 2.2278 | 60 | 4.0436 | 2.2263 |
| 15 | 4.0468 | 2.2267 | 65 | 4.0441 | 2.2265 |
| 20 | 4.0457 | 2.2264 | 70 | 4.0445 | 2.2269 |
| 25 | 4.0445 | 2.2260 | 75 | 4.0449 | 2.2273 |
| 30 | 4.0435 | 2.2258 | 90 | | 2.2284 |
| 35 | 4.0433 | 2.2257 | 110 | 4.0495 | |
| 40 | 4.0431 | 2.2256 | 125 | 4.0518 | 2.2323 |
| 45 | 4.0431 | 2.2256 | 170 | | 2.2383 |

In determining the above points the balance was left open for 3 minutes between weighings to allow free circulation of air to prevent the air in the balance from becoming saturated with volatile. The balance was kept open continuously between weighings made more than five minutes apart except while making the weighings, as before.

Opacity Section - Part 2.

Procedure:

Portions of the paints used for the reflectance determinations were used for these tests. A portion of the MacGregor paint was reduced until 0.003 inch draw-downs on a black and white contrast slide indicated the paint made with 18 dry to be equal in hiding power to the reduced paint made with MacGregor white lead. The reduction was made with mixed vehicle as described in part one.

Experimental Work - Tabulations and Results

Two fluid ounces of MacGregor paint were reduced with one fluid ounce of mixed vehicle equivalent to a 33-1/3% reduction.

18 dry formula comparable to MacGregor at 96% contrast.

| 17.41 lbs. | Gallon Weight | 14.16 lbs. |
|---|---|---|
| | Item | |
| 1110.89 lbs. | White Lead | 744.76 lbs. |
| 72 gals. | (44) | 78 gals. |
| 4 " | (1539) | 4-1/3 " |
| 4 " | (440) | 4-1/3 " |

Contrast data on spun films of the above paints:

| | | |
|---|---|---|
| 5.72% | Original | 5.05% |
| 96.66% | At start | 96.25% |
| 95.65% | At one hour | 96.19% |

Conclusions: (Part 2)

These paints are quite thin; however they indicate Mac Gregor to be approximately 50% better than 18 dry.

Opacity Section - Part 3.

Procedure:

Part 2 may be considered as affording preliminary exploration for the work done under this part. Having two definite indications, namely, that a 33-1/3% reduction of MacGregor makes it equivalent to 18 dry and that such paints, formulated as above, are too thin, we determined to start out with the original bases. Portions of the bases were obtained from Mr. Nelson. The base made with 18 dry was of suitable brushing consistency, therefore 2 fl. oz. of the base made with MacGregor was reduced with 1 fl. oz. of mixed vehicle - equivalent to a reduction of 33-1/3% by volume.

A portion of paint was measured out onto a gray contrast sheet by means of a syringe. The paint was distributed uniformly over the surface. A brush, one inch wide, was wet with some of the same paint and a preliminary contrast sheet brushed in order to determine the normal amount of paint left in the brush, then the brush was weighed and the weighed portion of paint measured out of the syringe was spread uniformly over the entire contrast surface, equivalent to one square foot. An attempt was made to have about the same amount of paint left in the brush after brushing out the sheet as was in the brush at the beginning.

Experimental Work - Tabulations and Results:

Base formulation calculated on the basis of 80 gallons of mixed vehicle for the sake of simplicity.

128 fl. oz./gal. x 80 gals. = 10,240 fl. oz.
10,240 " " ÷ 13.5 fl. oz. of mixed vehicle in laboratory formula = 758.52
758.52 x 45 av. oz. of pigment in laboratory formula = 34,133.4 av. oz. = 2133.33 lbs.

| | | |
|---|---|---|
| 2133.33 lbs. | 37.33 gals. | White Lead |
| 556.56 " | 72 " | (44) |
| 32.24 " | 4 " | (1539) |
| 29.92 " | 4 " | (440) |
| 2752.05 lbs. | 117.33 gals. | ∴ gal. wt. = 23.46 lbs. |

100 gals. x lbs./gal. = lbs./100 gals. = 2346.0 lbs.

| | | | | |
|---|---|---|---|---|
| 45 gals. | (44) | x 7.73 lbs./gal. | = 347.85 lbs. | |
| 2.5 " | (1539) | x 8.06 " " | = 20.15 " | |
| 2.5 " | (440) | x 7.48 " " | = 18.70 " | |
| 50 gals. | | | = 386.70 lbs. | 386.7 lbs. |
| | | 150 gals. | weighs | 2732.7 lbs. |

∴ 33-1/3% reduction yields a paint having a gal. wt. = 18.22 lbs.

1 gallon =≏= 3785.332 cc

Spread desired: - 450 to 500 sq. ft./gal.
1 sq. ft. requires 8.52 cc at 450
Used 8.4 cc

| 18 Dry | vs. | MacGregor |
|---|---|---|
| 2133.33 lbs. W. L. / 117.33 gals. = 18.18 lbs./gal. | | MacGregor contains 2/3 as much lead, or 12.12 lbs. |

| | | |
|---|---|---|
| Wt. of paint used | 22.6979 gms. | 17.6460 gms. |
| ÷ 453.59 gms./lb. | = 0.050 lbs./sq. ft. | 0.039 lbs./sq. ft. |
| Lbs./gal. ÷ lbs./sq. ft. | = 468.82 sq. ft./gal. | 467.18 sq. ft./gal. |
| Lbs. W. L./Gal. | = 0.039 lbs. sq. ft. | 0.0259 lbs./sq. ft. |
| Sq. ft./gal. | or 3.9 lbs./100 sq. ft. | 2.59 lbs./100 sq. ft. |

It was impossible to distinguish between the hiding power of the paints as brushed out on gray contrast sheets at equal spreading rates.

Conclusions - Part 3.

Opacity as determined by this method checks the results obtained with the reflectometer.

General Conclusions:

It is concluded that MacGregor white lead has at least 45% more hiding power than 18 dry.

That dry hiding power determinations are more accurate than wet ones. This would necessitate the use of the build-up method because heavy coats would wrinkle as has already been pointed out.

That comparing the contrast ratio of dried films with the apparent contrast of the wet films as a judgment of the agreement between reflectometer measurements and the usual visual impression is false. In order to check the agreement the reflectometer measurements must be made at the same time the visual judgment is made, except when judging a dried film. The contrasts shown by the spun films under part 2 shows the results that can be obtained.

That MacGregor settles much slower than 18 dry.

Special Note:

All data concerning reflectance measurements, brushing weights, etc. which does not appear in this report can be obtained by referring to the file copy held in the Allied files at Kensington.

| | | |
|---|---|---|
| Legion: | (44) | Alkali Refined Linseed Oil |
| | (1539) | Heat Bodied Perilla Oil |
| | (440) | Lead-Manganese Resinate Drier |

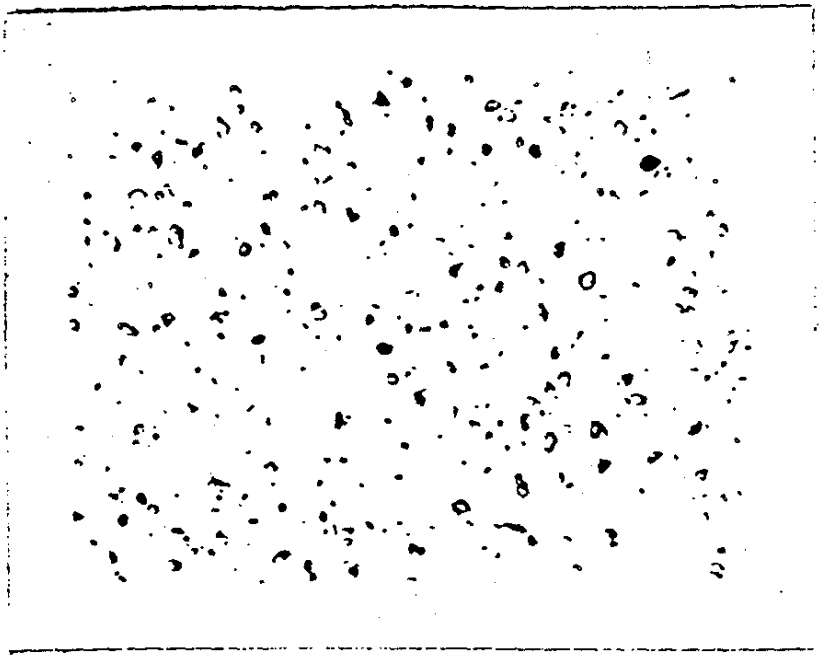
Photomicrographic Section:

The object of this section was to obtain representative pictures of the pigment and an indication of its structure.

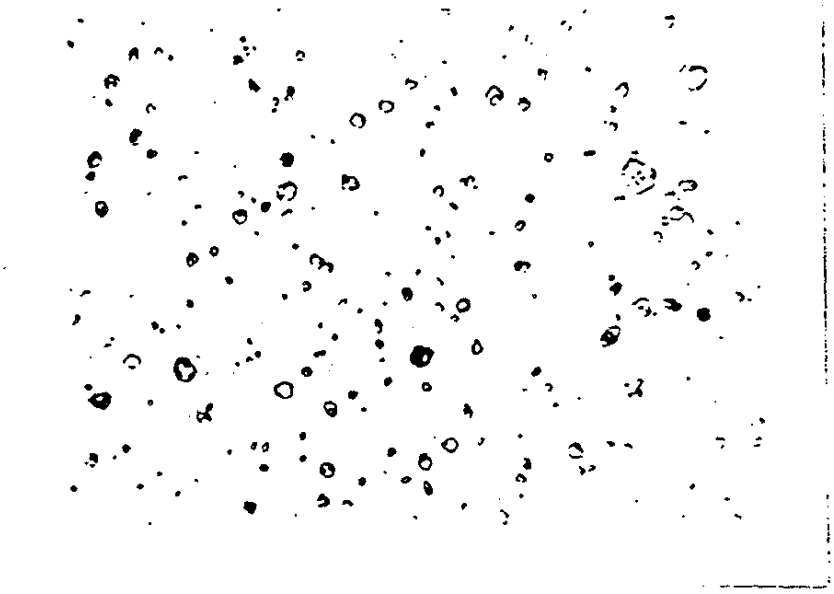
Discussion - General Notes:

Due to the difficulty normally encountered in dispersing white lead carbonate without a subsequent breaking down of large particles it was decided to try a new and different method of dispersion.

The pigments were dispersed in a solution of damar gum. The details of the procedure will be written at a later date and included under photomicrographic procedures.



MacGregor "Scotch Laddie" White Leed
x 1430 Film No. 1235



18 Dry - White Lead Carbonate
x 1430 Film No. 1226

Conclusions:

That MacGregor is more crystalline and generally more uniform and finer in particle size than 18 dry.

That the treating agent used on MacGregor or the nature of the particles themselves made it more difficult to photograph them. None of the pictures were clear and sharp.

It was very difficult to find a representative field suitable for photographing.

H. I. Tompkins

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